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SYNTHESIS OF 2,5-DIMERSAPTO-1,3,4-TIADIAZOLE DRESSING

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ANNOTATION
New dressing of 2,5-dimercapto-1,3,4-tiadiazole was synthesized. As a result of oxidation of 2,5-dimercapto-1,3,4-tiadiazole, 1,3,4-tiadiazole-2,5-disulfonic acid was obtained. Alkoloid dressing of this acid was obtained and confirmed by element Analysis of the structure of the obtained compounds, infrared IK, 1H - and 13C-NMR spectrometers.
KEYWORDS: *synthesis, 2,5-dimerkapto-1 3 4-tiadiazole, 1,3,4-tiadiazole-2,5-disulfonic acid.*

INTRODUCTION

Organic chemistry is considered one of the main tasks of the synthesis and production of substances with a new curative property. To date, the interest of chemists is of interest in heterocyclic compounds and their stumping of various dressing in the pharmaceutical and chemical industries.1,3,4-tiadiazole is a heterocyclic compound that is widely distributed and contains two nitrogen and sulfur atoms. There are several isomers of 1,3,4-tiadiazole, including 1,2,3-tiadiazole, 1,2,4-tiadiazole,1,2,5-tiadiazole,1,3, 4-tiadiazole[1]. Scheme 1

Tiadiazol flew in ditiol and dition tautically in the dressing literature, where sulfur was stored. In fact, the most numerous first structure of three tautomer structure uchiredi was recorded in the literature[2].

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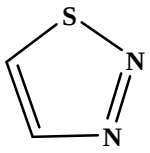
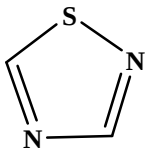
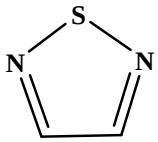
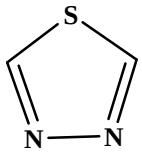
Scheme 1. Isomers of thiadiazole.

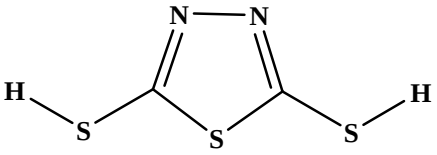

1,2,3 thiadiazole

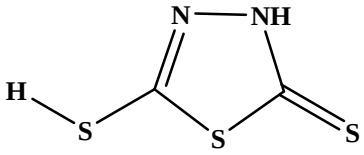

1,2,4 thiadiazole

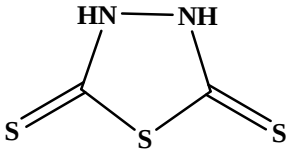

1,2,5 thiadiazole


1,3,4 thiadiazole

Scheme 2. Three tautomer shapes


-dithiol from


5-mercapto-1,3,4-thiadiazole-2(3H)-thione

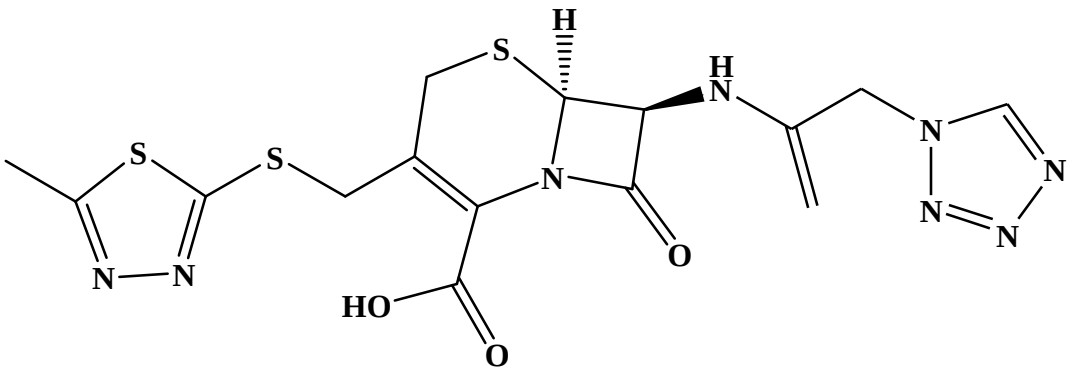

-dithione from

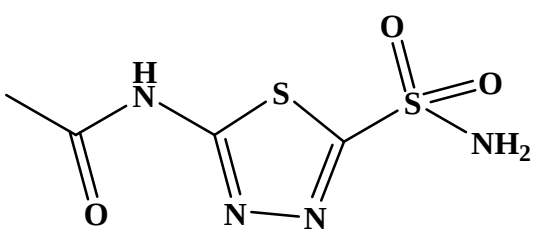
1,3,4-tiadiazole dressing is widely used in medicine,pharmaceuticals and agriculture[1]. 1,3,4-tiadiazol's new dressing is gaining etbor because it has a wide range of uses in different directions[3]. They synthesize new dressing 2,5-dimersapto-1,3,4-tiadiazol against microbes [4-6], against tuberculosis [7-11], against oxidation[12-15], against insulating[16-17], against seizures[18-20], antidepressants[21-22], against cancer[23-24], against fungi[25-26], against bacteria[27-28], against viruses[29-30] and others, so this study is has the wisdom in making.

