



Synthesis Identification of the New Heterocyclic System from Lactam

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Received: 7 March 2023

Accepted: 18 May 2023

Published: 20 July 2024

doi.org/10.30526/37.3.3318

Abstract

The importance of heterocyclic compounds has long been recognized, and the use of chemicals has remained constant, including in the manufacturing of pharmaceuticals. Several lactam synthesis methods have been developed. Among the essential compounds in our lives are lactams from amines and aldehydes and the preparation of Schiff bases. Schiff bases are prepared in two ways, including the reaction of hydrazine and various aldehydes with tetraethylamine and the production of a Schiff base, which leads to the formation of two types of lactams and the preparation of a kind of reaction of different amines and aldehydes. Three techniques prepared the lactams, the first being Schiff bases with acetyl chloride and triethylamine as catalysts and using dioxane as the solvent; The second is from Schiff bases with carboxylic acids; The third is aldehydes with amines. It produces beta-lactam derivatives (azetidines), and this research focuses on the production of lactams and their polymerization through their reaction with ethanol once and with ethanol and the base to form industrial polymers, which are polymers that are produced in the form of a final product with an active end and are used in many industries. These polymers have moderate to high antioxidant activity, and after being treated with safer natural materials, they are environmentally friendly compared to other materials. Its chemical composition was evaluated by the following required tests (FTIR, HNMR).

keywords: Acryle amide, β -lactam, poly vinyl alcohol, Schiff base.

1. Introduction

β -lactams are a well-known class of chemicals of great organic importance. Being an exciting catalyst, they also function as flexible organic chemical compounds. Indeed, due to its accessibility through multiple technologies and its inherent reactivity due to ring compaction, the β -lactam ring is the most sought-after substrate in the natural-synthetic chemists' armamentarium. Many reagent, heat, and light companies sell ring-forming and ring-creating merchandise [1]. Azole compounds are a family of substances that have a solid organic impact, and they are heterocyclic compounds containing nitrogen atoms [2], several nonantibiotics [4].



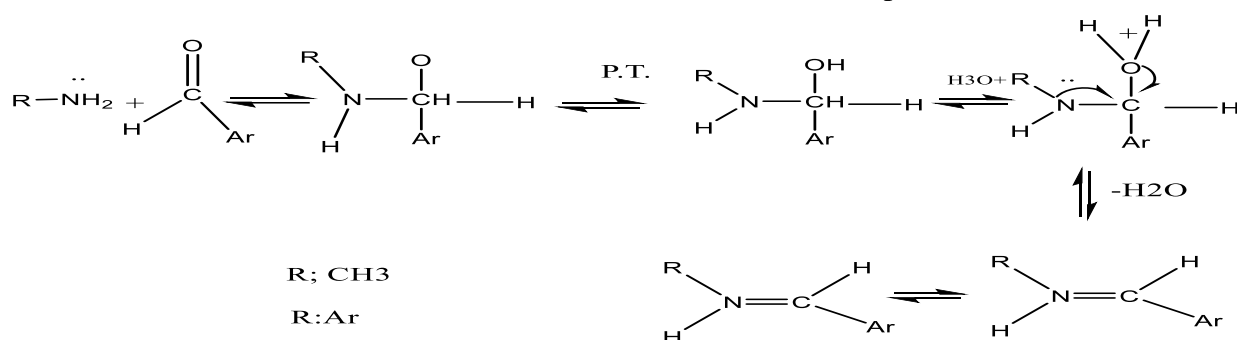
β Lactam, a type of Molecule, has influenced the advancement of chemical transformation tools [3]. The β -lactam-the-azetidinone ring technique is also employed in the creation of Tetrazoles are derivatives of oxazepines, which are a heterogeneous ring of (O and N) atoms to which an oxygen atom has been inserted. While the atom of nitrogen can be found at positions Tetrazoles are derivatives of oxazepines, which are a heterogeneous ring of (O and N) atoms to which an oxygen atom has been inserted. While the atom of nitrogen can be found at positions -2, -3, or -4 [5]. β -lactam is a heterocyclic amide ring composed of one nitrogen atom and three carbon atoms [6]. The synthetic route for azomethine compounds comprises the condensation of ammonia, first amines, and amino acids with carbonyl compounds using azeotropic distillation while simultaneously removing water [7]. The azomethine structural feature is recognized in these substances [8,9]. Schiff bases and their metal complexes played an essential role in our understanding of the coordination chemistry of transition metallic ions. Schiff bases and their structural counterparts, asligating compounds with acyclic and cyclicine C=N linkages, are essential in coordination chemistry [10,11].

2. Materials and Methods

All chemicals and solvents were obtained from the BPC-Analysis Center. FT-IR (Fourier Transform Infrared Spectrophotometer) measurements were made using a KBr disk on a SHIMADZU FT-IR-8300 spectrophotometer. The experiments were carried out at room temperature, and the FT-IR spectra were acquired in the 400–4000 cm^{-1} range to estimate the functional group of chemical compounds. The HNMR studies were performed at the Iran, University of Tehran.

2.1 Synthesizing Schiff (1-5)[6, 9,12,13]

About (0.5 g, 0.01 mol) of hydrazine hydrate with different aliphatic and aromatic aldehydes (formaldehyde, acetaldehyde, propionaldehyde, pnitrobenzalde, benzaldehyde) (0.3 g, 0.01 mol), (0.4 g, 0.0 mol), (0.58 g, 0.01 mol), (1.5 g, 0.01 mol), (1.3.0.01 mol) respectively in 25 mL absolute ethanol and then added few drops of glacial AcOH and refluxed at 0°C stirring for (6-8) hours This mix has been filtered, allowed to cool at the room temperature.

