

## Short Communication

Pak. j. sci. ind. res., vol. 36, no. 11, November 1993

## Synthesis of Some Diaroylhydrazines Bearing A Pyrazine Nucleus

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(Received January 28, 1993; revised July 10, 1993)

It has been reported that some pyrazine derivatives have flavouring properties. Thus, 5-alkyl-3-methyl-2(1H)-pyrazinones [1] have licorice - woody aroma which indicates that they could be used as food additives. Also some pyrazine derivatives were isolated [2] from the volatile components of roasted chickpea. These observations, coupled with our interest in the chemistry of diaroylhydrazines led us to synthesize some of these compounds bearing a pyrazine nucleus for testing their flavouring properties. All melting points reported are uncorrected.

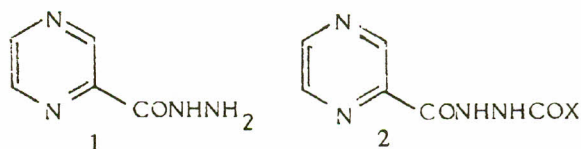
**Pyrazinoic acid hydrazide 1.** To a solution of pyrazinoyl chloride (1.43g., 10 m. mole) in methylene chloride (20 ml), anhydrous hydrazine (0.32 g; 10 m.mole) was added. The reaction mixture was refluxed for 2 hrs. On cooling yellow crystals separated out, filtered off and recrystallized from ethanol, m.p. 168 - 69°, yield 87%. This product was shown by direct comparison (m.p. and m.m.p.) to be identical with that reported by Spoerri *et al.* [3].

On repeating the above experiment with the molar ratio of the acid chloride and hydrazine (2 : 1 mole), *N,N'*-dipyrazinoyl hydrazine (2a) was obtained in pale yellow crystals (Table 1).

***N,N'* Diaroylhydrazines 2b-e.** To a solution of pyrazinoic acid hydrazide (1.38 g, 10 m.mole) in 30 ml methylene chloride, (10 m.mole) of the appropriate acid chloride were added. The reaction mixture was heated under reflux for 2 hrs. The product obtained was filtered off and recrystallized from the suitable solvent (Table 1).

**Pyrazinoyl semicarbazide 2f.** Pyrazinoyl chloride (1.43g, 10 m.mole) in 30 ml chloroform, was refluxed with a mixture of semicarbazide hydrochloride (1.49 g, 15 m.mole) and anhydrous sodium acetate (0.6 g) was added to give (2f) (Table 1).

Pyrazinoic acid hydrazide (1) was prepared [3] by the action of hydrazine on methyl pyrazinoate. In this investigation, we report the synthesis of this compound from the reaction between pyrazinoyl chloride and anhydrous hydrazine (1 : 1 mole). However, when the molar ratio of the acid



a; X = 2-Pyrazinyl, b; X = C<sub>6</sub>H<sub>5</sub>,  
c; X = 2-furyl, d; X = thienyl,  
e; X = CH<sub>3</sub>, f; X = -NH<sub>2</sub>

TABLE 1. PHYSICAL AND SPECTRAL DATA OF THE COMPOUNDS 2a-f.

Comp.	*M.p. (°C)	Yield %	<sup>1</sup> H-NMR, δ ppm	IR cm <sup>-1</sup>				EIMS
				ν C=O	ν C=N	ν NH	ν OH	
2a	214-15*	81	88.7 (m, 6H), 9.2 (2H)	1680	1642	3221	3429	(M <sup>+</sup> + 1, 257), 217, 213
2b	218-19**	75	in CH <sub>3</sub> OHd <sub>4</sub> 9.26 (s,2H), 8.71-8.82 (m,3H), 7.43- 7.93 (m, 5H)	1682	1637	3221	3436	(M <sup>+</sup> + 1,243), 163, 137
2c	228-29**	65	in DMSO 10.83 (s,1H) 10.51 (s,1H), 8.79-9 (m,3H), 6.68-7.93	1687	1641	3156	3451	(M <sup>+</sup> + 1, 233)
2d	243-44**	60	in DMSO 10.88(s,1H), 10.63(s,1H), 8.79-9(m,3H) 7.22-7.80 (m, 3H)	1679	1627	3080	3410	(M <sup>+</sup> + 1,249), 131, 79
2e	197-98**	63	in CH <sub>3</sub> OHd <sub>4</sub> 9.22(s,1H), 8.68-8.80(m,3H), 2.06 (s, 3H)	1698	1635	3076	3340	(M <sup>+</sup> + 1,181) (M <sup>+</sup> ,180), 107, 79, 52, 43
2f	153-54**	35.5	in CDCl <sub>3</sub> 8.9(s,1H) 8.4(s,1H),7.9(m, 3H) 5.3 (s, 2H)	1668	1635	3210 3260 (d)	3410	106,28

\* Solvent of crystallisation Benzene/Ethanol, \*\* Solvent of crystallisation Ethanol.

chloride to hydrazine is (2 : 1), N,N'-dipyrazinoylhydrazine (2a) is formed.

The structure of 2a is based upon spectral data including IR, <sup>1</sup>H-NMR and mass spectrum (Table 1).

The preparation of pyrazine derivatives using pyrazinoyl chloride was extended to the reaction with semicarbazide hydrochloride which led to the formation of pyrazinoyl semicarbazide (2f) as the sole product (Table 1).

All the products obtained were tested for their flavouring properties by Sniffing.

**Key words:** Diaroylhydrazine, Pyrazine nucleus, Flavouring properties.

#### References

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