

**How to Cite:**

Ismaeil, E. I. (2022). Synthesis, characterization of some derivations of 3-Nicotinic acid. *International Journal of Health Sciences*, 6(S9), 3783–3791. <https://doi.org/10.53730/ijhs.v6nS9.13469>

## Synthesis, characterization of some derivations of 3-Nicotinic acid

**Eman Ibrahim Ismaeil**

Nineveh Education, Mosul-Iraq

Email: [amysfalsalhy1254@gmail.com](mailto:amysfalsalhy1254@gmail.com)

**Abstract**---In This Research The Preparation Of Number heterocyclic compounds, Which include Seminars like compensators 1,3,4-oxadiazole -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-oxadiazoles by their reaction with carbon disulfide in potassium hydroxide, Use hydrazide To the preparation pyrazole during reactance with ethyl acetoacetate.

**Keywords**---Nicotinic acid, 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<sub>3</sub>) Which is found in food .Nicotinc acid (Niacine) is an important organic Compound Which is found as enzym and amide<sup>(1)</sup>, its decrease Causes pellagra Niacine vaises fatty protein and it is used to minize<sup>(2)</sup> the risk of getting diseases as Cardioras calar. Niacine is found as Nicotineamide dinucleotide and A denine Denucleotide phosphate<sup>(3)</sup> which play an important vole as an auxiliary factor for many dehy drogenose<sup>(4)</sup> enzymes such as lactate dehydrogenase. Niacine is used to decrease the level of cholesterol by inhibiting Amino Acids<sup>(5)</sup> in plasma It also reduces the risk of getting diabetes and osteoporosis<sup>(6)</sup> taking large amoumts of vitamin B<sub>3</sub> case nicin Red skin increased intracerebral blood flow<sup>(7)</sup>, 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 .

## Experimental

### Synthesis of Nicotnoyl Chloride

From thionyl Chloride (0.023 mol, 2.75 gm) is added as drops in a funnel to an acid about (0.024 mol, 3 gm) Continuously (15 minutes) to a solution of dry ether in a circular, Condensed ductile nicotinic (25cm<sup>2</sup>) flake in calcium chloride and the mixture is heated over a steam bath for one and a half hours, after with the solvent is evaporated using a rotating evaporator at temperature range (40-50m°) and the precipitate is recrystallized and the residue is obtained<sup>(8)</sup>

### Synthesis of ethyl Nicotinate

The addition of the absolute alcohol released ethanol is prepared gradually with degree stirring and cooling from acid chloride dry ammonia gas is passed in (0.02) suspended from (0.1 mol) (5-0m°) to the compound a chloride. the solution is leached to an ammonium chloride dispenser, the solvent is evaporated by rotary evaporation to obtain a liquid ester<sup>(9)</sup>

