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FULL PAPER

Synthesis and diagnosis of triazole and oxirane derivatives from hydroxyquioline with evaluating their biological and antioxidant activity

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Research involved preparing new compounds from hydroxyquioline with formaldehyde to form P1 and then reacted with benzylalcohol to form P2 reacted chloroaceticacid and $SOCl_2$ to form P3 and P4 with thiosemicarbazide to form P5 and P2 reacted with benzydehyde to form chalcone reacted with H_2O_2 to form P8. The compounds were identified via TLC, FTIR 1HNMR and 13CNMR. The measured the antibacterial and antioxidant activity of the prepared compounds, to give high resulte.

KEYWORDS

Hydroxyquioline; benzylalcohol; formaldehyde; thiosemicarbazide; benzydehyde antioxidant; biological activity.

Introduction

Heterocyclic compounds are found on different structure Hydroxyquinoline which good physical and chemical [1] and derivatives have a good and wide range of properties biological activities such as anti-viral [2,3] antimicrobial [4] anti-cancer [5]anti-Alzheimer [6] anti-fungal [7]and chemotherapy agents [8,9].

Chalcones are a component of and plants from damage caused by insects and other animals, reactive oxygen species be controlled [10].The Claisen Schmidt or Aldol condensation reaction, chalcone is discovered [11]. It possesses numerous significant qualities, including anti-bacterial, anti-fungal, diabetes-fighting and anti-cancer, also enhances joints, memory and kidneys include cancer prevention, immune system boosting and skin and hair [12,13].

It also serves a number of other purposes.

Aldehyde made from ligustrazine can be used to make novel chalcones. And found in nature in biological features including anti-cancer and pharmacologically active compounds [14-19].

The new chalcone was used to prepare the oxirane, and the effectiveness of all the prepared compounds was evaluated against two types of bacteria and antioxidant activity.

Five atoms, three nitrogens, two carbons, make up the heterocyclic chemical triazoles, which are triazoles [20]. It plays a significant role in interactions between medicine and agriculture [21]anti-viral, antibacterial, anti-fungal, and TB activity, as well as anti-oxidant, anti-tumor, analgesic, anti-

inflammatory [22-26] and anticancer agents [27].

Experimental Part

The chemical compounds used were from Merck and BDH. "Testseon and Shimadzu (FTIR 8400Series Japan)" instrument, and 1HNMR and 13CNMR spectra using DMSO solvent and with "Bruker, Ultra Shield 500 MHZ Switzerland."

Synthesis of compound P1 and P2 [28,29]

Compound (P) (1 mole) reacted with (0.131 g, 0.5 mole) and paraformaldehyde (0.045 g, 1.5 mol) in 1,4-dioxane (2 mL) and catalytic zinc chloride in concentrated HCl acid to form P1. Compound (P1) reacted with benzyl alcohol in the presence of sodium hydride in anhydrous THF and heated at 110 °C, dried the precipitate was recrystallized from absolute ethyl alcohol. Table1.

Synthesis of compound P3 and P4 [30,31]

Compound P2 (0.01 mole, 3.56 g) with (0.01 mole, 0.94 g) of chloroacetic acid added in around-bottom flask and headed for 3 hours to form P3, Compound (P3) reacted $SOCl_2$ (15 mL) and stirred (4h.), washed and recrystallized from ethanol to form (P4). Table1.

Synthesis of compound P5 [32]

Thiosemicarbazide (0.01mole, 0.18 g) and sodium hydroxide solution (10%) (50 mL) with (20 min.) stirring and (0.01 mole, 4.73 g,) of (P4) was added and dissolved in 50 mL of dioxane and stirred for (24 h.) added ice water, stirred for 30 minutes acidified with HCl concentrate. TLC was used to monitor reaction, and recrystallized from ethanol. Table1.

Synthesis of compound P6 [33]

Mixture of compound (P5), (0.01 mole, 0.63 g) and sodium hydroxide 4% (0.01 mole,) was refluxed with stirred for four hours. cooled and added HCl conc, the precipitate was recrystallized from absolute ethyl alcohol (Table1).

Synthesis of chalcone P7 [34]

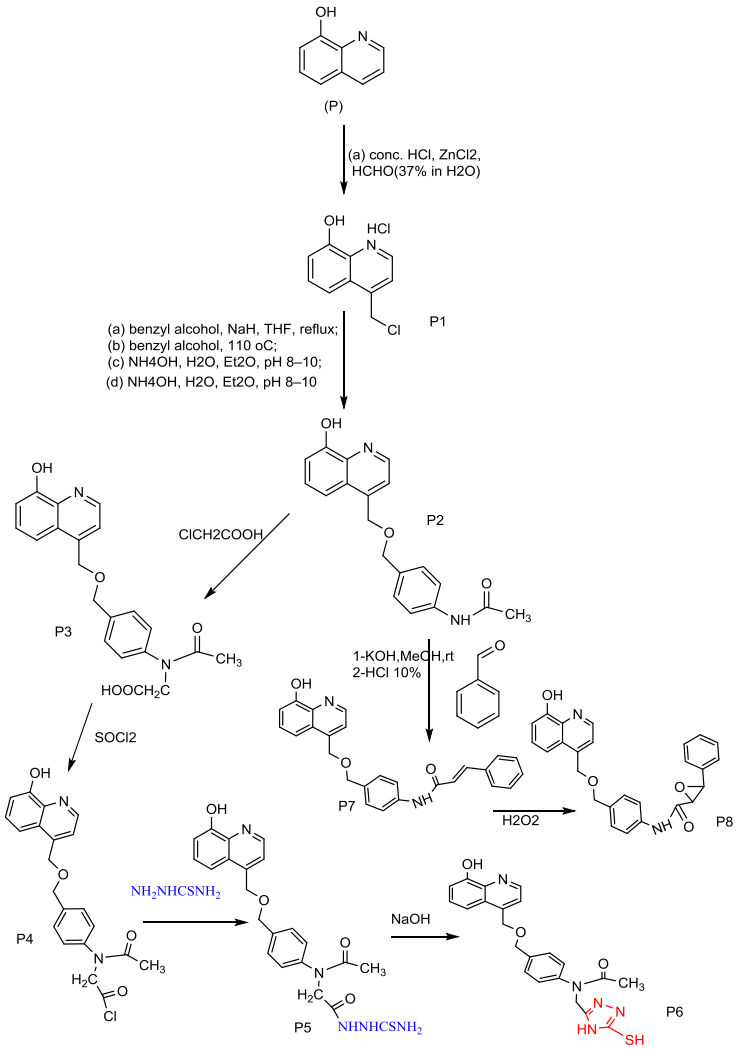
Compound P2 (1 mole) and benzaldehyde (1 mole) added in a round glass (50mL) with. Methanol (20 mL) was added with continuous stirring, and after dissolution completed, the concentrated base KOH (40%) (5 mL) added and the contents heated in a bath aqueous for 2 hours (40-45) °C and then kept in a cool place until the next day, the dark green chalcone salt. Mixture is acidified with concentrated hydrochloric acid (10%) (50 mL) to obtain a yellowish-orange precipitate. The precipitate was filtered and washed with cold water, dried and recrystallized with ethanol (Table1).

Synthesis of compound P8 [35]

Compound P7 (1 mole ,0.5 g) placed in a round glass (50 ml) dissolved in methanol (26.6 ml) with stirring and added hydrogen peroxide solution (30%)(9ml) with sodium hydroxide solution (16%) (15 mL), cooled with stirring for (5 hours) to give a dark green solution. The solution was acidified with hydrochloric acid (10%) to form chalcone epoxide, which has a dark orange color. The precipitate was filtered, washed with cold water, dried, and recrystallized from carbon tetrachloride solvent (Table1).

