

Synthesis, Characterization of Heterocyclic Compounds Containing Dapsone

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ABSTRACT

A series of new heterocyclic compounds have been synthesized by reaction of Schiff bases containing azo dyes with (maleic, phthalic anhydride) in the presence of toluene as solvent. The preparation procedure includes a series of steps, and the first step includes synthesizing azo compound from (4,4'-diamino diphenyl sulfone, Dapsone). The second step involved synthesizing Schiff bases by reaction of an azo compound with various substituted aniline derivatives. The third step involved a synthesis of heterocyclic compounds (oxazepine). Fourier transform infrared (FTIR), Proton nuclear magnetic resonance (¹H-NMR), ¹³ Carbon-13 (C13) nuclear magnetic resonance (C-NMR) spectra determined all compounds.

Keywords: Dapsone, heterocyclic, Schiff bases, oxazepine.

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Conflict of interest: None

INTRODUCTION

Heterocyclic compounds are cyclic constituents with atoms from on to the smallest two different substances acting as members from its ring.¹⁻³ These compounds have optimistic pharmacological and biological properties and have extended additional importance because of their physical subunits in many normal products, such as vitamins, hormones, and antibiotics.⁴⁻⁸ Heterocyclic has been found the main structural in medical chemistry⁹ and found in all compounds, including antifungal, anti-inflammatory, antibacterial, antioxidant, anti-HIV, antidiabetic and anticancer activity.^{10,11} Dapsone is the old antibiotic¹² used for the treatment of mycobacterial, protozoal infections, anti-glioma,¹³⁻¹⁵ anti-leprosy,¹⁶ antifungal,¹⁷ antibacterial,¹⁸ dermatologic disorders, and inhibits foliate.¹⁹⁻²³

MATERIALS AND METHODS

All chemicals secondhand were supplied from a fluke, Merck, and BDH chemical company; melting points were recorded

in open capillary tubes on the Electrothermal melting point apparatus, UK. Shimadzu 8400S recorded FT-IR spectra. Japan, using KBr disc. ¹H-NMR, and ¹³CNMR were recorded in DMSO-*d*₆ on Bruker Spectrophotometer at 500 MHz use TMS as an intern standard of reference.

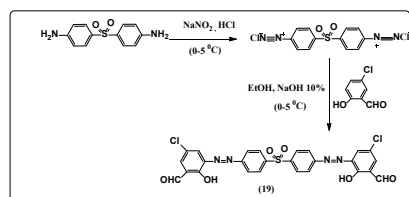
Synthesis and Characterization of Prepared Compounds:

Synthesis of 3, 3'-sulfonyl bis (1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxybenzaldehyde) (19)

Diapason (4,4'-diamino-diphenylsulfone) (2.5 gm, 0.01 mol.) was dissolved in hydrochloric acid(6 mL) and water distilled (20 mL) concentrated. At the ice-water bath, sodium nitrite (1.4/gm, 0.02 mol) was dissolved in 10 mL of distilled water and cold at (0–5°C) and, dropwise, was added to the solution stirring to form azo salt. 5-chloro salicylaldehyde (3.155 gm, 0.02mol) was dissolved in (25 mL) of ethanol and (10 mL) of sodium hydroxide 10% and was then cooled to (0–5°C), added to the diazonium solution is dropwise and stirring at (0–5°C) for (2hours.) for obtaining the coupling agent. The precipitated compounds were filtered, and the solution was again purified with water. The reaction pathway for the formation of azo compound [19] is shown by the Scheme 1.

Characterizations of Compound (19)

The result shows (83% yield), M. wt =583.4, m. p. =108-110 °C, the spectra exhibited the fundamental vibration modes



Scheme 1: Preparation of azo dye(19)

(cm^{-1}): -OH (3246 cm^{-1}), -C-H aromatic (3095), -CH aldehyde (2873 cm^{-1}), -C=O (1681 cm^{-1}), -C=C aromatic ($1571\text{--}1435\text{ cm}^{-1}$), -N=N (1471 cm^{-1}), -S=O (1305 cm^{-1}) and -C=Cl (767 cm^{-1}). $^1\text{H-NMR}$ (500 MHz, DMSO-d_6), $\delta=10.96\text{ ppm}$ [s, 1H, OH], $\delta=7.03\text{--}8.32\text{ ppm}$ [m, 12H, H aromatic], $\delta=10.41\text{ ppm}$ [s, 1H, H aldehyde], $^{13}\text{CNMR}$ (500 MHz, DMSO-d_6) $\delta=190.1\text{ ppm}$ [C=O aldehyde], $\delta=159.9$ [C-OH], $\delta=119.9\text{--}136.1$ [C-C aromatic].

