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Facile synthesis, characterization and spectral evaluation of some new pyrazolone derivatives

ALOK K. PAREEK*, P.E. JOSEPH and DAYA S.SETH

School of Chemical Sciences, Department of Chemistry,
ST. John's College Agra - 282 002 (India).

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ABSTRACT

The target compounds N^1-(2, 5-dichloro benzoyl)-3-methyl-4-(substituted phenyl hydrazono)-5-pyrazolone derivatives (2a-2t) have been synthesized by the condensation of different ethyl 2,3-dioxo butyrate-2-(substituted benzeneazo) phenyl hydrazone (1a-1t) with 2,5-dichloro phenyl hydrazine by using glacial acetic acid as a condensing agent. The structural assignment are based on their melting point, colour, elemental analysis, spectral data (IR) and chemical properties.

Key words: Pyrazolone, Subs. ethyl acetohydrazones, Subs. phenyl hydrazine, condensation, spectral data.

INTRODUCTION

Pyrazolone ring has fairly accessible properties and hence attracted much attention in the wide research area of synthetic chemistry. An intensive literature survey of the synthesis for various pyrazolone derivatives has been carried out,pyrazolone derivatives have been possess for their wide range of biological activity[1]. it also reported to have anaesthetic properties[2,3] and antibacterial activities[4,5]. Some derivatives of pyrazolone have also been found to possesses, antidiabetic[6],analgesic[7], diuretic[8], antitumor[9], anticonvulsant[10], and also antibacterial[11], antifingal[12] activity .

Pyrazolone have attractive target in organic chemistry because of their reactivity and biologi cal significance such as anticancer[13], antitubercular[14], antihypertensive[15], microbials. In continuation of our previous work[16,17]. keeping these facts in view we report here the synthesis of some new N^1(2,5 dichloro benzoyl)-3-methyl-4-(substituted phenyl hydrazono)- 5 -pyrazolones. The present communication deals with the reaction of subs. ethyl aceto hydrazones with 2,5-dichloro phenyl hydrazine in presence of glacial acetic acid.

EXPERIMENTAL

MATERIAL AND METHODS

All the chemicals were used for synthesis are of analytical grade and was obtained from Sigma-Aldrich Co. Melting points are taken in open capillery tubes and were uncorrected. Purity of the newly synthesized compounds was checked by TLC. IR spectra were recorded on Perkin-Elmer spectrum RX-1 FT-IR spectrophotometer using Kbr disc method. The physical and analytical data of the synthesized compounds is presented in the Table-1 and spectral evaluation are recorded in the Table-2.

Table 1: Physical & Analytical data of the Synthesized Compounds (2a-2t)

