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Synthesis and Characterization of 1,3-Oxazepindion Derivatives via Diformylbenzene Imines as Precursors

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Abstract

In this paper, 1,3-oxazepindione derivatives [D] and [E] were prepared by reacting Schiff bases di Azomethine [A], [B], and [C] with maleic anhydride and succinic anhydride in the presence of dried benzene as a solvent. Schiff bases di Azomethine [A], [B], and [C] were prepared by reacting Terephthalaldehyde, Isophthalaldehyde, or o-Phthalaldehyde with 5 drops of glacial acetic acid as a catalyst and halo-aniline derivatives in absolute ethanol as a solvent. The prepared compounds were diagnosed and confirmed by using infrared and nuclear magnetic resonance spectroscopies. All the reactions of the prepared compounds were monitored by TLC.

Introduction:

Schiff bases were produced for the first time by the scientist Hugo Schiff in 1864 by thermal condensation between each of the ketones or aldehydes and the primary amine [1]. Schiff bases were distinguished by their biological activities against cancer [2], bacteria [3], and fungi [4]. Schiff bases had given other names, like Azomethine and Imine [1]. The term "heterocyclic compounds" refers to cyclic organic compounds that contain at least one different atom (or more) within the ring. And the most common different atoms found in the structure of heterocyclic rings are nitrogen, phosphorous, oxygen, and sulfur atoms [5]. The synthesis of heterocyclic rings is important due to their biological activities against bacteria, viruses, and inflammation. Some of these rings have been considered precursors for many important biological molecules [6]. For instance, vitamins, hemoglobin, chlorophyll, RNA, DNA, and numerous other compounds and Heterocyclic rings have further uses as insecticides and disinfectants [7]. Oxazepine derivatives are seven-member heterocyclic organic compounds that consist of a nitrogen atom, an oxygen atom, and five carbon atoms. Oxazepine derivatives are known for their high level of stability, due to the conjugation between the double bonds in the ring. There are three isomers of oxazepines: 1,4-oxazepine, 1,3-

oxazepine, and 1,2-oxazepine [8] [9]. Oxazepine derivatives are produced via a cyclo-addition reaction (2+5) between Schiff bases and cyclic anhydrides [10]. Oxazepine derivatives are active against bacteria [11], fungi [12], microbials [13], and tumors [14], according to previous studies and literature [15]. This paper seeks to synthesize Schiff Bases di Azomethine and use them as precursors for the preparation of new 1,3-oxazepindione derivatives.

EXPERIMENTAL PART

Materials:

Merck, Sigma-Aldrich, and Scharlau supplied all of the chemicals and solvents used in this study. Bruker Ascend ™ 400 and Bruker Tensor 27 were used to record ^{13}C-NMR, ^{1}H-NMR, and FT-IR spectra. The Stuart SMP-10 equipment was used to determine melting point temperatures.

General Method for Preparing Schiff Bases di Azomethine (A_1-A_3) (B_1) (B_2) (C_1) (C_2):

In a 100-mL reaction flask containing 0.001 moles (2g) of Terephthalaldehyde, Isophthalaldehyde, or *o*-Phthalaldehyde in 20mL of hot absolute ethanol with 5 drops of glacial acetic acid. 0.002 moles of dissolved aniline substitutions in 30 mL of hot absolute ethanol were added. For 3–4 hours, the reaction mixture was left to reflux. The precipitate was filtered once the reaction's completion was verified, and recrystallized using absolute ethanol [1].

Characterizations of Schiff Bases di Azomethine (A_1-A_3) (B_1) (B_2) (C_1) (C_2):

[A_1] 1,1'-(1,4-phenylene))bis(N-(4-chlorophenyl)methanimine): color yellow, state solid, yield 83%, M.p. 182-183^{o}C, R*f* = 0.62 (benzene: acetone) (9:1). FT-IR (ν cm^{-1}): 3057cm^{-1} (C-H Aromatic), 1615cm^{-1} (C=N), 1581-1477cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, $CDCl_3$): 8.48 (s, C<u>H</u>=N), 8.00-7.18 (m, <u>H</u> Aromatic). ^{13}CNMR (400MHz, $CDCl_3$): 159.66 (<u>C</u>H=N), 150.11-120.31 (<u>C</u> Aromatic).

[A_2] 1,1'-(1,4-phenylene)bis(N-(4-bromophenyl)methanimine): color yellow, state solid, yield 88%, M.p. 121-122^{o}C, R*f* = 0.6 (benzene: acetone) (9:1). FT-IR (ν cm^{-1}): 3054cm^{-1} (C-H Aromatic), 1611cm^{-1} (C=N), 1562-1474cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 8.73 (s, C<u>H</u>=N), 8.09-7.28 (m, <u>H</u> Aromatic). ^{13}CNMR (400MHz, DMSO-*d*6): 161.39 (<u>C</u>H=N), 149.44-123.85 (<u>C</u> Aromatic).

[A_3] 1,1'-(1,4-phenylene)bis(N-(3-bromophenyl)methanimine): color greenish yellow, state solid, yield 91%, M.p. 178-179^{o}C, R*f* = 0.7 (benzene: acetone) (9:1). FT-IR (ν cm^{-1}): 3048cm^{-1} (C-H Aromatic), 1620cm^{-1} (C=N), 1560-1467cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 8.75 (s, C<u>H</u>=N), 8.16-7.32 (m, <u>H</u> Aromatic). ^{13}CNMR (400MHz, DMSO-*d*6): 162.20 (<u>C</u>H=N), 153.33-121.44 (<u>C</u> Aromatic).

[B_1] 1,1'-(1,3-phenylene)bis(N-(4-chlorophenyl)methanimine): color yellow, state solid, yield 83%, M.p. 108-109^{o}C, R*f* = 0.63 (benzene: acetone) (9:1). FT-IR (ν cm^{-1}): 3055cm^{-1} (C-H Aromatic), 1621cm^{-1} (C=N), 1577-1480cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz,

DMSO-*d*6): 8.74 (s, C<u>H</u>=N), 8.55-7.33 (m, <u>H</u> Aromatic). ^{13}CNMR (400MHz, DMSO-*d*6): 161.38 (<u>C</u>H=N), 150.39-123.41 (<u>C</u> Aromatic).

[B_2] 1,1'-(1,3-phenylene)bis(N-(4-bromophenyl)methanimine): color greenish white, state solid, yield 69%, M.p. 123-124^{o}C, R*f* = 0.6 (benzene: acetone) (9:1). FT-IR (ν cm^{-1}): 3078cm^{-1} (C-H Aromatic), 1626cm^{-1} (C=N), 1577-1479cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 8.74 (s, C<u>H</u>=N), 8.52-7.27 (m, <u>H</u> Aromatic). ^{13}CNMR (400MHz, DMSO-*d*6): 161.43 (<u>C</u>H=N), 150.80-119.30 (<u>C</u> Aromatic).

[C_1] 1,1'-(1,2-phenylene)bis(N-(4-chlorophenyl)methanimine) [16]: color yellow, state solid, yield 88%, M.p. 171-172^{o}C, R*f* = 0.73 (benzene: acetone) (9:1). FT-IR (ν cm^{-1}): 3078cm^{-1} (C-H Aromatic), 1611cm^{-1} (C=N), 1587-1483cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 8.08 (s, C<u>H</u>=N), 7.63-7-36 (m, <u>H</u> Aromatic). ^{13}CNMR (400MHz, DMSO-*d*6): 153.47 (<u>C</u>H=N), 141.40-121.94 (<u>C</u> Aromatic).

