Research Article

Synthesis, Chemistry of ٤-allyl-٥-(٣,٤-dimethoxyphenyl)-٤*H*-١,٢,٤-triazol-٣-thiol

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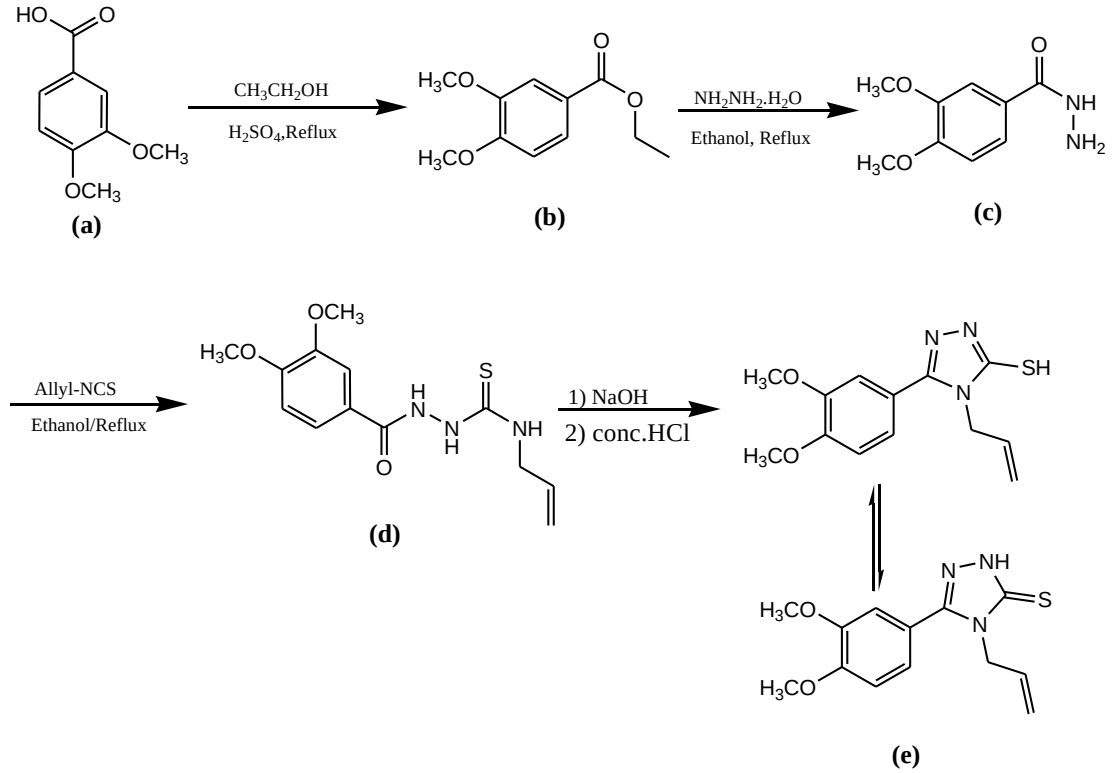
Abstract
Triazole ring moiety has a wide spectrum of biological activities. The synthesis, chemistry of ٤-allyl-٥-(٣,٤-dimethoxyphenyl)-٤*H*-١,٢,٤-triazol-٣-thiol was investigated in this study.
Key word: ١, ٢, ٤-triazole, allylisothiocyanate,

Introduction
Due to the different biological activities of ١,٢, ٤-triazole such as antimicrobial[١], anti-inflammatory[٢], antioxidant[٣], anti-convulsant[٤], antitubercular[٥] and anticancer[٦] activity. The utility of these compounds not only in biological activity but also in synthesis of other heterocyclic compounds for examples: thiazolotriazoles[٧], triazolothiadiazoles[٨], triazolothiazines[٩], and triazolothiadiazines[١٠]. There are several reported method for synthesis of triazole as Abdel Aziz H.A [١١]reaction, Ueda[١٢] method and Einhorn-Brunner[١٣] synthesis.

