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# Synthesis and Characterization of some New Oxazepine Compounds Derived from D-Erythroascorbic Acid

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## Abstract

This search include the synthesis of some new 1,3-oxazepine derivatives have been prepared, starting from reaction of L-ascorbic acid with dry acetone in presence of dry hydrogen chloride afforded the acetal (I). Treatment of the latter with *p*-nitrobenzoyl chloride in pyridine yielded the ester (II) which was dissolved in (65%) acetic acid in absolute ethanol yielded the glycol (III). The reaction of the glycol (III) with sodium periodate in distilled water at room temperature produced the aldehyde (IV).

The compound (V) [4-(1,3-dioxoisoindolin-2-yl)benzoic acid] was synthesized by reaction *p*-aminobenzoic acid and phthalic anhydride in presence of (gla. $CH_3COOH$). Reaction of compound (V) with thionyl chloride produced [4-(1,3-dioxoisoindolin-2-yl)benzoyl chloride]. Condensation of acid chloride with hydrazine hydrate afforded 4-(1,3-dioxoisoindolin-2-yl)benzohydrazide (VI).

The azomethine (VII) has been synthesized from the reaction between compounds (IV) and (VI). Moreover compounds (VIII-X) were synthesized from the cyclic condensation of Schiff base (VII) with (maleic, phthalic and 3-nitrophthalic) anhydride, the structures of the newly synthesized compounds have been confirmed by physical properties and spectral measurements such as (FTIR and some of them by $^{1}$H-NMR and $^{13}$C-NMR).

**Keywords:** Schiff base, 1,3-oxazepine, L-ascorbic acid.

## Introduction

L-Ascorbic acid, which is active form of vitamin C is a natural antioxidant that prevents the increase free radicals production which induced by oxidative damage to lipids in many cells and tissues. It has been reported that VC react with various kinds of radicals directly[1,2].

Imides are the compounds consists of nitrogen atom linked to two carbonyl groups. It is demonstrated that these compounds cross the biological membranes easily as they are neutral and hydrophobic in nature[3]. It is shown anti-inflammatory, antiviral, antibacterial and antitumor properties[4].

Schiff bases are an important type of organic compounds[5] that also shown a wide scope of biological activities as antiproliferiative, antimalarial, antibacterial, antipyretic properties, antifungal, antiviral and anti-inflammatory [6,7].

Oxazepine which is synthesized via the pericyclic cycloaddition of [maleic, pthalic, nitropthalic and succinic] anhydrides with Schiff bases [8], and exhibited a biological effectiveness as (antagonistic, hypnotic muscle relaxant, anti-bacterial, telomerase inhibitors, antifungal, antiepileptic, and anti-inflammatory) [9-11].

## Experimental

Melting points were determined by Dig melt MPA 161 (MSRS) electronic and are not corrected. FTIR spectra were recorded on FTIR-600 FTIR spectrometer and 8400s Shimadzu FT infrared spectrophotometer. $^{1}$H-NMR and $^{13}$C-NMR spectra were carried out by Ultra Shield 300 MHz spectrometer, DMSO-$d_6$ was used as a solvent with TMS as internal standard, at University of Al al-Bayt, Jordan. TLC was completed on plates of aluminum glaze with strata of (gel of silica), provided via (Merck). The spots have been recognized via (vapor of iodine). whole chemicals have been gained by (BDH and Sigma-Aldrich).

### Synthesis of 5,6-*O*-isopropylidene-L-AA (I)[12]

Dry (HCl) was quickly bubble with moving until (20 min) inside a (flask 250ml) including (100ml of dry $CH_3COCH_3$ & 10g of L-AA).

After addition (80ml of $C_6H_{14}$), moving and refrigeration in an (snow aqueous), decanted has been the supernatant. Washed has been the sediment (4 times) with (154 ml of hexane-acetone) mixture (7:4 v/v), refrigeration in an (snow aqueous) and elimination of supernatant next every addition. The final sediment has been dehydrated beneath miniature pressure to yield (I) (78%) as a white solid, melting point (210-212˚C). $R_f$ (0.68) (methanol: benzene, v/v, 1:1). FTIR (KBr, $cm^{-1}$): 3244 (O-H), 2993 (C-$H_{ali.}$), 2910 (C-$H_{ace.}$), 1751 (C=$O_{lac.}$), 1660 (C=C), 1435 ( C-$H_{asym}$), 1379 ( C-$H_{sym}$), 1140-900 (C-O), 768 δ(O-H) (O.O.P.)[13].

## Synthesis of 2,3-*O*-di(*p*-nitrobenzoyl)-5,6-*O*-isopropylidene-L-AA (II)

(10g, 46mmol) of compound (I) in dry pyridine (50ml) has been cooled, (24g, 129mmol) of (*p*-nitrobenzoyl chloride) was added with stirring. The subsequent blend was stirred for (2 hrs), subsequently preserved in murky venue at normal temperature to (22 hrs).

Poured has been the mixture inside (ice water) and moving until (20 min), extracted has been the oil layer by (chloroform 2×150 ml), laundered by water, (HCl 5%) (2×100ml), saturated watery $NaHCO_3$ (100ml) & water. Dehydrated over anhydrous $MgSO_4$. Evaporated has been Chloroform. The residue recrystallized from absolute ethanol to give (II) (44%) as a brown solid, melting point (102-104˚C). $R_f$ (0.76) (methanol: benzene, v/v, 1:1,). FTIR (KBr, $cm^{-1}$): 3078 (C-$H_{ar.}$), 2987 (C-$H_{ali.}$), 2943 (C-$H_{ace.}$), 1745 (C=$O_{lac.}$), 1693 (C=$O_{est.}$), 1606 (C=$C_{ali.}$), 1421 (C=$C_{ar.}$), 1531 ($NO_2$ $_{asym.}$), 1346 ($NO_2$ $_{sym.}$), 1263-1105 (C-$O_{est.}$), 900-600 δ(C-$H_{ar.}$) (O.O.P.)[13].

## Synthesis of 2,3-*O*-di(*p*-nitrobenzoyl)-L-AA (III)

(10g, 19.45mmol) of compound (II) was dissolved in blend of ($CH_3COOH$ 65%) (30ml) and (10ml) of (abs. $CH_3CH_2OH$) and stirred until (48 hrs) at normal temperature. The TLC appeared that the interaction has been accomplished (methanol: benzene: 1:1.5).