### Synthesis of Nicotino hydrazide

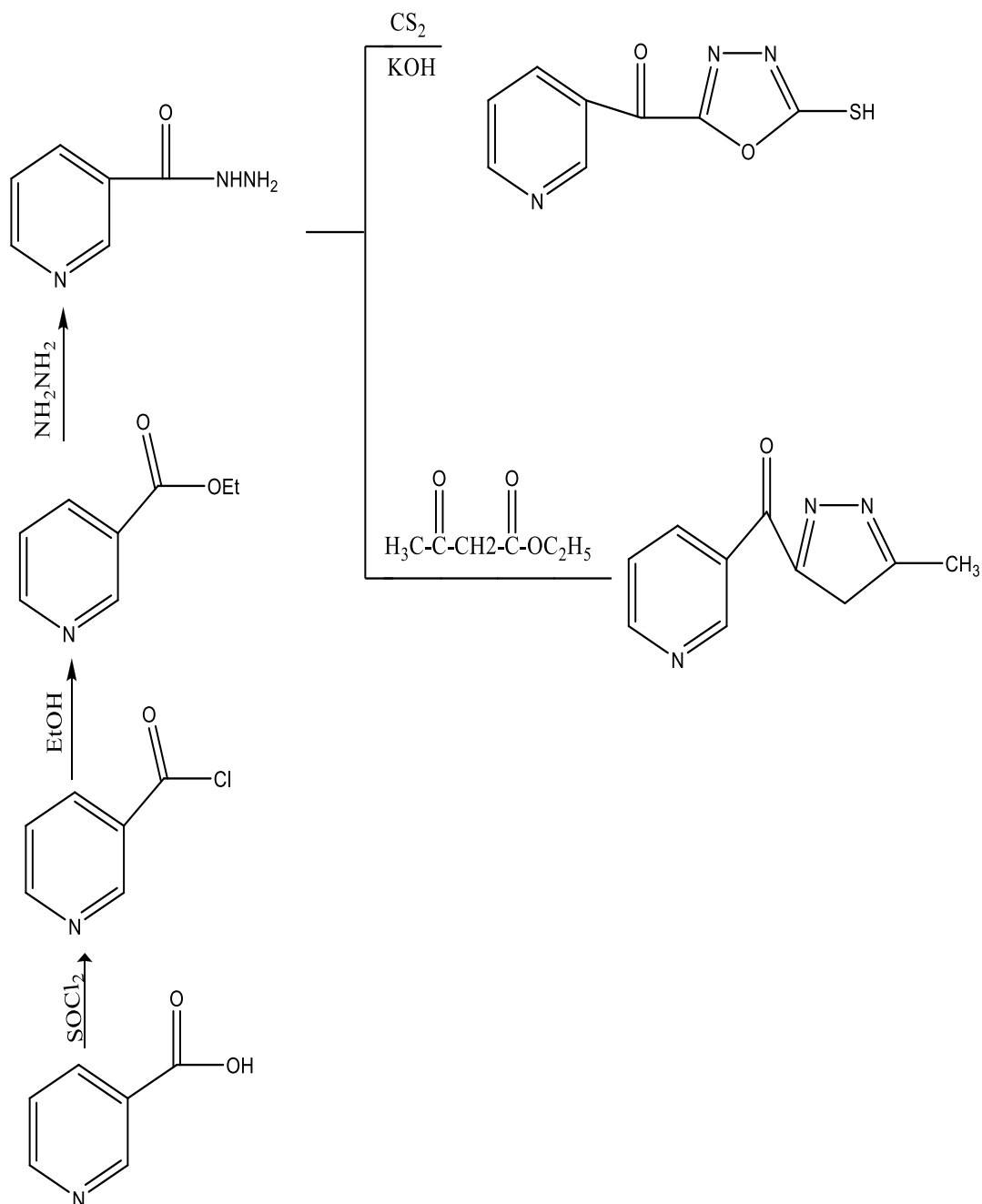
From the hydrazine (0.02 mol) of prepared ester and (0.004 mol) a mixture containing released methanol is ascended for five hours and then cooled to obtain (40 ml) water (99%) in (10) the methrazide residue, recrystallized from ethanol<sup>(10)</sup>

### 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 ethanol released for 15 minutes and then added to the mix gradually (0.15 mol, 12 ml) from Cs<sub>2</sub> until the emission of (H<sub>2</sub>S) is stopped the smell and blackening of a soaked paper with lead acetate liquid the solvent is vaporized the the release pressure and is added to the remaining crushed ice and then the mixture is acidified using hydrochloric acid – concentrated with cooling unit mixture is neutral and left for 24 hours to complete deposition and leaching the emulsion well washed and recrystallized using absolute ethanol to produce a precipitate<sup>(11)</sup>

