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Synthesis and characterization of some novel acid hydrazide and hydrazones

KAUSIK GHOSH, ARISTA CHAUDHARI* and D.S. SETH

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

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ABSTRACT

Synthesis of N-(4-butyl) phenyl malonamic acid hydrazide has been reported. When these hydrazides were condensed with various aldehydes, novel hydrazones were synthesized. Some of the hydrazones have been screened for biological screening

Key words: Synthesis, hydrazides, hydrazones, novel, aldehydes.

INTRODUCTION

The full therapeutic possibilities of acid hydrazides were realized after the discovery of Isonicotinic acid hydrazide (INH). The remarkable clinical value of INH[1] stimulated investigations of other heterocyclic hydrazide having mono cyclic nuclei such as furan, thiophene, pyrrole and dicyclic nuclei such as quinoline and isoquinoline.Acid hydrazides has been known to possess wide properties like anti-tubercular[2], anti-pyretic[3], fungistatic[4] and diuretic[5]. Some of the hydrazones exhibit antibacterial[5-9], antifungal [10-12], antiviral [13-14], as well as insecticidal activity [15-16]. Hydrazones were found to inhibit particularly or completely the growth of S.aureus, E.coli and B.subtilis and possess antimicrobial and anti-helminthic properties.

MATERIAL AND METHODS

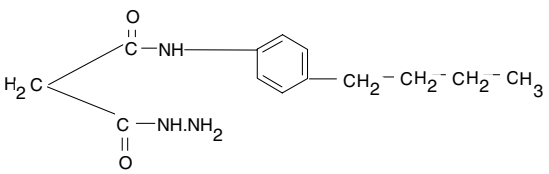
The chemicals employed were of A.R grade from Sigma Aldrich. The Melting points were determined in open capillaries on Electro thermal apparatus and were uncorrected. Infrared (IR) spectra were recorded on Perkin Elmer RX-1 using KBr wafers.

Synthesis of Ethyl N-(4-butyl) phenyl malonamate

3 ml of 4-butyl aniline and 6ml of diethylmalonate was refluxed for 1 hr. When dianillide get separated, filterate was collected in china dish and concenterated by heating on boiling water bath. White sticky mass of ethyl –N-4 butyl malonamate was obtained and finally treated with petroleum ether.

Synthesis of 4- butyl malonamic acid hydrazide

2.6 gm of ethyl -N-4 –butyl phenyl malonamate in 20 ml of ethanol was treated with 4 ml of hydrazine hydrate (98 %), white crystalline compound was obtained which was recrystallised from hot ethanol



4- Butyl phenyl malonamic acid hydrazide

Table 1: Hydrazones of 4 -butyl malonamic acid hydrazide with different aldehydes

S. No.	Aldehydes	Mol. Formula Hydrazone	M.P ºC	Yield %	Nitrogen% Found	Cal	I.R v cm^{-1}
1	Formaldehyde	$C_{14}H_{19}O_2N_3$	181	64.88	16.25	16.09	3280 cm^{-1} , (NH), 1664 cm^{-1} (CONH) , 1640 cm^{-1} (N=CH), 3086 cm^{-1} ,1569 cm^{-1} ,1490 cm^{-1} (aromatic characters), 825 cm^{-1} (1,4-substitution of benzene ring).
2	Benzaldehyde	$C_{20}H_{23}O_2N_3$	197	59.46	12.62	12.46	3200 cm^{-1} , (NH), 1654 cm^{-1} (CONH) , 1610 cm^{-1} (N=CH), 3066 cm^{-1} ,1560 cm^{-1} ,1487 cm^{-1} (aromatic characters), 815 cm^{-1} (1,4-substitution of benzene ring).
3	5- chloro Salicyaldehyde	$C_{20}H_{22}O_3N_3Cl$	201	53.98	10.96	10.83	3285 cm^{-1} , (NH), 1643cm^{-1} (CONH) , 1629 cm^{-1} (N=CH), 3061 cm^{-1} ,1560 cm^{-1} ,1987 cm^{-1} (aromatic characters), 835 cm^{-1} (1,4-substitution of benzene ring).
4	5-bromo salicyaldehyde	$C_{20}H_{22}O_3N_3Br$	199	45.02	10.01	9.72	3283 cm^{-1} , (NH), 1644 cm^{-1} (CONH) , 1629cm^{-1} (N=CH), 3071 cm^{-1} ,1569 cm^{-1},1493 cm^{-1} (aromatic characters), 827 cm^{-1} (1,4-substitution of benzene ring).
5	3, 5-dichlorosalicyaldehyde	$C_{20}H_{21}O_3N_3Cl_2$	204	42.55	10.12	9.95	3287 cm^{-1} , (NH), 1675 cm^{-1} (CONH) , 1631cm^{-1} (N=CH), 3085 cm^{-1} ,1560 cm^{-1} ,1487 cm^{-1} (aromatic characters), 843 cm^{-1} (1,4- substitution of benzene ring).
6	3, 5-dibromo alicyaldehyde	$C_{20}H_{21}O_3N_3Br_2$	216	40.93	8.32	8.21	3290cm^{-1}, (NH), 1684cm^{-1} (CONH) , 1645cm^{-1} (N=CH), 3076 cm^{-1} ,1560 cm^{-1} ,1497 cm^{-1} (aromatic characters), 851 cm^{-1} (1,4-substitution of benzene ring).