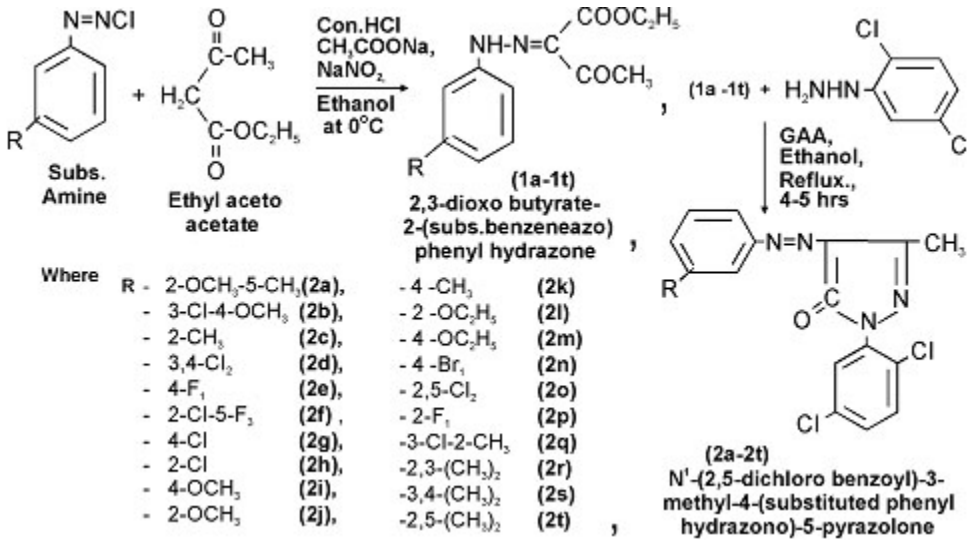
Codes	Molecular Formula	Molecular Weight	M.P (°C)	Yield %	Colour	% Analytical Data					
						C% cal.%	(found)	H% cal.%	(found)	N% cal.%	(found)
2a	$C_{18}H_{15}N_4O_2Cl_2$	390.26	125°	57.00	sunrise	55.39	(55.41)	3.87	(3.88)	14.35	(14.38)
2b	$C_{17}H_{12}N_4O_2Cl_3$	410.68	122°	64.28	light orange	49.72	(49.73)	2.94	(2.95)	13.64	(13.67)
2c	$C_{17}H_{13}N_4O_1Cl_2$	360.23	116°	62.44	dirty orange	56.68	(56.69)	3.63	(3.61)	15.55	(15.58)
2d	$C_{16}H_9N_4O_1Cl_4$	415.10	129°	66.73	light mango yellow	46.29	(46.31)	2.18	(2.20)	13.50	(13.53)
2e	$C_{16}H_{10}N_4O_1Cl_2F_1$	364.20	096°	80.62	mango yellow	52.76	(52.78)	2.76	(2.78)	15.38	(15.42)
2f	$C_{17}H_9N_4O_1Cl_3F_3$	448.66	156°	56.03	light orange	45.51	(45.53)	2.02	(2.03)	12.48	(12.52)
2g	$C_{16}H_{10}N_4O_1Cl_3$	380.66	132°	70.31	light mango yellow	50.48	(50.50)	2.64	(2.65)	14.71	(14.75)
2h	$C_{16}H_{10}N_4O_1Cl_3$	380.66	183°	62.04	dirty light yellow	50.48	(50.49)	2.64	(2.62)	14.71	(14.74)
2i	$C_{17}H_{13}N_4O_2Cl_2$	376.23	123°	65.15	orange	54.27	(54.28)	3.48	(3.46)	14.89	(14.91)
2j	$C_{17}H_{13}N_4O_2Cl_2$	376.23	120°	59.04	light buff	54.27	(54.29)	3.48	(3.49)	14.89	(14.93)
2k	$C_{17}H_{13}N_4O_1Cl_2$	360.23	108°	69.24	sporty yellow	56.68	(56.70)	3.63	(3.64)	15.55	(15.60)
2l	$C_{18}H_{15}N_4O_2Cl_2$	390.26	116°	62.93	dirty sunset	55.39	(55.40)	3.87	(3.86)	14.35	(14.37)
2m	$C_{18}H_{15}N_4O_2Cl_2$	390.26	128°	65.57	camp fire	55.39	(55.41)	3.87	(3.88)	14.35	(14.39)
2n	$C_{16}H_{10}N_4O_1Cl_2Br_1$	425.11	110°	68.63	dirty sporty yellow	45.20	(45.22)	2.37	(2.38)	13.18	(13.21)
2o	$C_{16}H_9N_4O_1Cl_4$	415.10	142°	62.57	light yellow	46.29	(46.30)	2.18	(2.19)	13.50	(13.54)
2p	$C_{16}H_{10}N_4O_1Cl_2F_1$	364.20	114°	74.34	orange	52.76	(52.77)	2.76	(2.74)	15.38	(15.40)
2q	$C_{17}H_{12}N_4O_1Cl_3$	394.68	154°	59.13	dirty orange	51.73	(51.74)	3.06	(3.04)	14.19	(14.22)
2r	$C_{18}H_{15}N_4O_1Cl_2$	374.26	109°	55.00	dark orange	57.76	(57.78)	4.04	(4.03)	14.97	(14.99)
2s	$C_{18}H_{15}N_4O_1Cl_2$	374.26	101°	55.90	sporty yellow	57.76	(57.78)	4.04	(4.02)	14.97	(15.00)
2t	$C_{18}H_{15}N_4O_1Cl_2$	374.26	121°	51.81	orange sunset	57.76	(57.74)	4.04	(4.05)	14.97	(15.02)

