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Two decades of the synthesis of mono- and bis-aminomercapto[1,2,4]triazoles

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4-Amino-5-mercapto[1,2,4]triazole and its 3-substituted derivatives have proven to be of biological interest and provide access to a new class of biologically active heterocyclic compounds for biomedical applications. This study will be helpful for scientific researchers interested in the chemistry of bifunctional versatile compounds as it provides a collection of all the methods for the preparation of 3-substituted-4-amino-5-mercapto[1,2,4]triazoles with aliphatic, aromatic, and heterocyclic moieties during the period from 2000 to mid-2020.

1. Introduction

[1,2,4]Triazoles and their fused heterocyclic derivatives have occupied a unique position as novel biologically active agents with remarkably diverse pharmacological properties such as antimicrobial, antifungal, anticancer, anticonvulsant, antiviral, anti-inflammatory, anti-HIV, and anti-mycobacterial activities.[1–8] A large number of ring systems containing [1,2,4]triazoles have been incorporated into a wide variety of therapeutically interesting drug candidates such as fluconazole, ravuconazole, itraconazole, voriconazole, posaconazole, vorozole, letrozole, ribavirin, triazolam, alprazolam, etizolam, furacylin, hexaconazole, triadimefon, myclobutanil, rizatriptan, propiconazole, and fluotrimazole (Chart 1).[9] Moreover, the synthesis of bis-heterocyclic compounds containing triazole rings has attracted attention due to the diverse applications of these compounds in numerous pharmacological and biological fields.[10–13]

Bis-[4-amino-5-mercapto[1,2,4]triazoles] (**1**) and 3-substituted-4-amino-5-mercapto[1,2,4] triazoles (**2–4**) (Chart 2) contain both amino and mercapto groups as ready-made nucleophilic centers for the synthesis of condensed heterocyclic rings. The introduction of these groups in different nuclei enhances their biological activities. Accordingly, the objective of the present review is to highlight the synthetic methods used to obtain 3-substituted-4-amino-5-mercapto[1,2,4]triazoles and bis-[4-amino-5-mercapto[1,2,4]triazoles] from 2000 until mid-2020.

2. Synthetic routes using thiocarbohydrazide as the precursor

2.1. Reactions with carboxylic acids

3-Substituted-4-amino-5-mercapto[1,2,4]triazoles **2–4** were prepared from the treatment of thiocarbohydrazide (**5**) with carboxylic acids (Scheme 1) (Table 1).

A series of dicarboxylic acids such as tartaric, malic,[41–43] succinic,[44] glutaric,[45] and others[46] were treated with thiocarbohydrazide (**5**) to afford the respective series of bis-(4-amino-5-mercapto[1,2,4]triazoles) **8**, **9** (Scheme 2).

Similarly, a condensation reaction between 5-(3-formyl-4-methoxybenzyl)-2-methoxybenzoic acid (**10**) and thiocarbohydrazide (**5**) at the melt temperature afforded bis[4-methoxy-3-[4-amino-5-sulfanyl-4*H*-1,2,4-triazol-3-yl]phenyl]methane (**11**) (Scheme 3).[47]

2.2. Reactions with esters

In addition, Demirbas *et al.*[48] reported the treatment of ethyl(3-alkyl-4-amino-5-oxo-4,5-dihydro-1*H*-1,2,4-triazol-1-yl) acetates (**12**) with thiocarbohydrazide (**5**), which furnished 5-alkyl-4-amino-2-[(4-amino-5-mercapto-4*H*-1,2,4-triazol-3-yl)methyl]-2,4-dihydro-3*H*-1,2,4-triazol-3-ones (**13**) (Scheme 4).

Moreover, refluxing thiocarbohydrazide (**5**) with diethyl terephthalate **14** using magnetic iron oxide (Fe_3O_4) nanoparticles as an eco-friendly catalyst yielded the respective 3,3′-(1,4-phenylene)bis(4-amino-1*H*-1,2,4-triazole-5(4*H*)-thione) (**15**) (Scheme 5).[49]

2.3. Reactions with lactones

4-Amino-3-(3-hydroxypropyl)-5-mercapto[1,2,4]triazole (**17**) was prepared *via* the treatment of thiocarbohydrazide (**5**) with lactone **16**, as reported by Zhang *et al.*[50] [Scheme 6].

The synthetic routes for the preparation of 4-amino-3-(D-galactopentitol-1-yl)-5-mercapto[1,2,4]triazole (**21**),[51] 4-

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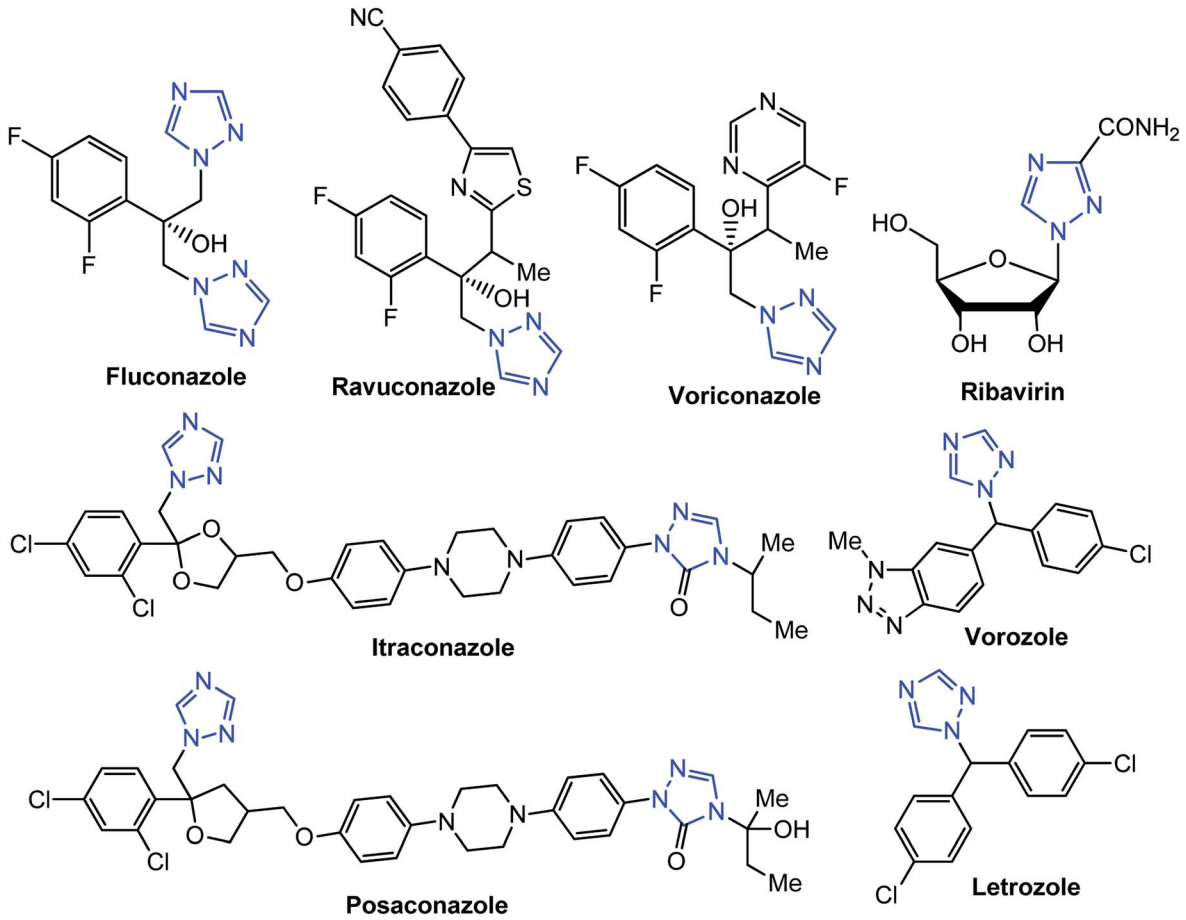


Chart 1 Examples of [1,2,4]triazole bearing drugs.