Filter the mixture, (40ml) of a benzene was added to the resulting solution and volatilized (reoccur this procedure four times).The residue recrystallized from absolute ethanol to yield (III) (74%) as a deep brown solid, m.p (122-124˚C), $R_f$ (0.46) (methanol: benzene, v/v, 1:1.5). FTIR (KBr, $cm^{-1}$): 3413 (O-H), 3078 (C-$H_{ar.}$), 2985 (C-$H_{ali.}$), 1720 (C=$O_{est.}$), 1603 (C=$C_{ali.}$), 1425 (C=$C_{ar.}$), 1520 ($NO_2$ $_{asym.}$), 1358 ($NO_2$ $_{sym.}$), 1275-1105 (C-$O_{est.}$), 900-600 δ(C-$H_{ar.}$) (O.O.P.)[13].

## Synthesis of pentulosono-γ-lactone-2,3-enedi(*p*-nitrobenzoate) (IV)

A solution of compound (III) (10g, 21mmol) in (abs. $CH_3CH_2OH$) (60ml) was added dropwise to the solution of ($NaIO_4$) (5.6g, 26mmol) in distilled water (60ml) at (0˚C) with stirring. (0.5ml) of ($HO\text{-}CH_2CH_2\text{-}OH$) ethylene glycol has been added as dropwise after stirring for (15 min). Stirring was went on at normal temperature for (1 h).

(40ml) of distilled water has been added to the mixture after has been filtered subsequently the yield was taken away by (ethyl acetate 3×50ml), the extracts dehydrated via (anhydrous $MgSO_4$), subsequently filtered and the solvent have been removed and recrystallized has been the residue by absolute ethanol to give the compound (IV) (54%) as a yellow solid, melting point (194-196˚C). $R_f$ (0.73) (methanol: benzene, v/v, 1:1.5). FTIR (KBr, $cm^{-1}$): 3068 (C-$H_{ar.}$), 2993 (C-$H_{ali.}$), 2673, 2548 ($CH_{ald.}$), 1697 (C=$O_{ald.}$), 1603 (C=$C_{ali.}$), 1423 (C=$C_{ar.}$), 1535 ($NO_2$ $_{asym.}$), 1356 ($NO_2$ $_{sym.}$), 1284-1105 (C-$O_{est.}$), 900-600 δ(C-$H_{ar.}$) (O.O.P.). $^1$H-NMR (DMSO-$d_6$ δ ppm): 13.70 (s, 1H, CHO), 8.15-8.34 (dd, 8H, aromatic), 3.92 (s, 1H, lactone ring H-4). $^{13}$C-NMR (DMSO-$d_6$ δ ppm): 178.58 (C=$O_{lac.}$), 165.75 (C=$O_{est.}$), 149.99 (C-3), 123.58-136.32

($C_{ar.}$), 105.85 (C-2), 47.99 (C-4)[13]. The sign of carbonyl aldehydic has been vanished because of it appeared outside the measure[14].

## Synthesis of 4-(1,3-dioxoisoindolin-2-yl)benzoic acid (V)[15]

A mixture of equimolar amounts (1mmol) of 4-aminobenzoic acid with phthalic anhydride in (15ml) of (gla. $CH_3COOH$).

The mixture was refluxed for (3 hrs). Ice distilled water (25ml) has been added to the medium of reaction and the compound was filtered, dried and recrystallized from absolute ethanol to give (V) (93%) as a white solid, m.p. (290-292˚C). FTIR (KBr, $cm^{-1}$): 3300-2500 (O-H), 3078 ($C\text{-}H_{ar.}$), 1716 ($C{=}O_{cyclicimide}$), 1701 ($C{=}O_{carb.}$), 1603 ($C{=}C_{ar.}$)[13].

## Synthesis of 4-(1,3-dioxoisoindolin-2-yl)benzohydrazide (VI)[16,17]

A mixture of compound (V) (10mmol) and (10ml) of ($SOCl_2$) in (5ml) of dry benzene was refluxed for (3 hrs). After cooling the overabundant of ($SOCl_2$) and (benzene) were isolated beneath vacuum. (50mmol) of (hydrazine hydrate 80%) and (dry benzene 15ml) was added to the yield.

The mixture was refluxed for (4 hrs). After cooling, the excess of hydrazine hydrate and solvent were isolated beneath reduce pressure, the residue was washed with ether, then recrystallized from absolute ethanol to give (VI) (60%) as a white solid, m. p. (270-274˚C). FTIR (KBr, $cm^{-1}$): 3429 (N-H), (3344, 3302) ($NH_2$), 3032 ($C\text{-}H_{ar.}$), 1624 ($C{=}O_{amide}$), 1604 ($C{=}C_{ar.}$). ($^1$H-NMR) (DMSO-$d_6$ δ ppm): 10.56 (1H, NH), 6.97-8.09 (m, 8H, aromatic), 4.10 (2H, $NH_2$)[13].

## Synthesis of Schiff base 4-[4-(1,3-dioxoisoindolin-2-yl)benzamido-imine]-pentulose-γ-lactone-2,3-enedi(*p*-nitrobenzoate) (VII)

A mixture of 4-(1,3-dioxoisoindolin-2-yl)benzohydrazide (VI) (0.13g, 0.5mmol), aldehyde (IV) (0.2g, 0.5mmol), (10ml) of absolute ethanol and glacial acetic acid 3 drops have been refluxed for (48 hrs). The solvent was evaporated and the residue recrystallized from absolute ethanol to yield (VII) (47%) as a pale brown, m.p. (208-210˚C). FTIR (KBr, $cm^{-1}$): 3425 (N-H), 3026 ($C\text{-}H_{ar.}$), 2920 ($C\text{-}H_{ali.}$), 1651 (C=N), 1606 ($C{=}C_{ali.}$), 1496 ($C{=}C_{ar.}$) 1523 ($NO_{2\ asym.}$), 1342 ($NO_{2\ sym.}$)[13].

## Synthesis of 1,3-oxazepines (VIII-X)

A mixture of equimolar amounts (0.1g, 0.14mmol) of Schiff base (VII) and different acid anhydrides such as (maleic, phthalic and 3-nitrophthalic) anhydride (0.14mmol) in (10ml) of dioxane was refluxed for (24 hrs). The solvent was removed and the resulting colored solid recrystalized from absolute ethanol to obtained 1,3-oxazepines (VIII-X). The nomenclature and physical properties of synthesized compounds are registered in Table (1). The FTIR absorption bands facts are fixed in Table (2).