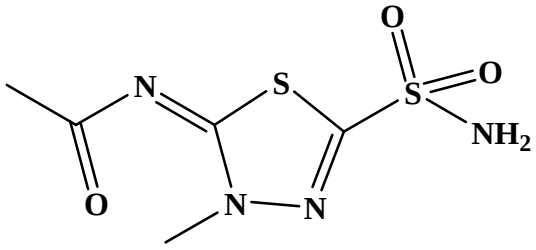
1,3,4-tiadiazole dressing has a place in agrochemistry as herbicides, fungicides, insecticides[31], pesticide, bactericides and plant growth regulator with high salinity[1].

In its composition, thiadiazol is part of the cefazolin-antibacterial property cephalorins, which are stored, acetazolamide and metazolamides are among the substances of sulfanamide nature, which are considered to be an exceptional shade as a drug[32]. Scheme 3.

Scheme 3. The ingredients are the ingredients of the drug for sale, which thiadiazol stores


Cefazolin


Acetazolamide


Metazolamide

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MATERIALS AND METHODS

Experimental

The percentage compositions of the elements (CHNS) for the compounds were determined using an elemental analyzer CHNS Model Fison EA 1108. The infrared spectra were recorded as potassium bromide discs using a Perkin-Elmer spectrophotometer GX. The ^{1}H (400 MHz) and ^{13}C (100 MHz) nuclear magnetic resonance spectra were recorded using the JEOL JNM-ECP 400 spectrometer. Ultraviolet spectra were recorded using Shimadsu UV-Vis spectrophotometer UV -2450, and EtOH was used as solvent. All reactions were monitored by TLC (aluminium foil-backed, 0.25mm silica gel 60 F254; Merck)(compound 1,2,3) **[33]:**.The IR spectra of the compounds were recorded on an Avatar-320 spectrometer in KBr pellets and mulls in mineral oil, and the ^{1}H and ^{13}C NMR spectra, on a Mercury-300 spectrometer with a working frequency of 300 MHz, solvent DMSO-d_6)(compound 4-11) **[35]**.

Synthesis of 2,5-dimercapto-1,3,4-thiadiazole (1)[33]:

A mixture of (99%) hydrazine hydrate (5 mL, 0.02 mol) and carbon disulfide (15 mL, 0.02 mol) with dry pyridine (50 mL) was refluxed for (5 h). Then the excess solvent was then distilled off, and the resulting solid was separated out by adding (25 mL) of water and (5 mL) of hydrochloric acid. The mixture was then filtered and the solid was recrystalized from ethanol.

Synthesis of 2,5-dihydrazino-1,3,4-thiadiazole (2) [33]:

To 2,5-dimercapto-1,3,4-thiadiazole (1) (1.5 g,0.01 mol) dissolved in ethanol, hydrazine hydrate (5 mL,0.02mol) was added dropwise with stirring and the mixture was then refluxed for (6 h), then the excess solvent was distilled off. Filtered the resulting solid which was separated out on cooling and recrystalized from ethanolto give the desired product.

Synthesis of 2,5-di(arylhydrazone)-1,3,4-thiadaizoles (3) [33]:

A mixture of (2) (1.46 g, 0.01 mol) in absolute ethanol (20 mL) and appropriate aldehyde (0.02 mol) was refluxed on water bath at (80 C) for (8 h). The crude product was isolated and recrystalized from ethanol.

Synthesis of 1,3,4-Thiadiazole-2,5-disulfonic acid(4) [35]:

A 2.5% aqueous solution of 6.32 g (0.04 mol) of KMnO4 was added dropwise with stirring at room temperature over a period of 3 h to an aqueous solution of 1.5 g (0.01 mol) of 2,5-dimercapto-1,3,4-thiadiazole. The mixture was heated with stirring on a water bath until its complete decolorization. The precipitate of manganese dioxide was filtered off, the filtrate was evaporated, and the dry residue was washed with alcohol. 1,3,4-Thiadiazole-2,5-disulfonic acid **4** was recrystallized from ethanol-water, 10 : 1.