### Synthesis of Methyl -4 H- pyrazal – 3-yl phridine -3-yl methanone

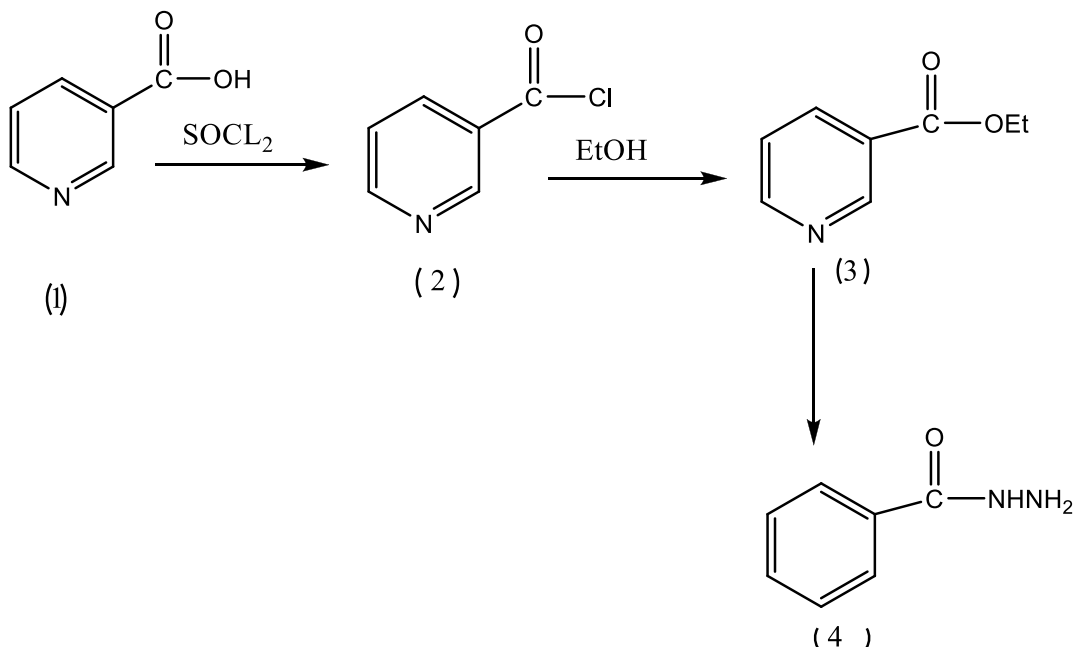
(0.05mol, 1.9 gm) from one of the components such as beta – dicaronylis added to solution of hydrazide (0.05mol, 1.9gm) in (30ml) of abs-EtoH, then, it is restricted for 24 hours where the solution is hot and then the crushed ice is added to precipitate which is separated by filtration and hydrated and recrystallized from ethanol<sup>(12)</sup>



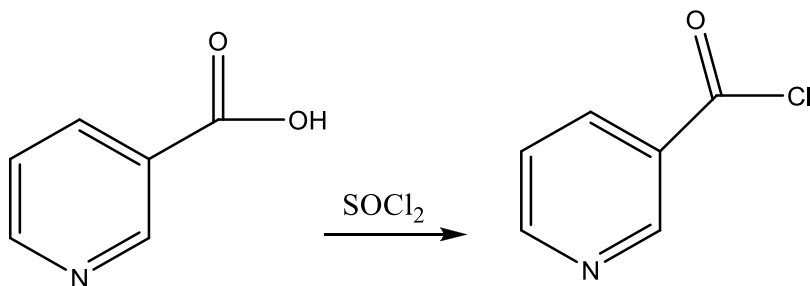
## Result and Discussion

Several studies have shown that heterocyclic compounds are important in medical field as it is used cure<sup>(13)</sup> as antibiotics, and has been used industrially, and has been used industrially in the manufacture of dyes<sup>(14)</sup>, as well as for anti-copper consumption due to their trophic importance in various fields of interest to researchers, these compounds are involved in the structure of chlorophyll

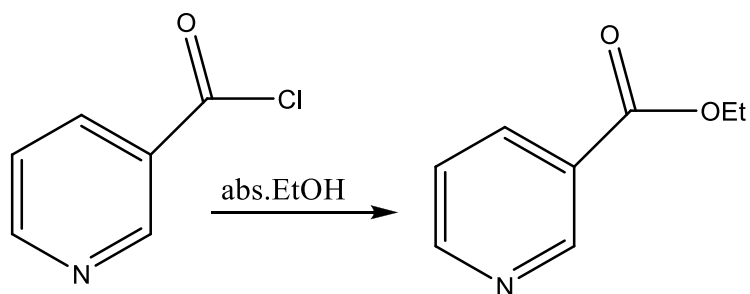
inplants oxadiazol and phrazole compounds are heterogeneous penta - compounds - in this study, nicotinic acid was used as anucleus to prepare non-penta circulating cyclocompounds for the purpose of producing compounds of expected biological in terest. The scheme(1) was shown the prepared hydrazide (4)