**Table (1): Nomenclature and physical properties of 1,3-oxazepine compounds (VIII-X)**

| Comp. No. | Nomenclature | Molecular formula | M.p. °C | Color | Yield % |
|---|---|---|---|---|---|
| VIII | 2-(Pentulose-γ-lactone-2,3-ene(*p*-nitrobenzoate)-3-[4-(1,3-dioxoisoindolin-2-yl)benzamide]-2,3-dihydro-[1,3]-oxazepine-4,7-dione | $C_{38}H_{21}O_{16}N_5$ | 240-242 | Deep brown | 73 |
| IX | 2-(Pentulose-γ-lactone-2,3-ene(*p*-nitrobenzoate)-3-[4-(1,3-dioxoisoindolin-2-yl)benzamide]-2,3-dihydrobenzo[1,2e][1,3]-oxazepine-4,7-dione | $C_{42}H_{23}O_{14}N_5$ | 178-180 | Pale brown | 75 |
| X | 2-(Pentulose-γ-lactone-2,3-ene(*p*-nitrobenzoate)-3-[4-(1,3-dioxoisoindolin-2-yl)benzamide]-2,3-dihydro(3-nitrobenzo)[1,2e][1,3]-oxazepine-4,7-dione | $C_{42}H_{22}O_{18}N_6$ | 180-182 | Yellow | 77 |

**Table (2): The FTIR absorption bands data of 1,3-oxazepine compounds (VIII-X)**

| Comp. No. | N-H | C-H ar. | C-H ali. | C=O lactone | C=O lactam | C=C ali. | C=C ar. | C-O-C est. | $NO_2$ asym. | $NO_2$ sym. |
|---|---|---|---|---|---|---|---|---|---|---|
| VIII | 3167 | 3020 | 2898 | 1712 | 1657 | 1603 | 1552 | 1298-1117 | 1493 | 1333 |
| IX | 3168 | 3014 | 2897 | 1738 | 1658 | 1603 | 1556 | 1302-1157 | 1496 | 1342 |
| X | 3369 | 3020 | 2902 | 1697 | 1660 | 1606 | 1489 | 1294-1113 | 1545 | 1340 |

## Results and Discussion

The aim of this work is synthesis of some new 1,3-oxazepine compounds derived from D-erythroascorbic acid, scheme (1). To obtain these compounds starting to prepare the acetal (I) from reaction of L-ascorbic acid with dry acetone in presence of HCl gas, following Salomon method[12], then esterification of hydroxyl groups at positions C-2 and C-3 for compound (I) by used *p*-nitrobenzoyl chloride in presence of dry pyridine yielded compound (II). The structure of compounds (I) and (II) were identified by FTIR spectroscopy. The FTIR spectrum of the ester (II) showed disappearance of absorption band belong to (O-H) at (3244 $cm^{-1}$) for compound (I) and

appearance of absorption band at (1693 $cm^{-1}$) assigned to carbonyl group of ester for compound (II).

Afforded to the glycol (III) from compound (II) by using (65%) acetic acid with absolute ethanol, the stirring until (48 hrs) at normal temperature. The structure of the glycol (III) was confirmed by FTIR spectrum which was shown appearance of absorption band at (3413 $cm^{-1}$) due to hydroxyl groups. Oxidation of the glycol (III) by sodium periodate yielded the aldehyde (IV), the FTIR spectrum of compound (IV), Fig. (1) showed the absorption band at (1697 $cm^{-1}$) assigned to (C=O) aldehydic. This compound (IV) appeared a positive Tolen's experiment by forming a mirror of silver [18]. The structure of (IV) was proven by $^{1}$H-NMR spectrum, whom offered a signal at δ(13.70) ppm to proton of (H-C=O), Fig. (2) and has been identified via $^{13}$C-NMR spectrum, Fig. (3).

To prepared the amide (VI), we needed compound (V), which was prepared from reaction of phthalic anhydride with 4-aminobenzoic acid in glacial acetic acid, the mixture was refluxed for 3 hrs, then converted to acid chloride by its reaction of thionyl chloride in dry benzene, the mixture was refluxed for 3 hrs. Finally, to obtain the amide (VI) by reaction of acid chloride with hydrazine hydrate in benzene, the mixture was refluxed for 4 hrs.

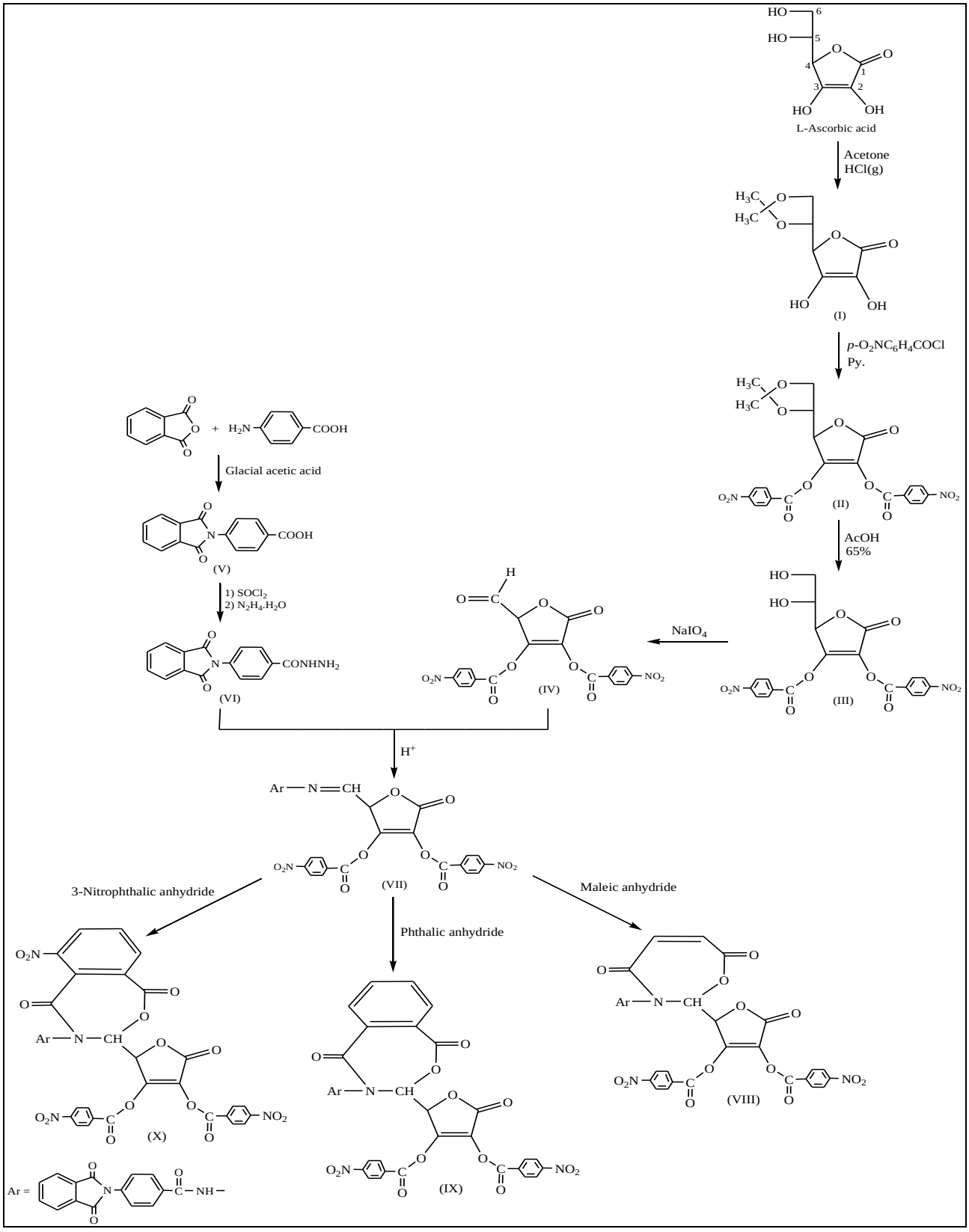
The structure of compounds (V) and (VI) were identified by FTIR spectra and $^{1}$H-NMR spectrum for compound (VI). The FTIR spectrum of compound (V) exhibited the absorption band at (1701 $cm^{-1}$) assigned to (COOH). The FTIR spectrum of compound (VI), Fig. (4) showed the absorptions band at (3345, 3302) $cm^{-1}$ assigned to asymmetric and symmetric stretching vibration of ($NH_2$) and at (1624 $cm^{-1}$) for (C=O) for amide. The $^{1}$H-NMR spectrum for compound (VI), Fig. (5) showed the signals at δ(10.56) ppm for (1H, NH), δ(6.67-8.09) ppm due to aromatic protons and δ(4.10) ppm for (2H, $NH_2$).

