

Synthesis and characterisation of some heterocyclic Compounds Containing more than two nitrogen

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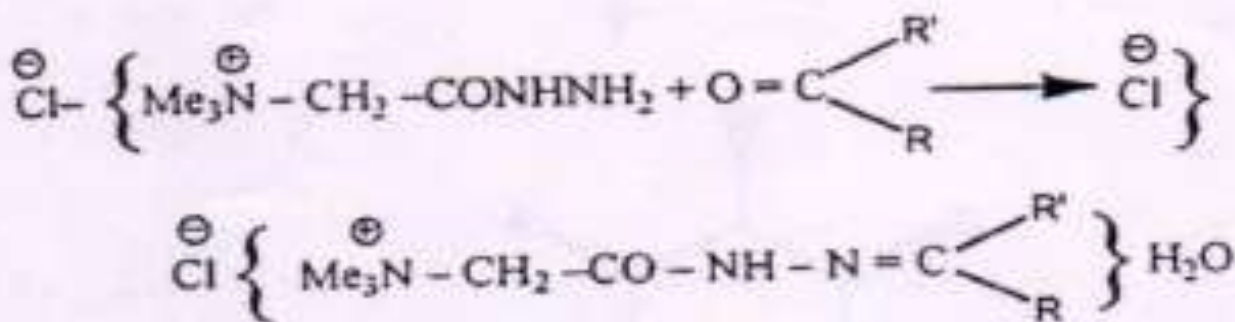
Abstract:Hydrazides undergo a role of variety of reactions giving different products which depends upon the nature of hydrazide. The reagent employed and the reaction conditions on treatment with into diacyhydrazides. The action heat of primary hydrazides on heating. Split out a molecule of hydrazine and are converted into diacyhydrazides. Diacyhydrazides, as well as acylhydrazides may, be cyclised to give variety of heterocyclic component containing five or six membered ring systems with three or four nitrogen atoms of azole or azine series. The results obtained were characterized and finally conformed alternative method of known synthesis.

(Key Words ; Hydrazides , Diacyhydrazides , azoles azines)

Introduction

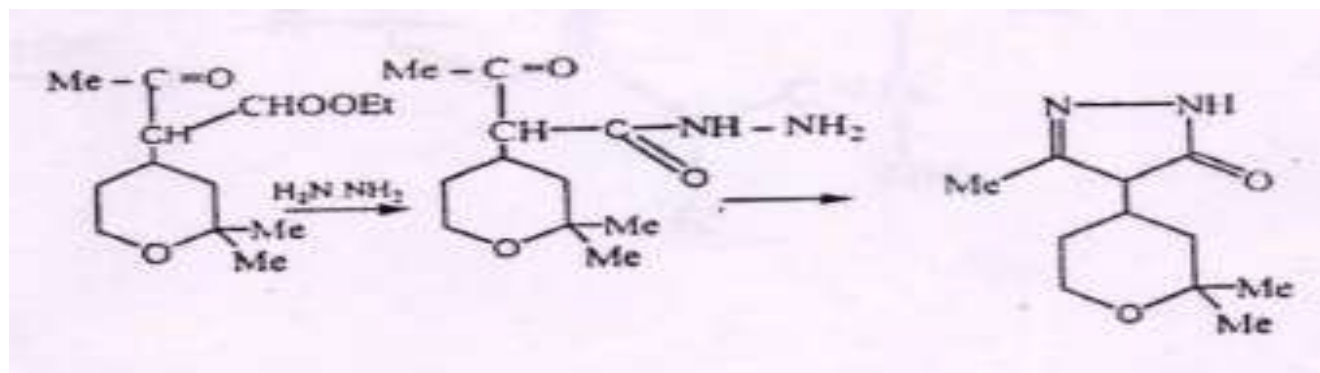
Carboxylic acid derivatives such as ester acid chloride, acid amide form acid hydrazide. Where treated with hydrazine under normal condition. Simple carbonyl compounds are usually crystalline with well-defined melting points. They are used sometimes for the identification of carbonyl compounds.

Hydrazides containing a quaternary ammonium group in the acid portion of the molecule condense with ketones to give condensation products which are soluble in water. Such hydrazides are called Girard Reagents. They are sometimes used in separating hormones as water soluble compounds from accompanying fats. The ketone is then recovered by hydrolysing the hydrazide.