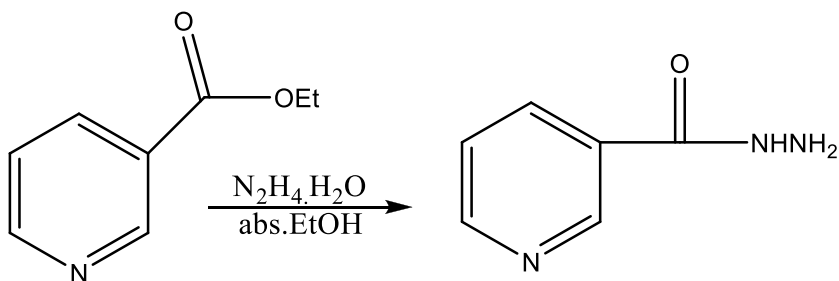
Nictinoyl chloride are Prepared by adding thionyl chloride to nicotine acid in etheridry:



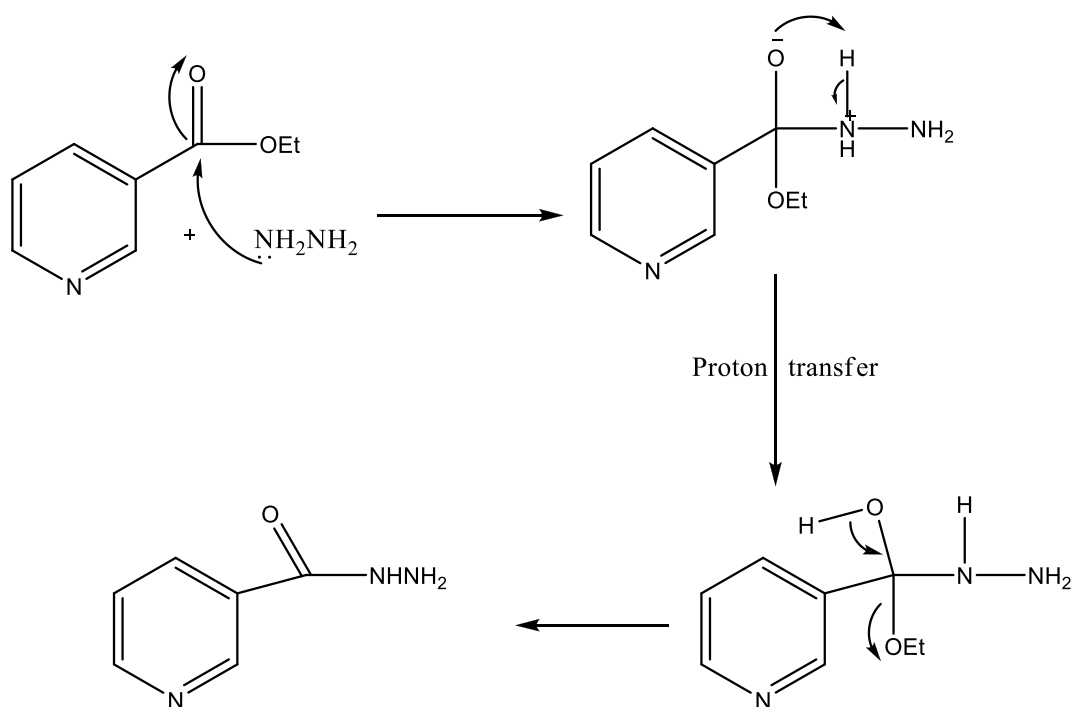
and a composite person with in frared spectros copy if the spectrum shows frequency bands at as asharp and strong (c-o) due to the karonel frequency (1742-1792  $\text{cm}^{-1}$ ) at the frequency (c = c ) and the absorption bands of the (O-H) bond absoption band ( 3068  $\text{cm}^{-1}$  ) Armenian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