General Procedure for Synthesis of Imines Compounds (20-22)

A round bottom flask (100 mL) was mixed together with 0.01 mol of Dapsone azo (0.01 mol), and 3-amino phenol, 4-methoxy aniline, and 4-chloro aniline (0.020 mol), which were each added into the ethanol (55 mL) while being stirred. The solution was synthesized using 4 drops from glacial acetic acid as a catalyst. A reflux was applied to the reaction mixture to achieve (12–14 hours.) and left to cool depressed to apartment temperature, hard produce (20-22) was obtained. The solvent was removed by filtration then the residual solid was recrystallized from absolute ethanol. The reaction pathway for forming Imine compounds [20-22] is shown by Scheme 2.

Characterizations (Figure 1 to 22)

6, 6' - sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(4-chloro-2-((E)-(3-hydroxyphenyl) imino methyl phenol) (20)

(77%yield), M.wt = 765.62, m. p. = 166–168 °C, the spectra exhibited the fundamental vibration modes (cm^{-1}): -OH ($3566\text{--}3586\text{ cm}^{-1}$), -C-H aromatic (3094 cm^{-1}), -CH=N- (1683 cm^{-1}), -C=C- aromatic ($1571\text{--}1435\text{ cm}^{-1}$), -N=N- (1506 cm^{-1}) and -S=O (1305 cm^{-1}). $^1\text{H-NMR}$ (500 MHz, DMSO-d_6), $\delta=13.11\text{ ppm}$ [s, 1H, OH], $\delta=9.79\text{ ppm}$ [s, 1H, OH], $\delta=8.85\text{ ppm}$ [s, 1H, H imine], $\delta=6.59\text{--}7.76\text{ ppm}$ [m, 20H, H aromatic], $^{13}\text{CNMR}$ (500 MHz, DMSO-d_6) $\delta=162.1\text{ ppm}$ [CH=N], $\delta=159.9$ [C-OH], $\delta=108.5\text{--}149.4$ [C-C aromatic].

6, 6' -sulfonyl bis-(4,1-phenylene)bis-(diazene-2,1-diyl) bis-(4-chloro-2-((E)-(4-methoxy phenyl) imino) methyl phenol) (21)

(68%yield), M.wt = 793.68, m. p. = 193–195 °C, the spectra exhibited the fundamental vibration modes (cm^{-1}): -OH (3438 cm^{-1}), -C-H aromatic (3083 cm^{-1}), -CH=N- imine (1683 cm^{-1}), C=C aromatic ($1577\text{--}1438\text{ cm}^{-1}$), -N=N- (1506 cm^{-1}) and -S=O (1325 cm^{-1}).

6, 6' - sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(4-chloro-2-((E)-(4-chloro phenyl) imino) methyl phenol) (22)

(71 %yield), M. wt = 802.51, m. p. = 185–187 °C, the spectra exhibited the fundamental vibration modes (cm^{-1}): -OH (3442 cm^{-1}), -C-H aromatic (3090 cm^{-1}), -CH=N- imine (1683 cm^{-1}), C=C aromatic ($1575\text{--}1490\text{ cm}^{-1}$), -N=N- (1471 cm^{-1}) and -S=O (1325 cm^{-1}).

General Procedure for 1,3-oxazepine derivatives [23-28]

In a round bottom flask (100 mL) armed with binary surface condenser tailored with an anhydrous calcium chloride

protector tube and stirring magnetic saloon, a combination of Imines (20,21,22) (0.01 mol) and maleic, phthalic anhydride (0.01 mol) in toluene (40 mL) was placed. The reaction mixture was refluxed for (20–28 hours) then the separated solid products [23-28] and was washed away, dried and recrystallized with distilled water twice with ethanol. The pathway of preparation of the target 1,3- oxazepine derivatives [23-28] were showed in scheme 2.

2,2' - sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(3-(3-hydroxy phenyl)-2,3-dihydr- 1,3-oxazepine-4,7-dione). (23)

(62%yield), M.wt = 961.74, m. p. = 214–216 °C, the spectra exhibited the fundamental vibration modes (v cm^{-1}): -OH (3317 cm^{-1}), -C-H aromatic (3062 cm^{-1}), -C=O lactam (1704 cm^{-1}), and -N=N- (1473 cm^{-1}). $^1\text{H-NMR}$ (DMSO-d_6 and 500MHz), $\delta=13.1\text{ ppm}$ [s, 1H, OH phenol], $\delta=10.3\text{ ppm}$ [s, 1H, OH], $\delta=6.7\text{--}9.6\text{ ppm}$ [m, 30H, H aromatic], $\delta=6.0\text{--}6.2\text{ ppm}$ [d, 2H, CH=CH], $^{13}\text{CNMR}$ (500 MHz, DMSO-d_6) $\delta=162.1\text{ ppm}$ [C=O lactam], $\delta=159.4$ [C-OH], $\delta=107.1\text{--}158.7$ [C-C aromatic].