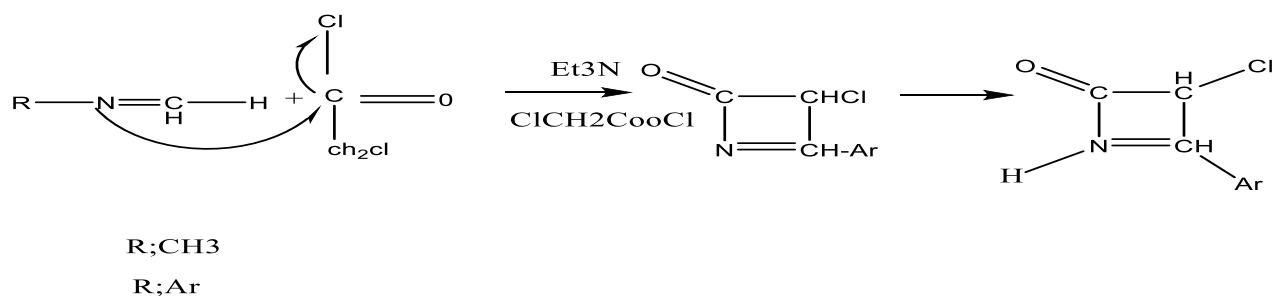


Scheme 1. Synthesis mechanism of Schiff base with glacial acetic acid catalysis (N'(benzylidene)hydrazide).

2.2 Synthesis of β -lactam from Schiff base and aldehyd compounds (6-10)[10,11,13,14]

About (0.1 g, 0.01 mol), (0.4 g, 0.01 mol), (0.45 g, 0.01 mol), (1.4 g, 0.01 mol), (1g, 0.01 mol) Schiff's base of (methylenehydrazine, ethylidenehydrazine, propylidenehydrazine, 4-nitrobenzylidene hydrazine, benzylidenehydrazine) respectively in 20 mL dioxane when applied to mixture a few drops of Et₃N, then (0.5 mL) of chloroacetyl chloride was added dropwise. The mixture was mixed well at -0°C, stirring for (6-8) hours; after that, the reaction mixture was kept at room temperature for two days, after which it was poured into crushed ice. Experimental 41

water. This product was filtered, followed by washing with water, and purified from methanol (1:1).



Scheme 2. Mechanism synthesis of β -lactam (1-amino-4-phenylazetidin-2-one).

2.3 Synthesis of B-lactam from Schiff base and carboxylic acid compound [12,13,15,16]

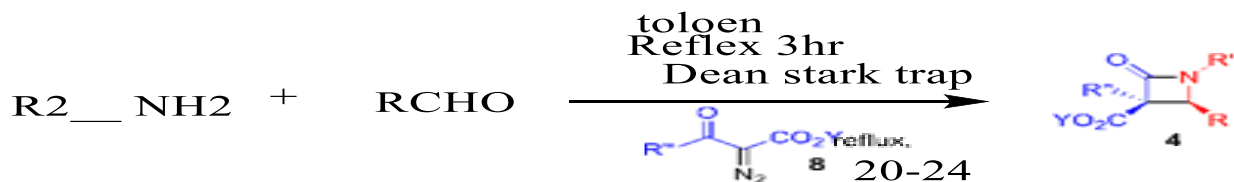
About (0.4 g, 0.01 mol) of Schiff bases (ethylidenehydrazine) and (0.46 g, 0.01 mol), (0.6 g, 0.01 mol), (0.8 g, 0.01 mol), (1.4 g, 0.01 mol), (2.6 g, 0.01 mol) respectively of (formic acid, acetic acid, butyric acid, octanoic acid, palmitic- Experimental 42 acid) derivatives were prepared, then 10 mL of DMF was added to (0.22 g, 1.2 mol) of TCT, cyanuric chloride-2,4,6-trichloro-1,3,5-triazine, and The resulting. The suspension had been agitated for 5 minutes at room T. Along with the Dry Et_3N , the required EtOH (1.2 mol) was added to the (TCT, DMF) solution (0.6 mL, 0.4 mol). Overnight, the reaction mixture was agitated at r.T. Before being dried over (Na_2SO_4) and filtered; the solution was washed with sat. 4 mL of $NaHCO_3$ and 4 mL marinade. The raw material was synthesized. After the solvent evaporated at low pressure. β -lactams were purified by recrystallization from EtOAc and by brief-column chromatography.



Scheme 3. Mechanism synthesis of β -lactam (1-methyl-4-propylazetidin-2-one).

2.4 Synthesis of β -lactam from amin and aldehyd compounds (16-18) [13,14-19]

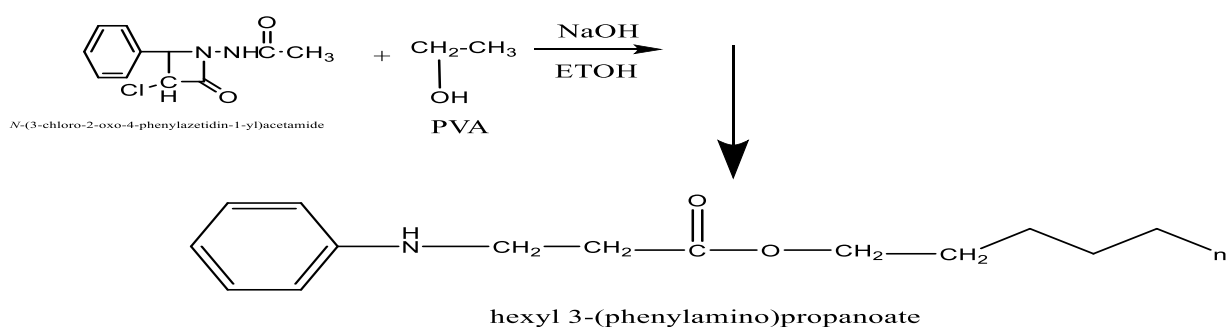
Preparation of (0.5 g, 0.01 mol) in hydrazinehydrate with different aliphatic and aromatic aldehydes (propionaldehyde, 3- methylbutanal, phenylacetaldehyde (0.3 g, 0.01 mol), and (0.4 g, 0.01 mol, 0.58 g) respectively in round-bottom flask, was toluene is dissolved in 10 mL and azeotropic water removal refluxed. Half of the solvent was distilled off after 1 hour, and acyl - diazoacetate (0.8 g, 0.1 mmol, 1.2 g) was included. After that, the mixture was refluxed overnight. Meanwhile, the reaction progress was observed (TLC). When the compound was no longer traceable (diazo), the solvent was evaporated in vacuo, and the remaining aggregate column chromatography on silica gel with a linear gradient was used to purify (0-25%) of (hexane in acetone overall. The amount of eluent required to produce natural compounds was 450 mL.



