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СИНТЕЗ И ХАРАКТЕРИСТИКА СЛОЖНОГО ЭФИРА НИКОТИНА ИЗ ДИНИКОТИНОВОЙ КИСЛОТЫ И 3-АМИНО-1-ПРОПАНОЛА

Аннотация: *В этой статье сложный эфир никотина был синтезирован прямой реакцией сложного эфира (сложный эфир Фишера) между 1 молями диникотиновой кислотой и 6 молями 3-амино-1 пропанола, используемого в качестве растворителя, и субстратом в присутствии кислой среды, Амберлист 15. Соединение охарактеризовано на основании 1H-ЯМР и инфракрасной спектроскопии.*

Ключевые слова: *Диникотиновая кислота, 3-амино-1-пропанол, Амберлист 15, гетероциклическое соединение.*

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SYNTHESIS AND CHARACTERIZATION OF NICOTINE ESTER FROM DINICOTINIC ACID AND 3-AMINO-1-PROPANOL

Abstract: *In this research, a nicotine ester was synthesized by a direct ester reaction (Fischer ester) between 1 mole of Dinicotinic acid with 6 moles of 3-amino-1 propanol used as a solvent and a substrate in the presence of an acidic medium, Amberlyst 15. The compound was characterized based on 1H-NMR and infrared spectroscopy.*

Key words: *Dinicotinic acid, 3-amino-1-propanol, nicotine ester, Amberlyst 15, heterocyclic compound.*

1. Introduction:

Dinicotinic acid, also called pyridine-3, 4_di carboxylic acid contains a heteroaromatic Pyridine ring and a two carboxylic acids functional group at site 3 and 4.

Dinicotinic acid is in use since 1955. It Contributes to cure of different diseases and used for multiple purposes, so it is an important compound in the chemical industry as

it is widely used in the manufacture of medicines, cosmetics, perfumes, flavors and resins as well as in other products and detergents. [1, 2]

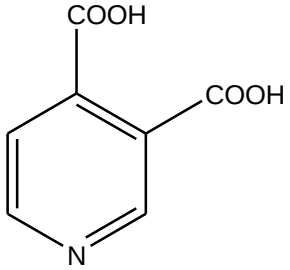


Figure 1. Structural formula of Dinicotinic acid

There are many analytical applications such as the use of nucleotides to treat stomach acidity and dilute it by modifying the acid structure, as well as in the field of perfumes and cosmetics. The nucleotides prepared in this article are esters resulting from the interaction of dinicotinic acid with an amino alcohol, where the reaction results an ester and water, which is a direct esterification reaction in the presence of a suitable catalyst as the below Chemical equation: [3,4,5]

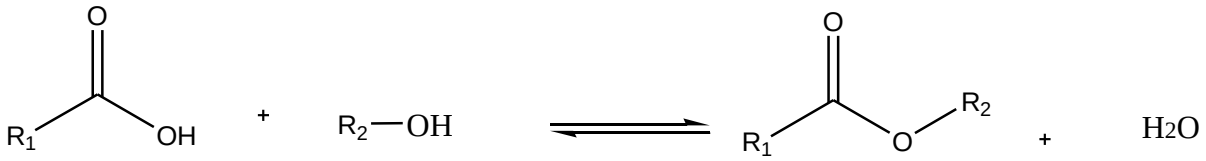


Figure 2. General reaction of a fisher ester

In this article we will synthesis and characterize nucleotide ester from the interaction of 1 mole of dinicotinic acid with 6 moles of 3-amino-1- propanol. [6]

2- Experimental:

2.1. Materials:

Dinicotinic acid, 3-amino-1-propanol and Amberlyst 15 was used as a heterogeneous acidic catalyst. All the solvents and chemicals were used of analytical reagent grade without further purification. Spectroscopic grade solvents (Merck-India, Ltd.) were used for UV–Visible, 1H-NMR spectroscopy and TLC Plates were used also from Merck-India.