2,2' - sulfonyl bis (1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(3-(4-methoxy phenyl)-2,3-dihydr- 1,3-oxazepine-4,7-dione) (24)

(63%yield), M.wt = 989.79 m. p. = 238–240 °C, the spectra exhibited the fundamental vibration modes (cm^{-1}): -OH (3263 cm^{-1}), C-H aromatic (3062 cm^{-1}), C=O lactam (1712 cm^{-1}) and -N=N- (1481 cm^{-1}).

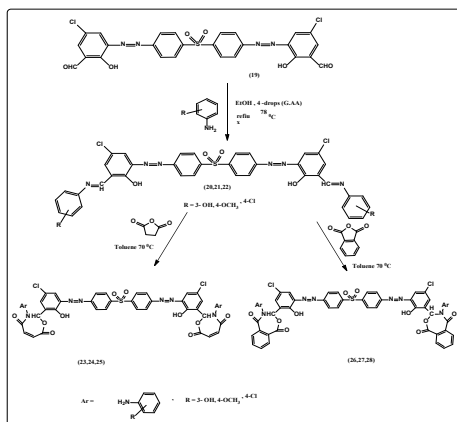
2,2' - sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene)-bis-(3-(4-chloro phenyl)-2,3-dihydr-1,3-oxazepine-4,7-dione). (25)

(78%yield), M.wt = 998.62 m. p. = 201–203 °C, the spectra exhibited the fundamental vibration modes (cm^{-1}): -OH (3394 cm^{-1}), C-H aromatic (3078 cm^{-1}), C=O lactam (1704 cm^{-1}) And -N=N- (1488 cm^{-1}). $^1\text{HNMR}$ (DMSO-d_6 and 500MHz), $\delta=10.9\text{ ppm}$ [s, 1H, OH], $\delta=6.4\text{--}8.9\text{ ppm}$ [m, 28H, H aromatic], $\delta=6.1\text{--}6.4\text{ ppm}$ [d, 2H, CH=CH], $^{13}\text{CNMR}$ (500 MHz, DMSO-d_6) $\delta=167.4\text{ ppm}$ [lacton], $\delta=163.8\text{ ppm}$ [lactam], $\delta=159.2$ [lacton], $\delta=112.9\text{--}138.4$ [C-C aromatic].

3,3' - sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(4-(3-hydroxy phenyl)-3,4-dihydrobenzo (e)(1,3)oxazepine - 1,5-dione) (26)

(54%yield), M.wt = 1061.86, m. p. = 254–256 °C, the spectra exhibited the fundamental vibration modes (cm^{-1}): -OH (3394 cm^{-1}), -C-H aromatic (3078 cm^{-1}), -C=O lactam (1689 cm^{-1}) and -N=N- (1483 cm^{-1}). $^1\text{H-NMR}$ (DMSO-d_6 and 500MHz), $\delta=13.1\text{ ppm}$ [s, 1H, OH phenol], $\delta=10.2\text{ ppm}$ [s, 1H, OH], $\delta=6.4\text{--}8.8\text{ ppm}$ [m, 34H, H aromatic], $^{13}\text{CNMR}$ (500 MHz, DMSO-d_6) $\delta=169.07\text{ ppm}$ [C=O lacton], $\delta=167.9\text{ ppm}$ [C=O lactam], $\delta=107.1\text{--}131.4$ [C-C aromatic].

3,3' - sulfonyl bis-(4,1-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene)-bis-



Scheme 2: Preparation of Schiff bases and 1,3-oxazepine compounds.

(4-(4-methoxy phenyl)-3,4-dihydrobenzo (e) (1,3) oxazepine-1,5-dione) (27)

(61% yield), M.wt=1089.91, m. p. =273–275 °C, the spectra exhibited the fundamental vibration modes (cm^{-1}): -OH (3394 cm^{-1}), aromatic CH (3070 cm^{-1}), lactam C=O (1712 cm^{-1}), -N=N- (1512 cm^{-1}).