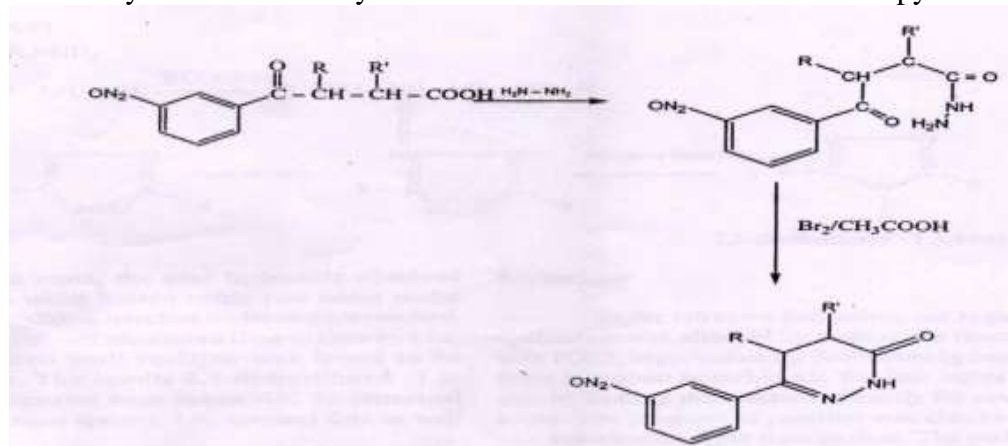


The reaction of hydrazides with other carbonyl compounds such as 1,3- and 1,4-diketones has not been investigated so thoroughly. In reaction with these diketones, the hydrazides form heterocyclic compounds of azole or azine series.

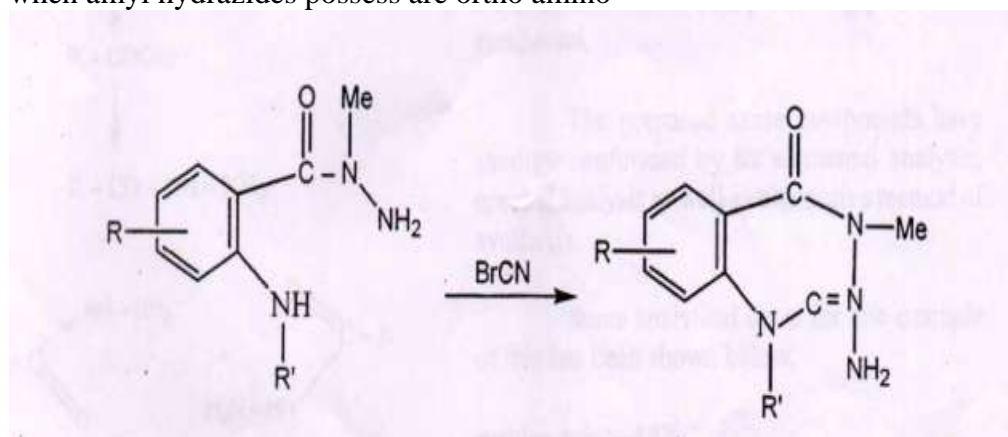
Hydrazides of β -keto acids may be cyclised intramolecularly to give heterocyclic compounds. Many of which possess biological activity. Several such compounds have been synthesized recently. For example β -ketoesters have been cyclised through the hydrazide into pyrazolone.



Similar cyclisation of the hydrazides of the α -Keto acid furnished the pyridazinoes.



The possibilities of cyclisation giving different types of ring system, increase considerably when amyl hydrazides possess are ortho amino