Scheme 4. Mechanism synthesis of β -lactam (1-aminoazetidin-2-one).

2.5 Synthesis of hexyl 3-phenylamino propanoate [15-20,23-25]

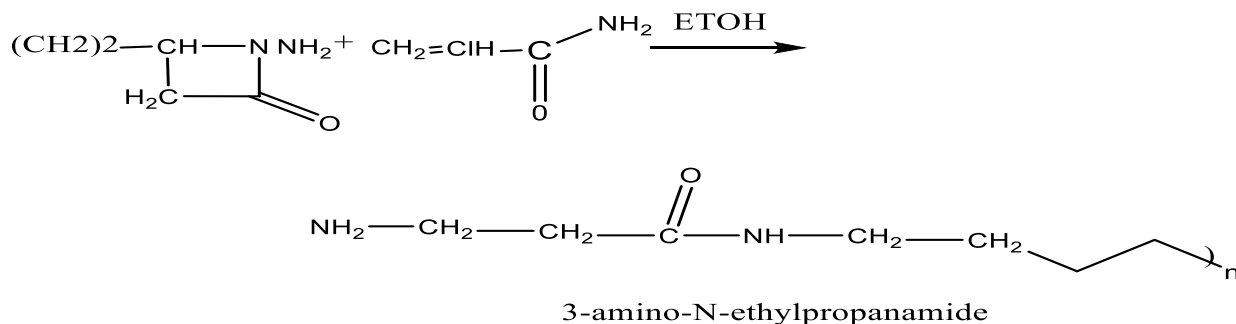
About (1 mol, 0.5 g), (1 mol, 0.6 g), (1 mol, 0.75 g), respectively of lactam about 1 g of polyvinyl alcohol, both dissolved in (EtOH, NaOH), have been refluxed for six hours at 70 °C, the crystalline substance became filtered cease detergent the result to (10 mL) diethyl welkin to oust unpolished last impurities, an apparent white precipitate turned into obtained. This was honored by filtering and drying.



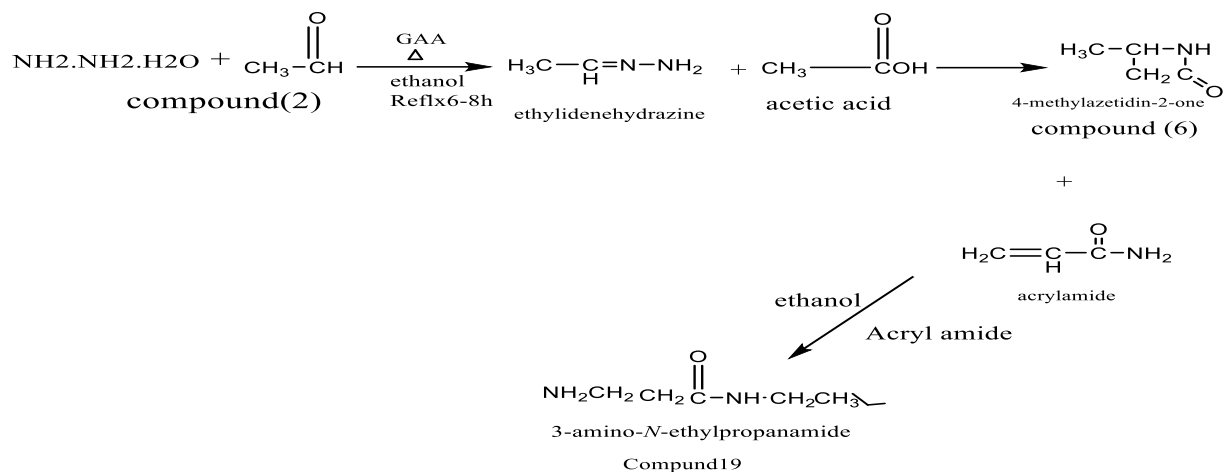
Scheme 5. Mechanism synthesis of poly hexyl 3-phenylaminopropanoate.

2.6 Synthesis of 3-amino-N-ethylpropanamide [25,26,28-31].

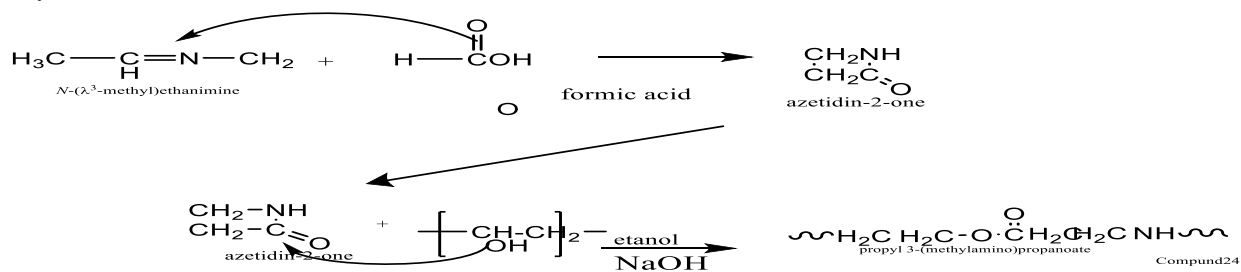
About (0.01 mol, 0.5 g) of lactam mixed with one gram of acryl amide, both dissolved in EtOH, had been refluxed for 6 hours at 70 °C. After washing the product with (10 mL) diethyl ether to remove any last impurities, an apparent white precipitate was received. This turned out to be accompanied by the filtering and drying method.



Scheme 6. Mechanism synthesis of poly 3-amino-N-ethylpropanamide.