3,3'-sulfonyl bis- (1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene)-bis-(4-(4-chloro phenyl)-3,4-dihydrobenzo (e) (1,3)oxazepine-1,5-dione) (28)

(64%yield), M.wt = 1098.74, m. p. =271–273°C, the spectra exhibited the fundamental vibration modes (cm^{-1}): -OH (3309 cm^{-1}), aromatic -CH (3020 cm^{-1}), C=O lactam (1697 cm^{-1}) and -N=N- (1510 cm^{-1}). $^1\text{H-NMR}$ (DMSO- d_6 and 500MHz), δ = 10.9 ppm [s, 1H,OH], δ =7-8.9 ppm [m, 32H, H aromatic], $^{13}\text{C-NMR}$ (500 MHz, DMSO- d_6) δ = 169 ppm [C=O lactam], δ = 167.9 ppm [C=O lactam], δ = 159.2 ppm [C-OH], δ = 119.1-147.4[C-C aromatic].

RESULTS AND DISCUSSION

The common method of diazotization of (4,4'-di amino di phenyl sulfone, Dapsone) usages hydrochloric acid. diazonium salt was reacted to 5-chlorine salicylaldehyde coupling in weak media of acid (pH=5-6) (Scheme 1). The disappearance of NH_2 group absorption bands in the FTIR absorption spectrum of (4,4'-di amino di phenyl sulfone, Dapsone) with the appearance of a new band assigned to azo group (N=N) and the appearance of a new absorption band which is allocated to aldehyde group (CHO). The FTIR spectra are listed in Table 1. Also, this compound's $^1\text{H-NMR}$ spectrum exhibited a singlets at δ 10.2 ppm integrating for the proton of the OH and a multiplet at δ 7.03–8.32 ppm integrating for the proton of the (H aromatic) and a singlet at δ 10.9 ppm integrating for the proton of the (H aldehyde). $^{13}\text{C-NMR}$ This spectrum shows that the signal is present at δ 190.1 ppm integrating for the carbon in (CHO) aldehyde and the signal at δ 159.9 integrating for the carbon in (C-OH) and the signal at δ 119.9-136.1 ppm integrating for the carbon in (C=C) aromatic. To make new Schiff bases [20-22], azo compound of aromatic aldehyde[19] and primary aromatic amine were refluxed in toluene with a few drops of glacial

acetic acid. By using an FT-IR absorption spectrum, we can see which of the starting materials' (NH_2) and (-CHO) groups are present as well as which additional absorption bands appear at 1683 cm^{-1} . which is assigned to imine ($\text{CH}=\text{N}$) and azo bands at $1471\text{--}1506 \text{ cm}^{-1}$. with bands of (-OH) at $3246\text{--}3587 \text{ cm}^{-1}$. The FT-IR spectra are listed in Table 1. Pericyclic reaction type [2+5] has been utilized to synthesize 1,3-oxazepine derivatives (23-28). In the cycloaddition reaction between imine groups ($\text{CH}=\text{N}$) in [20-22], maleic and phthalic anhydrides are used as five-membered components, while imine groups ($\text{CH}=\text{N}$) in [20-22] are used as two-membered components, giving a seven-membered structure. In the FT-IR spectrum of the 1,3-oxazepine derivative [23-28], the strong bands of imine ($\text{CH}=\text{N}$) groups were no longer detected and appearance of strong absorption bands of the (C=O) for lactam structures inside 1,3-oxazepine ring at $1688\text{--}1712 \text{ cm}^{-1}$ and azo group (N=N) at $1473\text{--}1512 \text{ cm}^{-1}$. The FTIR spectra are listed in Table 2. These compounds also have a singlet signal $^1\text{H-NMR}$ at δ 10.96 ppm integrating for the proton of the (-OH) and a multiplet signal at δ 7.03–8.32 ppm integrating for the proton of the (H aromatic), $^{13}\text{C-NMR}$ (500 MHz, DMSO- d_6) δ = 169-168 ppm integrating for (C=O lactam), and signal at δ = 164.9-162 ppm integrating for (C=O lactam), and at δ = 119.1-147.4 for (C-C aromatic).

Table 1: The spectra from the FT-IR measurements for synthesized compounds [19-22].

Comp. no.	V(OH)	(CH) arom	V (N=N)	V (O=S=O)	V (CH=N)	V (CHO)
19	3246	3095	1471	1305		1681
20	3566	3094	1505	1305	1683	
21	3438	3083	1506	1325	1683	
22	3442	3090	1471	1332	1683	

Table 2: The spectra from the FT-IR measurements for synthesized compounds [23-28].