TABLE 1 Physical properties of derivatives (P1-P8)

No. of Component	Color	M. formula	M.Wt	M.P	Yield %	Rf	Solvent
P1	Yellow	$C_{10}H_8ClNO$	193	146-148	75	0.74	DCM/diethyl ether 1:1
P2	Yellow	$C_{19}H_{18}N_2O_3$	322	163-165	84	0.96	DCM/diethyl ether 1:1
P3	Brown	$C_{21}H_{20}N_2O_5$	380	140-142	82	0.81	n-hexane: DCM 1:1
P4	Reddish-brown	$C_{21}H_{19}ClN_2O_4$	398	Oily	75	0.65	n-hexane: DCM 1:1
P5	Off - white	$C_{22}H_{23}N_5O_4S$	453	166-168	91	0.60	Acetone :n-hexane 1:2
P6	Light brown	$C23H_{25}N_5O_3S$	451	104-106	77	0.68	Benzene: acetone 1:1
P7	Yellowish -orange	$C_{26}H_{22}N_2O_3$	410	151-152	89	0.74	chloroform /diethyl ether 1:2
P8	Dark orange	$C_{26}H_{22}N_2O_4$	426	75-76	84	0.72	chloroform : petroleum ether 1:3



SCHEME 1 Synthesis of compounds (P1-P8)

Biological activity

Antibacterial

The effect of antimicrobial properties of the prepared compounds compared to two types of bacteria were studied in agar medium at 37 °C for 24 hours, and it gave good results, (Table 2) [36].

Antioxidants

DPPH (four mg) was used by dissolving it in 100 mL of methanol. Various concentrations (25, 50 and 100) ppm were attended. It was dissolved in (10 mL) of methanol, 3 mL of the sample was taken and 1 mL of DPPH was added to it in a tube, left for 30 minutes. In the dark at 37 °C, the absorbance was measured using a spectrophotometer at 517 nm.

Results and discussion

Synthesis and identification of compounds (P1-P8)

The FT-IR spectra (Vmax, cm-1) of P1:
3439 (OH), 3068 (CHar), 2926 (CH str.), 1633 (C=N), 1525 (C=C), 1226-1398 (C-O, C-N). 1H-NMR (δppm): 2.5 (DMSO), 5.18 (s, 2H, CH_2), 10.88 (s, 1H, OH), 7.25 -8.38 (m, 5H, CHar).^{13}C-NMR (125 MH, δppm): 170.49, 175.4 (C=N), 158.2- 107.6 (C-ar), 68.7 (C-OH), 71.1(CH_2) and 40.4 (DMSO) (Figures 1 and 2).

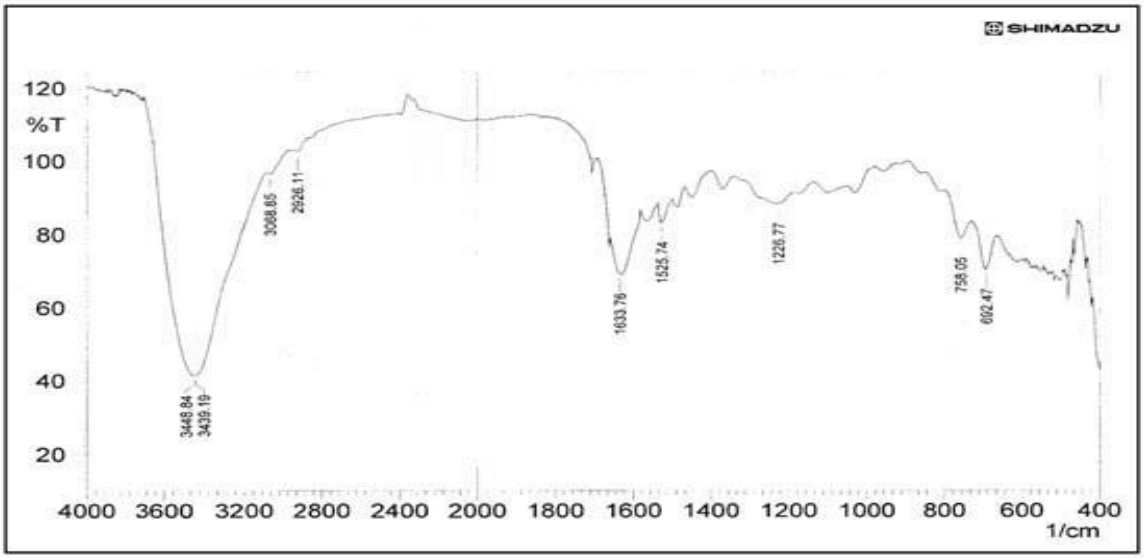


FIGURE 1 FT-IR for P1

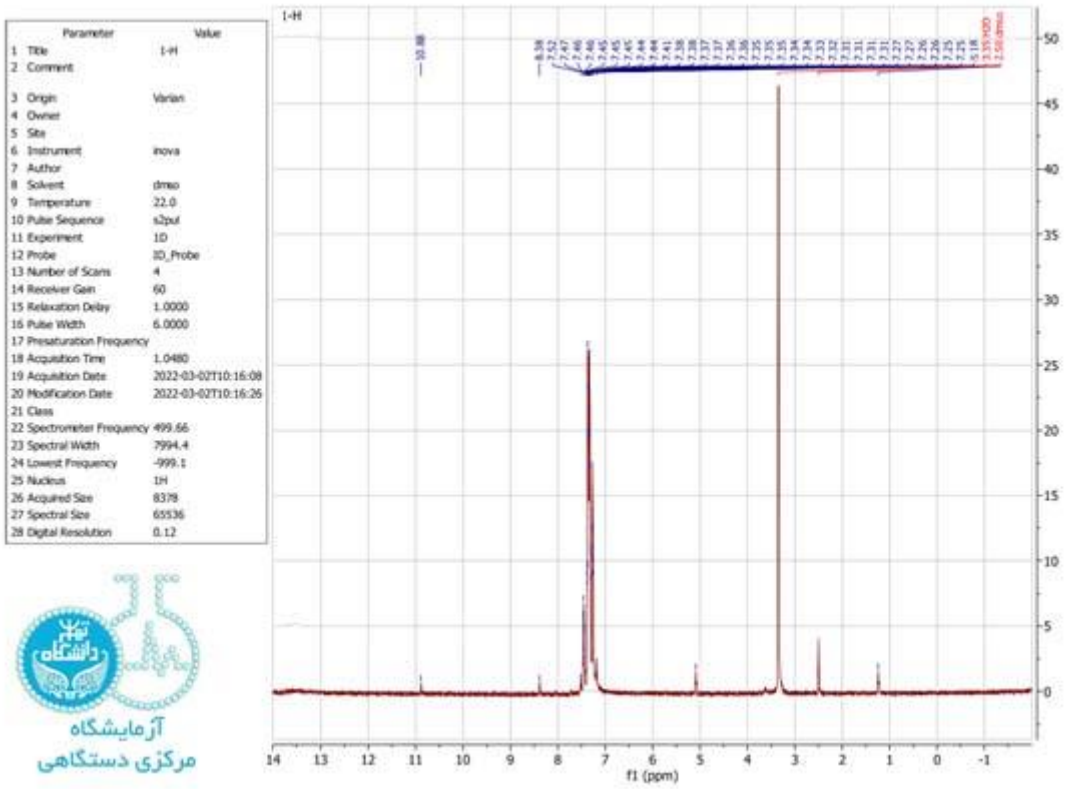


FIGURE 2 1HNMR for P1

The FT-IR spectra for P2: 3338 (OH), 3090 (CHar.), 2945(CH str.), 1716 (C=O amid), 1552 (C=C), 1612 (C=N), 1230-1309 (C-O, C-N). ^{1}H-NMR: 2.51 (DMSO), 3.24 (s, 3H, CH_3), 4.51-4.56 (s, 4H, CH_2), 10.05 (s,1H, OH), 7.19-7.57 (m, 9H, CHar), 10.10 (s, 1H, NH),^{13}C-NMR:159.1(C=O), 134.0-126.8 (C, Ar.), 142 (C-OH), 40.2 (DMSO), 82 (CH_2), 25.3 (CH_3). (Figures 3, 4 and 5).

