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Synthesis, characterization of some derivationes of 3-Nicotinc acide

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***Abstract*---**In This Research The Preparation Of Number heterocyclic compounds, Which include Seminars like compensators 1,3,4-oxadizole -2-thiol and AL Pyrazole -2- yl, Nicotinic Used as Vaw material in the Preparation .Attended ester During reactance nicotinic acid With thionyl Chloride in the presence of ethanol, The resultant ester were treated with hydrazine hydrate in ethanol the Corresponding hydrazide The synthesized hydrazides were converted to 1,3,4-oxadizoles by their reaction with carbon disulfide in potassium hydroxide, Use hydrazide To the praparation pyrazole during reactance with ethyl acetoacetate.

***Kewords*---**Nicotinic acide, Nicotonyl Chlorid, Nicotino hydrazide, oxadiazole.

Introduction

Nicotinic acid is on of the organic compounds which is found in all living Cells, it is also known as vitamin (B_3) Which is found in food .Nicotinc acid (Niacine) is an important organic Compound Which is found as enzym and amide(1), its decrease Causes pellagra Niacine vaises fatty protein and it is used to minize(2) the risk of getting diseases as Cardioras calar. Niacine is found as Nicotineamide dinucleotide and A denine Denucleotide phosphate(3) which play an important vole as an auxiliary factor for many dehy drogenose(4) enzymes such as lactate dehydrogenase. Niacine is used to decrease the level of cholesterol by inhibiting Amino Acids(5) in plasma It also reduces the risk of getting diabetes and osteoporosis(6) taking large amousts of vitamin B_3 case nicin Red skin increased intracerebral blood flow(7), diavrheq and vomiting Bile occuss and the liver will be dam aged (nicotinic) acid and yacin are converted to nicotine amide, which has the same enamel as the vitamin, but the pharma cological and toxic effect that occursin (3g) transitions to sleepers, so it does not penetrate choles terol and cause, supply but adose above daily for adults is toxic to the liver Bacterial hypergro with in the small in testine is aknw cause of the nicotine amid text .

Experimintal

Synthesis of Nicotnoyl Chloride

From thiongl Chloride (0.023 mol, 2.75 gm) is added as drops in a funnel to an acid about (0.024 mol, 3 gm) Continuosly (15 minutes) to asulution of dry ether in acircular, Condensed ductile nicotinic ($25cm^2$) flakein calcium chloride and the mixture is heated over asteam bath for one and ahalf hours, after with the solvent is evaporated using arotating eraporator at temperature range (40-50m°) and the precipitate is recrystallized and the residue is obtained[8]

Synthesis of ethyl Nicotinate

The addion of the absulue al cohol Creleased ethanol is prepared gradually with degree stirring and cooling fromacid chloride dry ammonia gas is possed in (0.02) supended from (0.1 mol) (5-0m°) to the compound a cid chloride. the solution is lea ched to an ammonium chloride dispenser , the solvent is evaporated by avotary evapov to abtain aliquid ester[9]

