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Synthesis and Characterization of Azopyrazole derivatives

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ABSTRACT

In present investigation 3-(2-(4-substitutedphenyl)-hydrazono)-pentane-2,4-dione synthesized by using substituted aniline and acetyl acetone in presence of sodium nitrite. The synthesized compound further reacted with isoanizide and semicarbazide to obtained 4-(4-substitutedphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanoneand4-(4substitutedphenyl) diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide. All the newly synthesized compounds were characterized on the basis of elemental analysis, respective melting point, IR and ^{1}H-NMR spectroscopic techniques.

Keywords: Aniline, Azopyrazole, IR, ^{1}H-NMR.

INTRODUCTION

Pyrazole is an important heterocyclic compound containing three carbon atom and two adjacent nitrogen atom. Synthesis of azopyrazole derivatives from aniline bears a great attention in recent years. Azo pyrazole derivative exhibeat wide variety of biological activities such as antibacterial, analgesic, antifungicidal anti-inflamatory [1-5]. By considering this point of view the objective of present work is to prepare new derivatives of azopyrazole and characterized by different spectroscopic techniques.

METHODOLOGY

All chemicals used were of the analytical reagent (AR) grade and of highest purity available and purchased from SD-Fine Chem Limited. Melting points were determined with an Electro thermal 9100 apparatus and are uncorrected.

Synthesis of 3-(2-(4-substitutedphenyl)-hydrazono)-pentane-2,4-dione

4-substituted aniline (0.01 mole) was dissolved in a mixture of concentrated HCl (8 ml) and water (6 ml) and cooled to ^{0}C on ice bath. The cold diazonium salt solution was filtered into a cooled solution of acetyl acetone in presence of sodium nitrite, sodium acetate (0.01 mole) in ethanol (20 ml) and stirred for 2 hrs and resulting solid was filtered, dried and recrystallized by ethanol. (2a-c)

Synthesis of 4-(4-substitutedphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanone

A mixture of 3-(2-(4-substitutedphenyl)-hydrazono)-pentane-2,4-dione (0.01 mole) and isoaniazide (0.015 mole) in glacial acetic acid (15 ml) is refluxed for 4-5 hrs. The resulting mixture was concentrated and allowed to cool. The resulting solid was filtered, washed, dried and recrystallised from ethanol to obtained pure compound. (3a-c)

Synthesis of 4-(4-substitutedphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide

A mixture of 3-(2-(4-substitutedphenyl)-hydrazono)-pentane-2,4-dione (0.01 mole) and semicarbazide (0.015 mole) in glacial acetic acid (15 ml) is refluxed for 4-5 hrs. The resulting mixture was concentrated and allowed to cool. The resulting solid was filtered, washed, dried and recrystallised from ethanol to obtained pure compound. (4a-c)

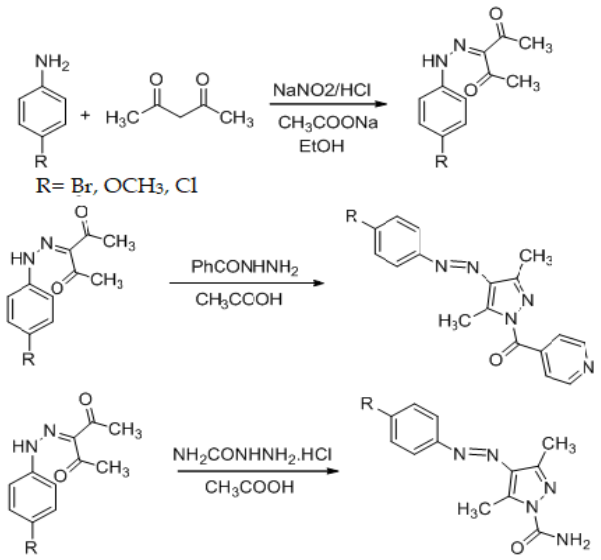

Fig 1: Scheme of Reaction

Table 1: Physiochemical data of all synthesized compounds.

Sr No	Comp.	R	Molecular Formula	Mol. Wt.	% Yield	Elemental Analysis			
						M. P. (^{0}C)	% C	% H	% N
1	2a	Br	$C_{11}H_{11}BrN_2O_2$	283.12	71%	110	46.66	3.92	9.89
2	2b	OCH_3	$C_{12}H_{14}N_2O_3$	234.25	69%	84	61.53	6.02	11.96
3	2c	Cl	$C_{11}H_{11}ClN_2O_2$	238.67	70%	107	55.36	4.65	11.74
4	3a	Br	$C_{17}H_{14}BrN_5O$	384.23	53%	171	53.14	3.67	18.23
5	3b	OCH_3	$C_{18}H_{17}N_5O_2$	335.36	59%	179	64.47	5.11	20.88
6	3c	Cl	$C_{14}H_{14}ClN_5O$	339.78	62%	169	60.09	4.15	20.61
7	4a	Br	$C_{12}H_{12}BrN_5O$	322.16	67%	132	44.74	3.75	21.74
8	4b	OCH_3	$C_{13}H_{15}N_5O_2$	273.29	65%	133	57.13	5.53	25.63
9	4c	Cl	$C_{12}H_{12}ClN_5O$	277.71	70%	129	51.90	4.36	25.22

RESULTS AND DISCUSSION

All the newlly synthesized compound were characterized on the basis of IR and ^{1}H-NMR spectroscopic techniques.