Synthesis of alkaloid- and amine-containing salts of 1,3,4-thiadiazole-2,5-disulfonic acid, (4--10) [35].

An aqueous solution of 0.02 mol of appropriate secondary amine or alkaloid was added dropwise with stirring over a period of 1 h to an aqueous solution of 2.46 g (0.01 mol) of 1,3,4-thiadiazole-2,5-disulfonic acid **4**. The mixture was stirred at room temperature for 12 h and left in a vacuum desiccator to remove the solvent. The solid residue was washed with alcohol, filtered off, and recrystallized from ethanol-water, 10 : 1.

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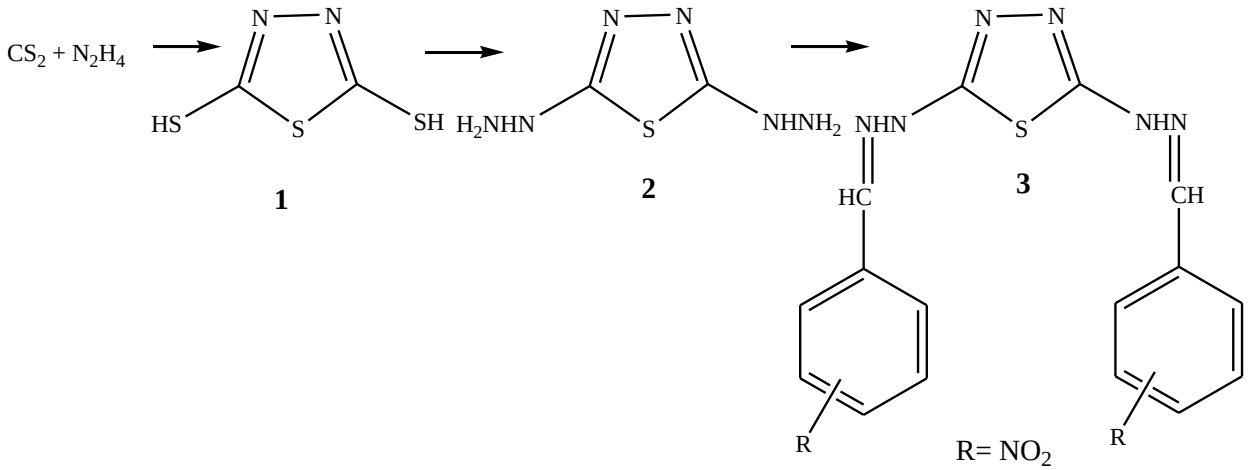
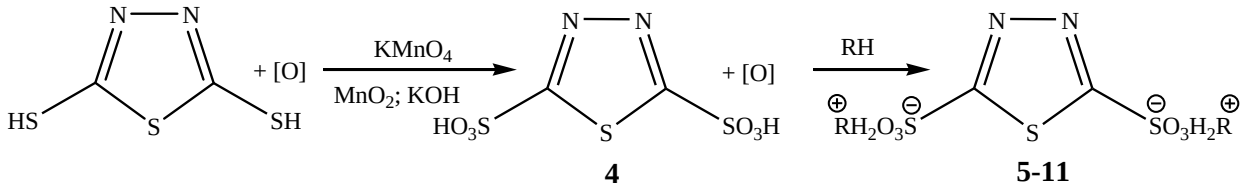
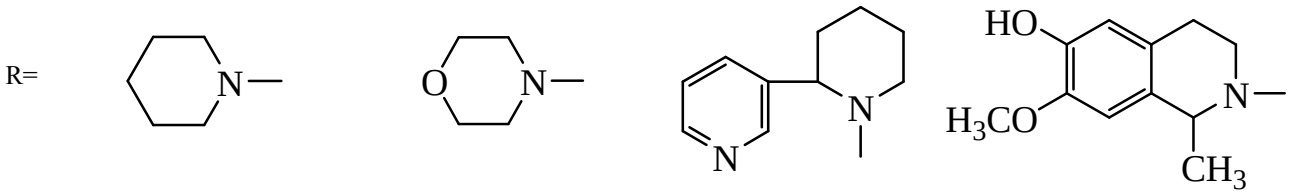
RESULTS AND DISCUSSION







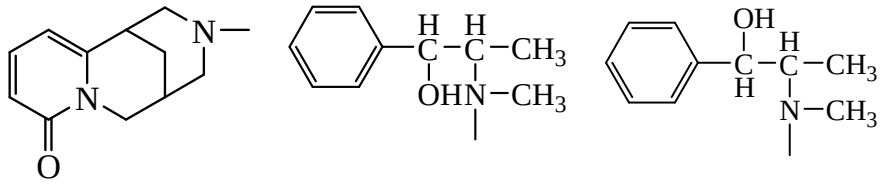


Table 1: Elemental analysis and physical properties of prepared compounds 1-11[33,35]