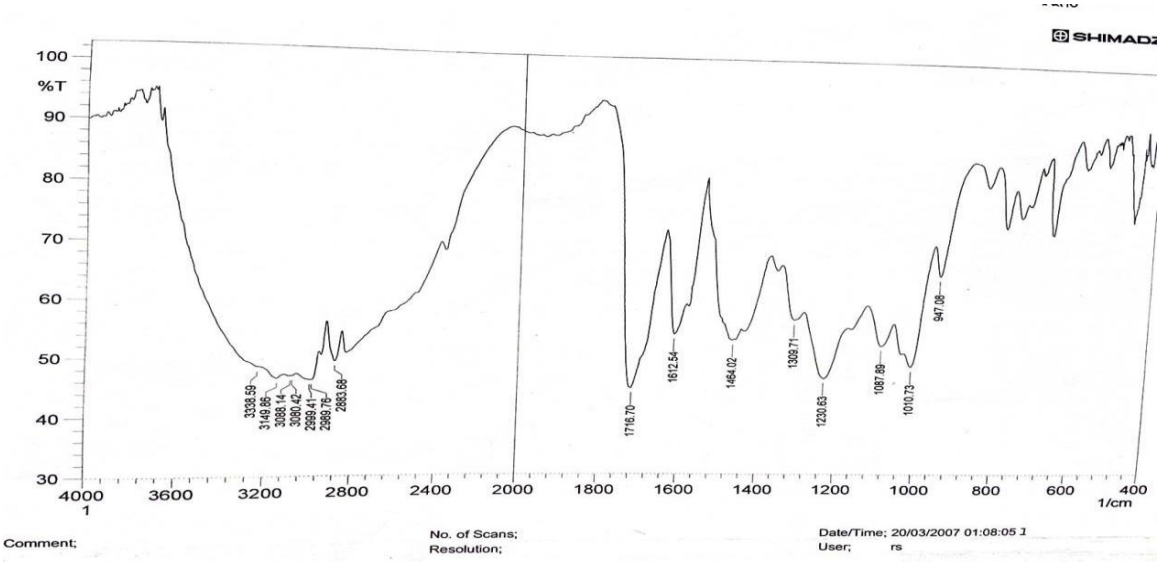


FIGURE 3 FT-IR for P2

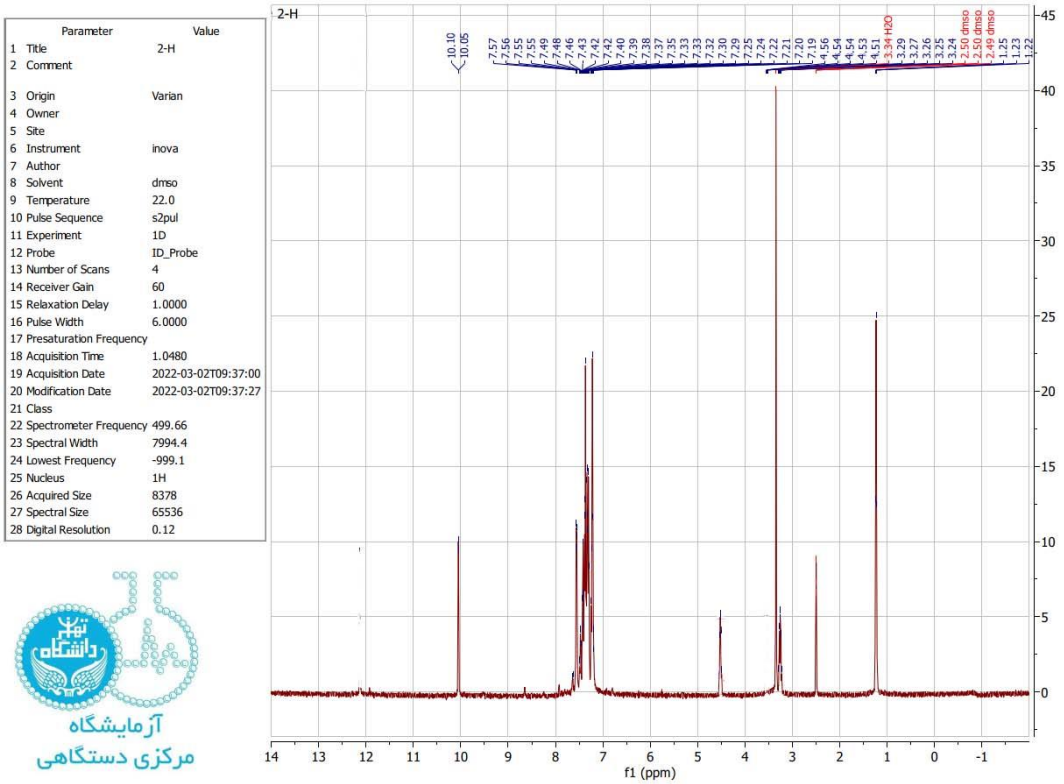


FIGURE 4 1HNMR for P2

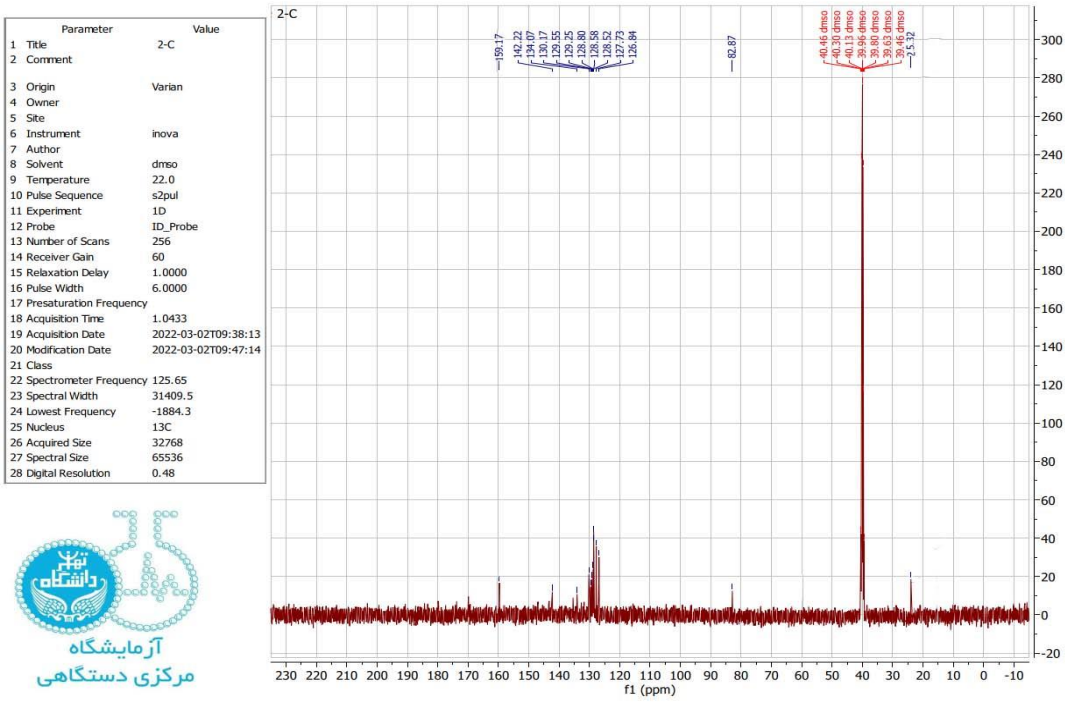


FIGURE 5 13CNMR of P2

The FT-IR spectra for P3: 262-3346 (COOH) 3051(CHar.), 2949 (CH str.), 1720 (C=O), 1606 (C=C),1658 (C=N) , 1213-1307 (C-N), 3315(OH). ^{1}H-NMR: 3.26 (s, 3H, CH_3),