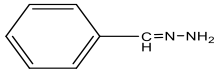
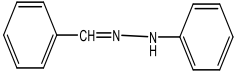
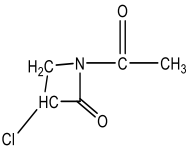
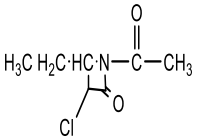
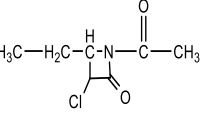
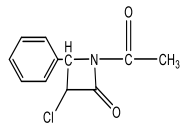
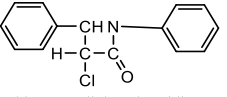
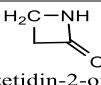
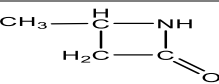


Scheme 7. Synthesis poly amid of lactam of hydrazine with aldehydes formed (Schiff base and carboxylic acid and acrylamide).



Scheme 8. Synthesis of Schiff base and formic acid to form a lactam and its conjugation with PVA.

Table 1. Synthesized chemicals' physicochemical characteristics and FT-IR spectral data cm⁻¹ (1-18).

| No. of comp. | physicochemical characteristics | | | | Major FT-IR absorption cm ⁻¹ | | | | | |
|--------------|---|---------------|--------------|--------|---|---------------|---------------------|------|------|------------------|
| | Compound composition | m.p. °C | Color | Yield% | (N-H) | (C-H) Arom. | C-H Aliph | C=O | C=N | Other bands |
| 1 | $\text{H}_2\text{C}=\text{N}-\text{NH}_2$ methylenediazine | 90 | Græ | 90 | 3414 | ... | 2974-2850 | - | 1624 | |
| 2 | $\text{H}_2\text{C}=\text{N}-\text{NH}_2-\text{CH}_3$ N'-ethylidenehydrazide | 80 | White | 82 | 3240 | ... | 2978 | - | 1627 | |
| 3 | $\text{H}_3\text{C}-\text{H}_2\text{C}=\text{N}-\text{NH}_2-\text{CH}_3$ N'-propylidenehydrazide | 95 | Dark brown | 74 | 3136 | ... | 2978 | - | 1627 | |
| 4 |  N'(benzylidene)hydrazid | 75 | Yellow | 97 | 3236 | 3047 | 2943-2858 | - | 1620 | |
| 5 |  1-benzylidene-2-phenylhydrazine | 73 | Light green | 76 | 3385 | 3059 | 2900-2885 | - | 1642 | |
| 6 |  1-acetyl-3-chloroazetidin-2-one | 77 | Græ white | 66 | 3410 | ... | 2924-2850 | 1678 | - | v(C-Cl) (756) |
| 7 |  1-acetyl-3-chloro-4-ethylazetidin-2-one | 60 | Light Brown | 67 | 3194 | - | 2924-2850 | 1674 | - | (C-Cl) 752 |
| 8 |  1-acetyl-3-chloro-4-ethylazetidin-2-one | 55 - 57 | Brown | 71 | 3460 | - | 2931-2873 | 1670 | - | v(C-Cl) 752 |
| 9 |  1-acetyl-3-chloro-4-phenylazetidin-2-one | 12 0 | Light yellwo | 80 | 3741 | 3194 | 2939 | 1678 | - | (C-Cl) 748 |
| 10 |  3-chloro-1,4-diphenylazetidin-2-one | 13 0 | Yellow gare | 78 | 3400 | 3059- 3024 | 2885 | 1624 | - | (C-Cl) 759 |
| 11 |  azetidin-2-one | 70 | White | 59 | 3387 | - | 2954.94- 2827.64 | 1670 | - | (C-N) 1346 |
| 12 |  4-methylazetidin-2-one | 88 | White | 80 | 3228.83 | - | 2954-2846 | 1670 | - | (C-N) 1346 |

| | | | | | | | | | |
|--------|--|---------|--------------|----|------|------|-----------------|------|------|
| PLS55 | | 165-159 | Light yellow | 77 | 3417 | 3059 | 2950-2885.51 | 1732 | 1311 |
| PLS66 | | 180-188 | Light green | 69 | 3456 | - | 2954-2831 | 1712 | 1346 |
| PLS77 | | 170-177 | white | 80 | 3456 | - | 2958.80-2846.93 | 1681 | 1346 |
| PLS108 | | 188-190 | Light white | 77 | 3417 | - | 2954.95-2846.93 | 1681 | 1350 |

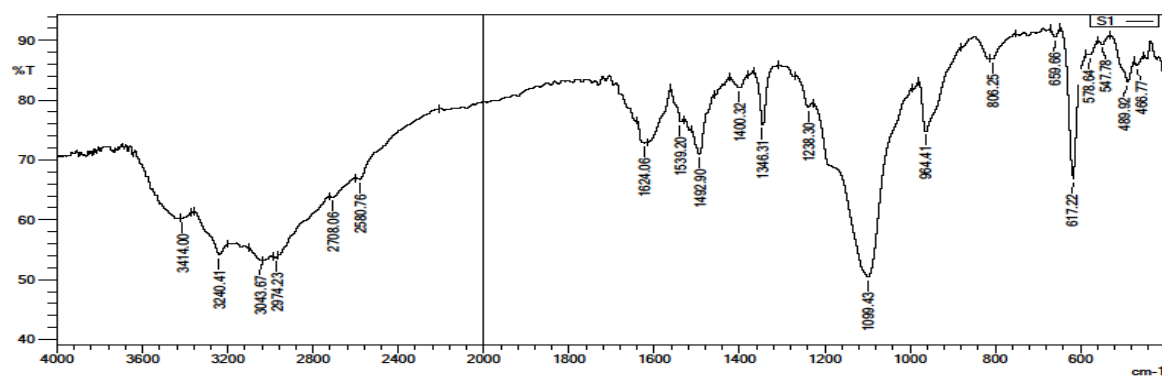
Table 3. The ¹H-NMR spectra (ppm) data for several substances.