2.2. Methods and instruments:

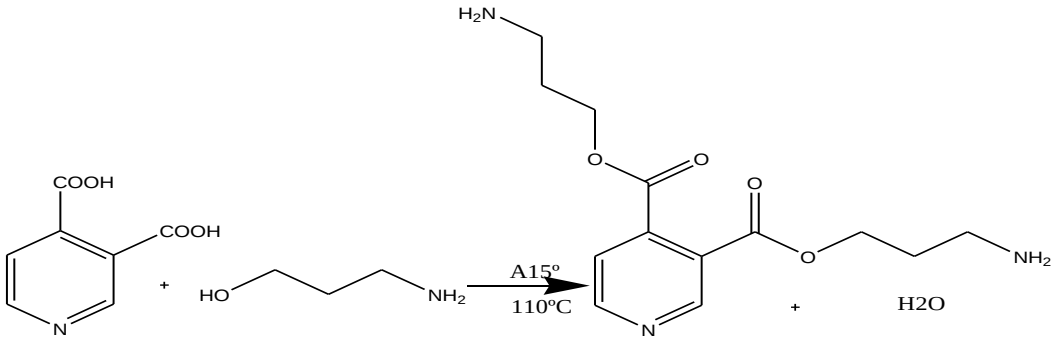
Melting points were determined by open capillary method, The Infrared spectra FT-IR were recorded in the range (400-4000 cm-1) by KBr pellet from the Japanese company Jasco spectrophotometer. The Proton nuclear magnetic resonance 1H-NMR and the Carbon-13 nuclear magnetic resonance 13C-NMR device 400 MHz model by Switzerland company, Rotary evaporator model 4.91 from Normschiff German company , thin layer chromatographic of aluminum coated by Silica Gel , Sheets chromatography thin layer glass coated with Silica Gel 60F254 measuring 20 x 20 from Merck German company.

2.3. The method of work:

Preparation of bis (3-aminopropyl) pyridine_3, 4-dicarboxylate:

(0.004mol, 0.5gr) of dinicotinic acid is added to a ball flask equipped with a magnetic stirrer, then (5% mol) of the acid medium is added. Then (1,37gr, 0,024mol) of 3-amino-1 propanol are added, then the reaction mixture is stirred at A temperature of 110 ° C while monitoring the reaction process by means of thin layer aluminum chromatography, using a dredging system consisting of (methanol: dichloroethane) (10%: 90%).

Then the reaction product was extracted with an ethanol extract several times, the remaining acid was deposited by cooling, and finally the organic phase was collected and evaporated by a rotary evaporator under vacuum, and it is purified by using glass plate chromatography to obtain a white solid product with yield (69%).



Scheme 1. Synthesis of bis (3-aminopropyl) pyridine_3, 4-dicarboxylate

Table 1. Shows some physical properties and yield of bis (3-aminopropyl) pyridine_3, 4-dicarboxylate

Nicotine ester	Melting point	Color	Yield
the product	149-150 ºC	White Powder	69%

3. Results and discussion:

3.1. Synthesis and characterization of bis (3-aminopropyl) pyridine_3, 4-dicarboxylate:

1H-NMR (400 MHz, Methanol):d= CH=N aromatic(s,1H) 8,7PPM ,C_H aromatic (d,1H) 7,9-6,5 PPM , CH2-O (m ,1H) 4,3 PPM ,CH2-NH2 (m,4H) 2,9 PPM , NH2(t,4H) 2,2 PPM, CH2 (m,4H) 1,9 PPM.

IR (KBr) cm-1: m = 2922 (Csp2_H), 1628 (C=O), 1386 (CO), 774 (C_H of aromatic ring).

(Scheme 1) shows the synthesis of bis (3-aminopropyl) pyridine_3, 4-dicarboxylate by the reaction: Dinicotinic acid with six moles of 3-amino-1- propanol, the use of 3-amino 1 propanol as a reactant and a solvent together in the presence of an acidic heterogeneous medium Amberlyst 15, which was extracted and purified to form white crystalline products. This ester is obtained well yields (69%). The structure of these ester was established by spectral studies (IR, 1H-NMR). The infrared spectra of the ester exhibit the carbonyl group shift property, it is also noted from the comparison of the spectra of both dinicotinic acid and the resulting ester a shift in the absorption value of each of the carbonyl and ester groups (1628cm-1) than they are for the carbonyl group in the acid (1700 cm-1) indicating the formation of the compound.

4. Conclusion:

In this research, an aromatic mono-ester was synthesized from dinicotinic acid with 3-amino-1-propanol, and the structures of the products were determined by modern spectroscopic methods.

An environmentally friendly synthesis method was used through the use of 3-amino-1 propanol as a reactant and a solvent together, the use of a heterogeneous medium that is easy to separate from the reaction medium.

5. Acknowledgment:

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