Synthesis of Nicotino hydrazide

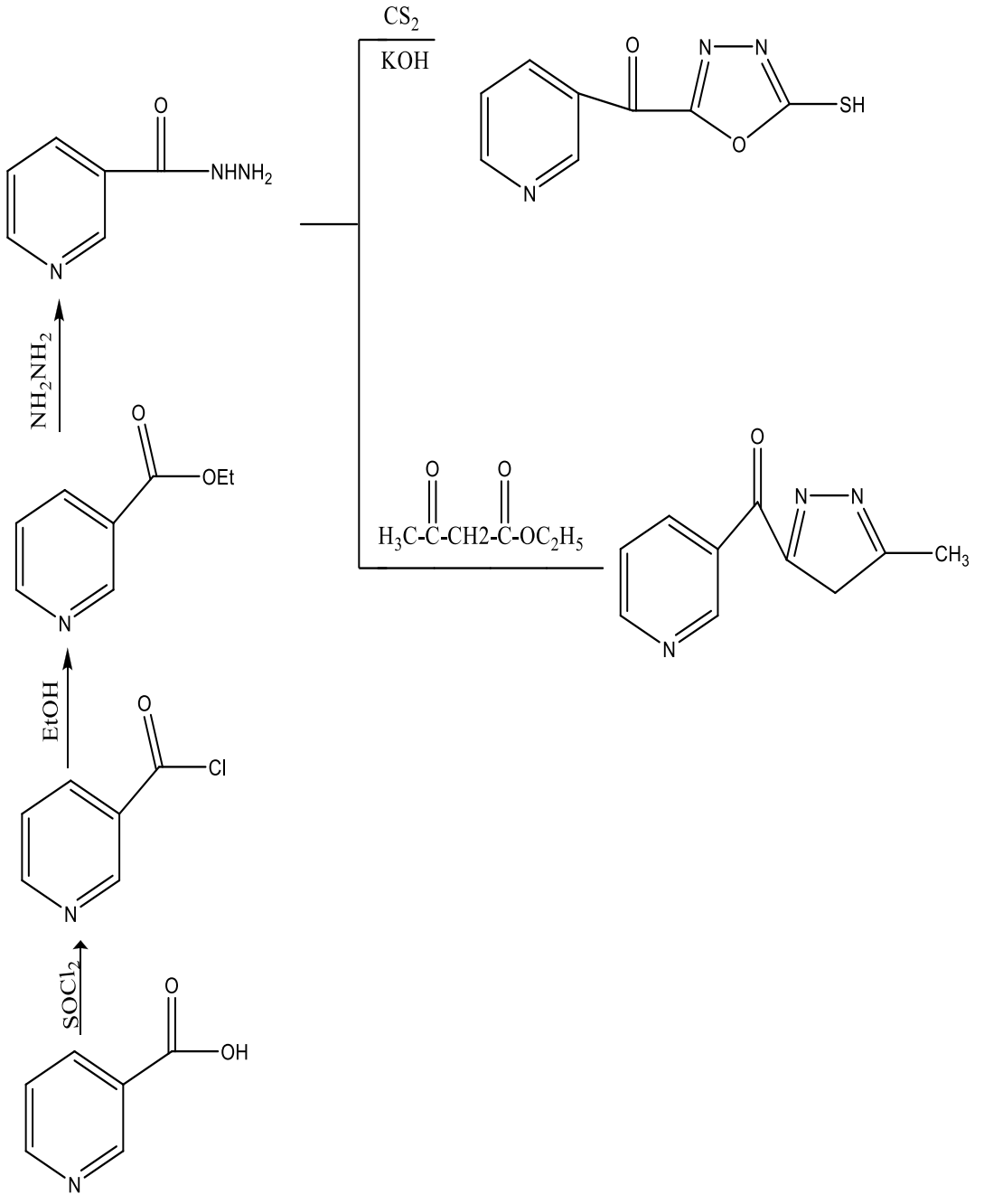
From the hydrazine (0.02 mol) of prepared ester and (0.004 mol) amixture containing released methanol is ascended for fire hours and then cooled to abtain (40 ml) water (99%) in (10) the methrazide residue, recury stallized from etharol[10] .

Synthesis of Mercopto -1,3,4-oxadiazol -2-yl) phridine -3-y)

Hydrazide in potassium hydroxide solution (0.05 mol,19 gm) in (100ml) of the rathanol released for 15 minutes and than added to the mix gradually (0.15 mol , 12 ml) from Cs_2 untill the emission of (H_2S) is slopped the smell and blackening of a soaked apaper with lead acetate liquid the solvent is vaporized the the release pressure and is added to the remaining crushed ice and than the mixture is acidified using hydrochloric acid – con centerata with cooling unit miture is neutral and left for 24 hours to complete deposition and leaching the emulaloris well washed and recrystallized usind abosulute ethanol to produce aprecipitate[11]