Table 1. Cont...

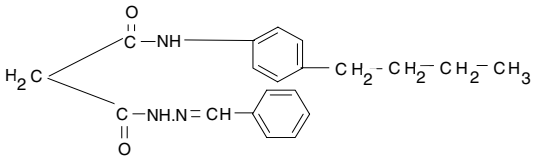
7	5-chloro-3-nitro salicyaldehyde	$C_{20}H_{21}O_4N_4Cl$	227	45.45	13.65	13.44	3289 cm^{-1}, (NH),1672 cm^{-1} (CONH), 1657 cm^{-1} (N=CH), 3111 cm^{-1} ,1594 cm^{-1} 1493 cm^{-1} (aromatic characters), 849 cm^{-1} (1,4- substitution of benzene ring).
8	5-bromo-3-nitro salicyaldehyde	$C_{20}H_{21}O_4N_4Br$	196	49.78	12.19	12.14	3293 cm^{-1}, (NH), 1678 cm^{-1} CONH), 1666 cm^{-1} (N=CH), 3085cm^{-1} ,1598 cm^{-1} 1494 cm^{-1} (aromatic characters), 858 malonamic acid hydrazidecm^{-1}
9	3, 5-dinitro salicyaldehyde	$C_{20}H_{21}O_6N_5$	226	49.06	16.55	16.39	3298 cm^{-1} , (NH), 1683 cm^{-1} (CONH) , 1679 cm^{-1} (N=CH), 30926cm^{-1} ,1581 cm^{-1} ,1491 cm^{-1} (aromatic characters), 863 cm^{-1} (1,4-substitution of benzene ring).
10	N –(4-butyl) phenyl malonamic acid hydrazide	$C_{13}H_{19}O_2N_3$	162	55.86	16.97	16.86	1605 cm^{-1} ($CONHNH_2$), 3290 cm^{-1}, 3275 cm^{-1} (NH stretching), 2960 cm^{-1} ,1515 cm^{-1} ,1418 cm^{-1} (aromatic characters), 813 cm^{-1} (1,4- substitution of benzene ring).

Table 2: Antibacterial activity screening of some of the compounds

S. No.	Compound No	ConcentrationIn H gm/ml	Sensitivity			
			Pseudomonas	***Staphylococcus***	***E.coli***	***B.subtilis***
1	5	20	R	R	R	R
		30	R	R	R	R
		40	R	R	R	R
		50	R	R	R	R
2	7	20	R	R	+1	R
		30	R	R	+1	R
		40	R	R	+2	R
		50	R	R	+2	R
3	8	20	R	R	R	R
		30	R	R	R	R
		40	R	R	R	R
		50	R	R	+1	+2
4	9	20	R	R	R	R
		30	R	R	R	R
		40	R	R	R	R
		50	+1	+1	R	+1

Synthesis of 4- butyl and 4-isopropyl malonamic acid hydrazone of different aldehydes and ketones

The mixture of aldehyde (1 mol) and phenyl malonamic acid hydrazide (1 mol) in 10 ml of abs. ethanol was gently refluxed for 2 hr. On cooling product separated, was filtered and recrystallised from ethanol.



4-butyl malonamic acid hydrazone of benzaldehyde

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