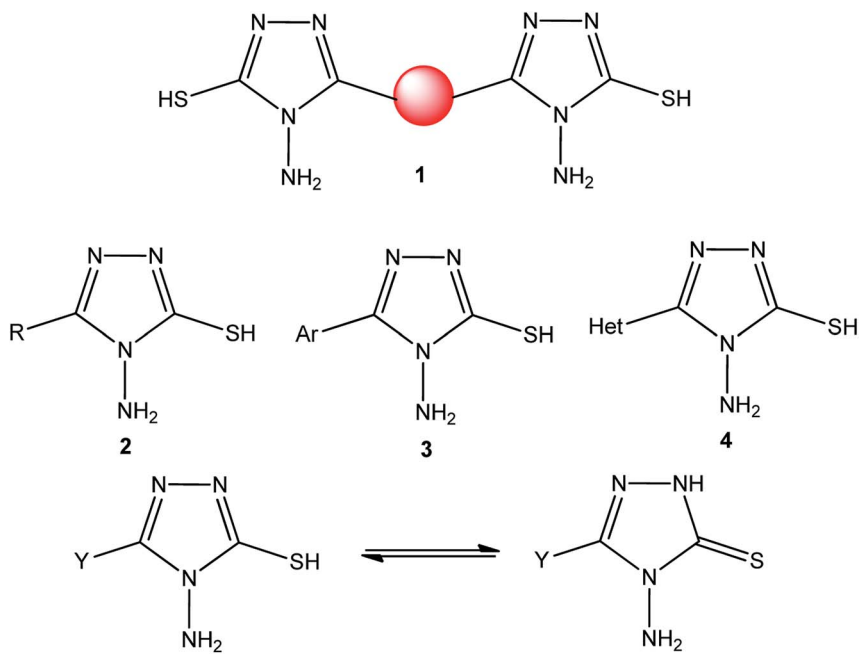
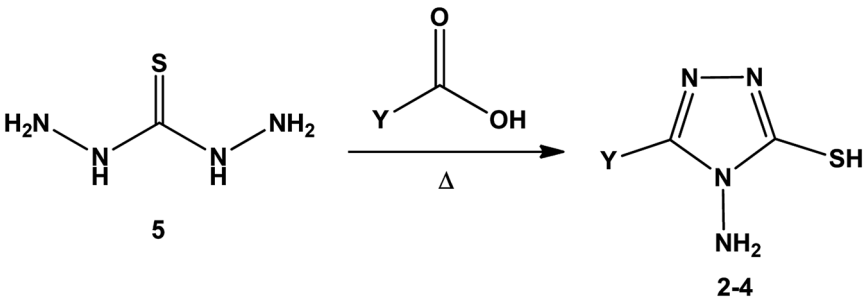


Chart 2 Structures of bis-[4-amino-5-mercapto[1,2,4]triazoles] (**1**) and 3-substituted-4-amino-5-mercapto[1,2,4]triazoles (**2**–**4**).



Scheme 1 Synthesis of triazoles 2–4.

Table 1 Derivatives of 3-substituted-4-amino-5-mercapto[1,2,4]triazoles

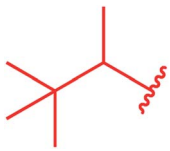
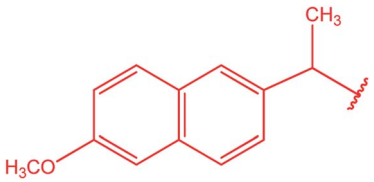
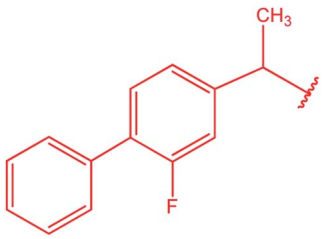
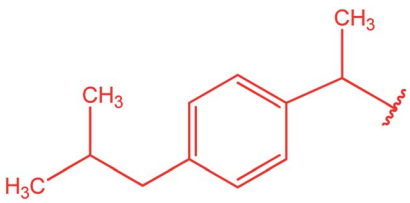
Y	Ref.
H, $-CH_3$, $-C_2H_5$	14
$-CH_3$	15 and 16
$-CH_3$, $-CF_3$	17
	18
	19
	20
Substituted phenyl	21–23
Ar–CH_2–CH_2– & cyclohexyl–CH_2–CH_2–	24
Ar–O–CH_2– & Ar–NH–CH_2– & Ar–S–CH_2– & Ar–SO_2NH–CH_2– & Ar–CONH–CH_2– & Ar–CH(CH_3)– & triazole–CH_2–	25
	26
	26
	26 and 27

Table 1 (*Contd.*)

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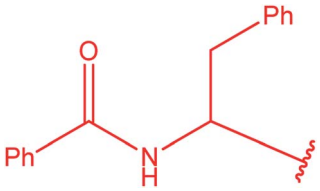
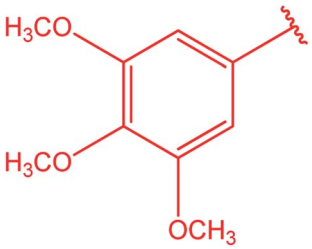
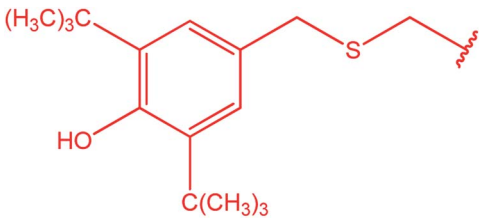
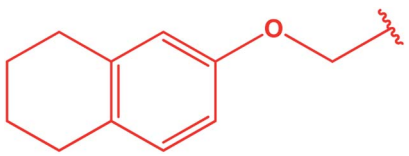
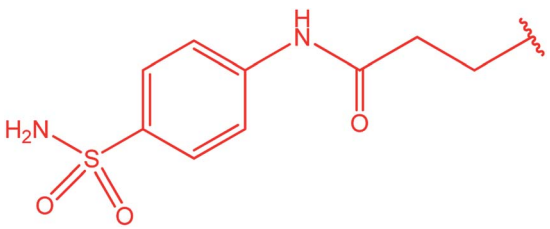
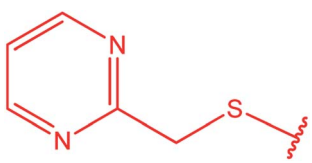
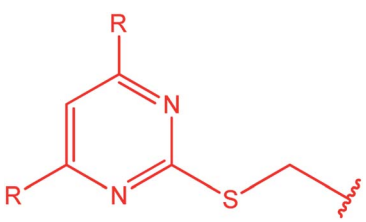
	28
	29
	30
	31
	32
	33
	34

Table 1 (*Contd.*)

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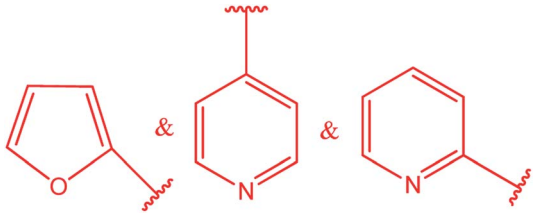
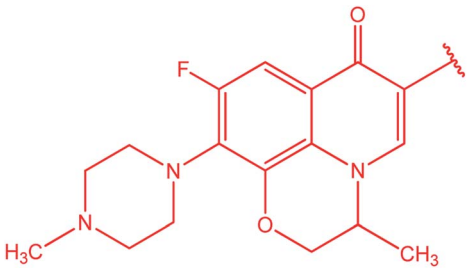
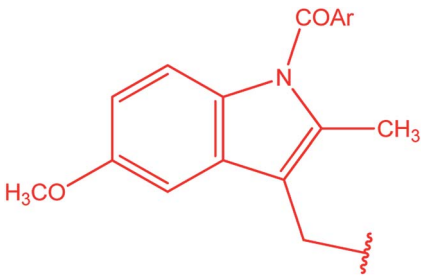
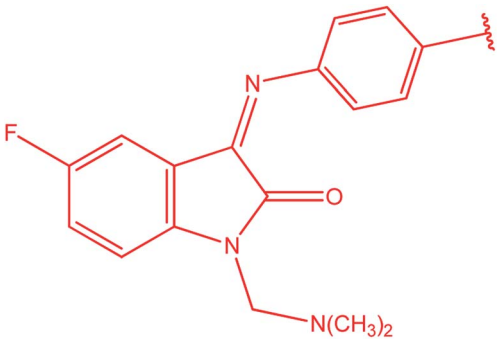
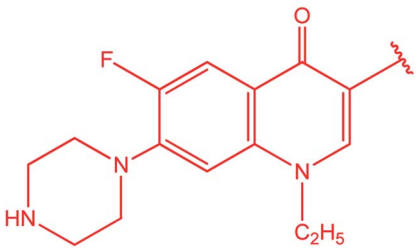
	35
	36
	37
	38
	39