2.51 (DMSO), 9.55 (s, 1H, OH), 4.55, 5.37 (s, 4H, CH_2), 12.13 (s, 1H, COOH), 7.19-7.57 (m, 9H, CHar.). ^{13}C-NMR: 134-126 (C-ar), 40.4(DMSO), 179, 172 (C=O), 84 (CH_2), 28 (CH_3), 159 (OH-C), 142 (C-N) (Figures 6, 7 and 8).

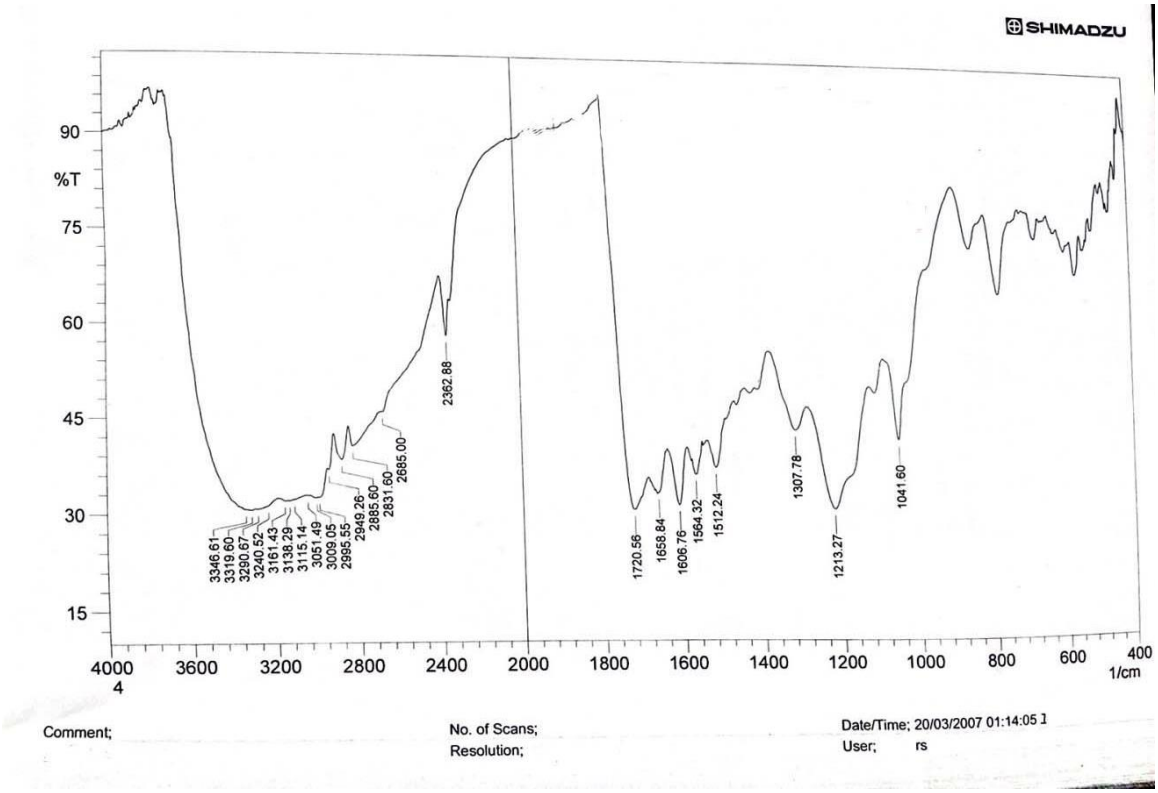


FIGURE 6 FT-IR for P3

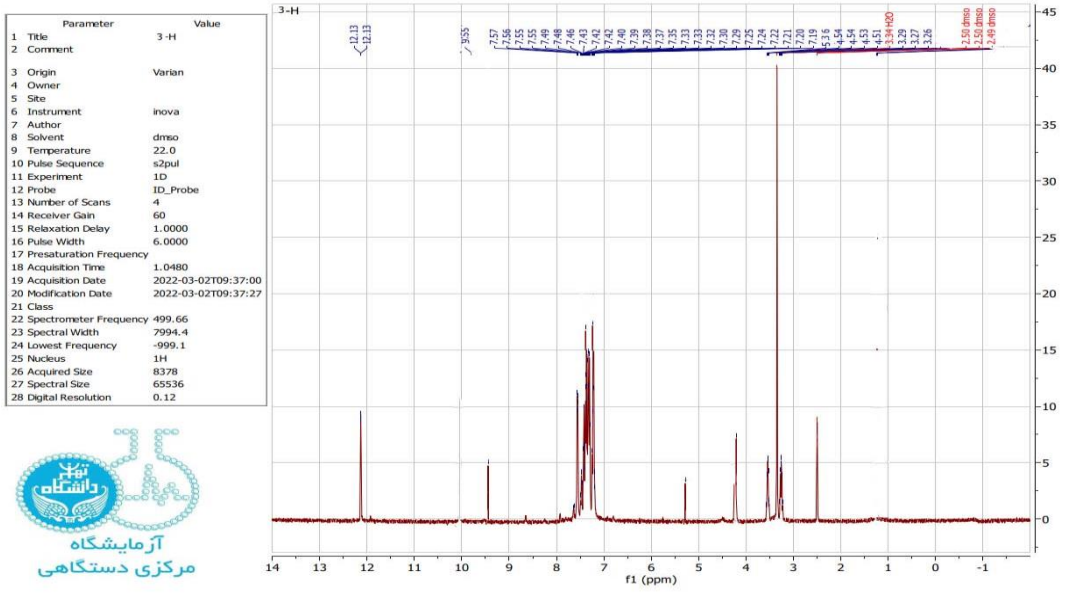


FIGURE 7 1HNMR for P3

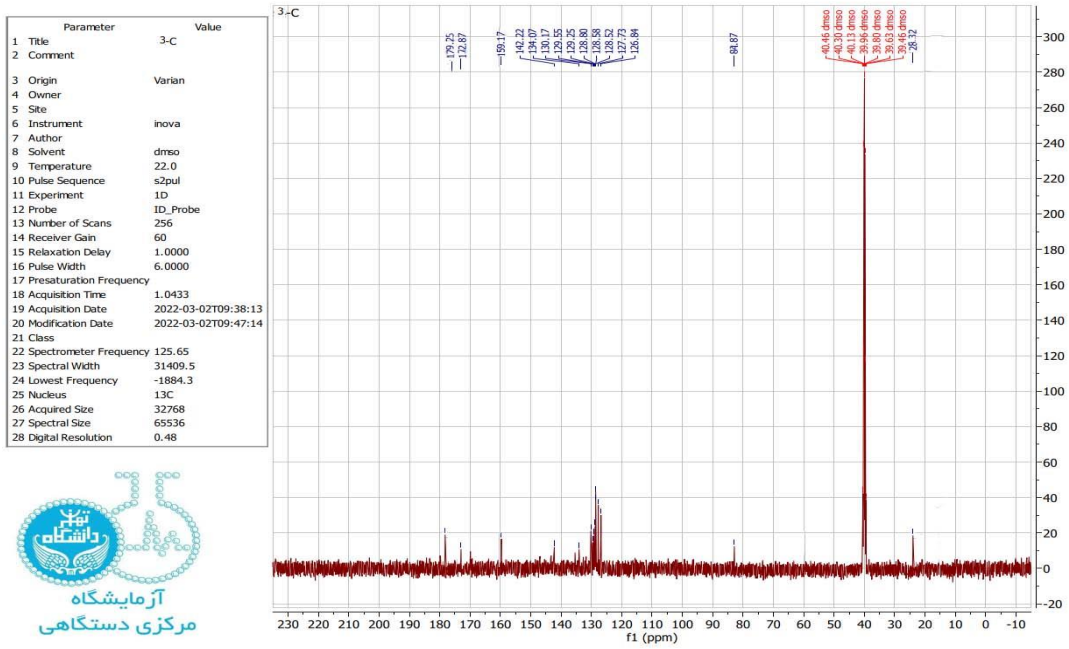


FIGURE 8 13CNMR for P3

The FT-IR spectra for P6:3416 (OH), 3296(NH), 2943 (CH str.), 1716 (C = O), 1661 (C=N), 1601 (C=C), 1209.4-1307.7(C-N). ^{1}H-NMR: 2.24 (s, 3H, CH_3), 2.49 (DMSO), 4.18, 4.25, 5.25 (CH_2), 9.58 (s, 1H, OH), 13.5(s, 1H, SH), 7.2-7.4 (m, 9H, CHar), 10.8 (s, 1H, NH). ^{13}C-NMR: 136.2-128.5 (C-ar), 40.4 (DMSO), 188.5 (C=O), 79.2 (CH_2), 29.2 (CH_3), 159 (OH-C), and 153 (C=N) (Figures 9, 10 and 11).