Comp. no.	V(OH)	(CH) arom	V (N=N)	V C=O	V (O=S=O)
23	3317	3062	1473	1704	1335
24	3263	3062	1481	1712	1304
25	3394	3078	1488	1704	1325
26	3394	3078	1483	1688	1312
27	3394	3070	1512	1712	1361
28	3309	3020	1510	1697	1330

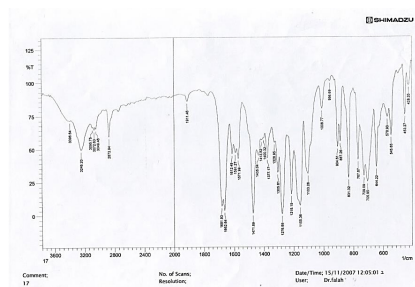


Figure 1: FT-IR spectrum of 3,3'-sulfonyl bis (1,4-phenylene) bis (diazene-2,1-diyl) bis (5-chloro-2-hydroxybenzaldehyde).

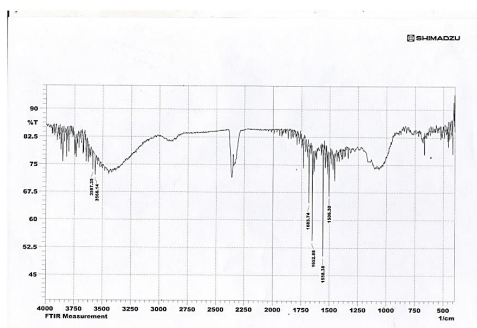


Figure 2: FT-IR spectrum of 6,6'-sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(4-chloro-2-((E)-(3-hydroxy phenyl) imino methyl phenol).

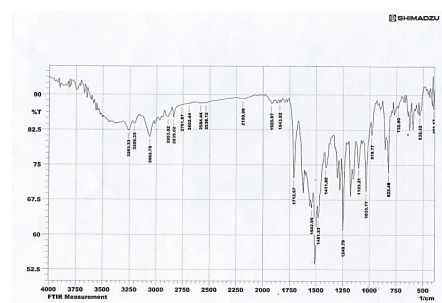


Figure 6: FT-IR spectrum of 2,2'-sulfonyl bis (1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(3-(4-methoxy phenyl)-2,3-dihydr-1,3-oxazepine-4,7-dione).

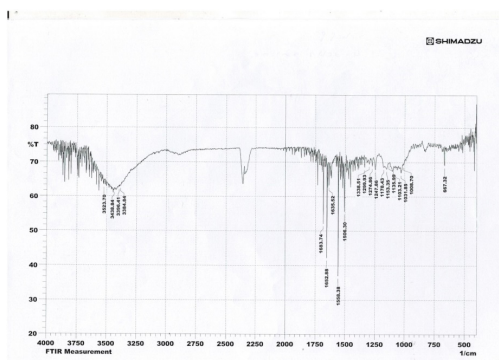


Figure 3: FT-IR spectrum of 6,6'-sulfonyl bis-(4,1-phenylene)bis-(diazene-2,1-diyl)bis-(4-chloro-2-((E)-(4-methoxy phenyl) imino methyl phenol).

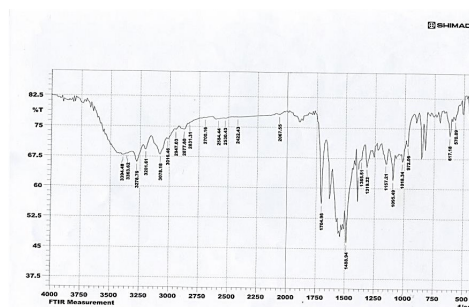


Figure 7: FT-IR spectrum of 2,2'-sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl)bis-(5-chloro-2-hydroxy-3,1-phenylene)-bis-(3-(4-chloro phenyl)-2,3-dihydr-1,3-oxazepine-4,7-dione).

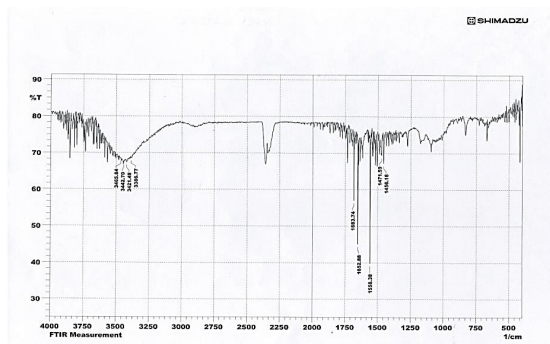


Figure 4: FT-IR spectrum of 6,6'-sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(4-chloro-2-((E)-(4-chloro phenyl) imino methyl phenol).