General procedure for the synthesis of 2,3-dioxo butyrate-2(substituted benzeneazo) phenylhydrazone (1a-1t)

To the substituted aniline (0.025 mole) was diazotised by adding concentrated HCl (8ml) and dis. water (6ml) cooled the solution at 0°C in an ice-bath, the cold aqueous solution of $NaNO_2$ was added to it, the prepared diazonium salt solution was added drop-wise in to the maintained at 0°C cooled solution of sodium acetate (0.12 mole) and ethyl aceto acetate (0.025 mole) in ethanol (20 ml) dissolved with minimum quantity of dis. water ,thus the product started separating, filtered, recrystallized with hot ethanol.

General procedure for the synthesis of N^1-(2,5-dichloro benzoyl) - 3 - methyl - 4 - (substituted phenyl hydrazono)-5-pyrazolone (2a-2t)

To (1a-1t ; 0.001 mole) was dissolved in 10 ml of ethanolic solution of 2,5-dichloro phenyl hydrazine (0.001 mole) and refluxed for about 4-5 hours in the presence of 4 - 5 drops glacial acetic acid, the obtained product was filtered, recrystallized with ethanol and identified to be N^1-(2,5-dichloro benzoyl)-3-methyl-4-(substituted phenyl hyrazono)-5-pyrazolone.



RESULTS AND DISCUSSION

The IR (Kbr) spectrum of newly synthesized pyrazolones have been recorded in the frequency region 4000-500 Cm^{-1} in the Table - 2.

The IR spectrum of N^1(2,5-dichloro benzoyl)-3-methyl-4(substituted phenyl hydrazono)-5-pyrazolone [2a-2f] shows the absorption in the range 3465.2-3481.2 cm^{-1} indicating the presence of -NH cm^{-1} stretching vibrations, and the other band are

Table 2: IR Absorption Bands

S.No.	Codes	NH cm^{-1} stretching	NH=C cm^{-1} stretching	Ar C=O cm^{-1} stretching	C=N cm^{-1} stretching	N-N cm^{-1} stretching	$-CH_3$ cm^{-1} stretching
1.	2a	3419.1	2373.8	1645.9	1541.8	1473.8	1421.9
2.	2b	3419.2	2374.3	1654.3	1566.7	1482.8	1422.0
3.	2c	3464.1	2370.2	1648.8	1560.1	1473.8	1419.8
4.	2d	3465.2	2363.7	1637.9	1564.6	1474.3	1419.8
5.	2e	3418.2	2367.6	1650.9	1561.6	1488.7	1419.1
6.	2f	3419.2	2371.6	1620.8	1565.6	1483.4	1423.7

appear in the region 2374.3-2363.7 cm^{-1} are confirm the -NH cm^{-1}, absorption in the range 1654.3-1620.8 cm^{-1} stretching vibrations are confirm the aromatic character -C=O cm^{-1}, absorption in the range 1566.7-1541.8 cm^{-1} indicates the C=N cm^{-1} stretching vibrations, and other band are appear in the range 1488.7-1473.8 cm^{-1} reveals the -N-N cm^{-1} stretching vibrations,the stretching vibrationsof $-CH_3$ group are appear in the range 1423.7- 1419.1 cm^{-1}.

These observations are lent support to the assigned structures of newly synthesized compounds 2a-2f and other compounds 2g-2t.

According to these facts Substituted Pyrazolones are stable solids, they have colouring properties, higher melting point shows thermal stability of the compounds.

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