Synthesis of Methl -4 H- pyrazal – 3-yl phridine -3-yl methanone

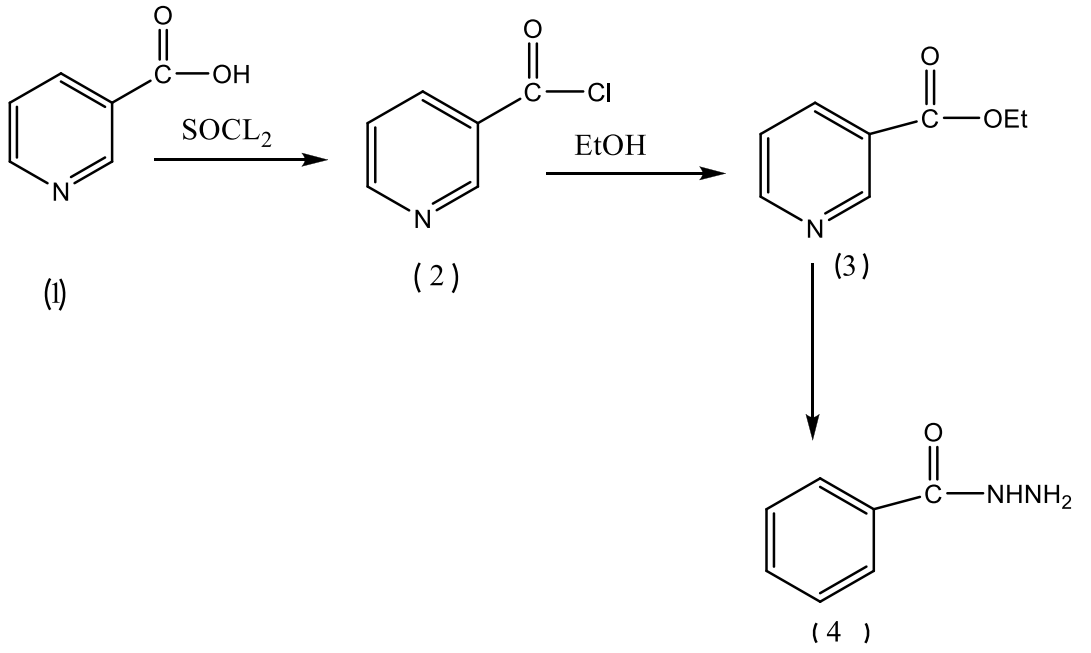
(0.05mol , 1.9 gm) from one of the componeds such as beta – dicaronylis addwd to solution of hydrazide (0.05mol ,1.9gm) in (30ml) of abs-Etoh ,then ,it is restrieted for 24 hours where the solution is hot and then the crushed ice is added to precipitate which is separated by filtrahen and hydrated and recrystalased from ethanaol[12] .



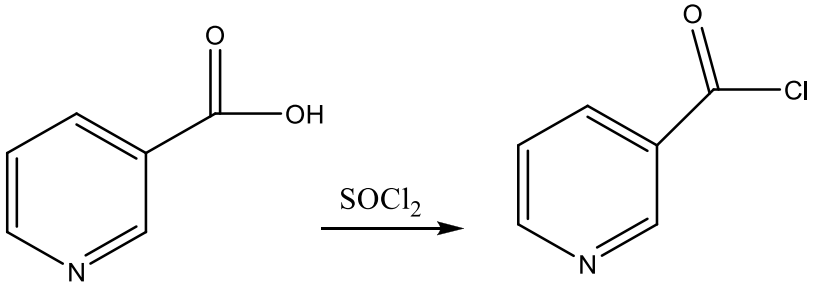
Result and Discussion

Several studies have shown that heterocyclic compounds are important in medical field as it is used cure[13] as antibiotcs ,and has been used induslrics ,and has been used industrially in the manuf acture of dyes[14], as well as for anti – copper consumption due to their trophic importance in various fileds of interest to researchers, these compounds are involved in the structure of chlorophyll

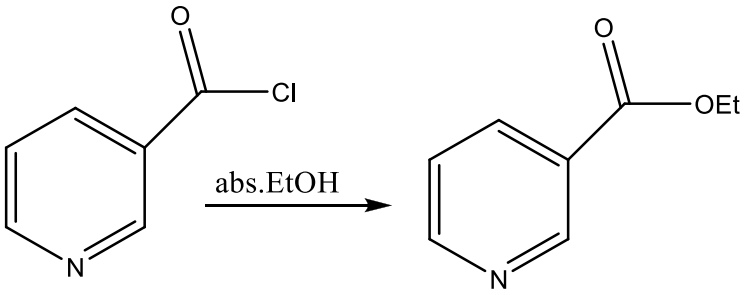
inplants oxadiazol and phrazole compounds are heterogeneous penta – compounds – in this study, nicotinic acid was used as anucleus to prepare non-penta circulating cyclocomponds for the purpose of producing compounds of expected biobgical in terest. The scheme(1) was shown the prepared hydrazide (4)