Schiff base (VII) was synthesized by condensation reaction of aldehyde (IV) with amide (VI) and few drops of glacial acetic acid in presence of absolute ethanol as a solvent. The structure of Schiff base was characterized by FTIR, $^{1}$H-NMR & $^{13}$C-NMR spectra. The FTIR spectrum of Schiff base, Fig. (6) showed disappearance of absorption bands at (3345, 3302) $cm^{-1}$ due to ($NH_2$) for amide (VI) and absorption band at (1697) $cm^{-1}$ due to (C=O) for aldehyde (IV) and appearance of absorption band at (1651) $cm^{-1}$ for (C=N). The $^{1}$H-NMR and $^{13}$C-NMR spectra for Schiff base (VII), Figs. (7) and (8) showed the following signals: 9.61 (br. s, 1H, CH=N), 6.31-9.01 (m, 16H, aromatic), 3.62 (br. s, 1H, lactone ring H-4), 195.75 (C=O lactone ring and cyclicimide), 165.91 (C=O ester and sec. amide), 151.81 (C-5), 149.89 (C-3), 123.62-136.77 (C aromatic), 112.57 (C-2), 41.23 (C-4).

Finally, 1,3-oxazepine compounds (VIII-X) were synthesized from 1,3-dipolar cycloaddition reaction of Schiff base (VII) with different acid anhydrides such as (maleic, phthalic and 3-nitrophthalic) anhydride in dioxane as a solvent.

The mechanism of the reaction is shown in scheme (2). The mechanism involves the addition of one σ-carbonyl to π-bond (N=C) to give 4-membered cyclic and 5-membered cyclic ring of anhydride in the same transition state $[T.S.]_a$, which opens into (maleic, phthalic and 3-nitrophthalic) anhydride to give 7-membered cyclic ring 1,3-oxazepine [C][19].

The FTIR spectra showed absorption bands at (1697-1738) $cm^{-1}$ for lactone and (1657-1660)
$cm^{-1}$ for lactam and evanescence of absorption band at (1651) $cm^{-1}$ to (C=N) of Schiff base (VII), FTIR spectrum for compound (X), Fig. (9). The $^{1}$H-NMR and $^{13}$C-NMR spectra for compound (X), Figs. (10) and (11) showed the following signals: 9.72 (s, 1H, NH sec. amide), 8.20-8.59 (dd, 12H, aromatic), 7.49-8.18 (m, 7H, aromatic), 6.55 (d, 1H, H-5), 3.50 (d, 1H, lactone ring H-4), 166.91 (C=O lactam), 166.43 (C=O lactone ring and cyclicimide), 166.27 (C=O sec. amide), 150.47 (C=O ester), 147.12 (C-3), 124.17-136.84 (C aromatic), 113.08 (C-2), 73.40 (C-5), 66.79 (C-4).