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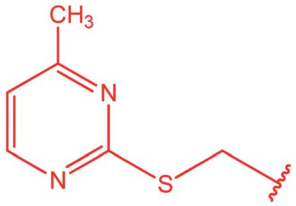
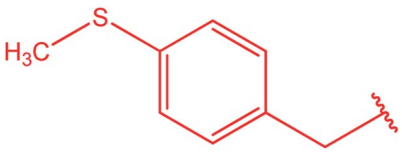
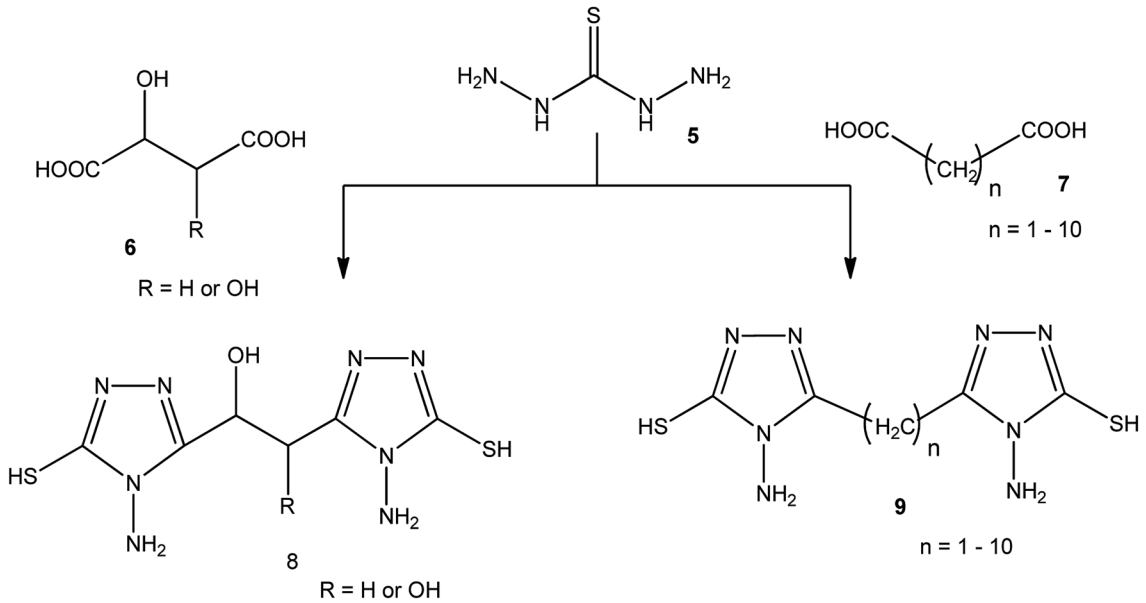
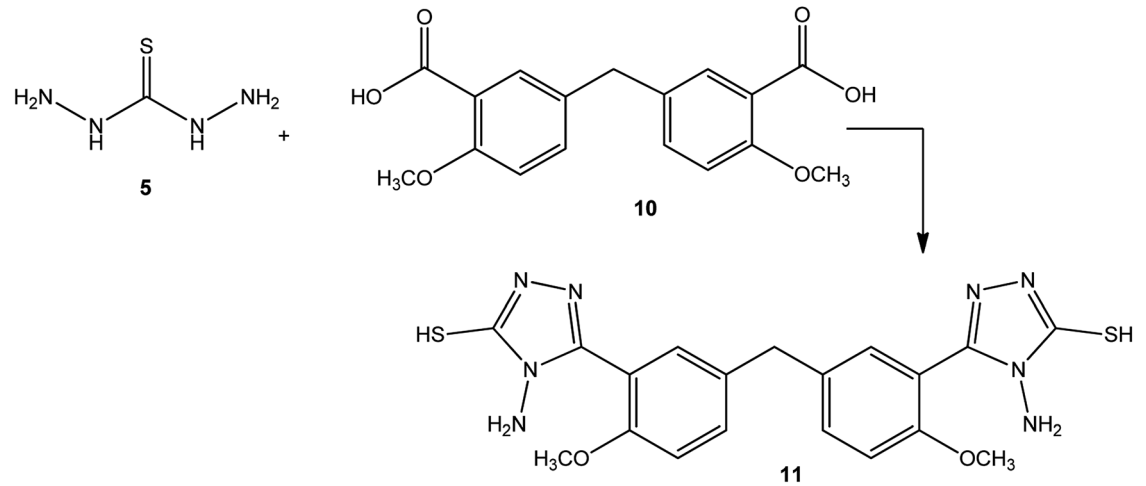
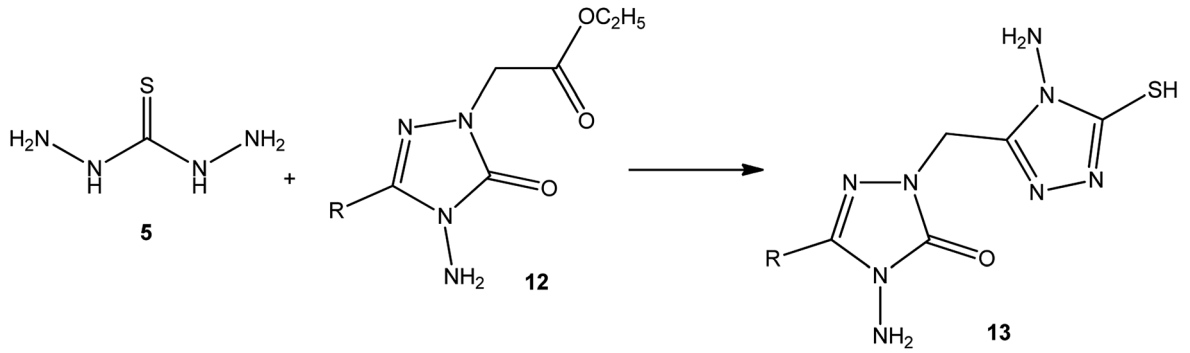
	40
	9



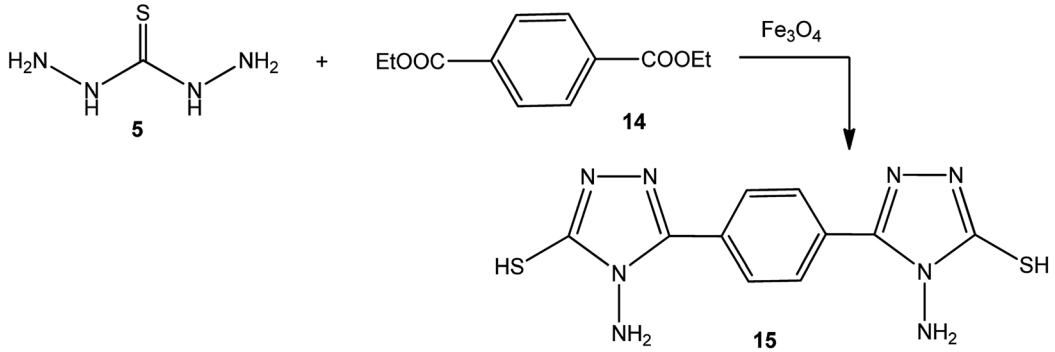
Scheme 2 Synthesis of bis-triazoles **8** and **9**.



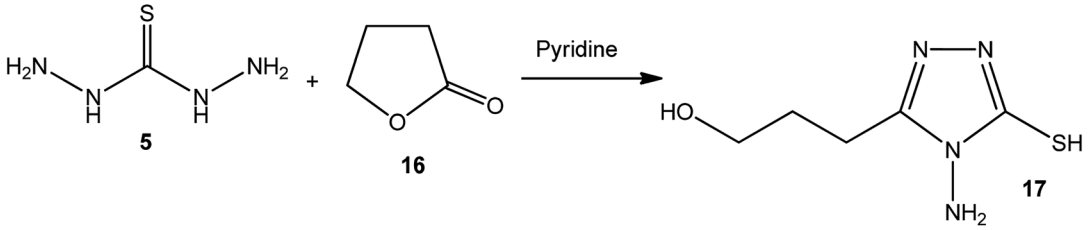
Scheme 3 Synthesis of bis-triazole **11**.