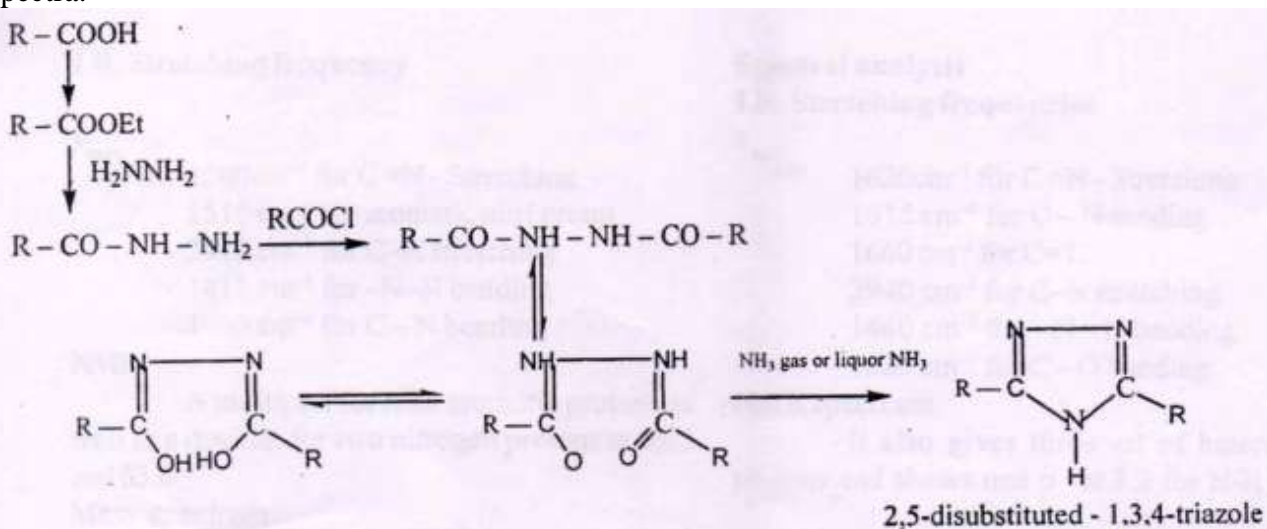


Experimental

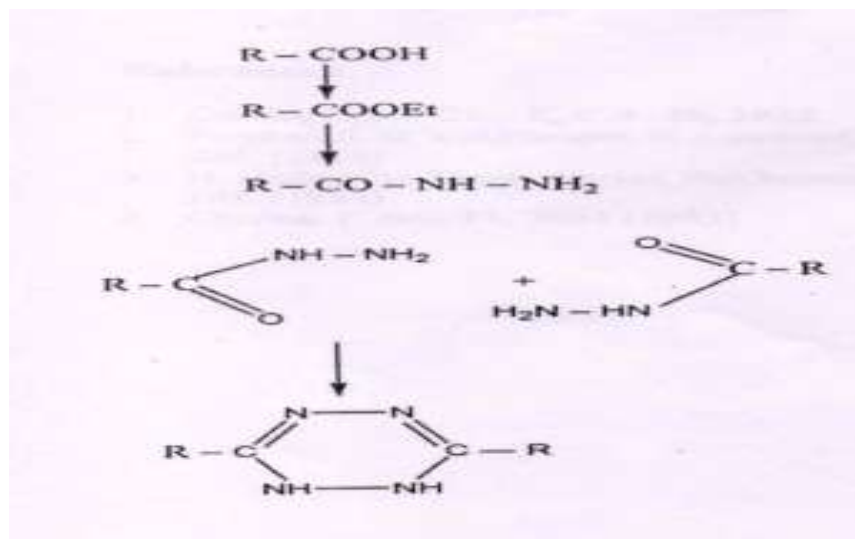
Carboxylic acids such as – picolinic acid, nicotinic acid, Furoic acid and phenyl acetic acid were easily available. They were firstly converted into their respective hydrazides and were again treated with corresponding acid chloride again treated the results obtained were diacyl hydrazide.

Substituted in the aromatic ring. Benzotriazepines were

These diacylhydrazides were forced to cyclise in the presence of ammonia gas to result 2,5-disubstituted 1,3,4-triazole compounds by the sequence of reaction carrying out alternative known method as well as elemental analysis and spectral data. E.g. uv. IR, NMR and mass spectra.



In second route, the acid hydrazide obtained earlier is when heated using two notes under normal condition, tetrazine molecules are resulted. The results 3,6-disubstituted -1,2- dehydrotetrazine were identified by elemental analysis, mass spectra, I.R.spectral data as well as nmr.



Explanation

In the tetrazine formation, the required cyclisation was affected by heating the reactants with $POCl_3$ in pyridine for four hours by heating them in carbon tetrachloride for four hours and also by heating the reactants for several hours. The progress of reaction was checked by t.l.c examination from time to time. The yield of the cyclised product in all cases were fairly good.

Since acidic reagents, are found to be more effective and at the same time basic reagents were also found good enough in bringing about such . The results had a sharp melting point pure compound.

The prepared azine compounds have shudure conformed by its elemental analysis, spectral analysis as well as alternative method of synthesis.

Some analytical dates for one example of this has been shown below.

Melting point – 172C

Mass spectra – (m/e)-295

Elemental analysis

Found N-23.80%

Calculated for $C_{15}H_{13}N_5O_2$ N-23.72%

I.R. Stretching frequency

1580 cm^{-1} for C=N- Stretching

1515 cm^{-1} for aromatic nitri group

2910 cm^{-1} for C-H stretching

1415 cm^{-1} for N-N bending

1450 cm^{-1} for C-N bending

NMR

A multiple for nine aeromatic protons as well as doublet for two nitrogen protons at 83.2 and 83.6

Mass spectrum

(m/e)-29

In the earlier case of synthesis of triazole cyclisation also occurs under same laboratory condition analytical data of one example for the given compound is mentioned as shown below.

2,5-bis(a-funyl)-1,3,4-triazole

Melting point – 11C

Elemental analysis

N- 20.76%

Spectral analysis

I.R. Stretching frequencies

1620cm⁻¹ for C=N-Stretching

1015cm⁻¹ for C-N sending

1660cm⁻¹ for C=C

2940cm⁻¹ for C-N stretching

1440cm⁻¹ for –N-N bending

NMR spectrum

It also gives three set of heterocyclic protons and shown one p at 3.2 for N-H proton

Mass spectra

[M/e]-201

All analytical dates are in the agreement of the structure of azine as well as azole compounds.

Acknowledgment

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