| Comp. No. | Compound composition | ¹ H-NMR (δ-H) data in ppm |
|-----------|--|---|
| S1 | methylenehydrazine | DMSO 300 MHz): NH 1.55 amine |
| S2 | N-methylmethanimine | Solvent= DMSO 300 MHz: NH ₂ 8.52 1.50 amine, CH ₃ 2.47 0.86 methyl, H-C-H 1.61 1 alpha - |
| S3 | methylenehydrazine ethane | 0.8-2.5 (d,2H,CH ₂),2.6DMSO (S3.38H,O=C-CH ₃);8.401H, CH ₂ N=NH) |
| S4 | benzylidenehydrazine | 2.50DMSO ,CH ₃ -C=O,H ₃)2.7 (S,1H,H-C=N-N-H),7.38-8.74(H,arom (m), 8.73 (S,H,HN=C-H) |
| S5 | 1-benzylidene-2-phenylhydrazine | Solvent=DMSO 300 MHz):NH 11.49, 1.50 sec amine,fromamineCH) 7.34 - 7.26 , 1-benzene, CH =7.76 - 7.62 benzylidenimin,1 -N-N=C 0.30 , benzylidenimin 7.5 - 7.29 |
| LS6 | azetidin-2-one | Solvent= DMSO 300 MHz: NH 7.1 (propiolactam) CH ₂ (3.08), β-lactam -N-C=O 0.46 |
| LS7 | 1-acetyl-3-chloro-4-ethylazetidin-2-one | Solvent-DMSO 300 MHz: (CH) 5.0, 3.08 propiolactam, 1.98 1 α-Cl from , methane CH 4.2, CH ₃ 3.42 ,CH ₂ 1.62 ,methylene, N(C=O) C=O 0.22 1 β- |
| LS8 | 1-acetyl-3-chloro-4-ethylazetidin-2-one | Solvent= DMSO 300 MHz: CH 5.0, 3.08 propiolactam,alpha -Cl from methane 1.98 Propiolactam 3.42 ,C(=O)R from N-CH 1, β-Lactam -N-C=O 0.35 |
| LS9 | 1-acetyl-3-chloro-4-phenylazetidin-2-one | 1.9 (s, 1H, CH);3.5(S, N-H,1H); 3.2 (S, 3H, CH ₃); 4.2(CH-Cl)7.5-8.5. (m, 4H, Ar-H),8.75(d,1H,NH-NH-C=O,β-Lactam -N-C=O 0.41 |
| LS10 | 3-chloro-1,4-diphenylazetidin-2-one | 1.2(s, 3H,CH ₃); 3. (S, 1H,CH ₂ =); 6.51 (S, 6H, N-(CH ₃)); 6.92-7.99 m, (7H, (Ar-H)) and 8.75,(NH, H,) 5.6 (CH-Cl),β-Lactam -N-C=O 0.32 |
| LS11 | azetidin-2-one | Solvent=DMSO 300 MHz): NH 7.1 propiolactamCH ₂ 3.42 , β-Lactam -N-C=O 0.37 |

| | |
|--|---|
| <p>LS12</p> <p>4-methylazetidin-2-one</p> | <p>Solvent=DMSO 300 MHz): NH =7.1,CH 3.52 , propiolactam , 3.42, CH2 3.15,CH3 1.31, 0.86 methyl 0.34 β-Lactam -N-C(=O)C 022</p> |
| <p>LS13</p> <p>1-methyl-4-propylazetidin-2-one</p> | <p>Solvent=DMSO 300 MHz):,CH 3.10 propiolactam CH2 2.88;2.63 3.08 propiolactamCH3 3.27 alpha(-N(C)-C=O) 0.34</p> |
| <p>LS14</p> <p>4-(2-methylpentan-3-yl)azetidin-2-one</p> | <p>Solvent=DMSO 300 MHz): NH 7.0 ,CH 3.22,propiolactam CH2 3.15;2.895 3.08 propiolact 1 β-C from -0.06 methylene CH 1.72 1.50 methine (0.62 , β-Lactam -N-C=</p> |
| <p>LS15</p> <p>4-butylazetidin-2-one</p> | <p>Solvent=DMSO 300 MHz):,NH 7.2 CH 3.3 propiolactamCH2 3.15;2.895 3.08 propiolactammethylene CH2 1.47 , 1.37 (CH=) methyle, β-Lactam -NC(=O)-C 0.38</p> |
| <p>LS16</p> <p>1-aminoazetidin-2-one</p> | <p>Solvent= DMSO 300 MHz): NH2 7.1CH2 3.4 3.42 propiolactam</p> |
| <p>LS17</p> <p>1-amino-4-ethylazetidin-2-one</p> | <p>Solvent=DMSO 300 MHz): NH2 =7.0 (CH) 3.3, 3.42 propiolactam (CH2) 1.62 1.37 methylene , β-Lactam -NC(=O)-C 0.24</p> |
| <p>LS18</p> <p>1-amino-4-ethylazetidin-2-one</p> | <p>Solvent=DMSO 300 MHz): NH2 7.0 ,CH 3.3, 3.42 propiolactam CH2 1.62 ,β-Lactam -NC(=O)-C 0.22 methyl β-Lactam -N-C=O 0.4</p> |
| <p>LS19</p> <p>1-amino-4-phenylazetidin-2-one</p> | <p>Solvent= DMSO 300 MHz): NH2 7.0 ,CH 4.77, 3.42 propiolacta CH 7.36 , 7.26 1-benzeneI CH 7.32 , 7.26 1-benzene2, β-Lactam</p> |

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BPC-Analysis Center



Baghdad- Adhamiyah near al Nu'man
 teaching hospital
 Cell Phone:- 07706064908- 07833056241

Figure 1. The FT-IR data Schiff base for compound 1.