Spectral data of 3-(2-(4-bromophenyl)-hydrazono)-pentane-2,4-dione (2a)
IR (KBr) ν_{max}: cm^{-1} : 3318(-NH), 3072(-Ar-CH), 2927(-Al-CH), 1683(-C=O), 1489(-C=N), 1584(-C=C), 1065(C-O), 741(C-Br): ^{1}H-NMR (DMSO-d_6) δ: 14.2(1H,s,NH), 7.6(2H,m,Ar-H), 7.3(2H,d,Ar-H), 2.6(3H,s,CH_3), 2.3(3H,s,CH_3)

Spectral data of 3-(2-(4-methoxyphenyl)-hydrazono)-pentane-2,4-dione (2b)
IR (KBr) ν_{max}: cm^{-1} : 3315(-NH), 3069(-Ar-CH), 2931(-Al-CH), 1680(-C=O), 1501(-C=N), 1590(-C=C), 170(C-O), 740(C-Br): ^{1}H-NMR (DMSO-d_6) δ: 14.3(1H,s,NH), 7.7(2H,m,Ar-H), 7.3(2H,d,Ar-H), 2.5(3H,s,CH_3), 2.3(3H,s,CH_3)

Spectral data of 3-(2-(4-chlorophenyl)-hydrazono)-pentane-2,4-dione (2c)
IR (KBr) ν_{max}: cm^{-1} : 3320(-NH), 3075(-Ar-CH), 2929(-Al-CH), 1681(-C=O), 1485(-C=N), 1584(-C=C), 1067(C-O), 741(C-Br): ^{1}H-NMR (DMSO-d_6) δ: 14.5(1H,s,NH), 7.5(2H,m,Ar-H), 7.4(2H,d,Ar-H), 2.6(3H,s,CH_3), 2.3(3H,s,CH_3)

Spectral data of 4-(4-bromophenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanone (3a)
IR (KBr) ν_{max}: cm^{-1} : 3060(-Ar-CH), 2921(-Al-CH), 1670(-C=O), 1514(-C=N), 1588(-C=C), 1171(C-O), 741(C-Br): ^{1}H-NMR (DMSO-d_6) δ: 7.8(4H,m,Ar-H), 7.5(4H,m,Ar-H), 2.5(3H,s,CH_3), 2.2(3H,s,CH_3)

Spectral data of 4-(4-methoxyphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanone (3b)
IR (KBr) ν_{max}: cm^{-1} : 3065(-Ar-CH), 2920(-Al-CH), 1674(-C=O), 1520(-C=N), 1590(-C=C), 1160(C-O): ^{1}H-NMR (DMSO-d_6) δ: 7.6(4H,m,Ar-H), 7.2(4H,m,Ar-H), 2.4(3H,s,CH_3), 2.2(3H,s,CH_3)

Spectral data of 4-(4-Chlorophenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-yl)(pyridine-4-yl)methanone (3c)
IR (KBr) ν_{max}: cm^{-1} : 3064(-Ar-CH), 2931(-Al-CH), 1672(-C=O), 1515(-C=N), 1591(-C=C), 1170(C-O): ^{1}H-NMR (DMSO-d_6) δ: 7.6(4H,m,Ar-H), 7.4(4H,m,Ar-H), 2.6(3H,s,CH_3), 2.4(3H,s,CH_3)

Spectral data of 4-(4-bromophenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide (4a)
IR (KBr) ν_{max}: cm^{-1} : 3062(-Ar-CH), 2935(-Al-CH), 1665(-C=O), 1540(-C=N), 1587(-C=C), 1165(C-O), 741(C-Br): ^{1}H-NMR (DMSO-d_6) δ: 7.2(2H,d,Ar-H), 7.8(2H,d,Ar-H), 2.2(3H,s,CH_3), 2.3(3H,s,CH_3)

Spectral data of 4-(4-methoxyphenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide (4b)
IR (KBr) ν_{max}: cm^{-1} : 3069(-Ar-CH), 2940(-Al-CH), 1664(-C=O), 1541(-C=N), 1585(-C=C), 1161(C-O), 735(C-Br): ^{1}H-NMR (DMSO-d_6) δ: 7.1(2H,d,Ar-H), 6.9(2H,d,Ar-H), 2.3(3H,s,CH_3), 2.5(3H,s,CH_3)

Spectral data of 4-(4-chlorophenyl)diazenyl)-3,5-dimethyl-1H-pyrazol-1-carboximide (4c)
IR (KBr) ν_{max}: cm^{-1} : 3070(-Ar-CH), 2933(-Al-CH), 1664(-C=O), 1543(-C=N), 1581(-C=C), 1169(C-O), 743(C-Br): ^{1}H-NMR (DMSO-d_6) δ: 7.4(2H,d,Ar-H), 7.6(2H,d,Ar-H), 2.5(3H,s,CH_3), 2.8(3H,s,CH_3)

CONCLUSION

During this study some azopyrazole have been synthesized by using isoaniazide and semicarbazide. Spectroscopic data obtained matches with the structure of compounds.

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Conflicts of interest: The authors stated that no conflicts of interest.

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