[C_2] 1,1'-(1,2-phenylene)bis(N-(4-bromophenyl)methanimine): color orange, state solid, yield 82%, M.p. 185-186^{o}C, R*f* = 0.7 (benzene: acetone) (9:1). FT-IR (ν cm^{-1}): 3075cm^{-1} (C-H Aromatic), 1611cm^{-1} (C=N), 1576-1489cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 8.01 (s, C<u>H</u>=N), 7.60-7.34 (m, <u>H</u> Aromatic). ^{13}CNMR (400MHz, DMSO-*d*6): 149.99 (<u>C</u>H=N), 140.84-122.32 (<u>C</u> Aromatic).

General Method for Preparing 1,3-Oxazepindion derivatives:

In a 50-mL reaction flask containing 0.001 moles (0.5g) of Schiff Bases di Azomethine in 15mL of hot-dried benzene. 0.002 moles of dissolved cyclic anhydrides (maleic and succinic anhydrides) in 15 mL of hot dried benzene were added. For 5–6 hours, the reaction mixture was left to reflux and then stirred at room temperature for an additional hour. The precipitate was filtered, and it was then recrystallized using dried benzene **[1]**.

Characterizations of 1,3-Oxazepindione derivatives (DA_1-DA_3) (DB_1) (DB_2) (DC_1) (DC_2):

[DA_1] 2,2'-(1,4-phenylene)bis(3-(4-chlorophenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione): color pale yellow, state solid, yield 68%, M.p. 202-203^{o}C, R*f* = 0.58 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3076cm^{-1} (C-H Aromatic), 3011cm^{-1} (C-H alkene), 1701cm^{-1} (C=O lactone), 1628cm^{-1} (C=O lactam), 1522-1483cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.48 (s, O–C<u>H</u>–N), 7.66-7.37 (m, <u>H</u> aromatic), 6.48-6.30 (m, C<u>H</u>=C<u>H</u>), ^{13}CNMR (400MHz, DMSO-*d*6): 167.37 (<u>C</u>O–O), 163.80 (<u>C</u>O–N), 138.08-129.20 (<u>C</u> aromatic), 130.65-127.79 (<u>C</u>H=<u>C</u>H), 121.43 (O–<u>C</u>H–N).

[DA_2] 2,2'-(1,4-phenylene)bis(3-(4-bromophenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione): color pale yellow, state solid, yield 69%, M.p. 200-201^{o}C, R*f* = 0.55 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3082cm^{-1} (C-H Aromatic), 3051cm^{-1} (C-H alkene), 1702cm^{-1} (C=O lactone), 1626cm^{-1} (C=O lactam), 1504-1483cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.47 (s, O–C<u>H</u>–N), 7.61-7.49 (m, <u>H</u> aromatic), 6.48-6.30 (m, C<u>H</u>=C<u>H</u>), ^{13}CNMR (400MHz, DMSO-*d*6): 167.37 (<u>C</u>O–O), 163.82 (<u>C</u>O–N), 138.51-129.20 (<u>C</u> aromatic), 130.65-122.05 (<u>C</u>H=<u>C</u>H), 115.84 (O–<u>C</u>H–N).

[DA_3] 2,2'-(1,4-phenylene)bis(3-(3-bromophenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione): color pale green, state solid, yield 63%, M.p. 199-200°C, R*f* = 0.66 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3079cm^{-1} (C-H Aromatic), 3050cm^{-1} (C-H alkene), 1712cm^{-1} (C=O lactone), 1631cm^{-1} (C=O lactam), 1530-1498cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.48 (s, O–C<u>H</u>–N), 7.98-7.26 (m, <u>H</u> aromatic), 6.48-6.31 (m, C<u>H</u>=C<u>H</u>), ^{13}CNMR (400MHz, DMSO-*d*6): 167.38 (<u>C</u>O–O), 164.01 (<u>C</u>O–N), 140.75-126.76 (<u>C</u> aromatic), 130.59-122.15 (<u>C</u>H=<u>C</u>H), 118.62 (O–<u>C</u>H–N).

[DB_1] 2,2'-(1,3-phenylene)bis(3-(4-chlorophenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione): color pale yellow, state solid, yield 64%, M.p. 206-207°C, R*f* = 0.6 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3078cm^{-1} (C-H Aromatic), 3055cm^{-1} (C-H alkene), 1702cm^{-1} (C=O lactone), 1629cm^{-1} (C=O lactam), 1515-1485cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.48 (s, O–C<u>H</u>–N), 7.67-7.37 (m, <u>H</u> aromatic), 6.48-6.30 (m, C<u>H</u>=C<u>H</u>), ^{13}CNMR (400MHz, DMSO-*d*6): 167.36 (<u>C</u>O–O), 163.81 (<u>C</u>O–N), 138.08-129.19 (<u>C</u> aromatic), 130.64-127.80 (<u>C</u>H=<u>C</u>H), 121.44 (O–<u>C</u>H–N).

[DB_2] 2,2'-(1,3-phenylene)bis(3-(4-bromophenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione): color off-white, state solid, yield 65%, M.p. 209-210°C, R*f* = 0.56 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3080cm^{-1} (C-H Aromatic), 3052cm^{-1} (C-H alkene), 1703cm^{-1} (C=O lactone), 1626cm^{-1} (C=O lactam), 1504-1484cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.47 (s, O–C<u>H</u>–N), 7.61-7.37 (m, <u>H</u> aromatic), 6.48-6.30 (m, C<u>H</u>=C<u>H</u>), ^{13}CNMR (400MHz, DMSO-*d*6): 167.37 (<u>C</u>O–O), 163.83 (<u>C</u>O–N), 138.50-129.33 (<u>C</u> aromatic), 130.65-122.05 (<u>C</u>H=<u>C</u>H), 115.85 (O–<u>C</u>H–N).

[DC_1] 2,2'-(1,2-phenylene)bis(3-(4-chlorophenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione): color pale yellow, state solid, yield 63%, M.p. 194-195°C, R*f* = 0.62 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3078cm^{-1} (C-H Aromatic), 3056cm^{-1} (C-H alkene), 1704cm^{-1} (C=O lactone), 1631cm^{-1} (C=O lactam), 1523-1488cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.48 (s, O–C<u>H</u>–N), 7.67-7.07 (m, <u>H</u> aromatic), 6.49-6.30 (m, C<u>H</u>=C<u>H</u>), ^{13}CNMR (400MHz, DMSO-*d*6): 167.36 (<u>C</u>O–O), 163.81 (<u>C</u>O–N), 138.07-129.19 (<u>C</u> aromatic), 130.66-127.81 (<u>C</u>H=<u>C</u>H), 121.44 (O–<u>C</u>H–N).

[DC_2] 2,2'-(1,2-phenylene)bis(3-(4-bromophenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione): color pale yellow, state solid, yield 66%, M.p. 205-206°C, R*f* = 0.59 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3082cm^{-1} (C-H Aromatic), 3052cm^{-1} (C-H alkene), 1705cm^{-1} (C=O lactone), 1628cm^{-1} (C=O lactam), 1504-1485cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.49 (s, O–C<u>H</u>–N), 7.62-7.23 (m, <u>H</u> aromatic), 6.49-6.31 (m, C<u>H</u>=C<u>H</u>), ^{13}CNMR (400MHz, DMSO-*d*6): 167.36 (<u>C</u>O–O), 163.83 (<u>C</u>O–N), 138.49-128.79 (<u>C</u> aromatic), 130.68-121.82 (<u>C</u>H=<u>C</u>H), 115.88 (O–<u>C</u>H–N).