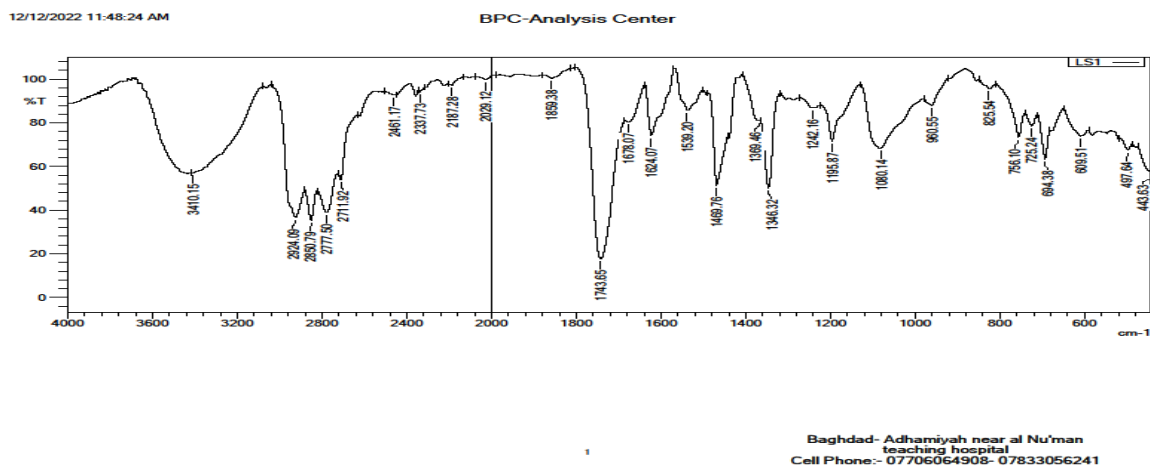


Figure 2. The FT-IR data lactam for compound 6.

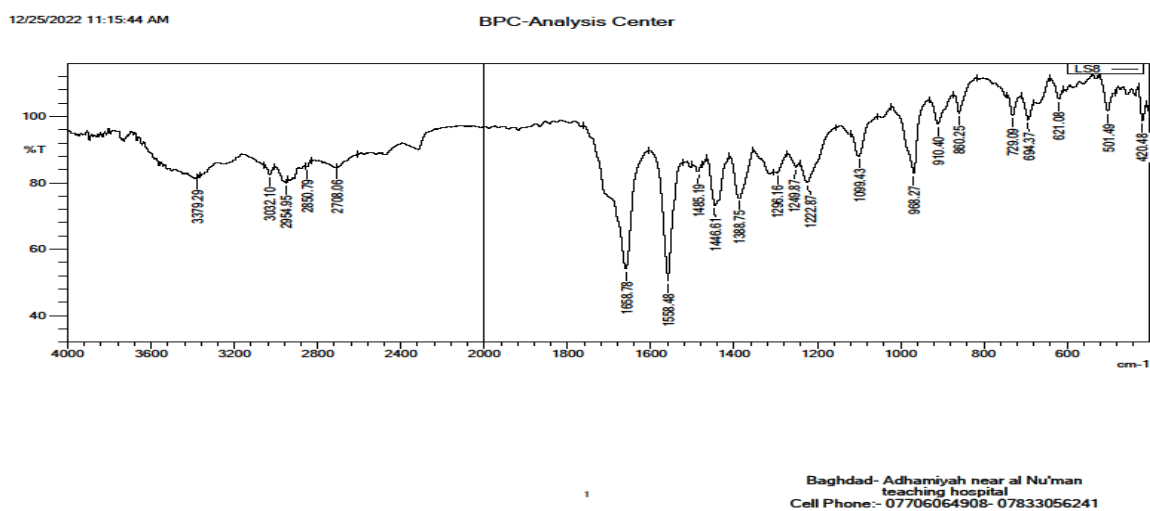


Figure 3. The FT-IR data lactam for compound 13.

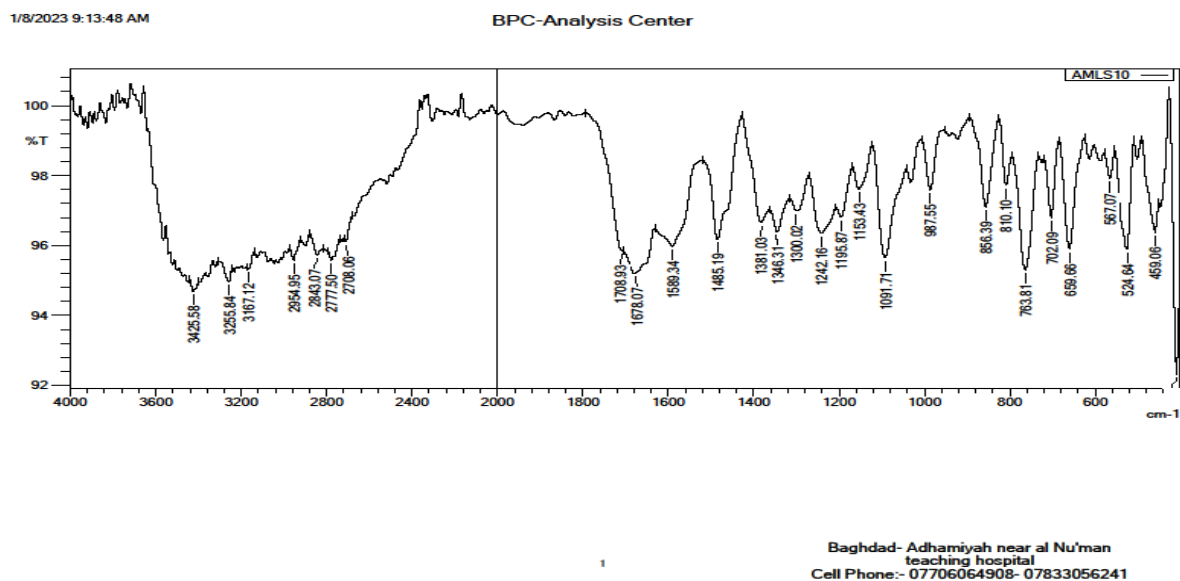


Figure 4. The FT-IR data for compound 27 (poly 3-amino-N-ethylpropanamide).