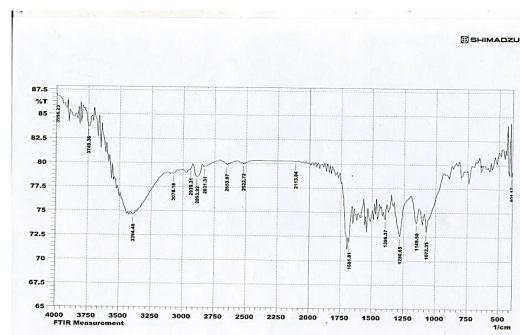


Figure 8: FT-IR spectrum of 3,3'-sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(4-(3-hydroxy phenyl)-3,4-dihydrobenzo(e)(1,3)oxazepine-1,5-dione).

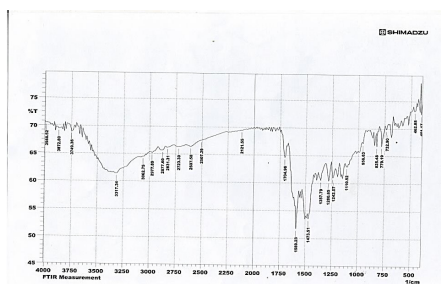


Figure 5: FT-IR spectrum of 2,2'-sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(3-(3-hydroxy phenyl)-2,3-dihydr-1,3-oxazepine-4,7-dione).

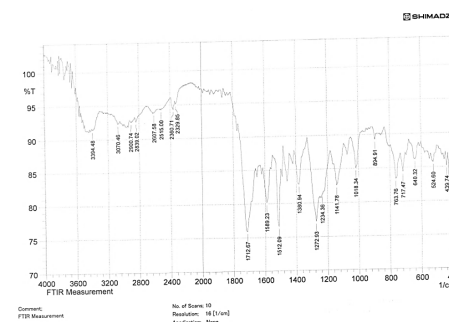


Figure 9: FT-IR spectrum of 3,3'-sulfonyl bis-(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(4-(4-methoxyphenyl)-3,4-dihydrobenzo(e)(1,3)oxazepine-1,5-dione).

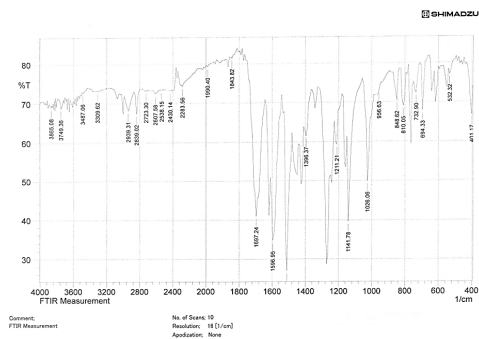


Figure 10: FT-IR spectrum of 3,3'-sulfonyl bis(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(4-(4-chloro phenyl)-3,4-dihydrobenzo(e)(1,3)oxazepine-1,5-dione).

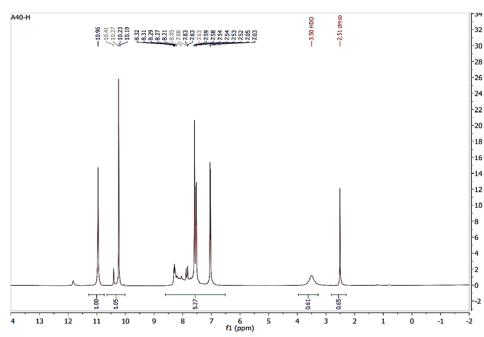


Figure 11: ¹H NMR spectrum of 3,3'-sulfonyl bis(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxybenzaldehyde).

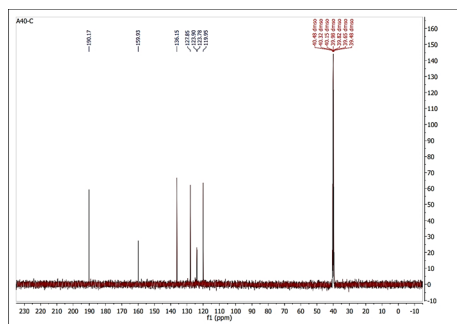


Figure 12: ¹³C NMR spectrum of 3,3'-sulfonyl bis(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxybenzaldehyde).

