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NOTE

Synthesis of Some Thiazolidinopyrazolines from Rhodanine

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Rhodanine is selected as a starting material due to the reason that at 5 position it contains an active methylene group which readily interacts with aromatic aldehydes to form benzylidene derivatives of rhodanine. The benzylidene compound derived from rhodanine possesses α : β unsaturated carbonyl group. They react with phenylhydrazine to form pyrazoline ring by cyclisation.

Rhodanine forms an important series of heterocyclic compounds. Rhodanine and its derivatives have been found to be fungicides,[1] insecticides[2] and mothicide[3]. Some rhodanine compounds have also been tested positive for amoebicidal activity[4] while some show anthelmintic activity[5].

Pyrazolines are another important series of heterocyclic compounds associated with a wide range of pharmaceutical properties such as antidiabetic[6], analgesic[7], antibacterial[8] and antitubercular[9] agents. Therefore, the compounds in which both the rhodamine and pyrazoline rings are fused together must have these properties. Taking this veiw we synthesised these types of compounds by more than one method. One important change was observed that in place of rhodaninopyrazoline the product obtained was 2-oxo-thiazolidinopyrazoline.

In order to synthesise rhodaninopyrazolines rhodanine(I) was condensed separately with *o*-methoxybenzaldehyde and *p*-methoxybenzaldehyde in presence of fused sodium acetate and glacial acetic acid and to give the benzylidenes (II) and (IV). Which on reaction with hydrazine, phenylhydrazine and 2 : 4 dinitrophenylhydrazine gave 2-oxothiazolidinopyrazolines (X)–(XV) respectively. The structure of the compounds are supported by their analytical and spectral results.

Compounds (X–XV) were screened for their antifungal activity against *Aspergillus flavus* Link ex Fries as the test fungi by Food Poison Technique at (0.05, 0.10, 0.15) concentration in Czapak Dox Agar medium. Since the diameters of the colonies are lesser, all the compounds are active.

All the melting points are uncorrected. The IR spectrum was recorded on Beckman DU-2.

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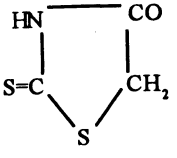
TABLE-1
ANALYTICAL AND SPECTRAL DATA OF 2-OXOTHIAZOLIDINOPYRAZOLINES

Compound No.	m.p. (°C)	Yield (%)	m.f.	I.R. peaks (cm^{-1})
(X)	325	70	$C_{11}H_{11}O_2N_3S$	1580 ν(C=N), 1680 ν(C=O), 3109 ν(NH)
(XI)	215	60	$C_{17}H_{15}O_2N_3S$	1590 ν(C=N), 1695, ν(C=O), 3280 ν(NH)
(XII)	226	65	$C_{17}H_{14}O_5N_6S$	1230 ν(C—NO_2), 1360 ν(C—NO_2) 1590 ν(C=N), 3330ν (NH)
(XIII)	311	70	$C_{11}H_{11}N_3O_2S$	1585 ν(C=N), 1680 ν(C=O), 3120 ν(NH)
(XIV)	276	65	$C_{17}H_{15}N_3OS$	1580 ν(C=N), 1680 ν(C=O), 3290 ν(NH)
(XV)	304	65	$C_{17}H_{14}N_6O_5S$	1260 ν (C—NO_2), 1365 ν(C—NO_2), 1590 ν(C=N), 3190ν (NH)

All the compounds gave satisfactory C, H and N-analysis



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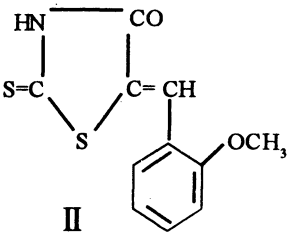

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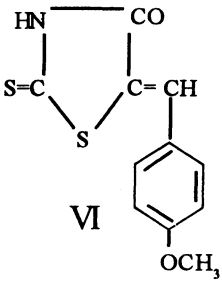

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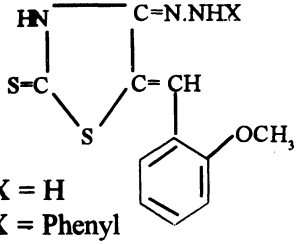
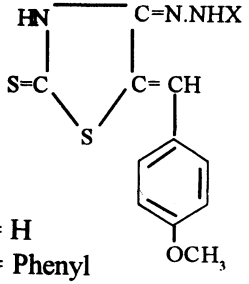
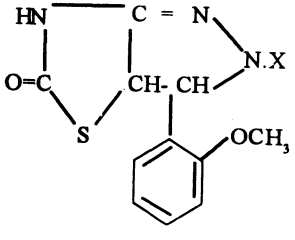


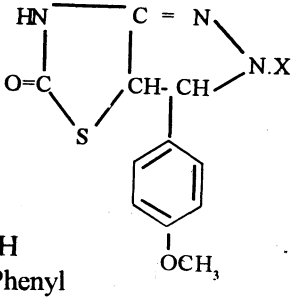
III. X = H
IV. X = Phenyl
V. X = 2:4 dinitrophenylhydrazine



VII. X = H
VIII. X = Phenyl
IX. X = 2:4 dinitrophenylhydrazine



X X = H
XI. X = Phenyl
XII. X = 2:4 dinitrophenylhydrazine



XIII. X = H
XIV. X = Phenyl
XV. X = 2:4 dinitrophenylhydrazine

Benzylideno Rhodanine (II): Rhodanine (5.5 g), fused sodium acetate (3 g) and *o*-methoxybenzaldehyde (5 mL) in glacial acid (20 mL) were refluxed for 1 h. A solid started appearing within 0.5 h. When the reaction was complete the mixture was cooled and the solid separated, washed with water and alcohol. It was crystallised from glacial acetic acid.

Benzylidene Rhodanine (VI): Rhodanine (5.5 g), fused sodium acetate (3 g) and *o*-methoxybenzaldehyde (5 mL) in glacial acetic acid (20 mL) were refluxed for 1 h and the product obtained was washed with alcohol and water and crystallised from glacial acetic acid.

2-Oxothiazolidinopyrazolines (X–XII): A mixture of benzylidene rhodanine(II) and three different hydrazines, *viz.*, hydrazine (2 g), phenylhydrazine (2 g) and 2 : 4 dinitrophenylhydrazine (2 g) for three different sets of experiments along with sodium acetate (2 g) in absolute ethanol (20 mL) were refluxed for 2 h and then cooled and kept overnight to form (X), (XI) and (XII) respectively. They were separated out, washed and crystallised from glacial acetic acid.

2-Oxothiazolidinopyrazolines (XIII–XV): A mixture of benzylidene rhodanine (VI) and three different hydrazine derivatives (2 g), *viz.*, hydrazine, phenylhydrazine and 2 : 4 dinitrophenyl hydrazine for three different sets of products (XIII, XIV and XV) were refluxed with sodium acetate (2 g) in absolute ethanol (20 mL). The product so obtained was, separated, washed and crystallised from glacial acetic acid.

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