№	Formula	% Yield	m.p(C)	Elemental analysis calc. (found)			
				%C	%H	%N	%S
1	$C_2H_2N_2S_3$	62	164-166	15.99 (15.98)	1.34 (1.35)	18.65 (18.64)	64.03 (64.04)
2	$C_2H_6N_6S$	60	198-200	16.43 (16.44)	4.14 (4.15)	57.49 (57.50)	21.94 (21.93)
3	$C_{16}H_{12}N_8O_4S$	65	187-189	46.60 (46.61)	2.93 (2.92)	27.17 (27.18)	7.78 (7.79)
-4	$C_2H_2N_2O_6S_3$	98.0	>350	9.67 (9.76)	0.90(0.81)	11.30(11.38)	-
5	$C_{12}H_{24}N_4O_6S_3$	76.9	>350	34.7(34.62)	5.72(5.77)	13.35(13.46)	-
6	$C_{10}H_{20}N_4O_8S_3$	66.9	>350	28.5(28.57)	4.64(4.76)	13.25(13.33)	-
7	$C_{22}H_{30}O_6S_3$	42.8	>350	46.39(46.32)	5.20(5.26)	14.84(14.74)	-
8	$C_{24}H_{30}N_4O_6S_3$	52.6	>350	50.81(50.88)	5.38(5.30)	9.81(9.89)	-
9	$C_{24}H_{30}N_6O_8S_3$	70.0	>350	46.00(46.01)	4.70(4.79)	13.51(13.42)	-
10	$C_{22}H_{32}N_4O_8S_3$	50.1	>350	45.89(45.83)	5.50(5.56)	9.82(9.72)	-
11	$C_{22}H_{32}N_4O_8S_3$	56.1	>350	45.77(45.83)	5.44(5.56)	9.70(9.72)	-

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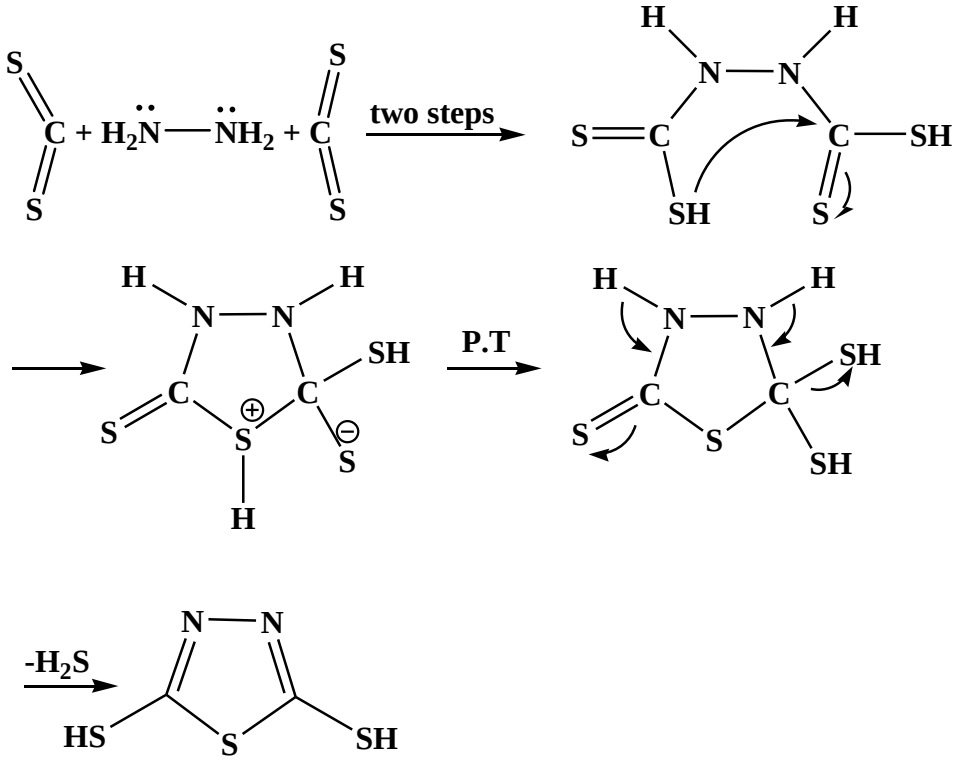
The mechanism for the formation of 2,5-dimercapto-1,3,4-thiadiazole (1) is shown in Scheme 5:



Scheme 5. Reaction mechanism for the preparation of compound 1.