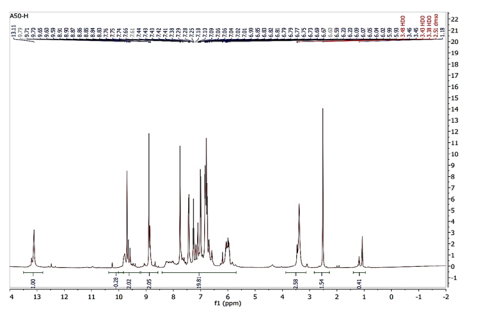


Figure 13: ¹H NMR spectrum of 6,6'-sulfonyl bis(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(4-chloro-2-(E)-(3-hydroxy phenyl) imino methyl phenol).

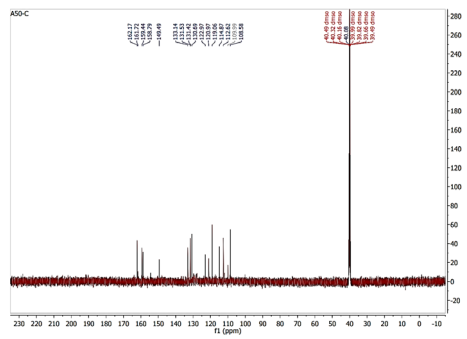


Figure 14: ¹³C NMR spectrum of 6,6'-sulfonyl bis(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(4-chloro-2-(E)-(3-hydroxy phenyl) imino methyl phenol).

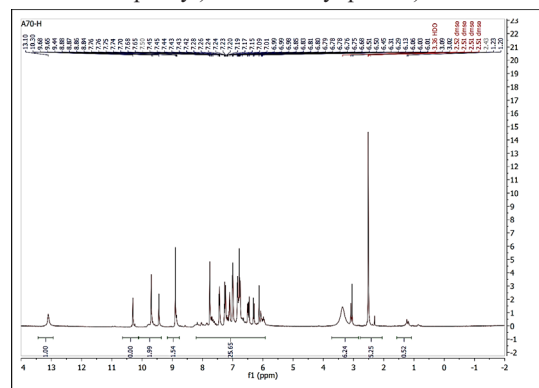


Figure 15: ¹H NMR spectrum of 2,2'-(sulfonylbis(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(3-(3-hydroxy phenyl)-2,3-dihydr-1,3-oxazepine-4,7-dione).

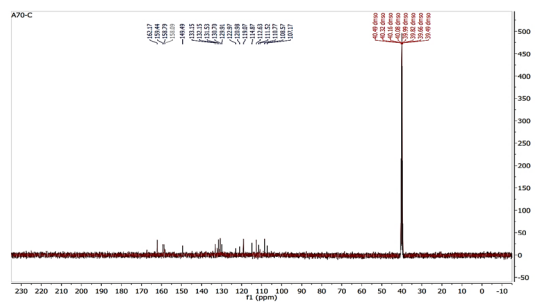


Figure 16: ¹³C NMR spectrum of 2,2'-(sulfonylbis(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(3-(3-hydroxyphenyl)-2,3-dihydr-1,3-oxazepine-4,7-dione).

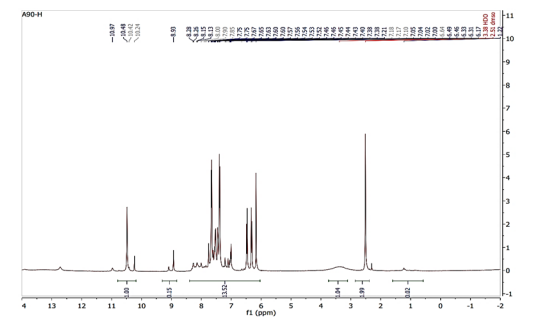


Figure 17: ¹H NMR spectrum of 2,2'-(sulfonylbis(1,4-phenylene) bis-(diazene-2,1-diyl) bis-(5-chloro-2-hydroxy-3,1-phenylene) bis-(3-(4-chlorophenyl)-2,3-dihydr-1,3-oxazepine-4,7-dione).