**Scheme (1): The scheme of prepared compounds**

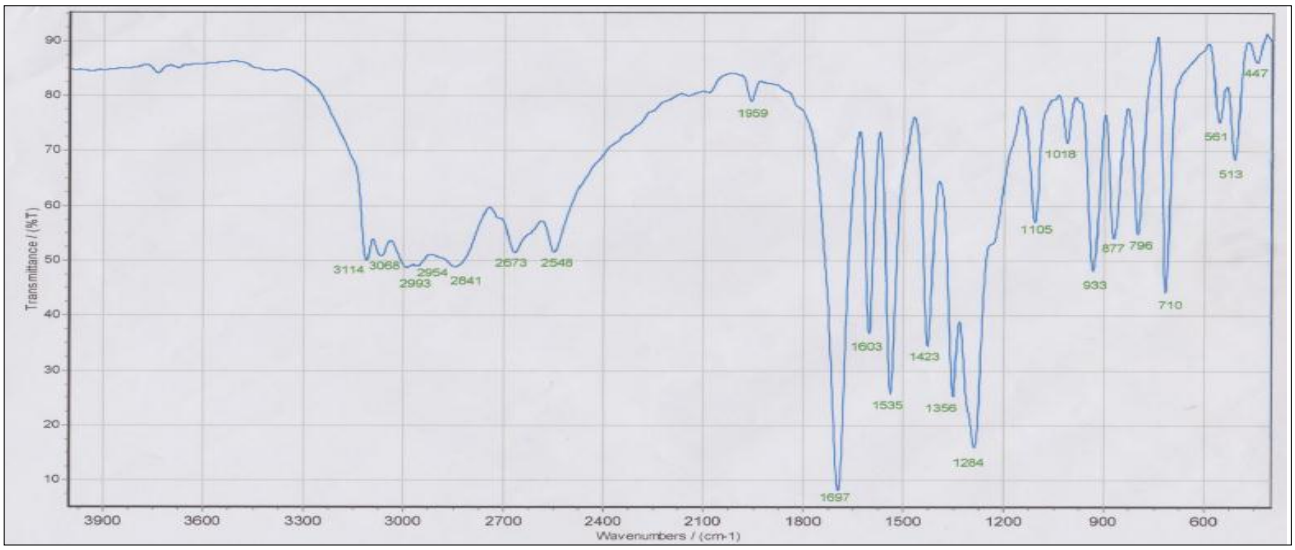



**Fig. (1): FTIR spectrum of aldehyde (IV)**

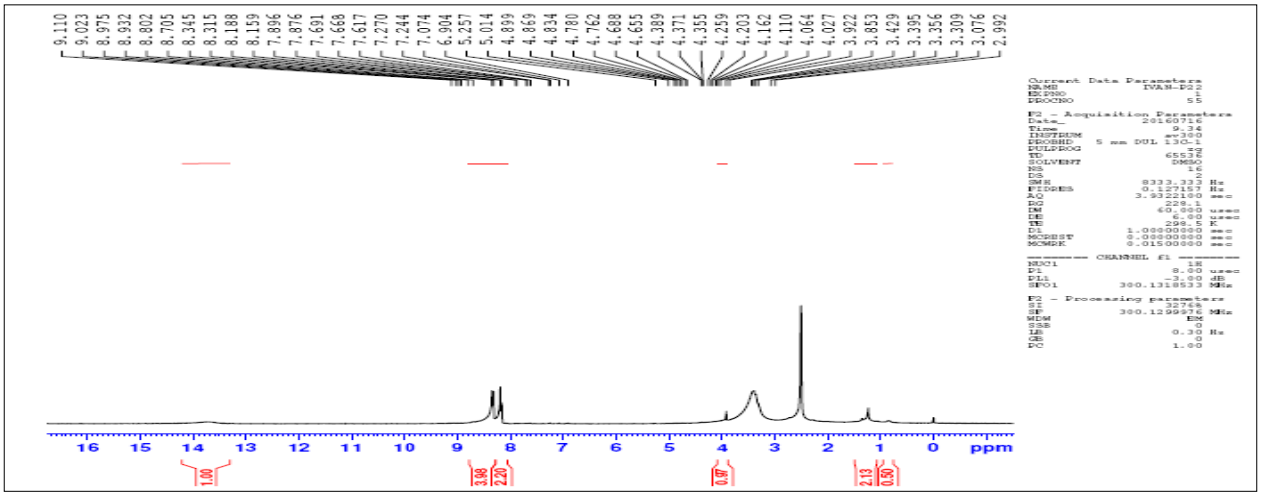


**Fig. (2): $^{1}$H-NMR spectrum of aldehyde (IV)**

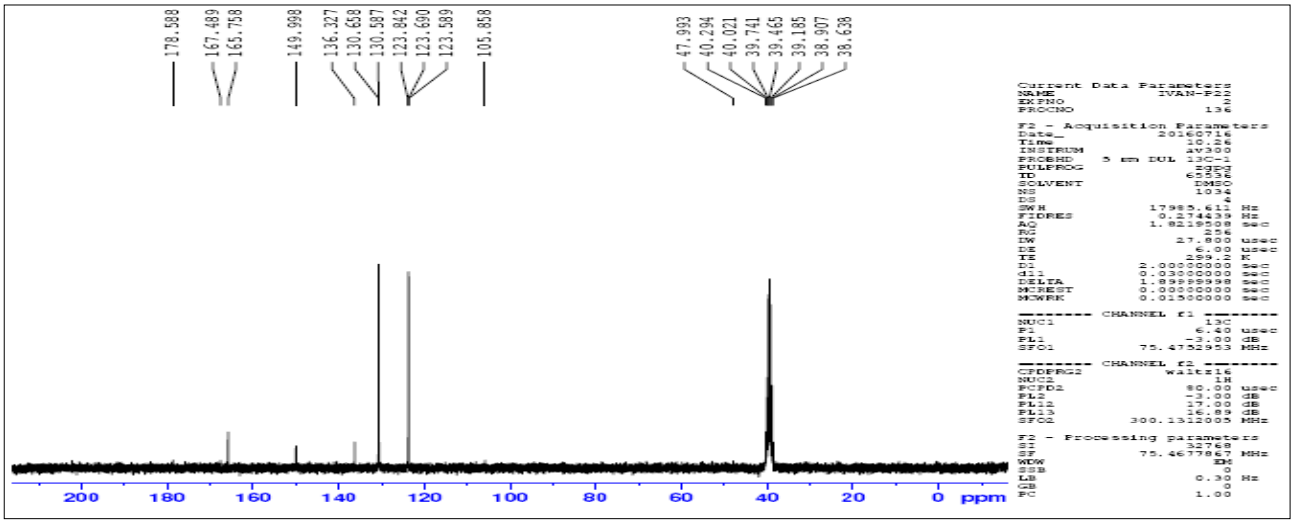


**Fig. (3): $^{13}$C-NMR spectrum of aldehyde (IV)**

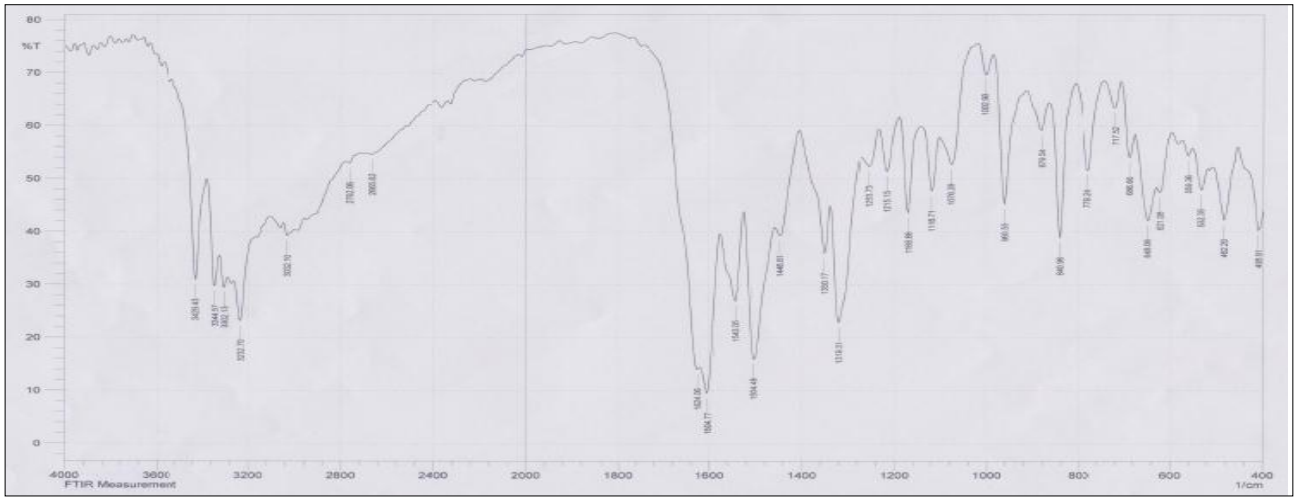