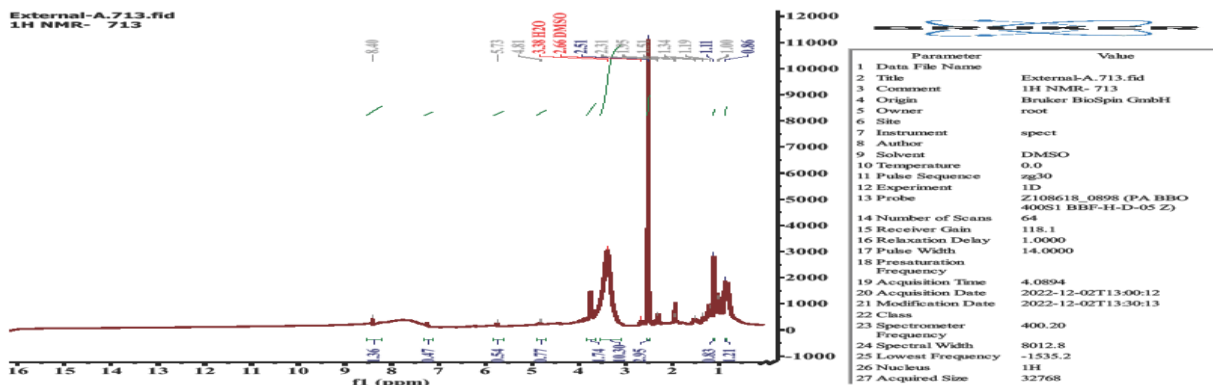


Figure 5. Compound 3 (H-NMR) spectrum.

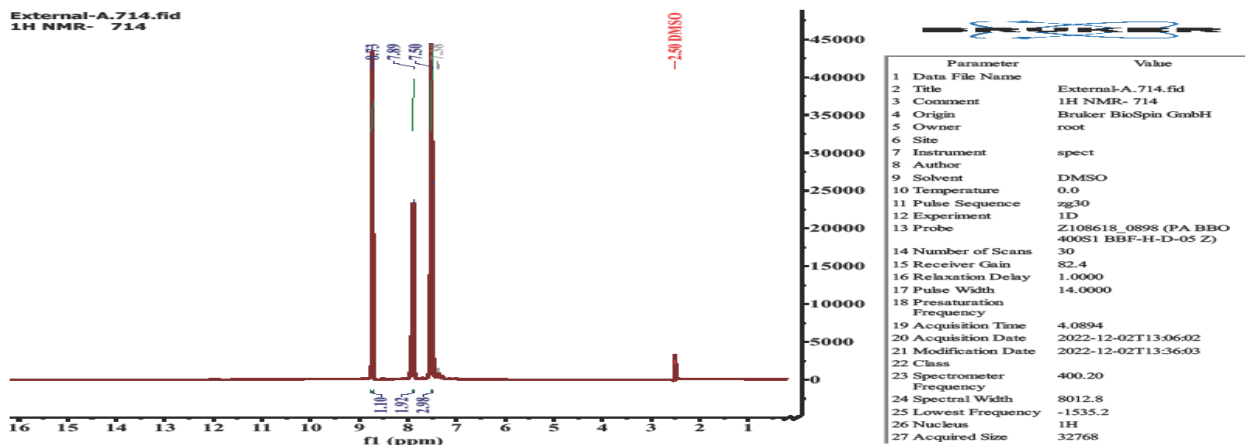


Figure 6. Compound 4 (H-NMR) spectrum.

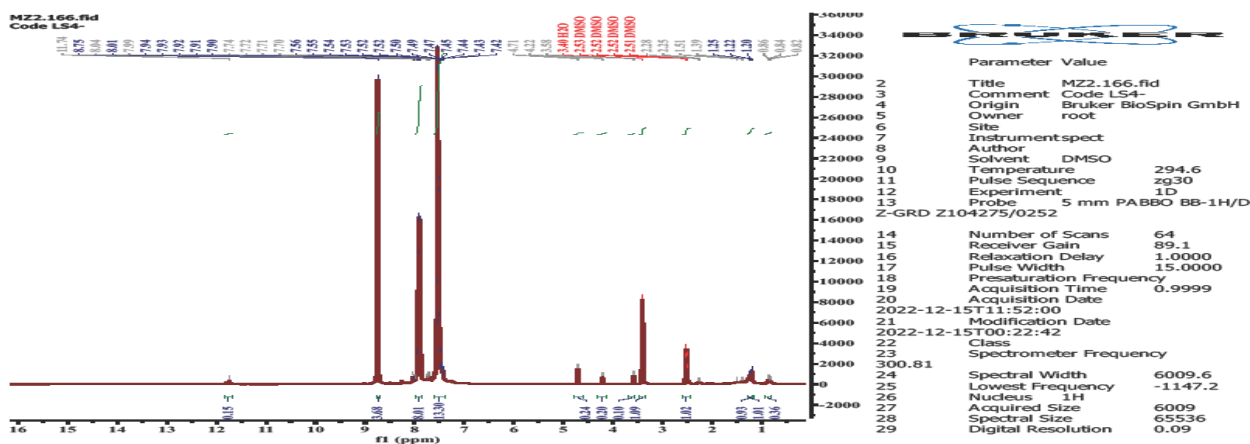


Figure 7. Compound 9 (H-NMR) spectrum.

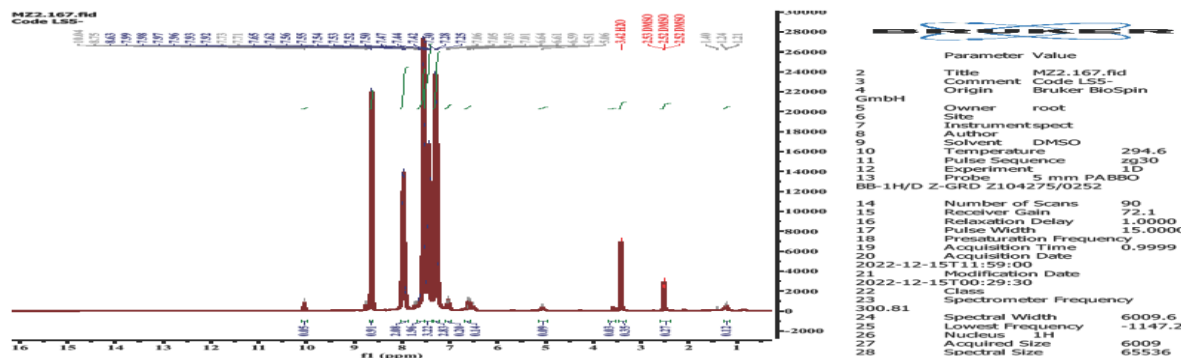


Figure 8. Compound 10 (H-NMR) spectrum.