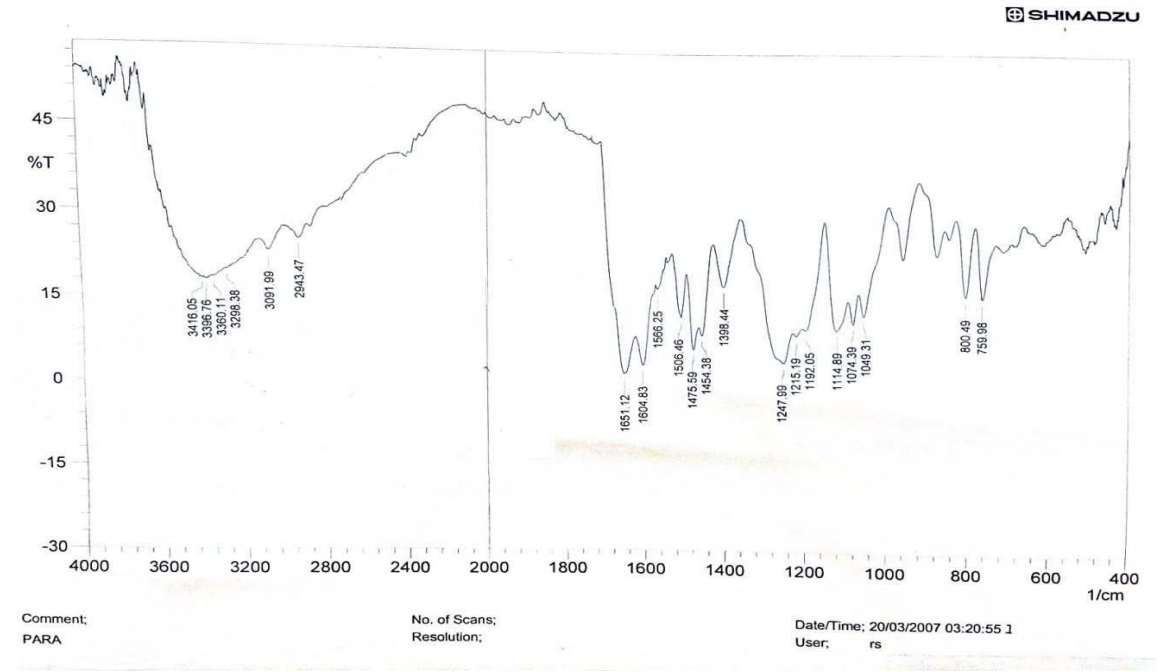


FIGURE 9 FT-IR for P6

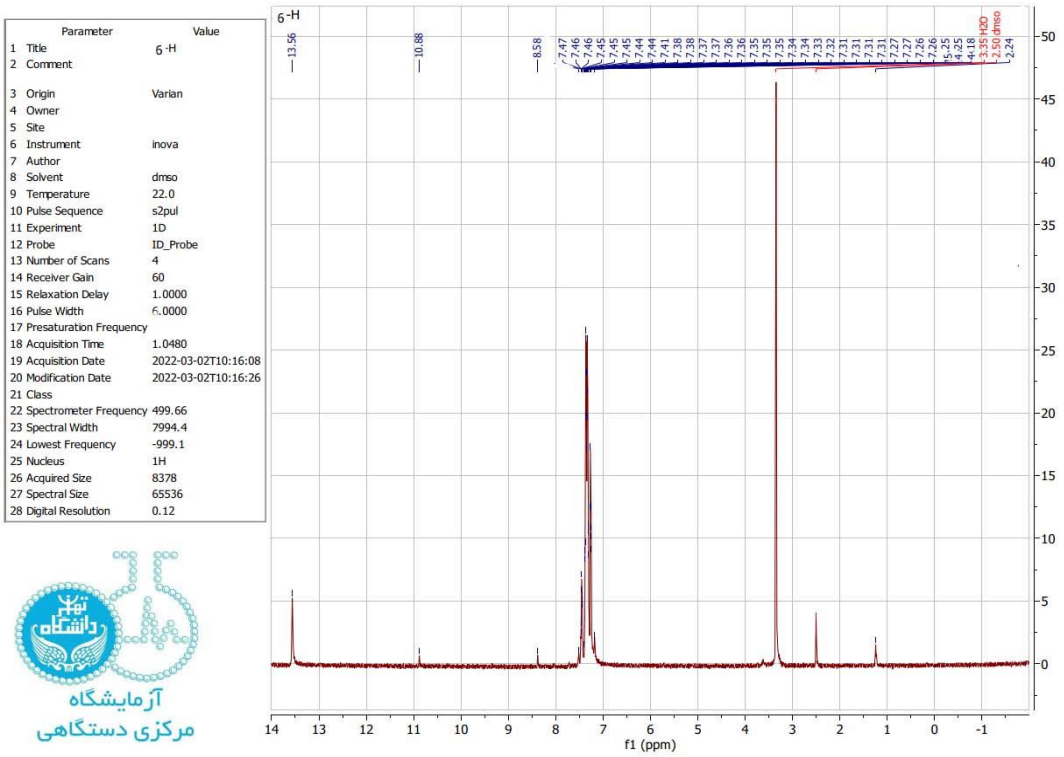


FIGURE 10 ^{1}H-NMR for P6

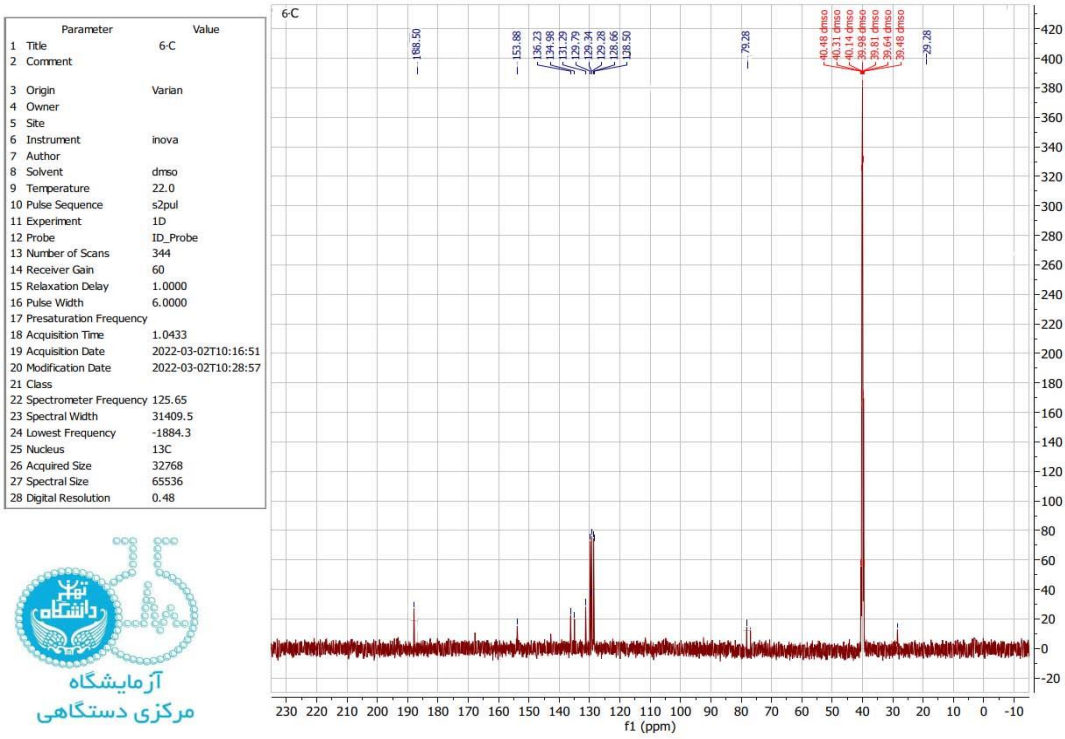


FIGURE 11 ^{13}C-NMR for P6

The FT-IR spectra for P7: 3346(OH), 3240 (NH), 2810 (CH str.), 2991(CH Ar.), 1699 (C=O), 1654 (C=N), 1608 (C=C), 1217-1387 (C-N). ^{1}H-NMR: 2.49 (DMSO), 5.45 (s, 2H,

CH_2), 8.0-8.9 (d, 2H, CH=CH), 9.09 (s, 1H, OH), 6.53-7.61 (m, 14H, CHar), 9.25 (s, 1H, NH). ^{13}C-NMR: 162.2 (C=O), 134-122 (Car), 152, 154 (C-O), 147 (C-N), 40.4 (DMSO), 84.09 (CH_2), 118 and 144 (CH=CH) (Figures 12, 13 and 14).