Characterizations of 1,3-Oxazepindion derivatives (EA_1-EA_3) (EB_1) (EB_2) (EC_1) (EC_2):

[EA_1] 2,2'-(1,4-phenylene)bis(3-(4-chlorophenyl)-1,3-oxazepane-4,7-dione): color off-white, state solid, yield 68%, M.p. 172-173°C, R*f* = 0.54 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3046cm^{-1} (C-H Aromatic), 2965-2842cm^{-1} (C-H aliphatic), 1693cm^{-1} (C=O lactone), 1656cm^{-1} (C=O lactam), 1593-1527cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-

*d*6): 10.11 (s, O–C<u>H</u>–N), 7.62-7.34 (m, <u>H</u> aromatic), 2.59-2.53 (m, C<u>H</u>$_2$-C<u>H</u>$_2$), ^{13}CNMR (400MHz, DMSO-*d*6): 174.27 (<u>C</u>O–O), 170.73 (<u>C</u>O–N), 138.71-123.46 (<u>C</u> aromatic), 31.49-29.17 (<u>C</u>H$_2$-<u>C</u>H$_2$), 120.86 (O–<u>C</u>H–N).

[EA$_2$] 2,2'-(1,4-phenylene)bis(3-(4-bromophenyl)-1,3-oxazepane-4,7-dione): color pale yellow, state solid, yield 71%, M.p. 198-199^{o}C, R*f* = 0.51 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3080cm^{-1} (C-H Aromatic), 2980-2872cm^{-1} (C-H aliphatic), 1696cm^{-1} (C=O lactone), 1655cm^{-1} (C=O lactam), 1563-1503cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.10 (s, O–C<u>H</u>–N), 8.15-7.30 (m, <u>H</u> aromatic), 2.50-2.41 (m, C<u>H</u>$_2$-C<u>H</u>$_2$), ^{13}CNMR (400MHz, DMSO-*d*6): 174.11 (<u>C</u>O–O), 170.26 (<u>C</u>O–N), 138.55-123.85 (<u>C</u> aromatic), 31.60-29.34 (<u>C</u>H$_2$-<u>C</u>H$_2$), 119.43 (O–<u>C</u>H–N).

[EA$_3$] 2,2'-(1,4-phenylene)bis(3-(3-bromophenyl)-1,3-oxazepane-4,7-dione): color pale yellow, state solid, yield 66%, M.p. 164-165^{o}C, R*f* = 0.64 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3069cm^{-1} (C-H Aromatic), 2959-2874cm^{-1} (C-H aliphatic), 1695cm^{-1} (C=O lactone), 1656cm^{-1} (C=O lactam), 1583-1529cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.10 (s, O–C<u>H</u>–N), 8.09-7.24 (m, <u>H</u> aromatic), 2.57-2.50 (m, C<u>H</u>$_2$-C<u>H</u>$_2$), ^{13}CNMR (400MHz, DMSO-*d*6): 174.23 (<u>C</u>O–O), 170.99 (<u>C</u>O–N), 138.83-123.84 (<u>C</u> aromatic), 31.51-29.11 (<u>C</u>H$_2$-<u>C</u>H$_2$), 118.06 (O–<u>C</u>H–N).

[EB$_1$] 2,2'-(1,3-phenylene)bis(3-(4-chlorophenyl)-1,3-oxazepane-4,7-dione): color white, state solid, yield 67%, M.p. 160-165^{o}C, R*f* = 0.55 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3045cm^{-1} (C-H Aromatic), 2936-2840cm^{-1} (C-H aliphatic), 1693cm^{-1} (C=O lactone), 1656cm^{-1} (C=O lactam), 1593-1527cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.10 (s, O–C<u>H</u>–N), 7.63-7.32 (m, <u>H</u> aromatic), 2.58-2.52 (m, C<u>H</u>$_2$-C<u>H</u>$_2$), ^{13}CNMR (400MHz, DMSO-*d*6): 174.26 (<u>C</u>O–O), 170.73 (<u>C</u>O–N), 138.71-126.88 (<u>C</u> aromatic), 31.49-29.16 (<u>C</u>H$_2$-<u>C</u>H$_2$), 120.86 (O–<u>C</u>H–N).

[EB$_2$] 2,2'-(1,3-phenylene)bis(3-(4-bromophenyl)-1,3-oxazepane-4,7-dione): color white, state solid, yield 66%, M.p. 191-192^{o}C, R*f* = 0.54 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3068cm^{-1} (C-H Aromatic), 2972-2870cm^{-1} (C-H aliphatic), 1695cm^{-1} (C=O lactone), 1661cm^{-1} (C=O lactam), 1588-1518cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.10 (s, O–C<u>H</u>–N), 7.64-7.37 (m, <u>H</u> aromatic), 2.58-2.53 (m, C<u>H</u>$_2$-C<u>H</u>$_2$), ^{13}CNMR (400MHz, DMSO-*d*6): 174.25 (<u>C</u>O–O), 170.76 (<u>C</u>O–N), 139.12-121.26 (<u>C</u> aromatic), 31.52-29.15 (<u>C</u>H$_2$-<u>C</u>H$_2$), 114.88 (O–<u>C</u>H–N).

[EC$_1$] 2,2'-(1,2-phenylene)bis(3-(4-chlorophenyl)-1,3-oxazepane-4,7-dione): color Pale yellow, state solid, yield 63%, M.p. 150-151^{o}C, R*f* = 0.58 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3027cm^{-1} (C-H Aromatic), 2980-2868cm^{-1} (C-H aliphatic), 1685cm^{-1} (C=O lactone), 1655cm^{-1} (C=O lactam), 1560-1506cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.11 (s, O–C<u>H</u>–N), 7.62-7.33 (m, <u>H</u> aromatic), 2.60-2.55 (m, C<u>H</u>$_2$-C<u>H</u>$_2$), ^{13}CNMR (400MHz, DMSO-*d*6): 174.09 (<u>C</u>O–O), 170.74 (<u>C</u>O–N), 138.12-122.03 (<u>C</u> aromatic), 31.45-29.20 (<u>C</u>H$_2$-<u>C</u>H$_2$), 120.85 (O–<u>C</u>H–N).

[EC$_2$] 2,2'-(1,2-phenylene)bis(3-(4-bromophenyl)-1,3-oxazepane-4,7-dione): color off-white, state solid, yield 64%, M.p. 162-163^{o}C, R*f* = 0. 0.57 (n-hexane: ethyl acetate) (6:4). FT-IR (ν cm^{-1}): 3028cm^{-1} (C-H Aromatic), 2978-2869cm^{-1} (C-H aliphatic), 1689cm^{-1} (C=O

lactone), 1656cm^{-1} (C=O lactam), 1588-1524cm^{-1} (C=C Aromatic), ^{1}HNMR (400MHz, DMSO-*d*6): 10.10 (s, O–C<u>H</u>–N), 7.57-7.37 (m, <u>H</u> aromatic), 2.58-2.53 (m, C<u>H</u>$_2$-C<u>H</u>$_2$), ^{13}CNMR (400MHz, DMSO-*d*6): 174.25 (<u>C</u>O–O), 170.76 (<u>C</u>O–N), 139.12-121.28 (<u>C</u> aromatic), 31.51-29.15 (<u>C</u>H$_2$-<u>C</u>H$_2$), 114.87 (O–<u>C</u>H–N).

RESULTS AND DISCUSSION

Schiff Bases di Azomethine (A_1-A_3) (B_1) (B_2) (C_1) (C_2) were prepared by thermal condensation between Terephthalaldehyde, Isophthalaldehyde, or *o*-Phthalaldehyde and aromatic amines (halo aniline) with 5 drops of glacial acetic acid and use absolute ethanol as a solvent. Scheme 1 shows the paths for preparing Schiff Bases di Azomethine, and Scheme 2 shows the mechanism of reaction for the preparation of Schiff Bases di Azomethine [10].