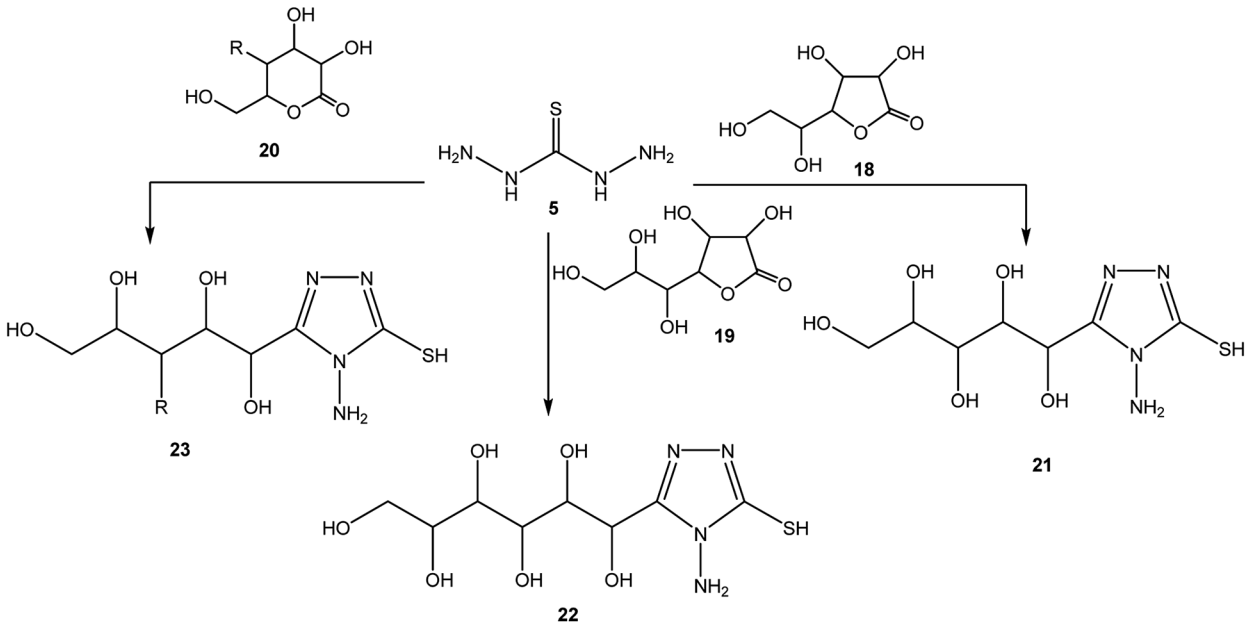
Scheme 4 Synthesis of triazoles **13**.



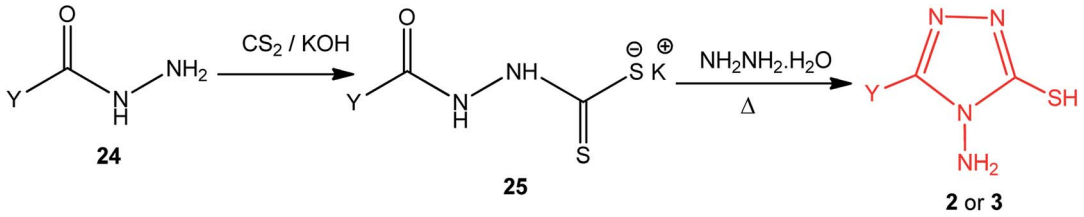
Scheme 5 Synthesis of bis-triazole **15**.



Scheme 6 Synthesis of triazole **17**.

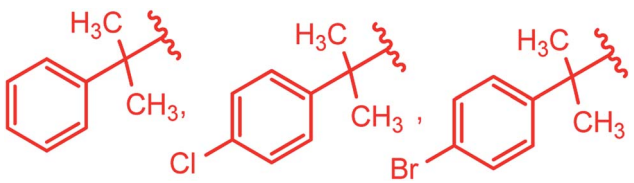
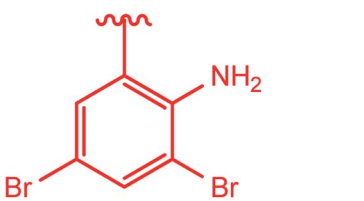
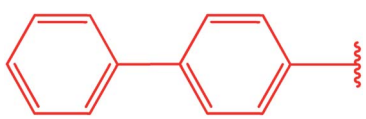
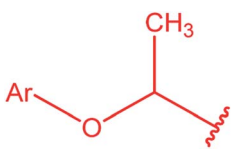
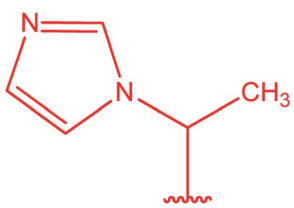


Scheme 7 Synthesis of triazoles **21–23**.



Scheme 8 Synthesis of triazoles 2 and 3.

Table 2 Derivatives of 3-substituted-4-amino-5-mercapto[1,2,4]triazoles

Y	Ref.
$-CH_3$, $-C_2H_5$, $-C_3H_7$	54
$CH_3-(CH_2)_{13}-CH_2-$	55
$CH_3-(CH_2)_{15}-CH(SO_3Na)-$	56
C_6H_5-	57–60
$3\text{-}ClC_6H_4-$	8
$4\text{-}CH_3OC_6H_4-$	61
$2\text{-}HOC_6H_4-$	62
$2\text{-}CH_3C_6H_4-$ & $2\text{-}CH_3\text{-}4\text{-}ClC_6H_3-$	63
C_6H_5- & $2\text{-}HOC_6H_4-$	64
$2\text{-}C_2H_5OC_6H_4-$	65
$3\text{-}Br\text{-}4\text{-}CH_3OC_6H_3-$	66
$2\text{-}HOC_6H_4-$ & $4\text{-}HOC_6H_4-$ & $4\text{-}C_2H_5OC_6H_4-$ & $2\text{-}HO\text{-}5\text{-}ClC_6H_3-$ & $4\text{-}HOC_6H_4-CH_2-$ & $4\text{-}C_2H_5OC_6H_4-CH_2-$	67
$2\text{-}FC_6H_4-CH_2-$ & $2\text{-}BrC_6H_4-CH_2-$ & $4\text{-}HOC_6H_4-CH_2-$ & $2\text{-}CH_3OC_6H_4-CH_2-$ & $4\text{-}NO_2C_6H_4-CH_2-$	68
C_6H_5- & $2\text{-}ClC_6H_4-$ & $2\text{-}NO_2C_6H_4-$ & $2\text{-}HOC_6H_4-$ & 2-furyl	69
C_6H_5- & $4\text{-}ClC_6H_4-$ & $4\text{-}BrC_6H_4-$ & $4\text{-}CH_3OC_6H_4-$ & 2-naphthyl–CH_2-	70
$2\text{-}HOC_6H_4-$ & $4\text{-}HOC_6H_4-$ & $2\text{-}NH_2C_6H_4-$ & $4\text{-}NH_2C_6H_4-$ & $3,4,5\text{-}(HO)_3C_6H_2-$	71
$C_6H_5-CH_2-CH_2-$	72
$2\text{-}(CH_3)_2NC_6H_4-$ & $4\text{-}CH_3NHC_6H_4-$ & 1-naphthyl–CH_2-	73
	74
	75
	76
	77
	77