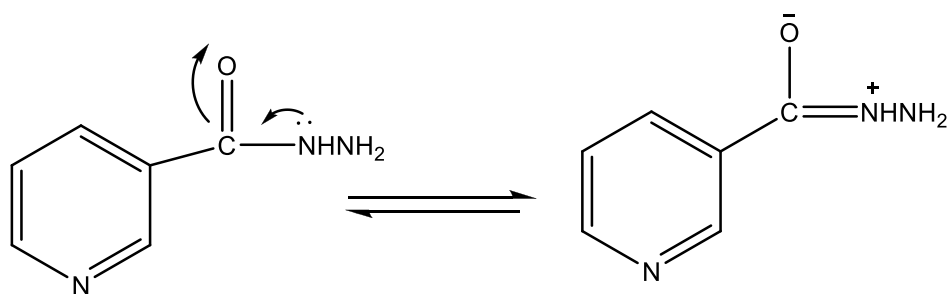
The resulting ester showed a clear detection using ferric hydroxamate detection using ferric hydroxamate detection indicating <sup>(16)</sup> that (IR) the ester set and the ester person produced with infrared spectroscopy are the carbon strand band (1745 $\text{cm}^{-1}$ ) the band of maths at (3071 $\text{cm}^{-1}$ ) frequency (2980-2830 $\text{cm}^{-1}$ ) the symmetric and asymmetric C-H of the (C-H) band. While hydrazides are biologically active compounds and have been used as a starting material to prepare new high value compounds by turning the main to the heterogeneous pentacyclic compounds preparation of hydrazides by ester reaction with absolute watery hydrazine in ethanol as shown in the following equation:<sup>(17)</sup>



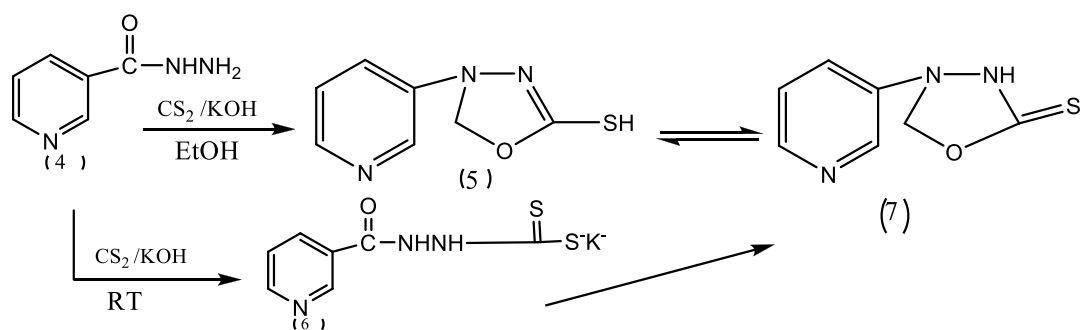
The reaction of hydrazine with ester is based on nucleophilic substitution on a carbon atom the ester carbonyl group is reacted by a nucleophilic mechanism by means of a double electron attack of the amine group in the hydrazine on the ester group, as a hybridization of an atom that forms an active intermediate with  $\text{sp}^3$  deletion is converted to a (C-O) carbon in the carbonyl group, the ethoxy group gives the hydrazide, as described in the following mechanism:



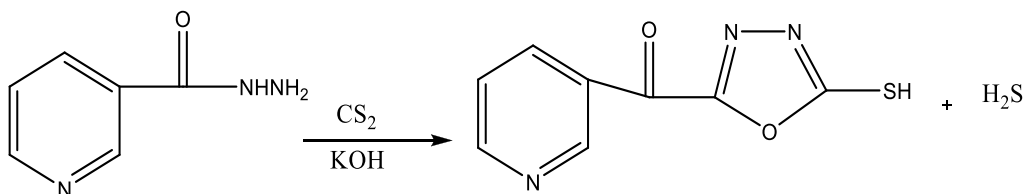
hydrazide was diagnosed as spectros by the infrared spectrum ,it goes back to ( $1664- 1623 \text{ cm}^{-1}$ ) and (N-H) Bear and ( $3300\text{cm}^{-1}$ ) the two carbonyl amide groups attached to the cyclic and terminal respectively the absorption of hyrazide by the karionel group has been abser red to have low - frequency displacement compared to the carbonyl group in the ester<sup>(18)</sup>, 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.