All FT-IR spectra for synthesized Schiff Bases di Azomethine showed the disappearance of the absorption bands of the (C=O) group in aldehydes and the (-NH$_2$) group in amines and the appearance of the absorption bands of the (C-H) aromatic at (3078–3048 cm^{-1}), Azomethine groups (C=N) at (1617–1612 cm^{-1}), and (C=C) aromatic at (1587–1467 cm^{-1}) [17] [18]. Figure 1 shows FT-IR spectra for [A_2] compound.

All ^{1}H-NMR spectra of the synthesized Schiff Bases di Azomethine showed signals of (N=C-<u>H</u>) (2H, s) at (δ=8.75-8.01ppm) and aromatic protons (12H, m) at (δ=8.55-7.18ppm) [19]. Figure 2 shows ^{1}H-NMR spectra for [A_1] compound.

All ^{13}C-NMR spectra of the synthesized Schiff Bases di Azomethine showed signals of (N=<u>C</u>-H) (2C) at (δ=162.20-149.99ppm) and aromatic carbons (18C) at (δ=153.33-119.30ppm). Figure 3 shows ^{13}C-NMR spectra for [B_1] compound.

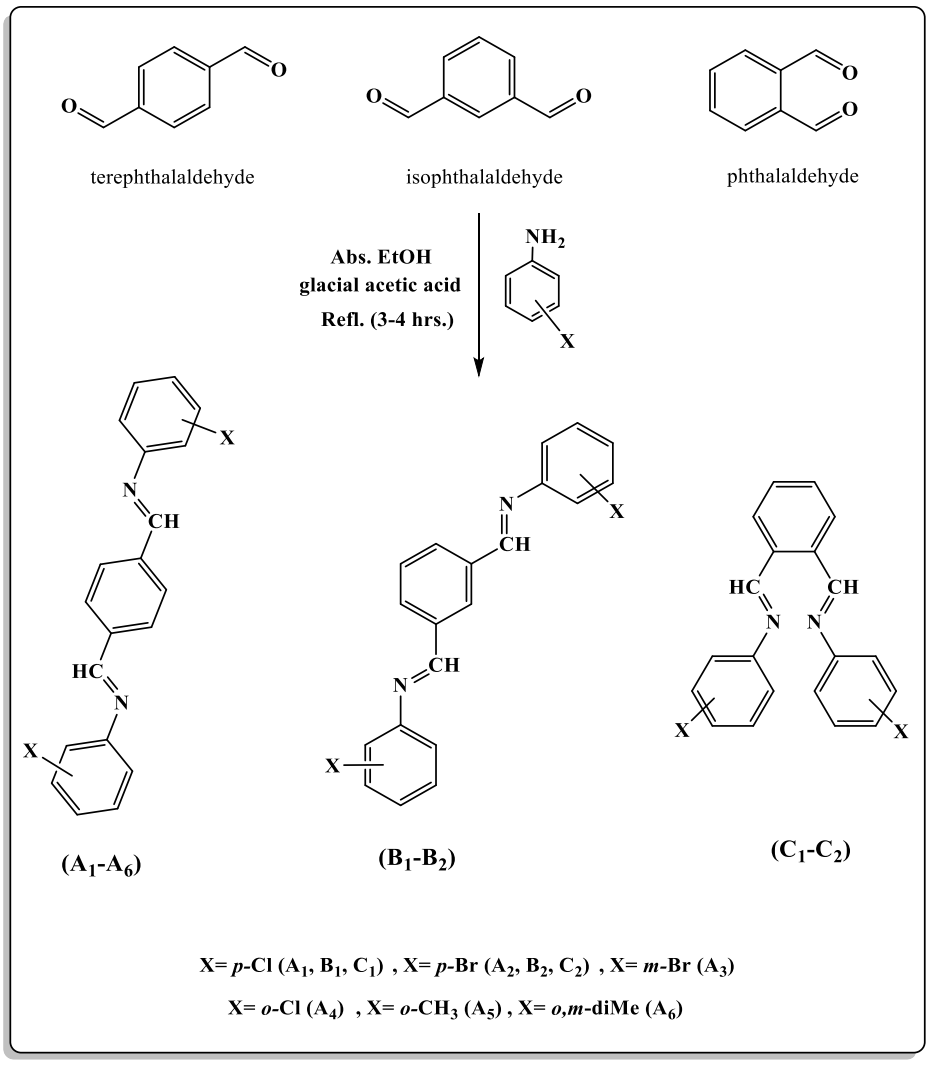
1,3-Oxazepindion derivatives were obtained by cycloaddition reactions between prepared Schiff Bases di Azomethine with Maleic anhydride or Succinic anhydride using dried benzene as a solvent. Schemes 3a and 3b show the paths for preparing 1,3-Oxazepindione derivatives, and Scheme 4 shows the mechanism of reaction for the preparation of 1,3-Oxazepindione derivatives [20].

All FT-IR spectra for synthesized 1,3-Oxazepindione derivatives showed the disappearance of the absorption band of Azomethine groups (C=N) at (1617–1612 cm^{-1}) and the appearance of the absorption bands of the (C-H) aromatic at (3082–3027 cm^{-1}), (CH=CH) at (3056–3011 cm^{-1}), (CH$_2$-CH$_2$) at (2980–2840 cm^{-1}), (C=O) lactone at (1712–1685 cm^{-1}), (C=O) lactam at (1655–1626 cm^{-1}) and (C=C) aromatic at (1593–1483 cm^{-1}) (17) (18). Figure 4 shows FT-IR spectra for [DB_2] compound.

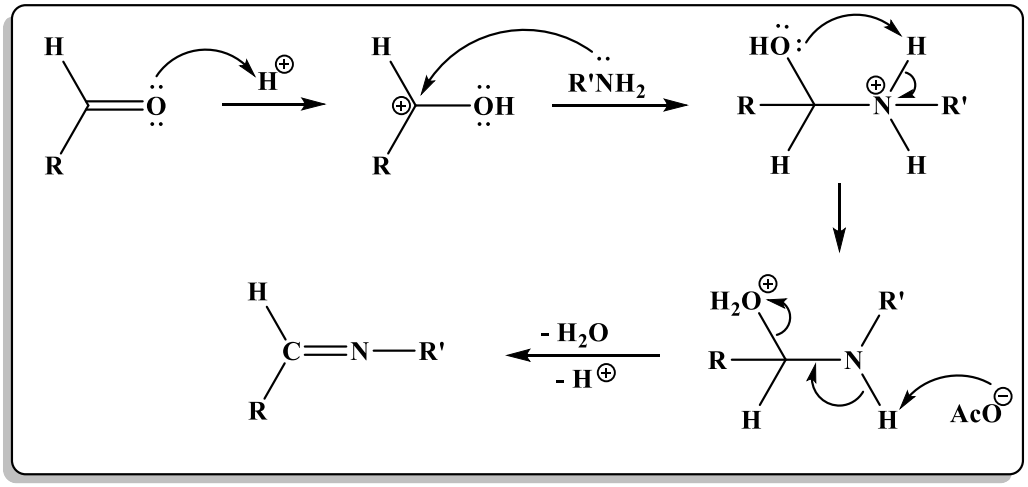
All ^{1}H-NMR spectra of synthesized 1,3-Oxazepindione derivatives showed signals of (N-C<u>H</u>-O) (2H, s) at (δ=10.49-10.10ppm), aromatic protons (12H, m) at (δ=8.15-7.07ppm), (C<u>H</u>=C<u>H</u>) (4H, d) at (δ=6.49-6.30ppm) and (C<u>H</u>$_2$-C<u>H</u>$_2$) (8H, t) at (δ=2.60-2.41ppm) (19). Figure 5 shows ^{1}H-NMR spectra for [EC_2] compound.