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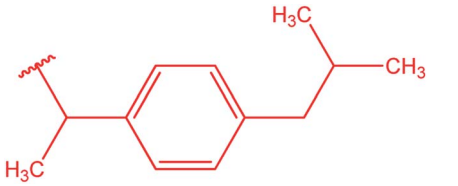
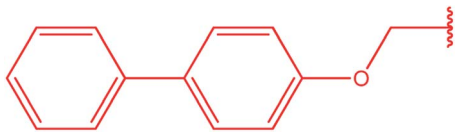
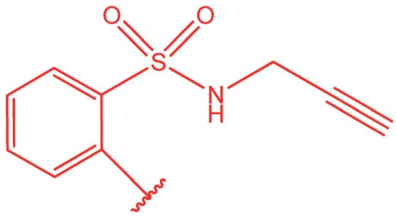
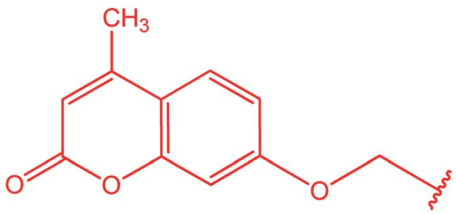
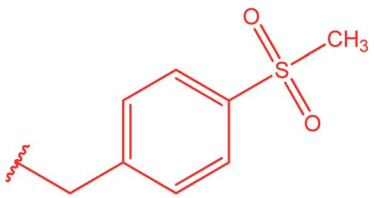
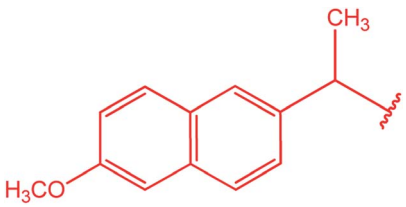
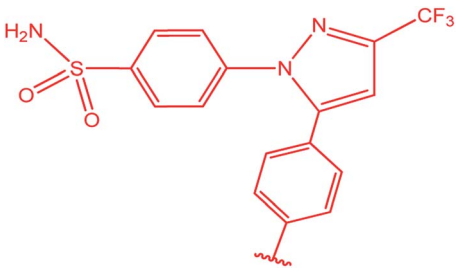
	78 and 79
	79
	80
	81
	82
	83
	84

Table 2 (*Contd.*)

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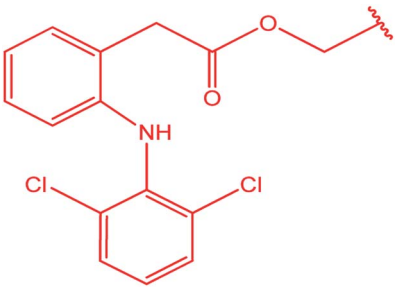
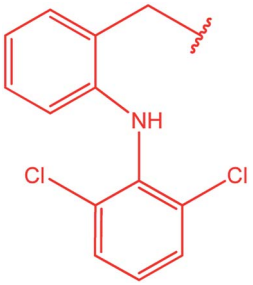
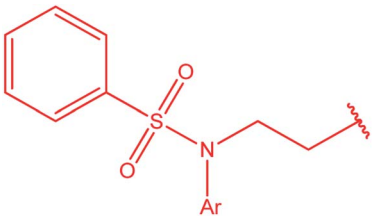
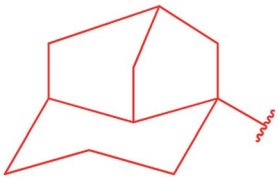
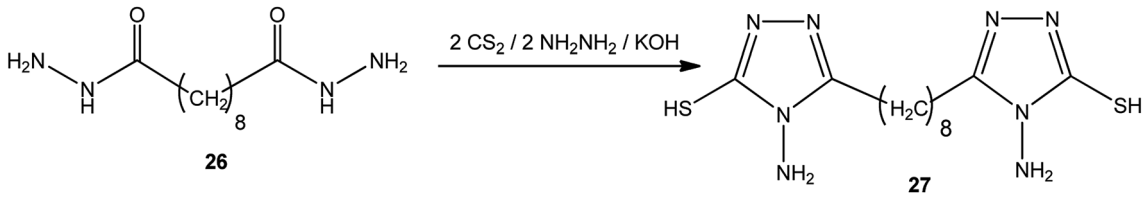
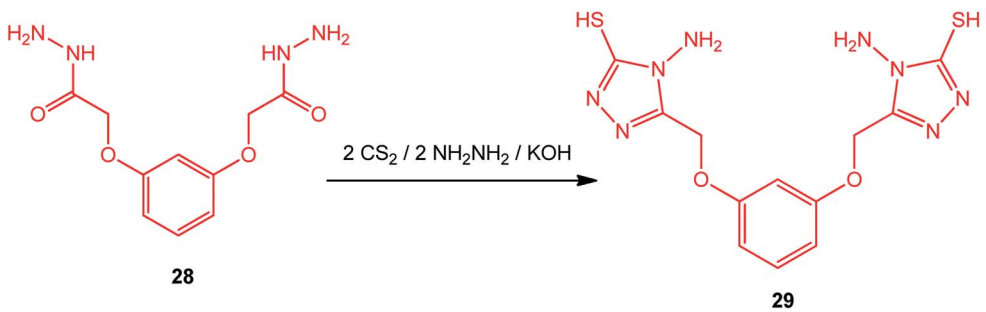
	85
	85 and 86
	87
	88



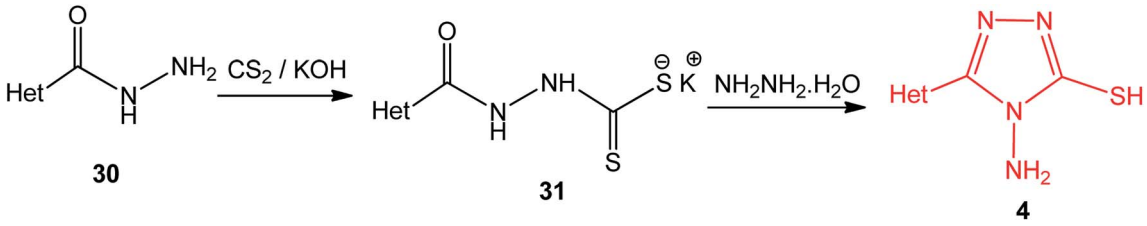
26 27

Scheme 9 Synthesis of bis-triazole **27**.



28 29

Scheme 10 Synthesis of bis-triazole **29**.



Scheme 11 Synthesis of triazoles **4**.

Table 3 Derivatives of 3-substituted-4-amino-5-mercapto[1,2,4] triazoles

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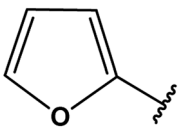
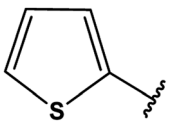
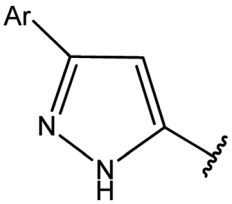
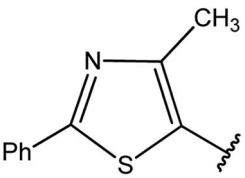
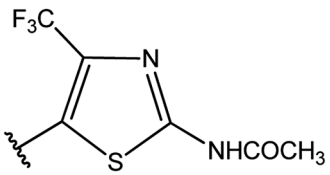
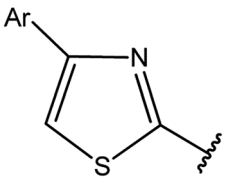
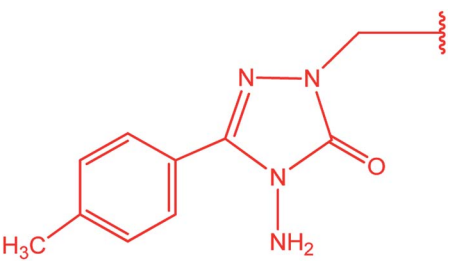
	91
	92
	93
	94
	95
	96
	97

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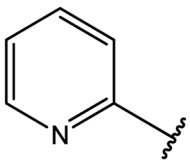
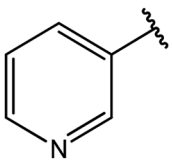
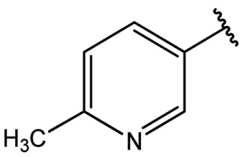
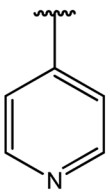
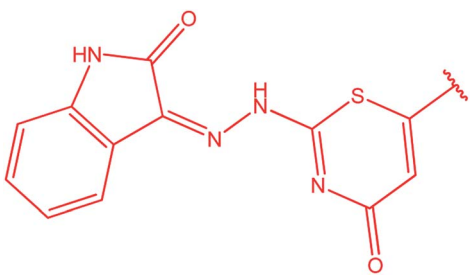
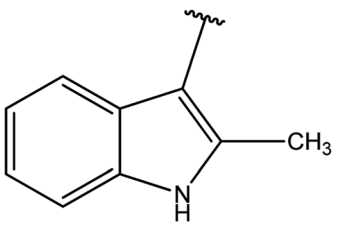
	98
	99
	20
	100–106
	107
	108