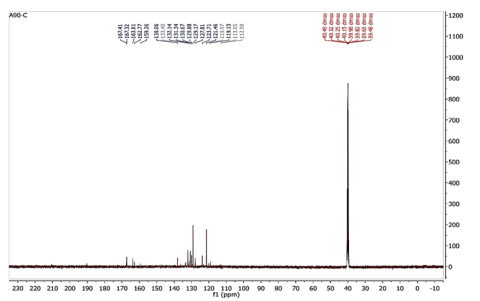


Figure 18: ^{13}C -NMR spectrum of 2,2'-sulfonylbis(1,4-phenylene)-bis-(diazene-2,1-diyl)-bis-(5-chloro-2-hydroxy-3,1-phenylene)-bis-3-(4-chlorophenyl)-2,3-dihydr-1,3-oxazepine-4,7-dione

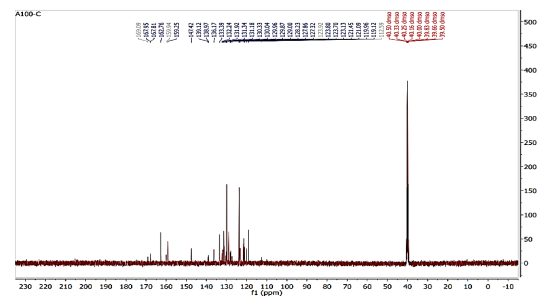


Figure 22: ^{13}C -NMR spectrum of 3,3'-sulfonyl bis-(1,4-phenylene)-bis-(diazene-2,1-diyl)-bis-(5-chloro-2-hydroxy-3,1-phenylene)-bis-(4-(4-chloro-phenyl)-3,4-dihydrobenzo(e)(1,3)oxazepine-1,5-dione)

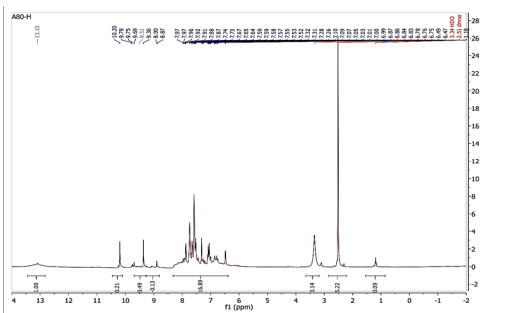


Figure 19: ^1H -NMR spectrum of 3,3'-sulfonyl bis-(1,4-phenylene)-bis-(diazene-2,1-diyl) bis (5-chloro-2-hydroxy-3,1-phenylene)-bis-(4-(4-chlorophenyl)-3,4-dihydrobenzo(e)(1,3)oxazepine-1,5-dione).

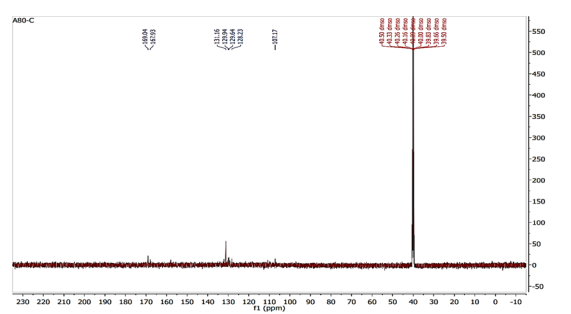


Figure 20: ^{13}C -NMR spectrum of 2,2'-sulfonylbis(1,4-phenylene)-bis-(diazene-2,1-diyl)- bis-(5-chloro-2-hydroxy-3,1-phenylene)-bis-3-(3-hydroxy phenyl)-2,3-dihydr- 1,3-oxazepine-4,7-dione.

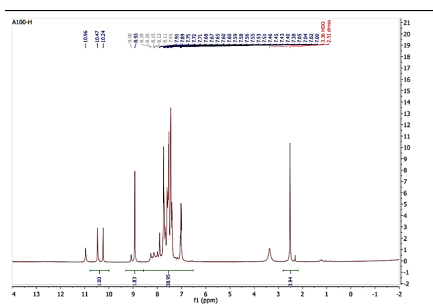


Figure 21: ^1H -NMR spectrum of 3,3'-sulfonyl bis-(1,4-phenylene)-bis-(diazene-2,1-diyl) bis (5-chloro-2-hydroxy-3,1-phenylene)-bis-(4-(3-hydroxyphenyl)-3,4-dihydrobenzo(e)(1,3)oxazepine-1,5-dione).

CONCLUSIONS

In conclusion, 3,3'-sulfonylbis-(1,4-phenylene)-bis-(diazene-2,1-diyl)bis(5-chloro-2-hydroxybenzaldehyde) (19) and imines

compounds (20-22) then heterocycle 1,3 oxazepine (23-28) derivatives were synthesized positively. All results, FT-IR, ^1H -NMR, and ^{13}C -NMR analysis show that the objective molecules were formed due to the least obtrusive result in all preparation methods by (5+2) cycloaddition reaction of maleic, phthalic anhydrides and imine group.

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