All ^{13}C-NMR spectra of the synthesized 1,3-Oxazepindione derivatives showed signals of (O=<u>C</u>-O) (2C) at (δ=174.27-167.36ppm), (O=<u>C</u>-N) (2C) at (δ=170.99-163.80ppm), aromatic carbons (18C) at (δ=140.75-121.26ppm), (N-<u>C</u>H-O) (2C) at (δ=121.44-114.87ppm),

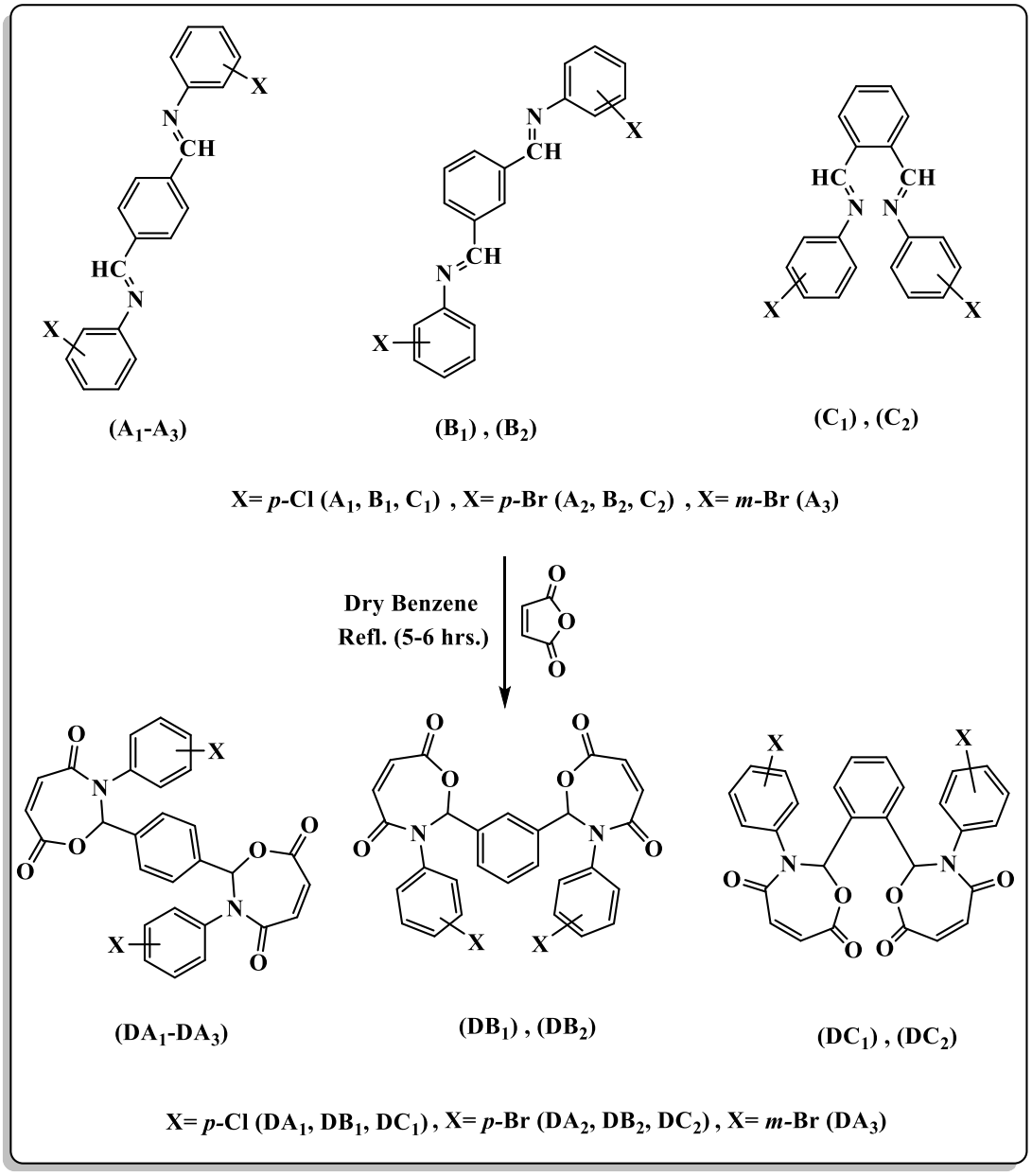
(C̲H2=C̲H2) (4C) at (δ=130.68-121.82ppm), and (C̲H2-C̲H2) (4C) at (δ=31.60-29.11ppm). Figure 6 shows ^{13}C-NMR spectra for [DA1] compound.



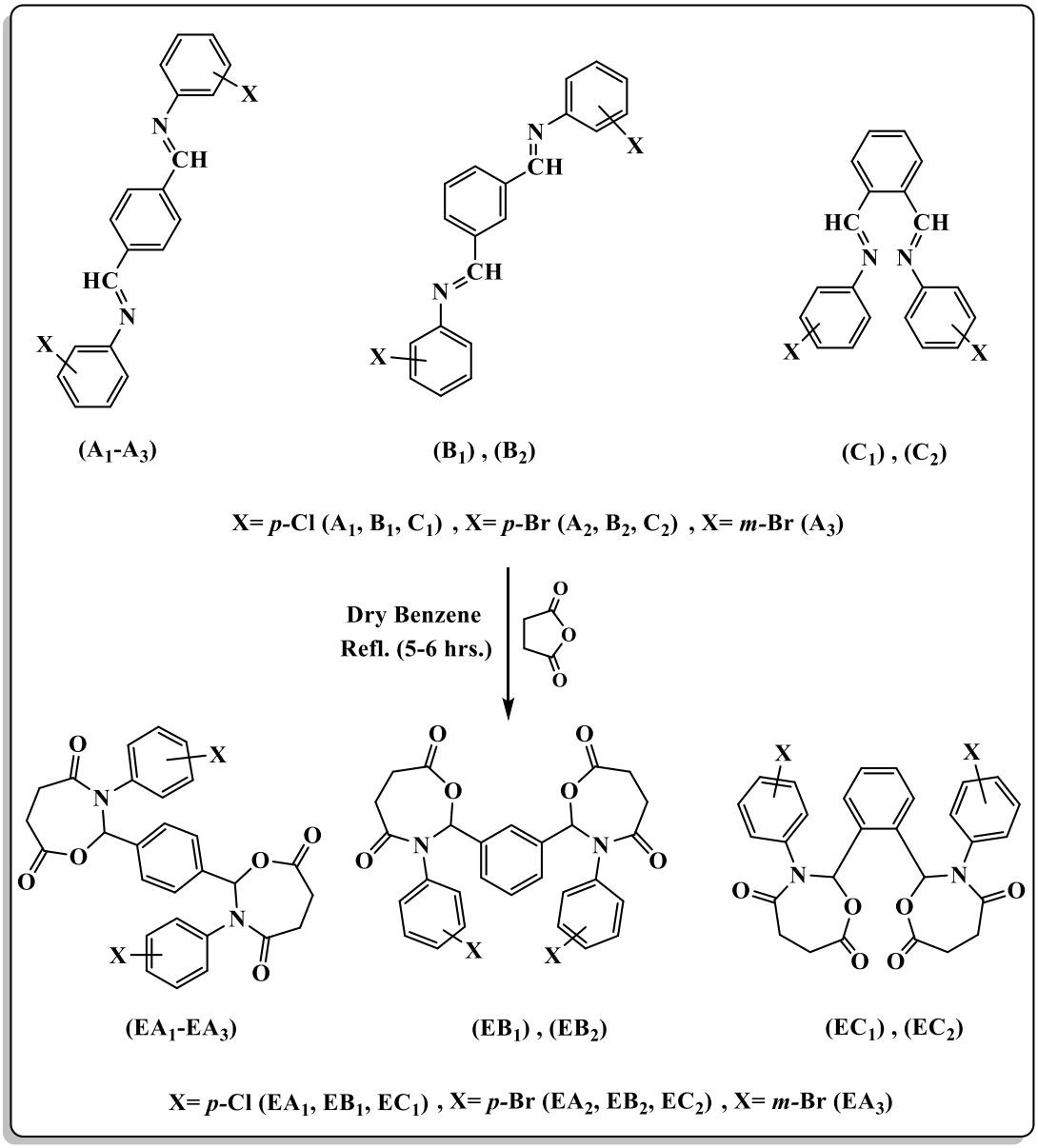
Scheme 1: the paths for preparing Schiff Bases di Azomethine **[A][B][C]**.