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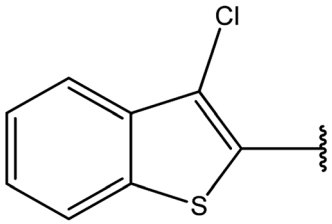
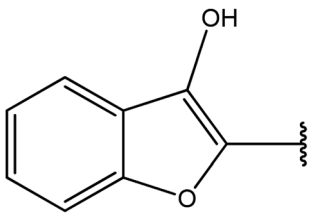
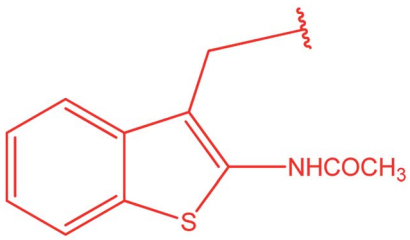
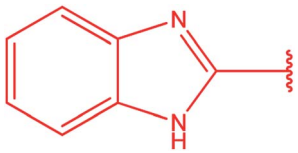
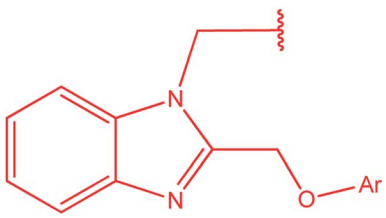
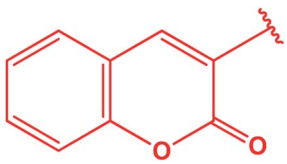
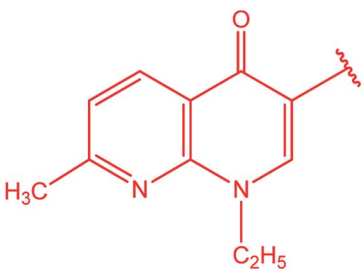
	109
	110
	111
	112
	113
	114
	115

Table 3 (*Contd.*)

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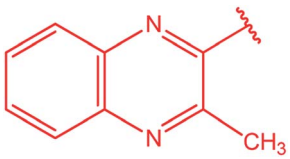
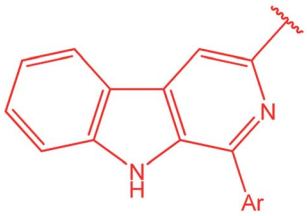
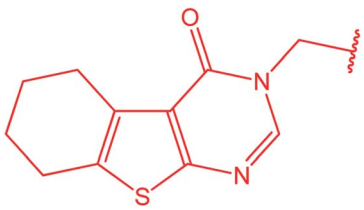
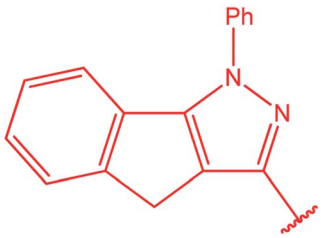
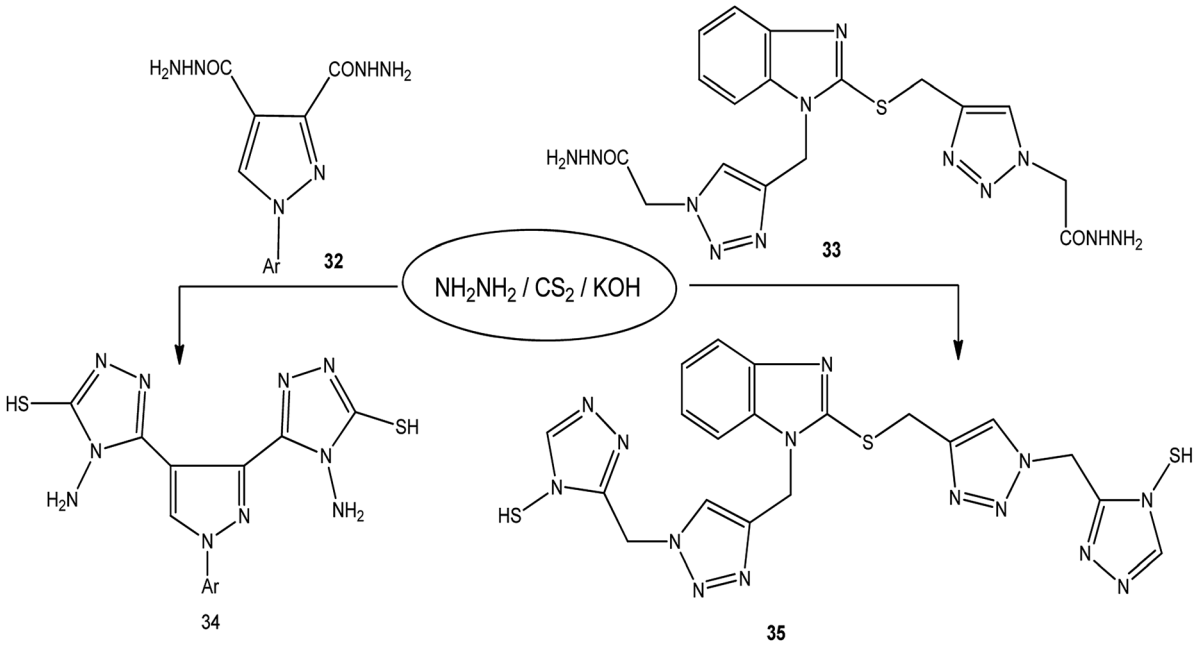
	117
	118
	119
	120

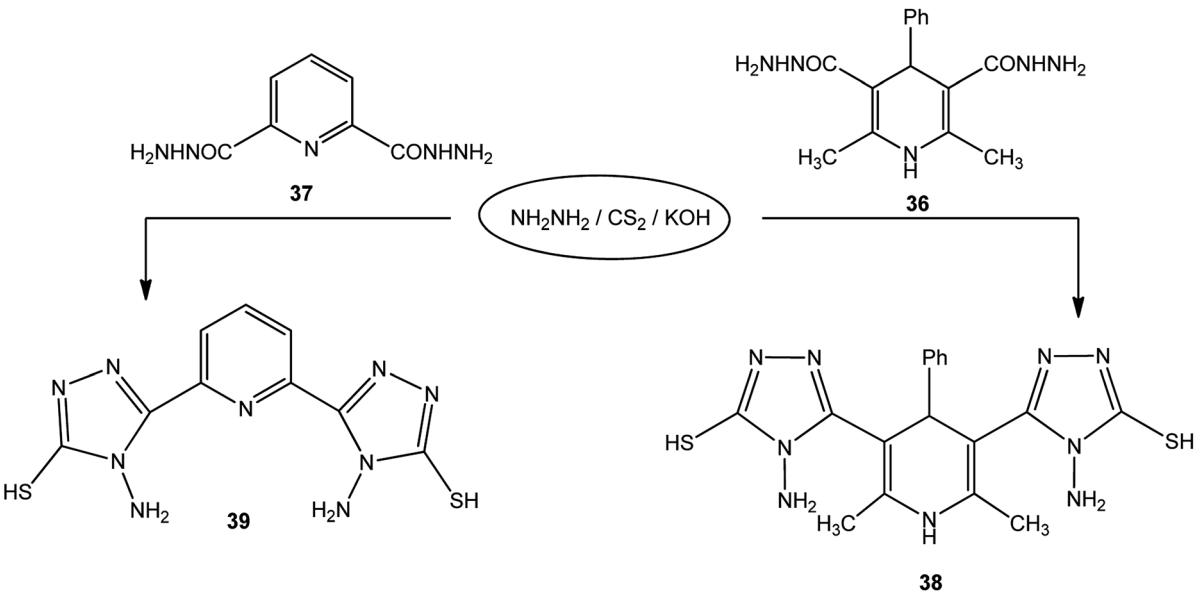
amino-3-(D-glucoheptonic-hexitol-1-yl)-1*H*-[1,2,4]triazole-5-thione (**22**),[52] and 3-(D-alditol-1-yl)-4-amino-5-mercapto-[1,2,4]triazole (**23**)[53] were reported through reactions of thiocarbohydrazide (**5**) with D(−)galactono-1,4-lactone (**18**), D-glucoheptonic-γ-lactone (**19**), and D-galactono-1,5-lactones (**20**), respectively (Scheme 7).