**Fig. (4): FTIR spectrum of amide (VI)**

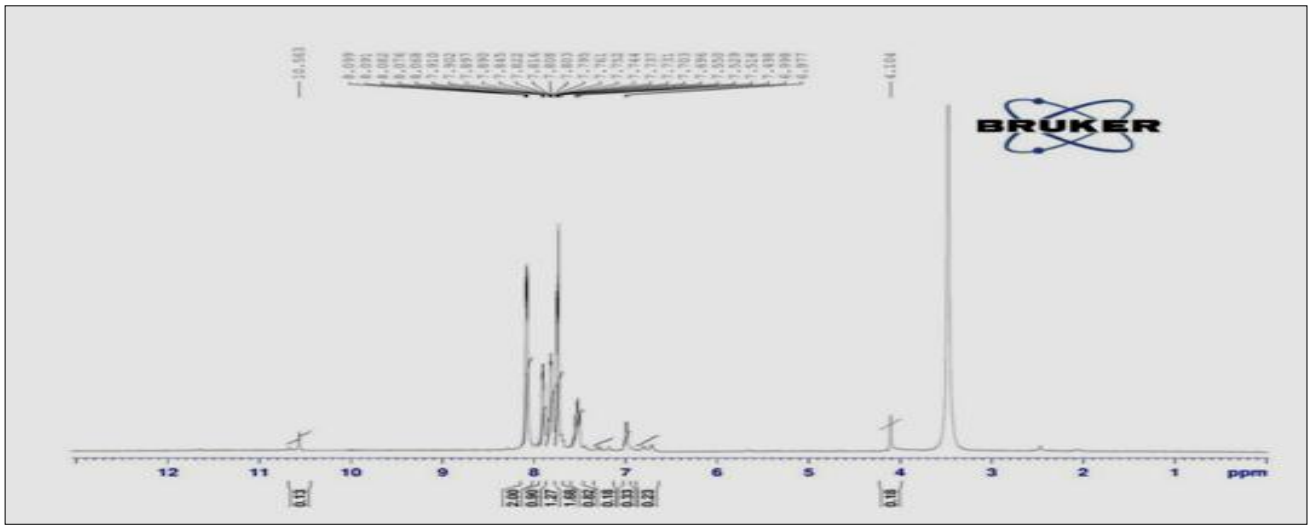



**Fig. (5): $^{1}$H-NMR spectrum of amide (VI)**

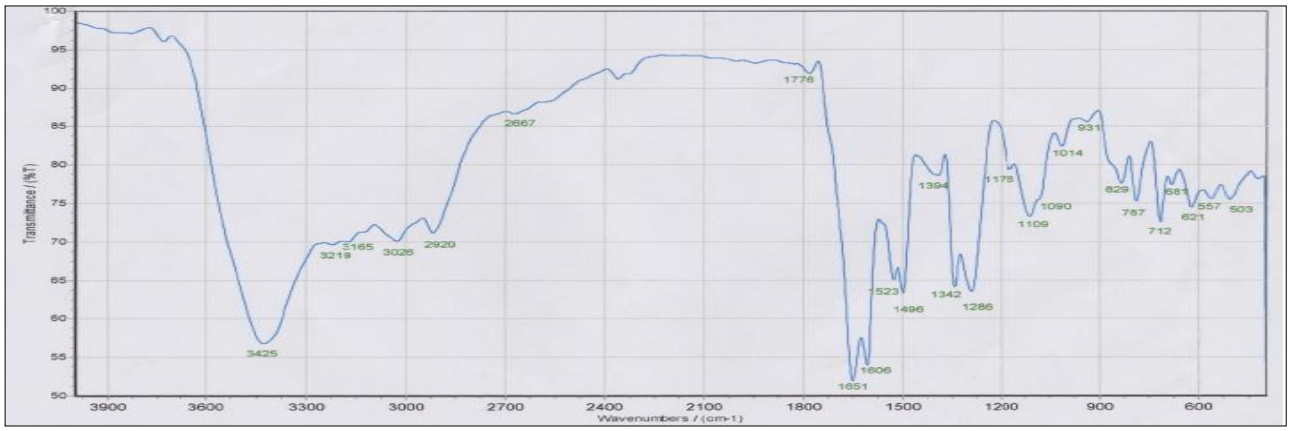



**Fig. (6): FTIR spectrum of Schiff base (VII)**

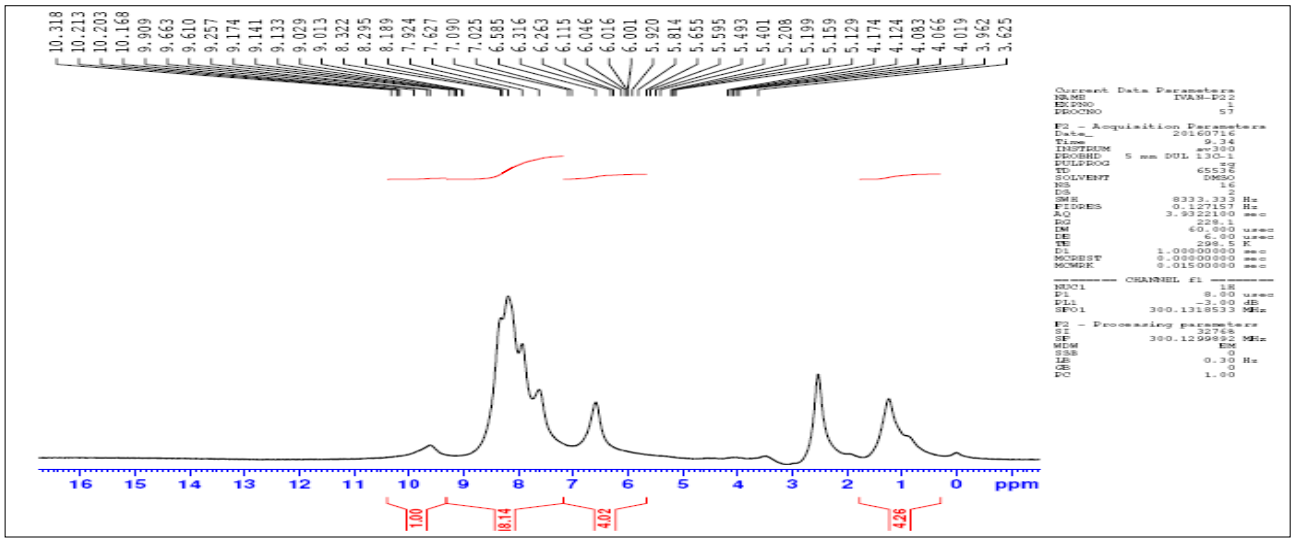


Fig. (7): $^{1}$H-NMR spectrum of Schiff base (VII)