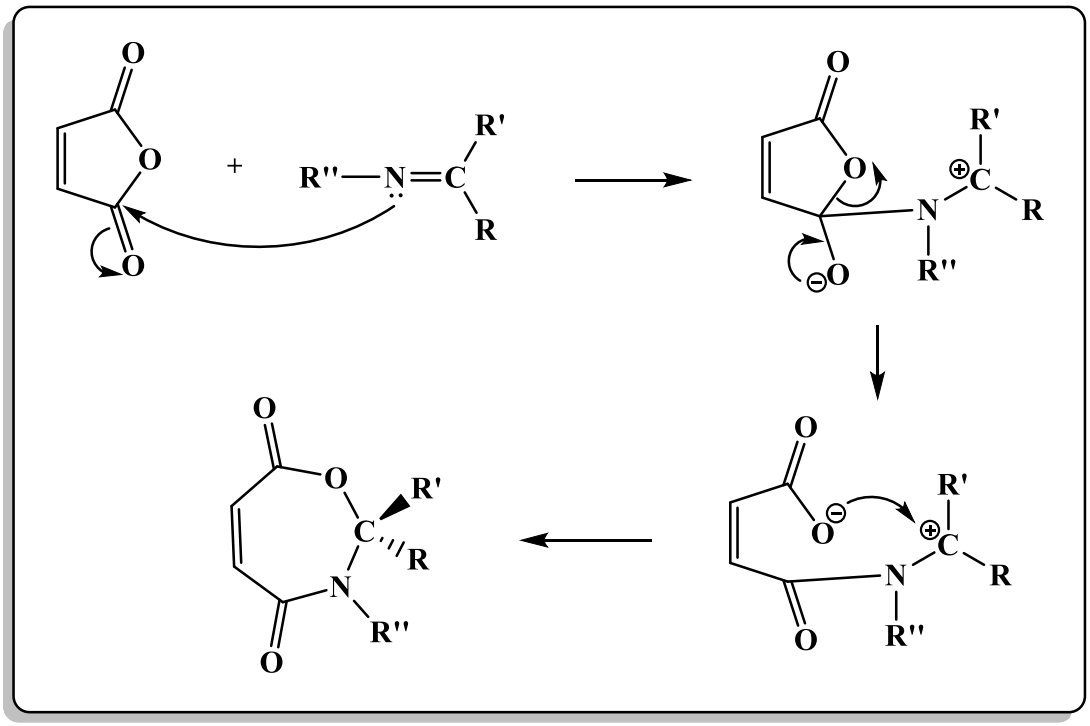
Scheme 2: the mechanism of reaction for the preparation of Schiff Bases di Azomethine.



Schemes 3a: the paths for preparing 1,3-Oxazepindione derivatives **[D].**



Schemes 3b: the paths for preparing 1,3-Oxazepindione derivatives **[E]**



Scheme 4: the mechanism of reaction for the preparation of 1,3-Oxazepindione derivatives.