Electronic Absorption Spectra:

The electronic spectra of the synthesized diaryl hydrazone compounds (3-12) dissolved in (EtOH) gave the (λ_{max}) absorption bands at about (224-497 nm) for all compounds. The (λ_{max}) are listed in Table 2:

Table 2: Electronic data of compounds 3

№	Ar group	λmax (nm)/ Ethanol
1	3-nitrophenyl	266

The spectral pattern data (Table 2) is found to be quite similar to other 1,3,4-thiadiazole derivatives reported in earlier literature [34].

FTIR spectra

The important diagnostic bands in the IR spectra were assigned and the bands positions are compiled in Table 3. The FTIR spectrum of compound (1) showed a medium intensity band at 1624 cm^{-1} that could correspond with (C=N) stretching in the vicinity of 1,3,4-thiadiazole ring. In this spectrum there are two other characteristic bands at 3200 and 2550 cm^{-1} due to (N-H) and (S-H) stretching
vibrations, respectively. From this we can say that this compound can exist in the thiol and thion form. On the other hand, the FTIR spectrum of 2,5-dihydrazino-1,3,4-thiadiazole (**2**) showed the disappearance of band at 2550 cm^{-1} for υ(C-S) with a new band at 3396, 3271 and 3200 cm^{-1} which are assigned as υ_{asym} (NH_2), υ sym (NH_2) and υ(NH) group, respectively. These bands proved the conversion of compound (**1**) to (**2**). Moreover,compound (**2**) showed characteristic IR bands at 1284cm^{-1} υ(N-C=C), 1109cm^{-1} υ(C-S-C) thion ether linkage and 1247cm^{-1} υ(N-N). A comparison of the FTIR spectrum of compound (**2**) with the spectra of compounds (**3**) revealed in the case of these compounds (υ_{asym} , υ_{sym} NH_2) were absent. The other characteristic bands are listed in Table 3.

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Table 3: FTIR data of compounds 3

№	Ar group		FTIR bands (cm^{-1}) υ (C=N)
3	3-nitrophenyl	1623	Asymmetric ($ArNO_2$)N-O stretching.,1523, Symmetric ($ArNO_2$)N-O stretching., 1350

Table 4: 1H- and 13C-NMR data of compounds 3

№	^{1}H- and ^{13}C-NMR spectral data
3	.^{1}H-NMR (400 MHz, DMSO-d_6): δ 5.43, 5.87 (2H, 2s, 2 N=CH-), 7.78-8.52 (8H, m, Ar-H), 9.74, 10.06 (2H, 2s, 2-NH,exchangeable with D_2O). ^{13}C-NMR (100MHz, DMSO-d_6): 115.65, 117.32 (2C, thiadiazole carbons), 122.61, 125.42 (2C, 2 N=CH-),134.11-140.76 (12C, aromatic carbons).

The structure and composition of 4-11 were confirmed by IR, ^{1}H NMR, and ^{13}C NMR spectroscopy,and also by elemental analysis[35].

The IR spectra of all the onium salts synthesized contain characteristic absorption bands of the thiadiazole ring at 680, 730, 1400, and 1500 cm^{-1},bands in the range 656-649 cm^{-1} assigned to -SO3 – vibrations, and absorption bands at 3501+/-3421 cm^{-1}characteristic of secondary ammonium salts [36].

In the ^{1}H NMR spectra of **4-11**, recorded in DMSO-d_6, protons of the ammonium group appear as an ill-resolved multiplet centered at 14.6 ppm. The chemical shifts of signals from protons of alkaloid and secondary amine fragments have typical values [37].

In the 13C NMR spectrum of 1,3,4-thiadiazole-2,5-disulfonic acid 4, the heterocyclic carbon atom appears as a singlet at 168.0 ppm. In the ^{13}C NMR spectrum of piperidinium salt of 1,3,4-thiadiazole-2,5-disulfonic acid **5**, the heterocyclic carbon atom appears as a singlet at 158.4 ppm, and carbon atoms of the piperidine fragment give signals at 23.1, 25.5, 26.0, 42.5,and 45.5 ppm[38]

CONCLUSION

This article examined the substances synthesized, studied the reaction time was synthesized in 2,5-dimercapto-1,3,4-tiadiazole dressing. The resulting substance was confirmed in a spectroscopic analysis. As a result of oxidation of 2,5-dimercapto-1,3,4-tiadiazole 1,3,4-tiadiazole-2,5-disulfonic acid is obtained and the synthesis of alkaloid dressing of this compound.

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