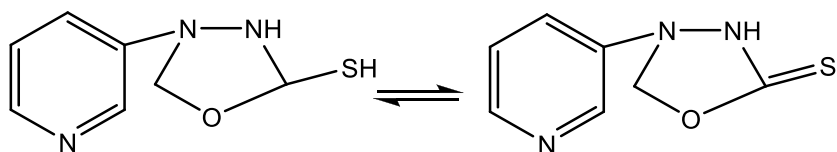
Hydrazide (4) was used to synthesis of substituted 1,3,4- oxadiazole (7) as shown in scheme (2)



In CS<sub>2</sub>, the compound was prepared by the reaction of the hydrazide with the carbon disulfide Alcoholic potassium hydroxide.

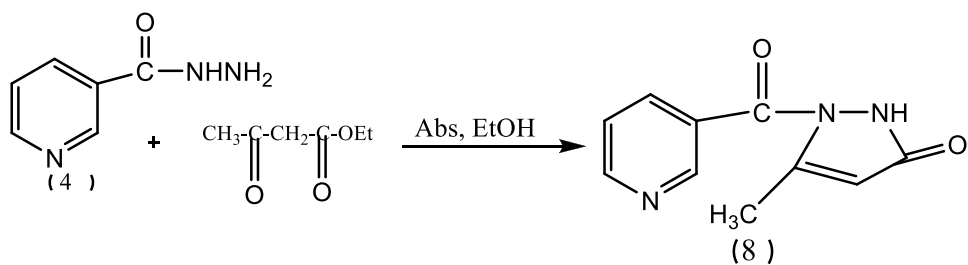


This interaction is an important reaction in the preparation of oxydiazol compounds containing a thiol group. It says a gas molecule, hydrogen sulfide, forming the oxydiazol-2-thion ring:

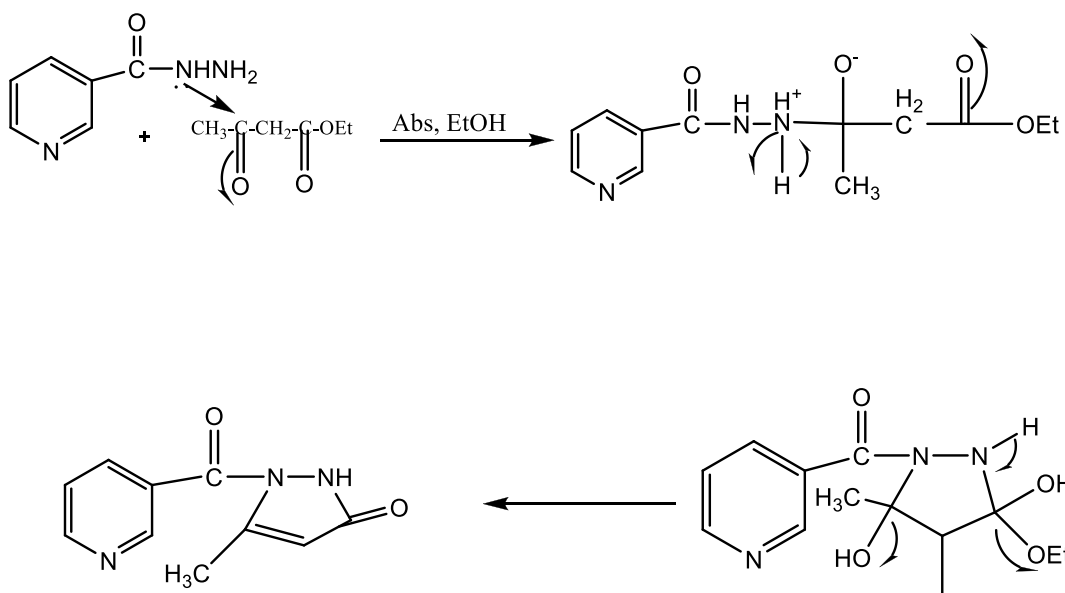


The interaction is done by nucleophilic addition mechanism that involves an attack carbon-based nucleophile of the imino, giving a carbon-carbonyl molecule to a thio carbazide<sup>(19)</sup> and then an implicit nucleophilic attack followed by the loss of a molecule. In the oxydiazol folds using infrared spectroscopy Exemption packets appear at the thion group gave absorption bands at C-C (1595cm<sup>-1</sup>) the frequency and showed pattern bands returning which confirms the state of the nanometer of the ion and thiol forms (1135cm<sup>-1</sup>)<sup>(20)</sup> it goes back to the pattern at (1424cm<sup>-1</sup>) at the frequency (N-N)<sup>(21)</sup> of (C=N).