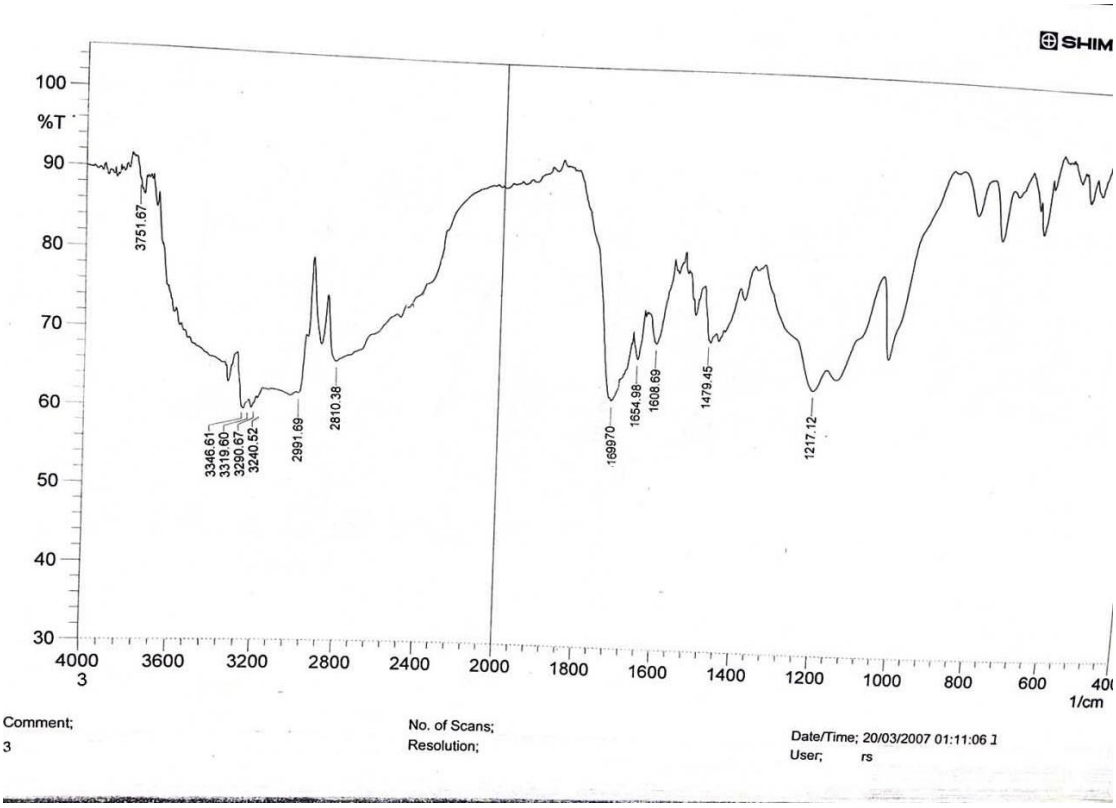


FIGURE 12 FTIR for P7

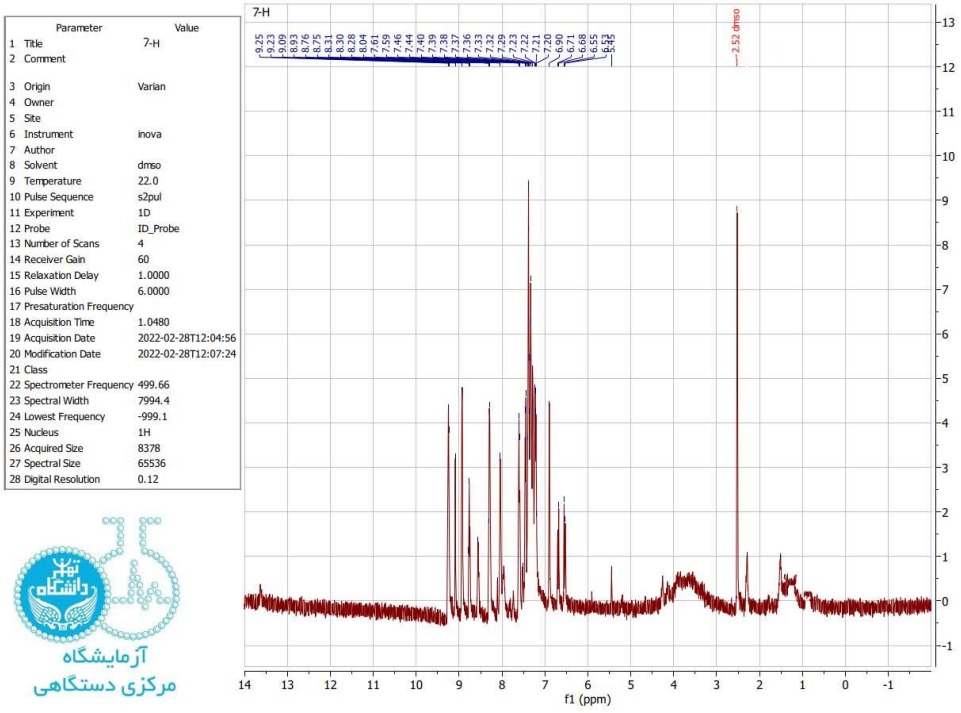


FIGURE 13 ^{1}H-NMR for P7

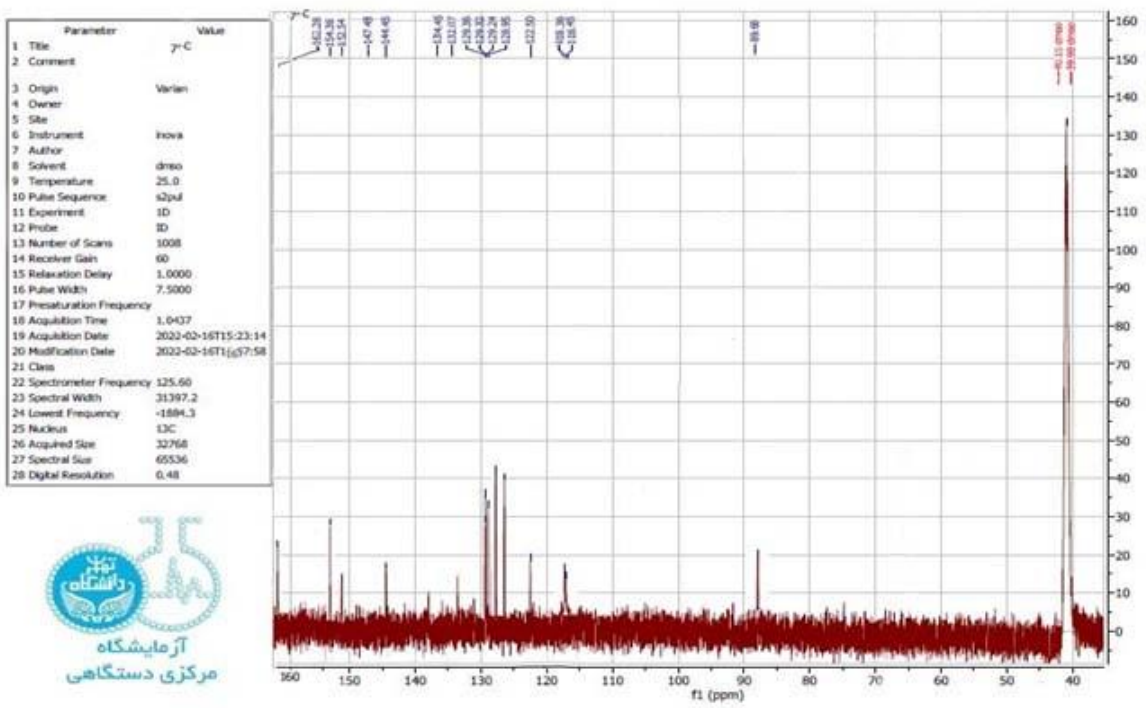


FIGURE 14 ^{13}C-NMR for P7

The FT-IR spectra for P8: 3421(OH), 3404 (NH), 3060 (CHar.), 2945 (CH str.), 1732 (C=O), 1651 (C=N), 1602 (C=Car), 1265-1394 (C-N). ^{1}H-NMR: 2.49 (DMSO), 4.1, 4.20 (d,2H,CHring), 4.50, 5.6 (s,4H,CH_2), 8.87 (s,1H,OH, 8.88 (s,1H,NH), 7.2-8.01(m,14H CHar), ^{13}C-NMR: 164.4 (C=O), 135.3-122.6 (Car), 154, 158 (C-O), 144 (C-N), 40.4 (DMSO), 81.4, 79 (CH_2), 61.12, and 68.4 (CH) (Figures 15, 16 and 17).