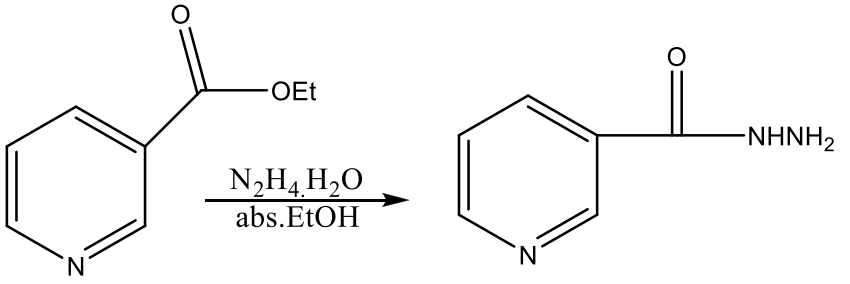
Nictinoyl chloride are Prepared by adding thionyl chloride to nicotine acid in etheridry:



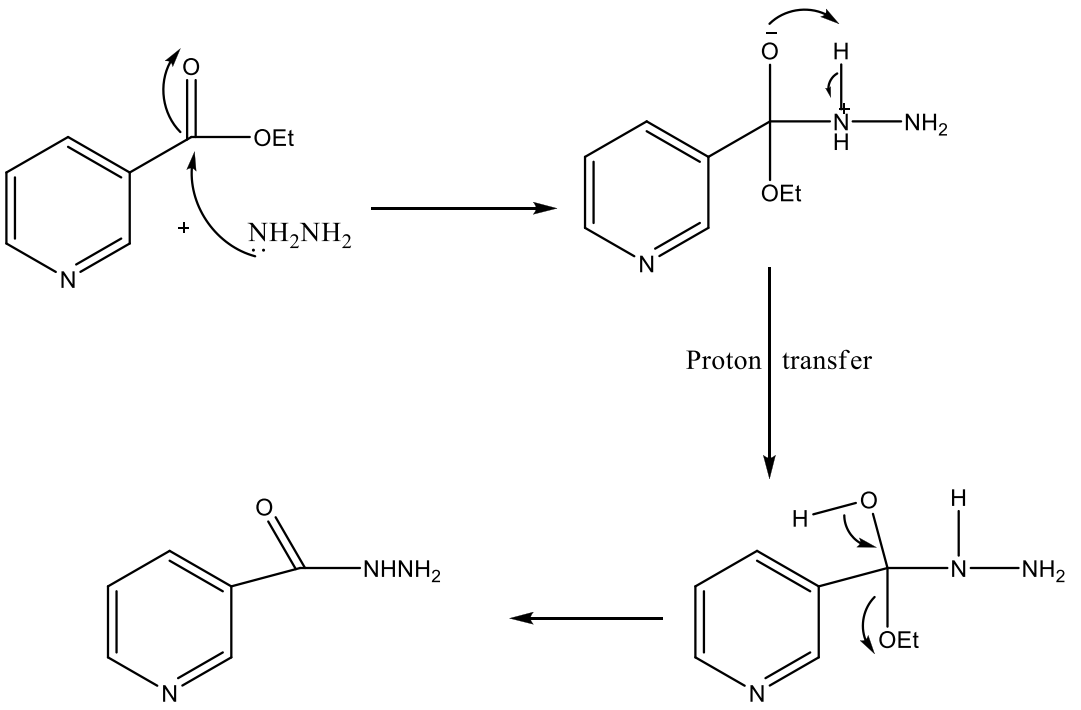
and a composite person with in frared spectros copy if the spectrum shows frequency bands at as asharp and strong (c-o) due to the karonel frequency (1742-1792 cm^{-1}) at the frequency (c = c) and the absorption bands of the (O-H) bond absoption band (3068 cm^{-1}) Armenian frequency band (1559-1574cm^{-1})[15] . are Prepared from the addition of free ethanol to acid chloride and abtion an ester asin the equation:



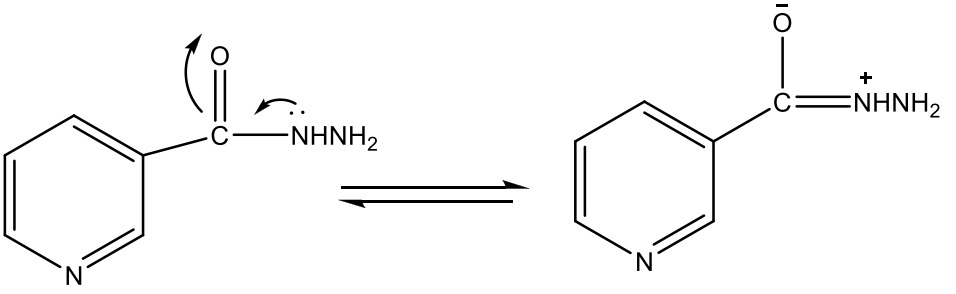
There sulting ester showed aware detection using ferric hydroxamate dete ctiong using ferric hyroxamate detection indicating [16] that (IR) the ester set and the ester person produced with infrared spectroscopy are the carbon strand band ($1745cm^{-1}$) the band of maths at ($3071cm^{-1}$) frequency ($2980\text{-}2830cm^{-1}$) the symmetric and asy mmetric fiber (C-H) of the (C-H) band. While hydrazides are biologically active compounds and hare been used as astavting material to perepare new high value compounds by turning the min to the heterogeneous pente cyclic compounds preparation of hydrazides by ester re action with absoluite watery hydrazinein ethanol as shown in the following equation:[17]



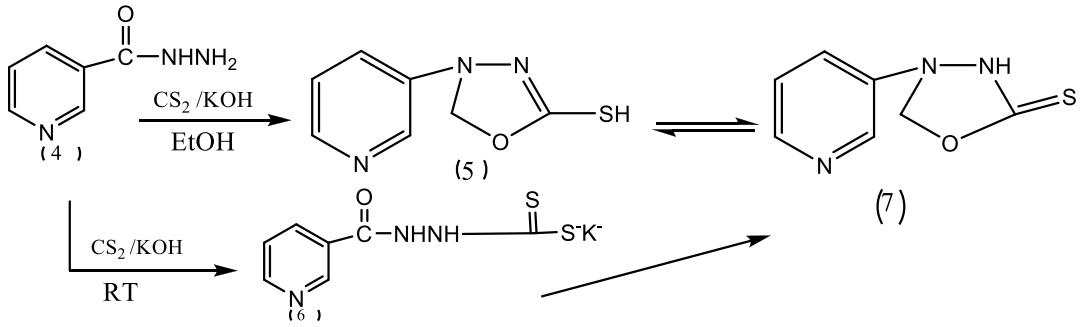
the re action of hydrazine with ester is bosed on aneuk lophilic substitution on acarbon atom the ester carbaonyl group is reacted by aterracoid al mechanism by means of abouble electron attack of the amine group in the hyrodrazine on the ester group, as a hybridization of an aton that forms an actire intermediate with sp^3 deletion is converted to a (C-O) carbon in the carbonyl group, the eoxy group gives the hydrazide, as decribed in the following mechanism:



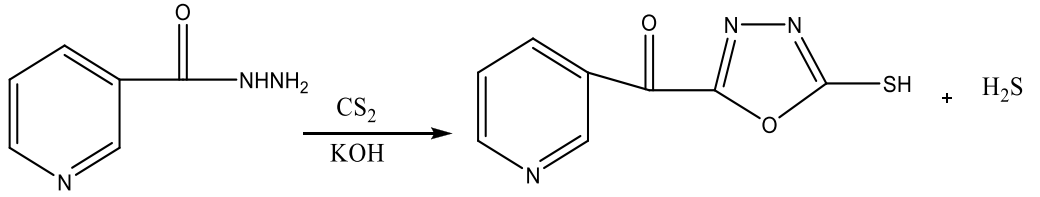
hydrazide was diagnosed as spectros by the infrared spectrum ,it goes back to (1664- 1623 cm^{-1}) and (N-H) Bear and (3300cm^{-1}) the two carbonyl amide groups atta ched to the cyclic and terminal respactirely the absorption of hyrazide by the karionel group has been abser red to have low – frequency displacement compared to the carbonyl group in the ester[18], this is due to the presence of the seeing phenomenon in hydrazine and hance strong constant of this, which reduces the carbonyl double – bond character and decreases its frequency.



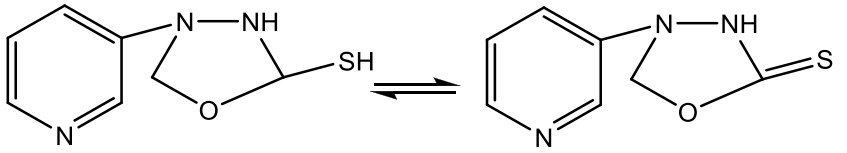
Hydrazide (4) was used to synthesis of substituted 1,3,4- oxadiazole (7) as shown in scheme (2)



In CS_2, the compound was prepared by the reaction of the hydrazide with the carbon ddisufide Alcoholic potassium hydroxide.