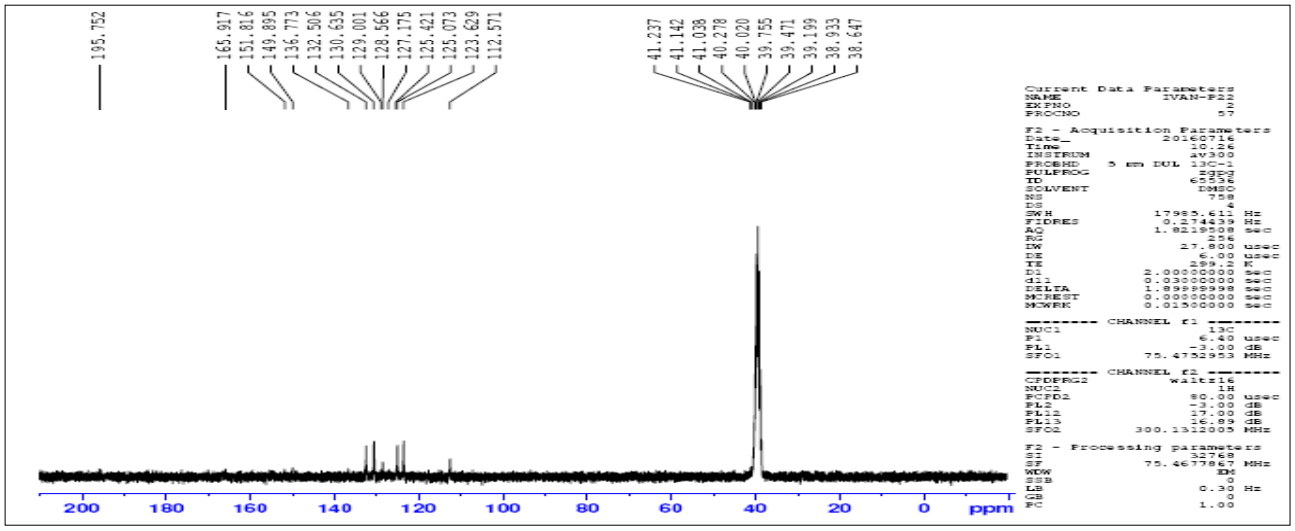


Fig. (8): $^{13}$C-NMR spectrum of Schiff base (VII)