3. Use of potassium acyldithiocarbazates with hydrazine hydrate

Potassium acyldithiocarbazates **25** is usually prepared by a reaction between the corresponding acid hydrazides **24** and carbon disulfide in an ethanolic potassium hydroxide solution.



Scheme 12 Synthesis of bis-triazoles **34** and **35**.

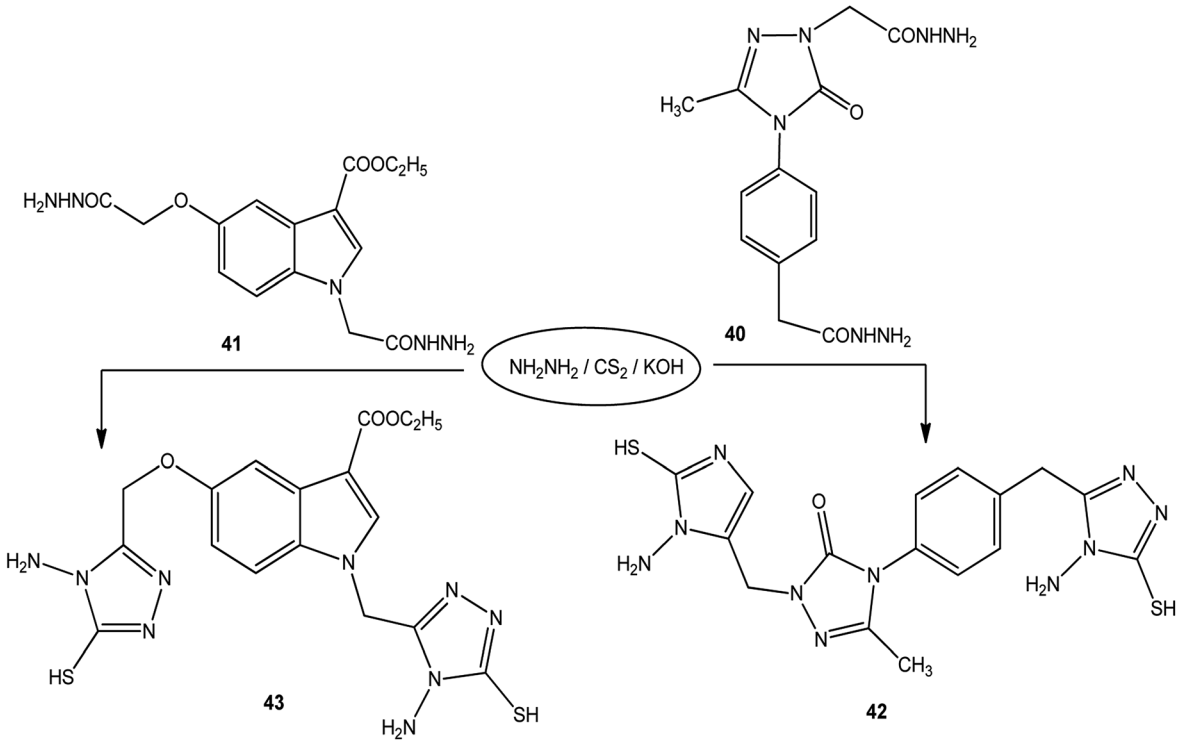


Scheme 13 Synthesis of bis-triazoles **38** and **39**.

This method was extensively used in the synthesis of numerous derivatives of 4-amino-5-mercapto[1,2,4]triazoles **2** (**3**) upon treatment with hydrazine hydrate (Scheme 8) (Table 2).

1,8-Bis-(3-mercapto-4-amino-[1,2,4]-triazol-5-yl)-octane (**27**) was achieved *via* the reaction of sebacic acid dihydrazide (**26**) with carbon disulfide and hydrazine hydrate in a molar ratio of 1 : 2 : 2 in the presence of potassium hydroxide[89] (Scheme 9).

Bis-(3-mercapto-4-amino-[1,2,4]-triazole) with an aromatic moiety was prepared under similar conditions by Zhao *et al.*[90] Thus, the reaction of 2,2′-[1,3-phenylenebis(oxy)]bis-acetic hydrazide (**28**) with CS_2/NH_2NH_2 afforded 2,2′-[1,3-



Scheme 14 Synthesis of bis-triazoles **42** and **43**.

Table 4 Derivatives of 3-substituted-4-amino-5-mercapto[1,2,4]triazoles

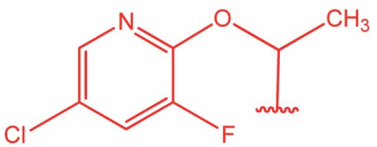
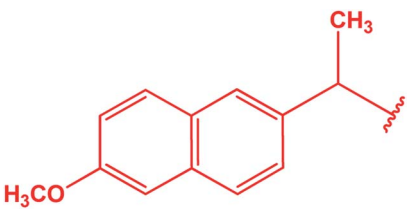
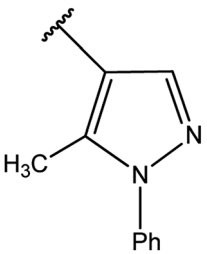
Y	Ref.
CH_3–CH_2–CH_2– & CH_3–$(CH_2)_4$–CH_2– & CH_3–$(CH_2)_5$–CH_2– & CH_3–$(CH_2)_6$–CH_2–	128
C_6H_5– & 4-$NO_2C_6H_4$– & 3-$NO_2C_6H_4$– & 3-NO_2-4-ClC_6H_3– & 2-NH_2-5-ClC_6H_3– & 4-$CH_3OC_6H_4$– & 3,4,5-$(OCH_3)_3C_6H_2$– & $C_6H_5CH_2$– & 1-naphthyl	128
	129
	130
	131

Table 4 (*Contd.*)

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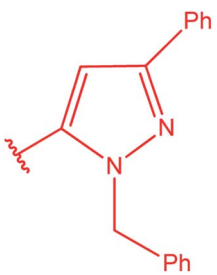
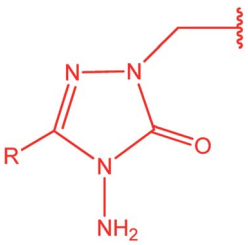
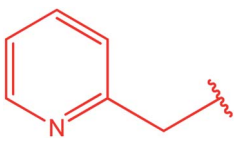
	132
	48
	133

Table 4 (*Contd.*)

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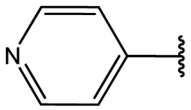
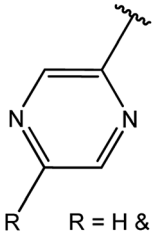
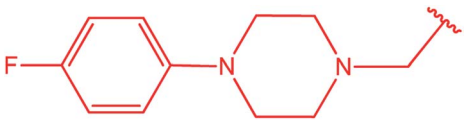
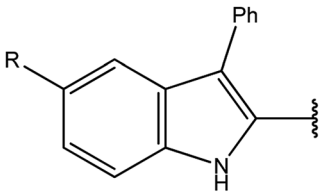
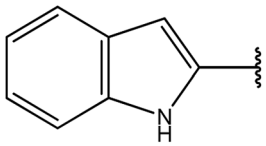
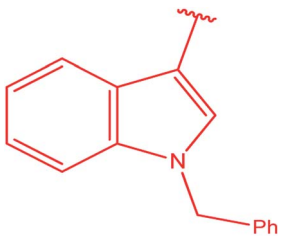
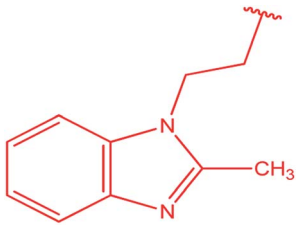
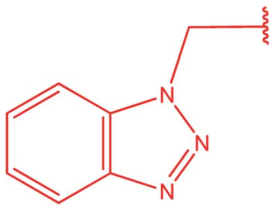
	134
 R = H & CH_3	135
	136
 R = CH_3 & OCH_3 & Cl & Br	137
	138
	139
	140
	141

phenylenebis(oxymethylene)]bis-(4-amino-3-mercapto-[1,2,4]triazole) (**29**) (Scheme 10).