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



The compound was prepared from the medrize reaction with 1,3 -dicaronyl compounds such as acetoacetyl<sup>(22)</sup> acetate and acetylasetone as in the following equation:



An absorption band at the infrared (IR) pyrazole person<sup>(23)</sup> afrequency band for the carneill group (CN) of the band bands (1580-1639 $\text{cm}^{-1}$ ) at the band frequen cies (1057-1027 $\text{cm}^{-1}$ ) at (N-N) and the band (1666 $\text{cm}^{-1}$ ) ving at the band<sup>(24)</sup> (3335 $\text{cm}^{-1}$ ) at the band (N-H) showed the pattern band return.

## Conclusion

We conclude that the Nicotinic acid 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

## References

1. Niacin"Drug Bank :a know lodge base for drugs" drug actions and drug targets,48, (2019)

2. Niacin-pubchem public chemical Database" The pub chem. project use National center for Biotechnology Information,84. (2014)
3. M. ElBairi, K, Amzazi .s. Toxicological properties of fenugreek "Trigonella foenum graecum ", 159 (2016)
4. D.Hathcock, J. Guarneri "Niacin: chemical forms, bioavailability, and health effects" 345-295 (2016)
5. Rolfe, Heidi M " Journal of cosmetic Dermatology, 324-328 .(2017)
6. Schulze and Trier, Zeit, physiol, chem, (40) (2020)
7. B.Valerie, (Neuronal nicotinic receptors from protein structure to function) (2001)
8. H. Gilman, Org.Syn.Coll.,Vol.1,John Wiley and sons, New York, p394 (1941)
9. M.N. Ibrahim, M.S. Saeed and S. Fadhil, J .Iraqi Chem. Soc. P159.(1987)
10. Ayman El-Faham, Zainab Al-Marhoom, Ahmed Abdel-Megeed and, Mohamed. S, Journal of Chemistry, Saudi University, 1,6, (2013)
11. M.Amir, K. Shika, J. Europ. Med. Chem. 39,535, (2004)
12. M.I.EL-Zahar,s.s.El-Karim, M.E .Haiba, World Journal of Chemistry,182-194 (2009)
13. A. S. Founda, M.M. Gouda and S. I. Abed EL-rahman, Bull .Korean chem. Soc, 21(11),1085-82,(2000)
14. L. Zhang, A. Zhang, X.X. Chen, X.X. Lei, X.Y. Nan, D. Y. Chen and Z.Y.Zhang,Molecules,7,681-689,(2000).
15. F.C. Charles and F.W. Mary, Experimental organic chemistry-A Small Scale Approach, Prentice Hall, Englewood Cliffs, New jersey, 2ed ed, USA,59, (1995)
16. A Shish Kumar Tewari and Anil Mishra, Indian Journal of chemistry, 8,234, (2006)
17. Ayman El-faham, Zainab Al-Marhoom Ahmed Abdel-Megeedand, Mohamed.s, Journal of chemistry, Saudi University, (2013)
18. E.Diez-Cecilica, B.Kelly, and I. Rozas, Tetrahedron Letters (2011)
19. S.J,Sinclair, R. Johnson and J.D, Hamil, Analysis of activities compound in Nicotiana species with contrasting alkaloid profiles functional plant Biology,(2004)
20. J.P Greenstein , M. Wintz (chemistry of the amino acids) John Wiley and Sons, INC , New york, (1986)
21. Rajeer Bhimwal, Anil .k. Sharma, Ankit Jain, Journal of Advanced pharmacy Education and Research, 251-258, (2011)
22. L.J. Bellomy. "The origin of group frequency shift abnormal frequencies in multiple bonds" Spectrochimica Acta, pergamon press, ltd, London,(66), (1958)
23. F.E.M.EL. Baih, J. saudi chem. Soc ,( 59) (2004)
24. K.M. Daoud, A.K. Ahmed J. educ. sci. (40). (2000)