3. Results

Infrared radiation absorption in chemical bonds is the basis spectroscopy. The bond is connected, to changes in the constant, dipole moment (such as stretching and bending). The FTIR is frequently used in product identification to determine certain functional groups or chemical compound bonds.

4. Discussion

The characteristic peaks in the FTIR spectra of all produced derivatives are presented below. **Table 1** and **Figures 1-4**. The structural equation, yield, percentage, melting point, and color were all displayed. This chemical has the highest yield. Compounds (1–5) melting point was 73–90 °C. The melting point was compounds (6–18). 60–90 °C Som stik The components (1–5) were created for reacting (hydrazine and anilen) with various chemicals (aliphatic and aromatic). In the presence of a solvent (ethanol), aldehydes (acetyldehyde, formaldehyde, propionaldehyde, and benzaldehyde) were produced. The produced chemicals (1–5) were identified using the FTIR spectrum shown in **Table 1**. These are the spectra. Showed absorption, (NH), respectively at (3240.41, 3240.41) cm^{-1} , $\nu(\text{C}=\text{O})$ respectively at (1624.06, 1627.92) cm^{-1} . **Table 1** contains other absorption chemicals. By reacting Schiff bases (1–5), Et₃N, and chloroacetyl chloride, the compound (6–10) was created. These spectra revealed (NH) at bsorption (3410.15, 3194.12) cm^{-1} . $\nu(\text{C}=\text{O})$ at (1678.07, 1624.06) cm^{-1} , $\nu(\text{C}-\text{H})$ aliphatic at (2954-2885.64) cm^{-1} , (CH aromatic) at (3091) cm^{-1} , $\nu(\text{C}=\text{N})$ at (1597.06). Other absorptions and compounds are found in **Table 1**. Compounds 11–15 were made by reacting Schiff base with acetic acid in In the case of DMF, Et₃N as a solvent, and CH₂Cl₂, ring of absorption (3387.00, 3390.85) cm^{-1} belong to $\nu(\text{NH})$ and show (C=O) at (1670.35, 1670.35) cm^{-1} also ν and other, responding compounds or (16-18) are found in **Table 1**.

The compounds were created by combining hydrazine with aldehyde in existence as a solvent and showed responding $\nu(\text{NH})$ at (3441.01, 3332.99, and 332.32) cm^{-1} , the (CH aliphatic) in (2920–2854) cm^{-1} activation of the C=O group¹³. $\nu(\text{C}=\text{O})$ at (1678.07, 1593.20, and 1620.21) cm^{-1} and other responding compounds are found in **Table 2**. Because its backbone has numerous primary amido corporations. Polymer PAA provides support with respect to one-of-a-kind second-choice chattels organizations, including COOH, NH₂, and -C=O. Poly (acrylamide) is a hydrophilic, high-molecular-weight fabricated polymer with NH₂ businesses on its up-chain drift, similar to chitosan¹⁵. Softening point (110–190) of the compounds in **Table 2** (1-3) lactam and acrylamide, both dissolved in EtOH. These spectra revealed absorption. $\nu(\text{NH})$ at (3425.58, 3425, and 3483.44) cm^{-1} . $\nu(\text{C}=\text{O})$ at (1681.93, 1678.07, and 1693.50) cm^{-1} , $\nu(\text{C}-\text{H})$ aliphatic at (2947.23-2885, 2954.95-2843.07, 2927, and 94-2854.65) cm^{-1} , $\nu(\text{C}-\text{N})$ at (1311.59, 1300.02, and 1303.88) cm^{-1} . The compounds in **Tables 2**. (4–8) were prepared by reacting lactam with polyvinyl alcohol and EtOH NaOH in ethanol's existence as a solvent. These spectra showed responding $\nu(\text{NH})$ at These spectra showed a responding $\nu(\text{NH})$ at (3429.43, 3417.86, 3456.44, 3456.44, and 3417.86) cm^{-1} , $\nu(\text{C}=\text{O})$ at (1739, 1732, 1712.79, 1681.93, and 1681.93) cm^{-1} , $\nu(\text{C}-\text{H})$ aliphatic at (2947.23-2850.79, 2950-2885.51, 2954-2831, and 2958.80-2846.93) cm^{-1} , $\nu(\text{C}-\text{N})$ at (1303, 1311.59, 1346, 1346.31, and 1350) cm^{-1} . For compound (4,5) (CH aromatic) at (3051.39, 3059.10-3028.24) cm^{-1} . The H-NMR chemical spectra of compounds in solvent ethanol is illustrated in **Table 3** and **Figures 5-8**.

5. Conclusion

Heterocyclic compounds are molecules with organic activity. Beta-lactam is a type of molecule discovered in chemical improvements and its effectiveness with many compounds to prepare new polymers of lactam prepared in different ways linked to heterocyclic rings. It can be used in various industries, including industrial and pharmaceutical, after preparing from several materials and forming it with lactam. They are manufactured materials that are safer and more environmentally friendly compared to other various materials. Different lactams have been prepared and polymerized for subsequent cross-linking to different synthetic polymers. In this research, light was shed on the preparation of lactams in various ways and their polymerization in two ways: the first with vinyl chloride alcohol and the base, and the second with acrylamide, where only alcohol was used.

Acknowledgment

The authors would like to thank everyone who contributed to the success of this review article. Department of Chemistry, College of Science for Women, Baghdad University, Prof. Dr. Sanaa Abdul Sahib Ministry of Higher Education & Scientific Research and Ministry of Science.

Conflict of Interest

None

Funding

There is no financial support.

Ethical Clearance

This work has been approved by the Scientific Committee at the University of Baghdad/ College of Science for Women.

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