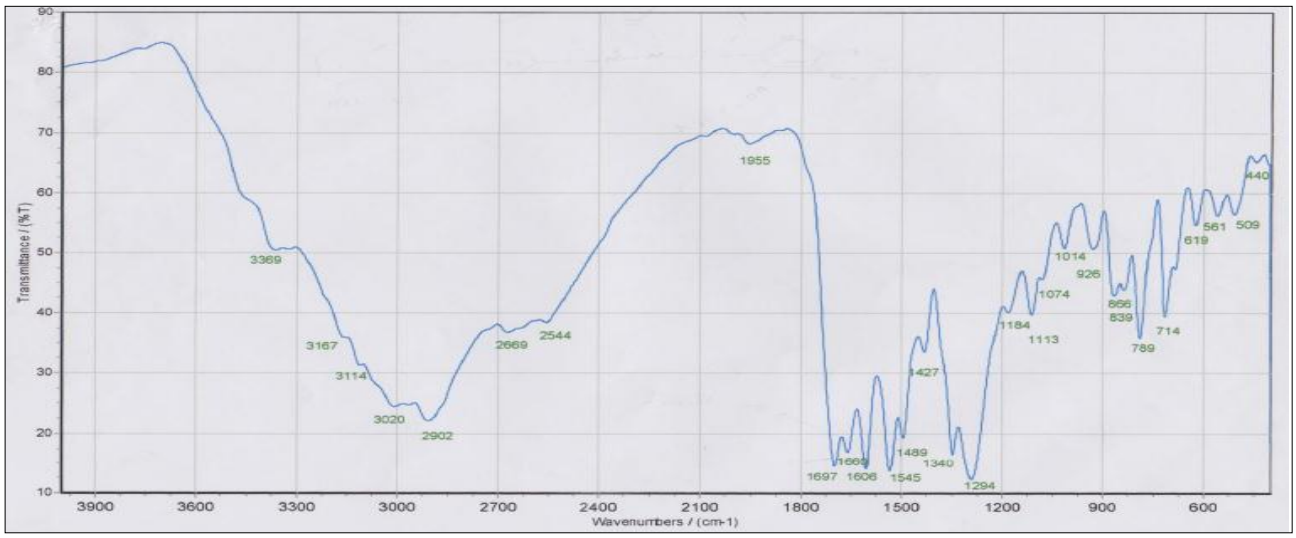


**Fig. (9): FTIR spectrum of compound (X)**

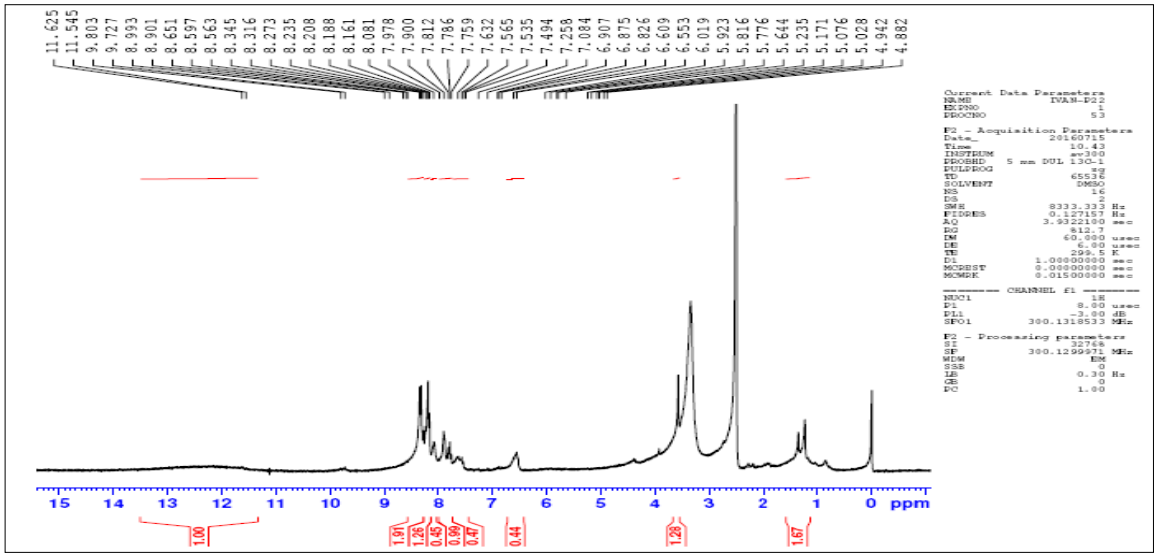


**Fig. (10): $^{1}$H-NMR spectrum of compound (X)**

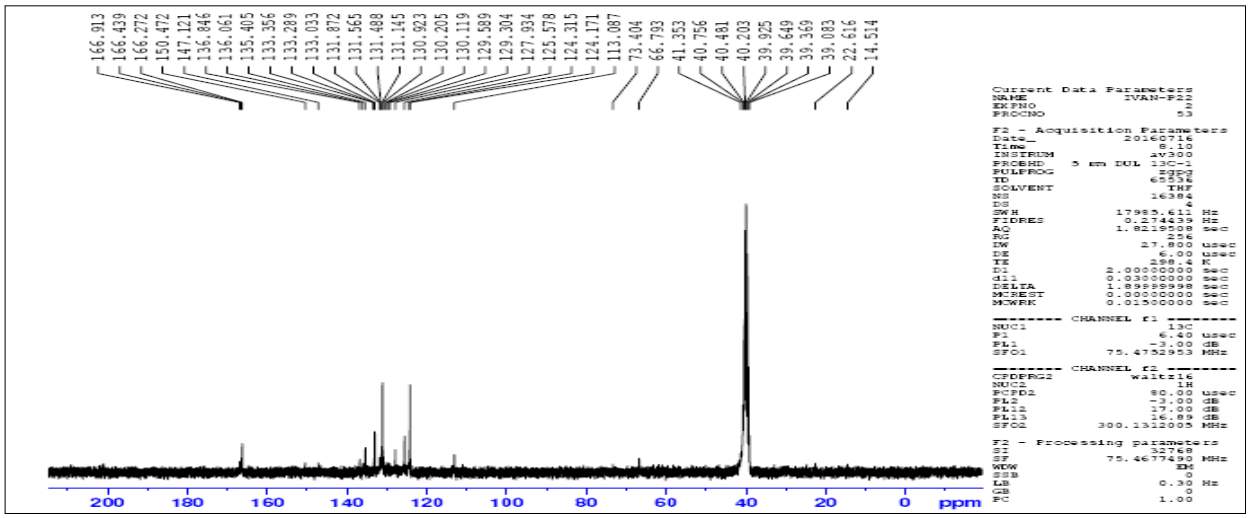
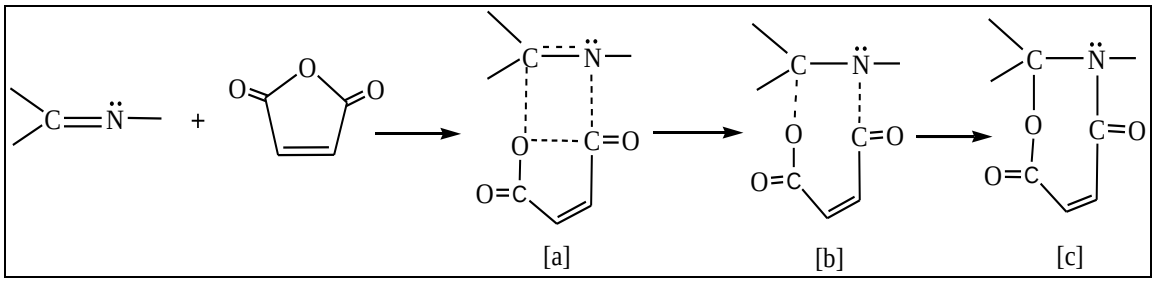


**Fig. (11): $^{13}$C-NMR spectrum of compound (X)**




**Scheme (2): The mechanism of formation of 1,3-oxazepine**

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# تحضير وتشخيص بعض مركبات الاوكسازبين الجديدة المشتقة من D- حامض الارثرواسكوربيك

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## الخلاصة

يتضمن هذا البحث تحضير بعض مشتقات ٣،١- اوكسازبين، حيث بدأنا بتحضير الاسيتال (I) من تفاعل L- حامض الاسكوربيك مع الاسيتون الجاف بوجود كلوريد الهيدروجين الجاف. وعند معاملة الاخير مع بارا- نايتروبنزويل كلورايد في البيريدين حصلنا على الاستر (II) الذي أذيب في (٦٥٪) من حامض الخليك في الايثانول المطلق وأعطى الكلايكول (III). تم الحصول على الالديهايد (IV) من تفاعل الكلايكول (III) مع بيرأيودات الصوديوم في الماء المقطر.

حضر المركب ٤-(٣،١- ثنائي اوكسو ازواندولين-٢- يل) حامض البنزويك (V) من تفاعل انهيدريد الفثاليك مع بارا- امينو حامض البنزويك بوجود حامض الخليك الثلجي. ان تفاعل المركب (V) مع كلوريد الثايونيل ينتج ٤-(٣،١- ثنائي اوكسو ازواندولين-٢- يل) كلوريد البنزويل الذي يتكاثف مع الهيدرازين المائي ليعطي ٤-(٣،١- ثنائي اوكسو ازواندولين-٢- يل) بنزوهيدرازيد (VI).

حضر مركب الازوميثين (VII) من التفاعل بين مركب (IV) و (VI). اضافة الى ذلك حضرت المركبات (X-VIII) من التكاثف الحلقي لقاعدة شف (VII) مع انهيدريد الماليك، الفثاليك و ٣- نايتروفثاليك وتم اثبات تراكيب المركبات الجديدة بواسطة قياسات الخواص الفيزيائية والطيفية مثل ( الاشعة تحت الحمراء وتم قياس لبعضها بروتون وكاربون الرنين النووي المغناطيسي).

الكلمات المفتاحية: قاعدة شف، ٣،١- اوكسازبين، L- حامض الاسكوربيك