3-Heteroaryl-4-amino-5-mercapto[1,2,4]triazoles (**4**) were synthesized by the treatment of the corresponding dithiocarbazate **31** with hydrazine hydrate (Scheme 11) (Table 3).

The treatment of dicarbohydrazides **32** (ref. 121) and **33** (ref. 122) with CS_2/NH_2NH_2 in the presence of KOH proceeded smoothly to afford the respective bis-triazoles **34** and **35** (Scheme 12).

In addition, pyridine dicarbohydrazide derivatives **36** (ref. 123) and **37** (ref. 124 and 125) were reacted with the above reagents under similar conditions to give **38** and **39**, respectively (Scheme 13).

Moreover, the reactions of dicarbohydrazide of triazole **40** (ref. 126) or indole derivatives **41** (ref. 127) with the same reagents in an alkaline solution furnished **42** or **43**, respectively (Scheme 14).

4. Synthesis of 5-mercapto[1,3,4] oxadiazoles with hydrazine hydrate *via* ring transformation reactions

An alcoholic solution of hydrazine hydrate achieves the ring transformation of 3-substituted-5-mercapto[1,3,4]oxadiazoles (**43**) to 3-substituted-4-amino-5-mercapto[1,3,4]triazoles **2–4** (Scheme 15) (Table 4).

5,5′-[1,4-Phenylenebis(oxymethylene)]-bis(1,3,4-oxadiazole-2-thiol) (**44**) was converted into 5,5′-[(1,4-phenylenebis(oxymethylene)]-bis(4-amino-4*H*-1,2,4-triazole-3-thiol) (**45**) upon treatment with hydrazine hydrate in dry pyridine under thermal conditions (Scheme 16).[142]

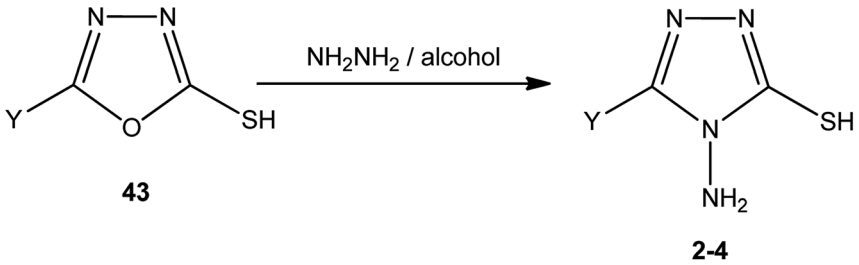
Similarly, the conversion of 5,5′-methylenebis(1,3,4-oxadiazole-2-thiol) (**46**) into 5,5′-methylenebis(4-amino-4*H*-1,2,4-triazole-3-thiol) (**47**) was achieved using an alcoholic hydrazine solution under refluxing conditions (Scheme 17).[1]

In addition, the same procedure (alcoholic hydrazine solution) was applied to the conversion of 1,4-bis(2-mercapto-1,3,4-oxadiazol-5-yl)butane-1,2,3,4-tetrol (**48**) to 1,4-bis(4-amino-5-mercapto-4*H*-1,2,4-triazol-3-yl)butane-1,2,3,4-tetrol (**49**) (Scheme 18).[143]

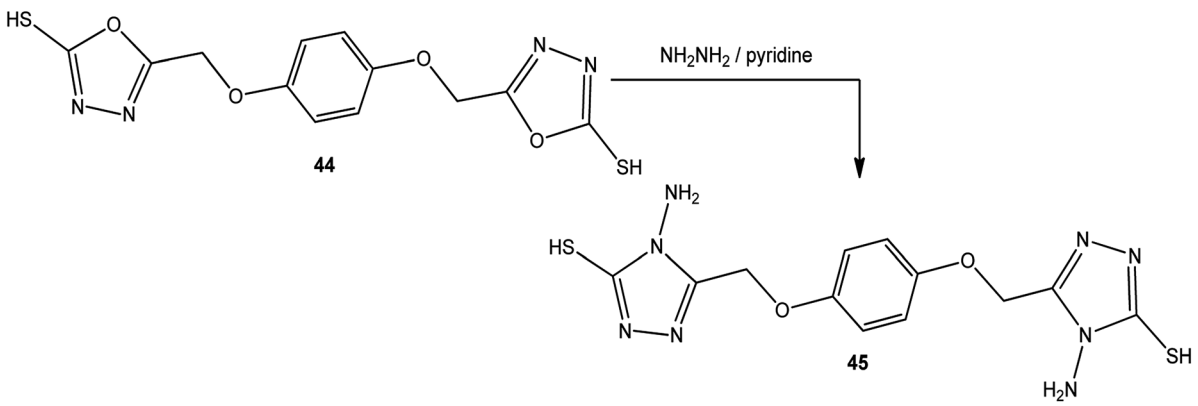
4-Amino-5-mercapto[1,2,4]triazole **1** and its 3-substituted derivatives **2–4** (Chart 2) contain both amino and mercapto groups as ready-made nucleophilic centers for the synthesis of condensed heterocyclic rings.

5. Conclusions and future directions

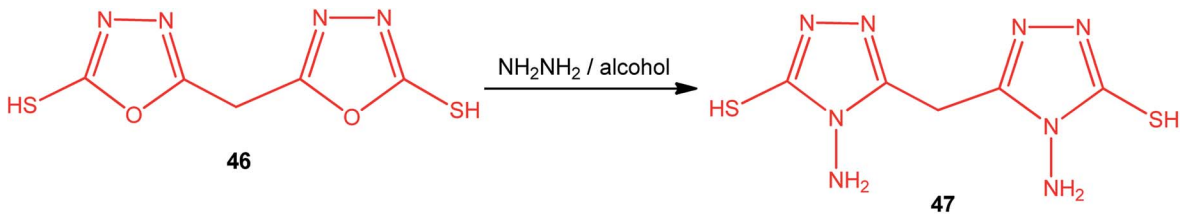
The reports in this review clearly demonstrate the elevated synthetic potential of 3-substituted-4-amino-5-mercapto[1,2,4]triazoles and bis-[4-amino-5-mercapto[1,2,4]triazoles]. Numerous scientific researchers in the fields of chemistry and pharmaceutical science are interested in the study and utilization of these compounds as building blocks in the synthesis of important bioactive compounds.



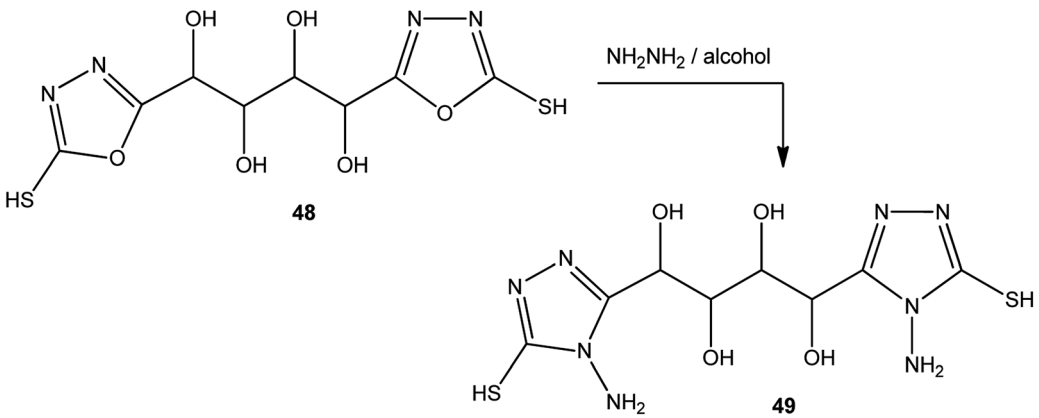
Scheme 15 Synthesis of triazoles **2–4**.



Scheme 16 Synthesis of bis-triazole **45**.



Scheme 17 Synthesis of bis-triazole **47**.



Scheme 18 Synthesis of bis-triazole **49**.

Conflicts of interest

The authors declare that there is no conflict of interests regarding the publication of this paper.

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