Results and discussion
٤-Allyl-٥-(٣, ٤-dimethoxyphenyl)-٤*H*-١,٢,٤-triazol-٣-thiol was prepared according to Scheme ١. Esterification of ٣,٤-dimethoxybenzoic acid according to Fischer esterification[١٤] using ethanol in presence of conc. H_2SO_4, treatment of ester **b** with hydrazine monohydrate ٩٥٪ give hydrazide **c**. Reaction of of hydrazide **c** with allyl isothiocyanate in presence of absolute ethanol afforded the corresponding carbazide **d.** Refluxing of carbazide **d** with ٢N NaOH followed by acidification with conc.HCl afforded the target compound **e.**



Scheme ١: Synthesis of ٤-allyl-٥-(٣,٤-dimethoxyphenyl)-٤*H*-١,٢,٤-triazol-٣-thiol

In the present study, the ١,٢,٤-triazole-٣-thiol derivatives was prepared by intramolecular cyclization of the ١,٤-disubstituted thiosemicarbazides.

Experimental

chemistry

Chemical reactions were monitored by TLC, using Merck ٩٣٨٥ pre-coated aluminum plate silica gel (Kiesel gel ٦٠) ٥ cm×٢٠ cm plates with a layer thickness of ٠.٢ mm. The spots were detected by exposure to UV-lamp at λ=٢٥٤ nm. Melting points were determined on Stuart electrothermal melting point apparatus and were uncorrected**.** NMR spectra (٥٠٠ MHz for ^{1}H, ١٢٥ MHz for ^{13}C) were observed in $CHCl_3$ on Bruker AM٤٠٠ spectrometer with tetramethylsilane as the internal standard. Splitting patterns are designated as follows: s, singlet; d, doublet; m, multiplet.

General procedure for synthesis synthesis of ٤-allyl-٥-(٣,٤-dimethoxyphenyl)-٤*H*-١,٢,٤-triazol-٣-thiol.

Heating at reflux of benzoic ٣,٤-dimethoxybenzoic acid with ethanol in the presence of concentrated sulphuric acid as a dehydrating agent afforded the corresponding esters. Hydrazinolysis of the ethyl ester derivatives with hydrazine monohydrate in refluxing ethanol afforded the corresponding carbohydrazides. The structure of the formed[١] hydrazides was confirmed by their reported melting points.

Heating at reflux of equimolar amounts of the hydrazides and allyl isothiocyanate in ethanol afforded the corresponding ١,٤-disubstituted thiosemicarbazides which was used as a crude products for the next step.

Allyl-٥-(٣,٤-dimethoxyphenyl)-٤*H*-١,٢,٤-triazol-٣-thiol.

White crystal, ٠.٣٤gm, ٧٨.٩٠٪; m.p: ١٢٤-١٢٥°C(Reported ١٢١-١٢٣°C)[٢], ^{1}H NMR (٥٠٠ MHz, $CDCl_3$) δ= ٣.٩١ (٣H, s, -OC*H*$_3$), ٣.٩٥ (٣H, s, OC*H*$_3$), ٤.٠٩-٤.١١ (٢H, m, NC*H*$_2$), ٤.٨٥ (١H, d, J_{trans}=١٧.٢٠ Hz, N-CH_2CH=C*H*$_2$), ٥.٤٠ (١H, d, *J*=١٠.٤٠ Hz, N-CH_2CH=C*H*$_2$), ٥.٩٤-٦.٠٣ (١H, m, N-CH_2C*H*=CH_2), ٦.٩٩ (١H, d, *J*=٨.٣٠ Hz, Ar-*H*) , ٧.١٩ (١H, d, *J*=٨.٣٠ Hz, Ar-*H*), ٧.٢٣ (١H, s, Ar-*H*), ١١.٢٢ (١H, s, N*H*); ^{13}C-NMR(١٢٥ MHz, $CDCl_3$) δ ٤٦.٢٥, ٥٦.٠٥, ٥٦.١٣, ١١٢.٧١, ١١٣.٩٨, ١١٧.٩٢, ١١٩.٢٦, ١٢٨.٩٦, ١٣٠.٧٨, ١٤٤.٤١, ١٤٩.٤٥, ١٥١.٢٢, ١٥٢.٧٠.

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