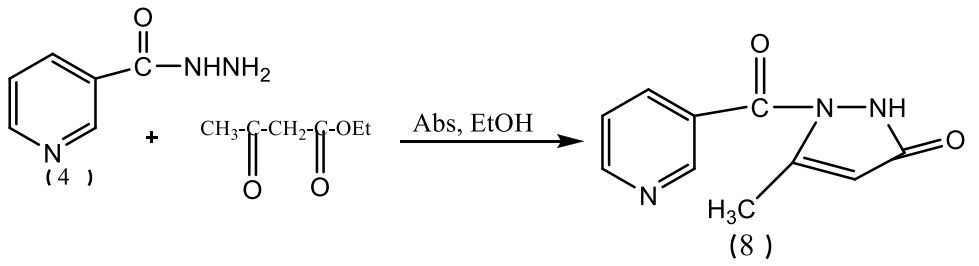


This interaction an important reaction in the preparation of oxydizol compounds containin athiol group. It says agas molecule, hydrogen sulfide, forming the oxidiazol -2-thion ring:

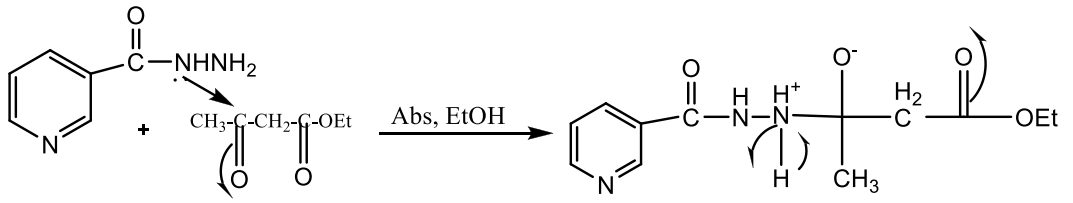


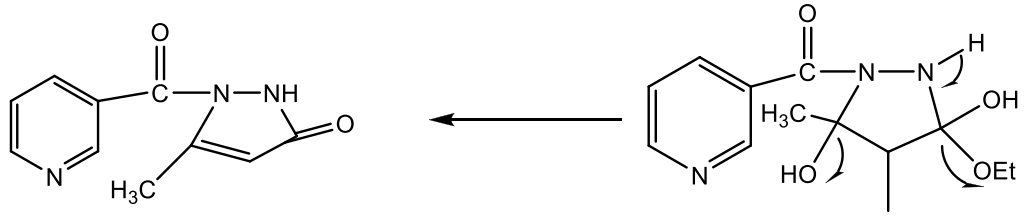
The interaction is done by anucleophilic addition anucleophilic addition mechanisn that involres an attckcarbon – based neokove lli of the mino , giring acarbon – carbeptide molecule to athio carbazide[19] and then an implicit euclophilic attack followed by the loss of amolecule. In gare oxydizol afolds using infrared spectroscopy Exemption packets appear at the thion group gare absorption bonds at C-C (1595cm^{-1}) the frequency and showed patten bands returning which confirms the state of the nanometer of the ion and tiol forms (1135cm^{-1})[20] it goseback to the patternat (1424cm^{-1}) at the frequency (N-N)[21] of (C=N) .

Finally, Hydrazide (7) was used the substituted pyrazotine-one (8) as shown in schem (3)



The compound was prepared from the medrizide reaction with 1,3 –dircaronyl compounds such as acettoacetyl [22] acetate and acetylasetone as in the following equation:





An absorption band at the infrared (IR) pyrazole person[23] afreaquency band for the carneill group (CN) of the band bands (1580-1639cm^{-1}) at the band frequen cies (1057-1027cm^{-1}) at (N-N) and the band (1666cm^{-1}) ving at the band [24] (3335cm^{-1}) at the band (N-H) showed the pattern band veturn.

Conclusion

We conclude that the Nicotinic acide was used as starting material to prepare some of Derivatines especially, Nicotionyl chloride, hydrazide and other heterocyclic compounds such as 1,3,4-oxadiazole and pyrazoline-2- one

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