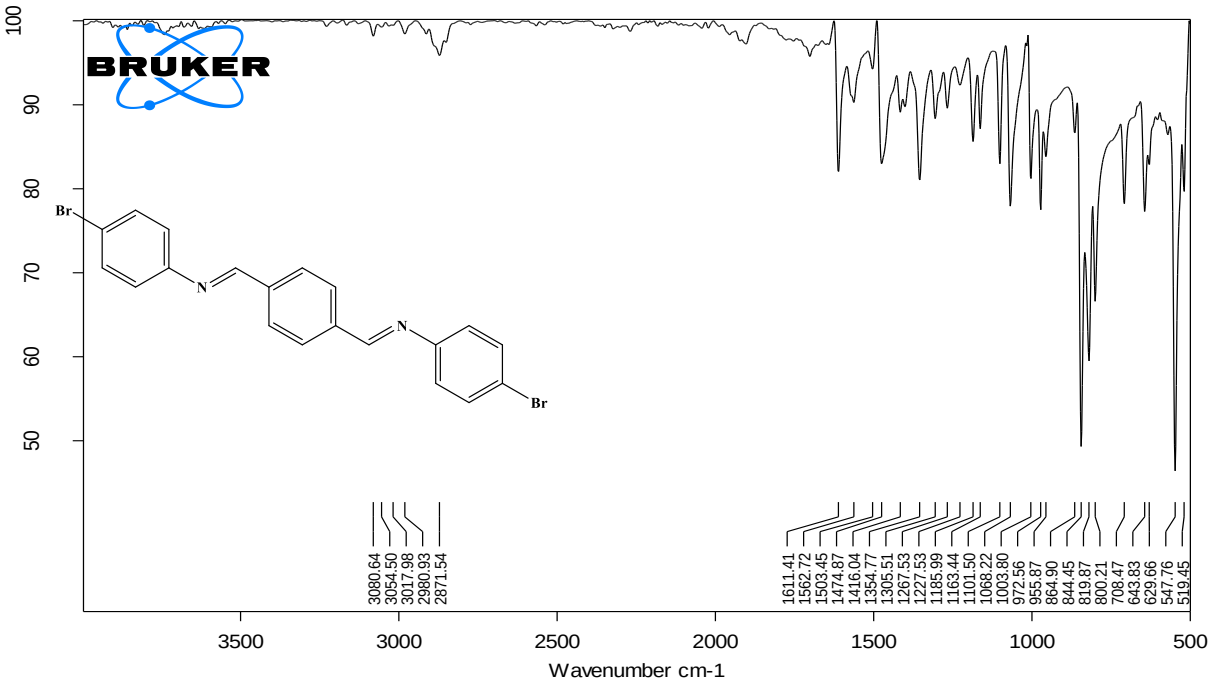


Fig. 1: shows FT-IR spectra for **[A_2].**

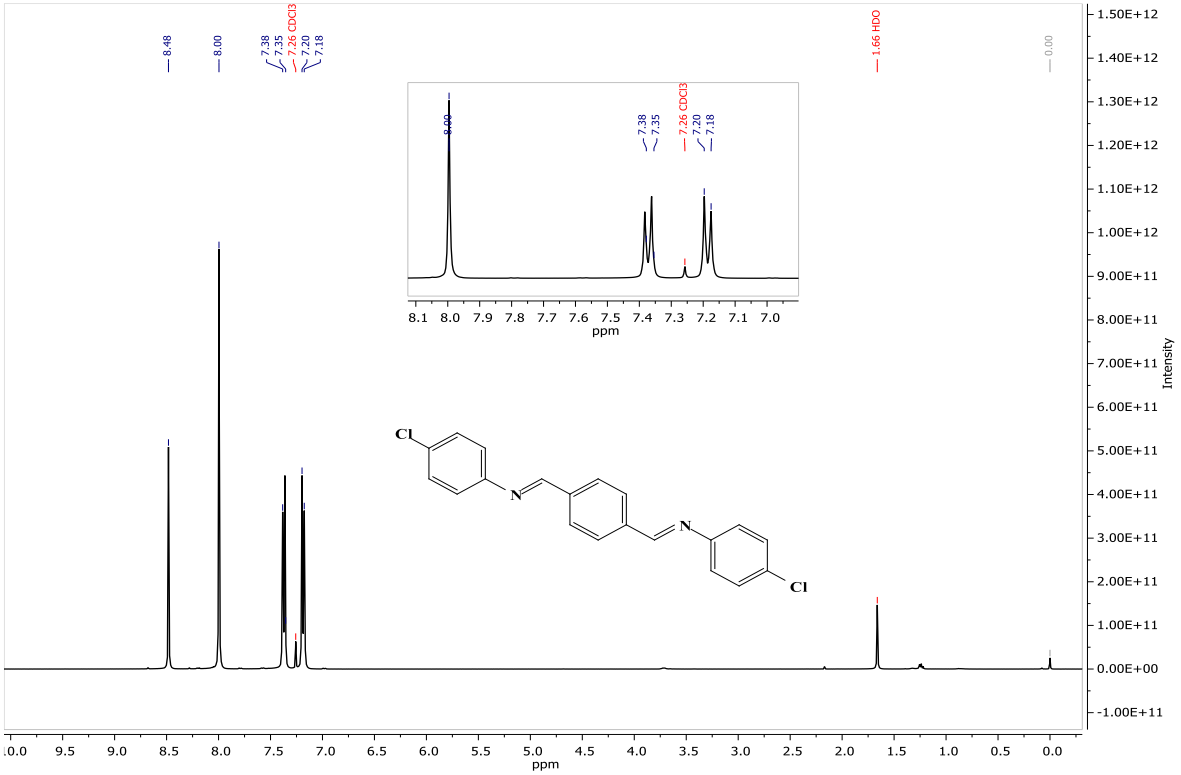


Fig. 2: shows ^{1}H-NMR spectra for **[A$_1$].**

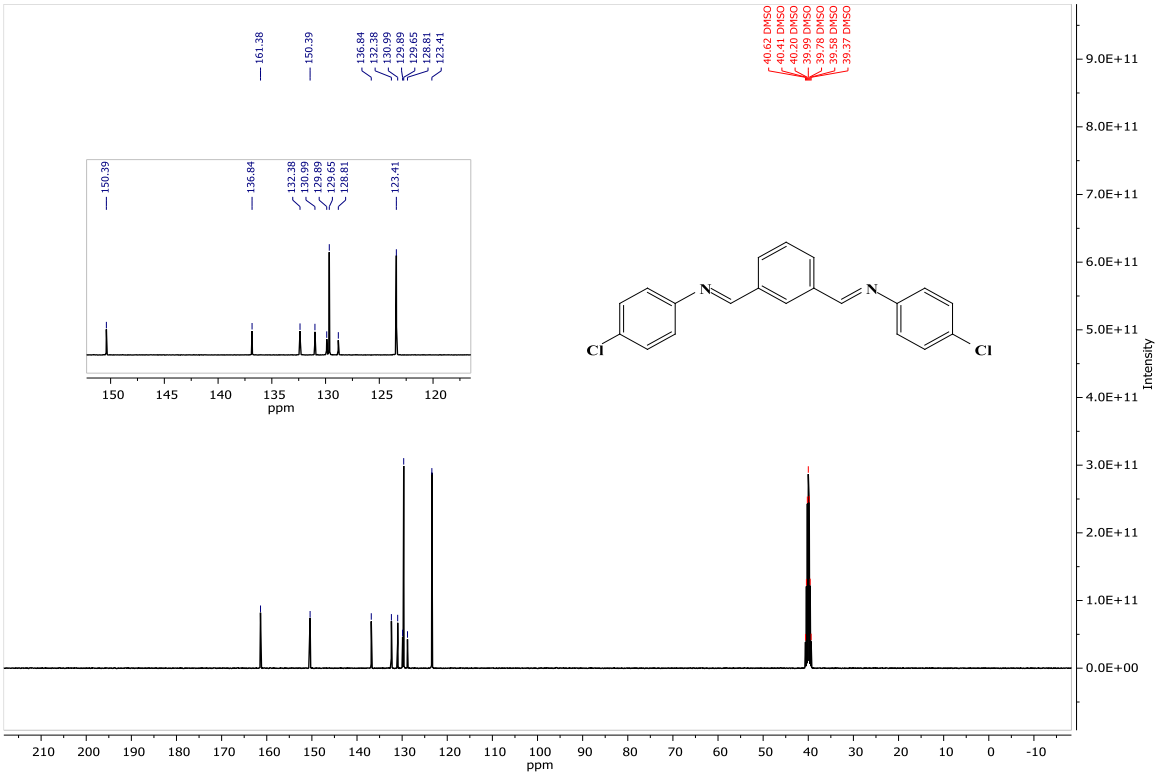


Fig. 3: shows ^{13}C-NMR spectra for **[B$_1$].**

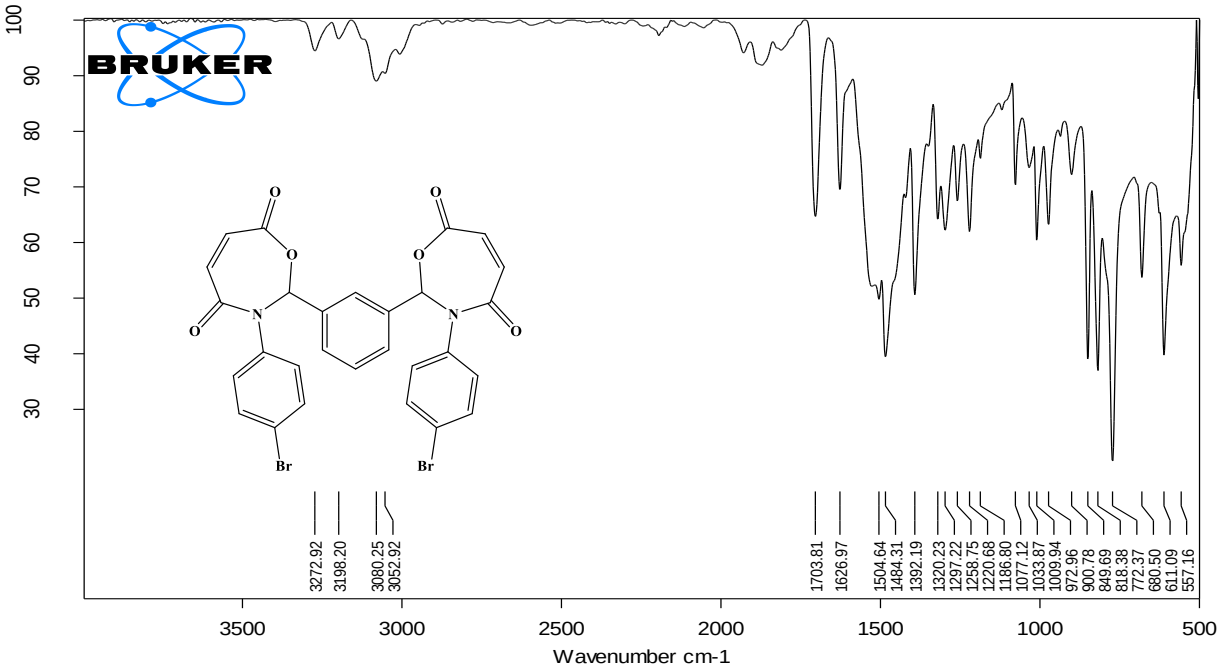


Fig. 4: shows FT-IR spectra for **[DB$_2$].**

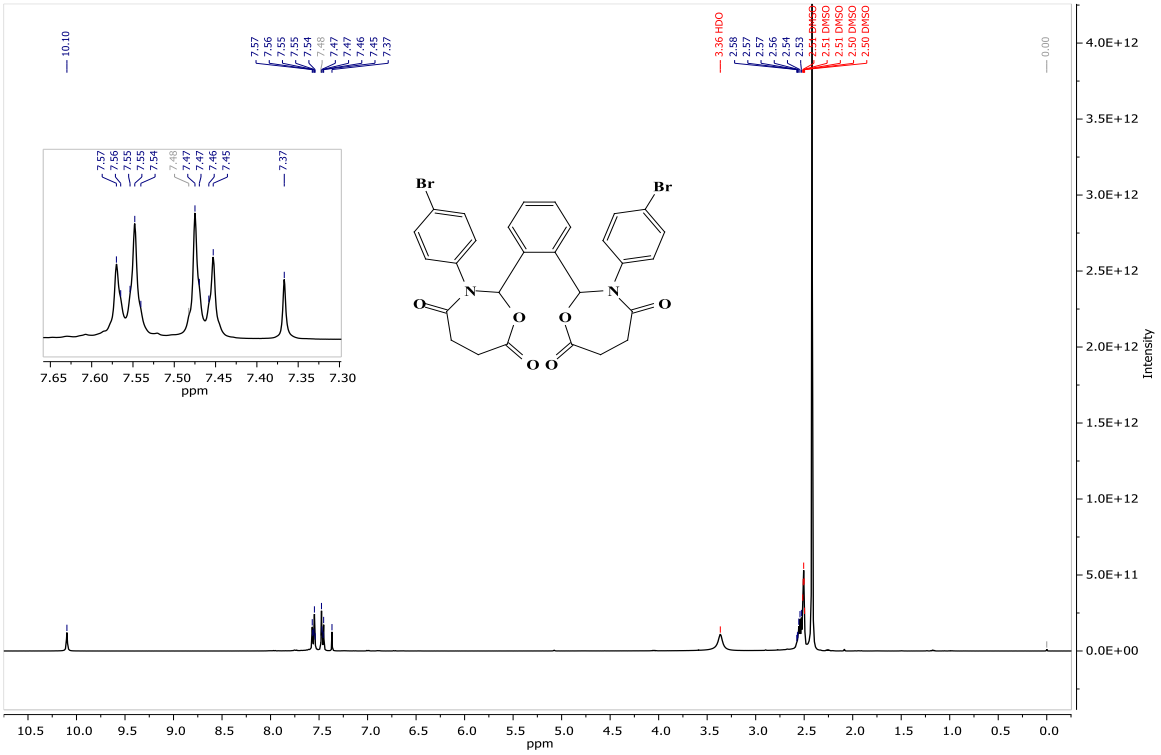


Fig. 5: shows ^{1}H-NMR spectra for **[EC$_2$].**

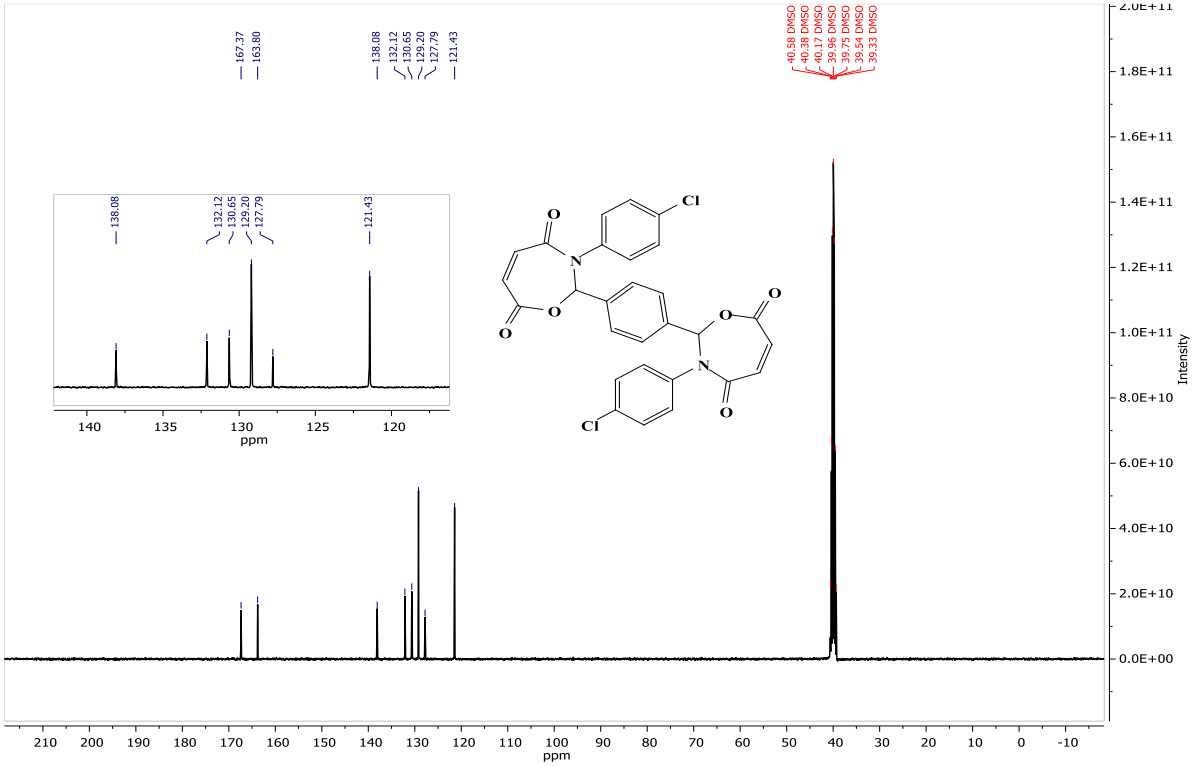


Fig. 6: shows ^{13}C-NMR spectra for **[DA$_1$].**

CONCLUSION

All compounds required for this scientific study were successfully prepared. Nuclear magnetic resonance spectroscopy (^{1}HNMR and ^{13}CNMR) and infrared (FT-IR) spectroscopy were used to characterize the prepared compounds. Using thin layer chromatography (TLC), all reactions were monitored.

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تحضير وتشخيص مشتقات 3،1-اوكسازبيندايون عن طريق إيمينات ثنائي فورميل البنزين كمادة أولية

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الخلاصة:
في هذا البحث، حضرت مشتقات 3،1-اوكسازبيندايون [D] و[E] عن طريق تفاعل قواعد شف ثنائية الازوميثين [[A، B]] و[C] مع أنهدريد الماليك وأنهدريد السكسنيك بوجود البنزين الجاف كمذيب. وحضرت قواعد شف ثنائية الازوميثين [A]، [B] و[C] عن طريق تفاعل ترفثالديهايد او ايزوفثالديهايد او اورثو-فثالديهايد مع 5 قطرات من حاض الخليك الثلجي كعامل مساعد ومشتقات الانيلين الهالوجينية في الايثانول المطلق كمذيب. تم تشخيص المركبات المحضرة واثباتها باستخدام مطيافية الاشعة تحت الحمراء والرنين النووي المغناطيسي. جميع تفاعلات المركبات المحضرة تمت متابعتها عن طريق تنقية كروماتوغرافيا الطبقة الرقيقة.