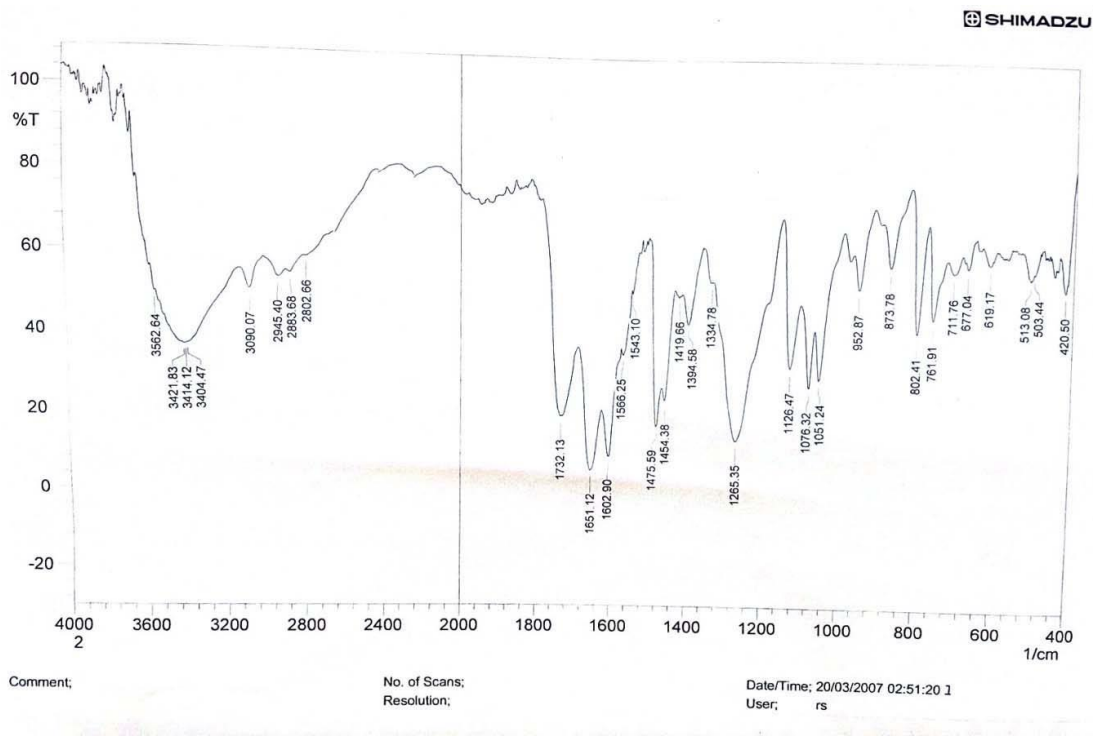


FIGURE 15 FT-IR for P8

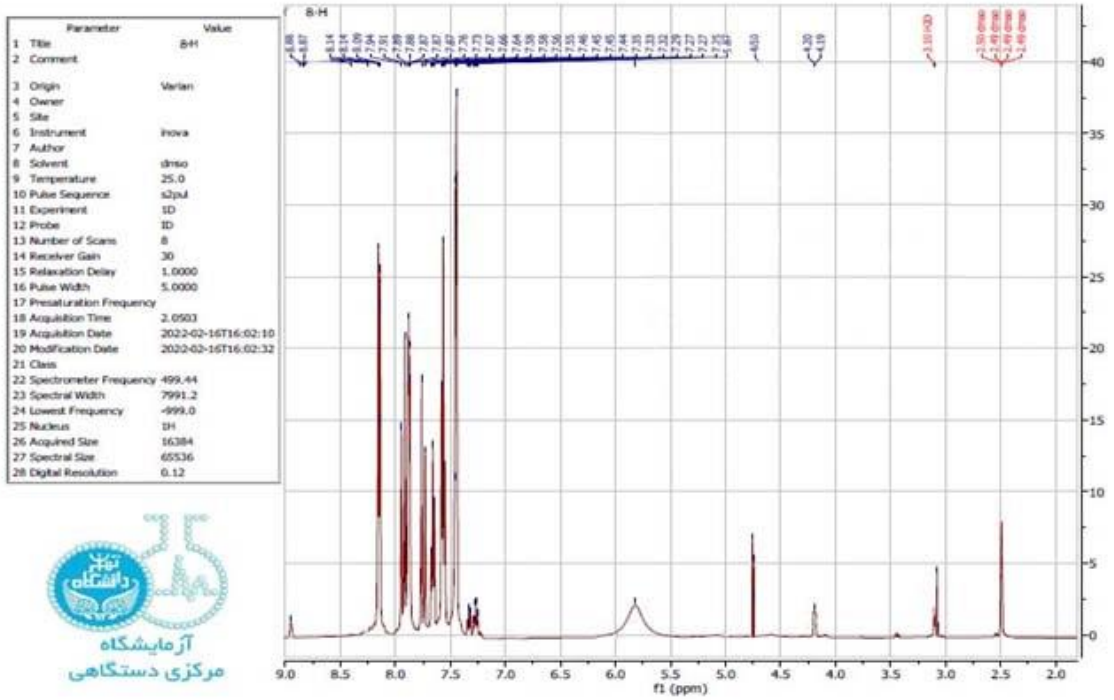


FIGURE 16 ^{1}H-NMR for P8

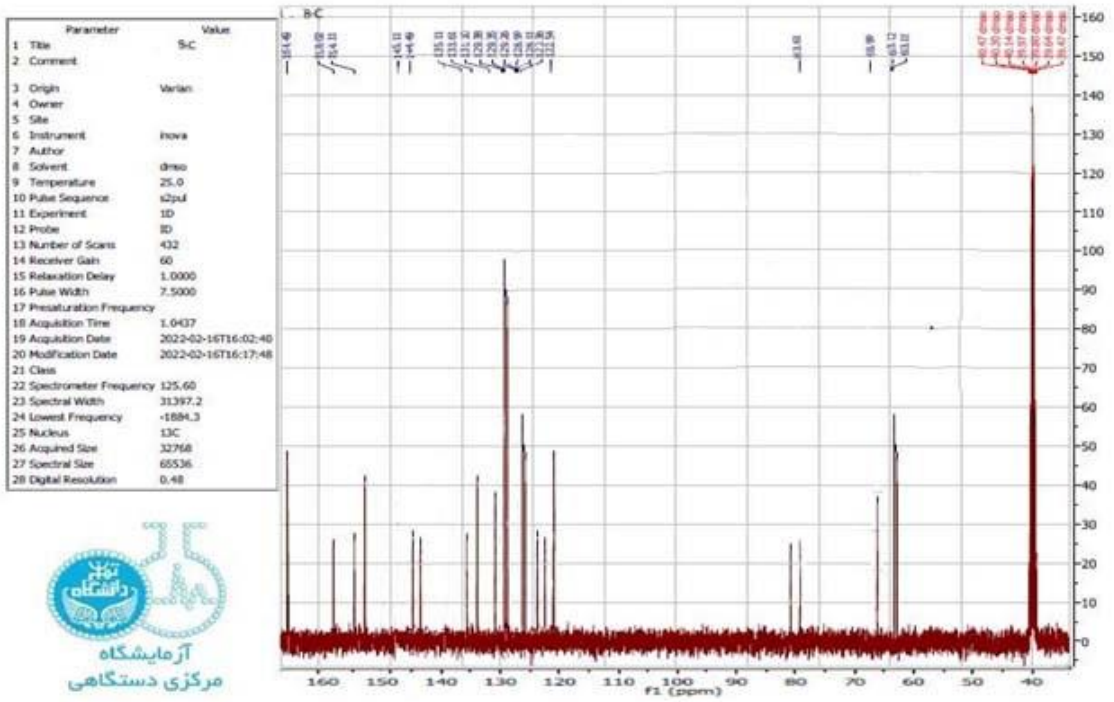


FIGURE 17 ^{13}C-NMR for P8

Biological activity

Anti-bacterial

The results of synthesis compounds show good antibacterial activity (Table 2). And importance in the clinical field to resistance of chemical drugs and few. Figures 18 and 19.

TABLE 2 Anti-bacterial activity for (P1-P8)

No. of Compound	Anti-bacterial activity test	
	klebsilla pneumonia (G-)	*Staphylcoccus aureus* (G+)
Entocid	11	18
P1	13	21
P2	9	11
P3	25	19
P4	16	22
P5	12	24
P6	22	21
P7	11	23
P8	13	20

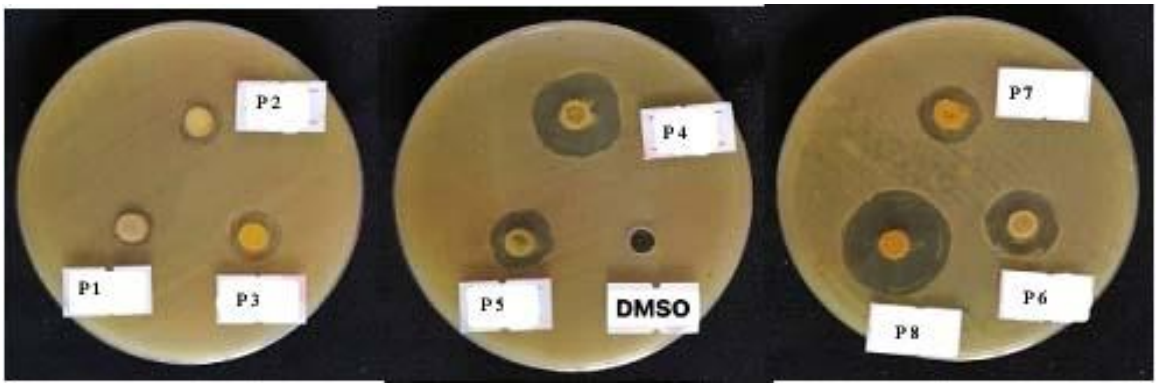


FIGURE 18 *Staphylococcus aureus* activity test

FIGURE 19 *Klebsiella pneumonia* activity test

Anti-oxidant activity

The DPPH method was used as demonstrated in Figure 20 and Table 3 compared with (ascorbic acid) (IC50 = 1.65 mg/ml), and the results were high to medium becouse OH group in compounds (P1-P8) as ordered compared with ascorbic acid: P8>P6>P7>P5>P4>P3>P2>P1.

TABLE 3 Anti-oxidants activity for (P1-P8)

Comp. No.	Inhibition %			IC50 mg/mL
	25 mg/mL	50mg/mL	100mg/mL	
P1	40.41	45.11	48.21	3.41
P2	47.75	49.02	54.16	3.18
P3	40.83	45.11	50.22	3
P4	38.04	43.15	52.12	2.79
P5	46.46	50.02	55.16	1.9
P6	44.76	45.83	49.99	1.1
P7	44.22	51.44	61.03	1.71
P8	48.06	52.56	56.91	0.25
Ascorbic acid(STD)	48.02	50.14	55.91	1.65

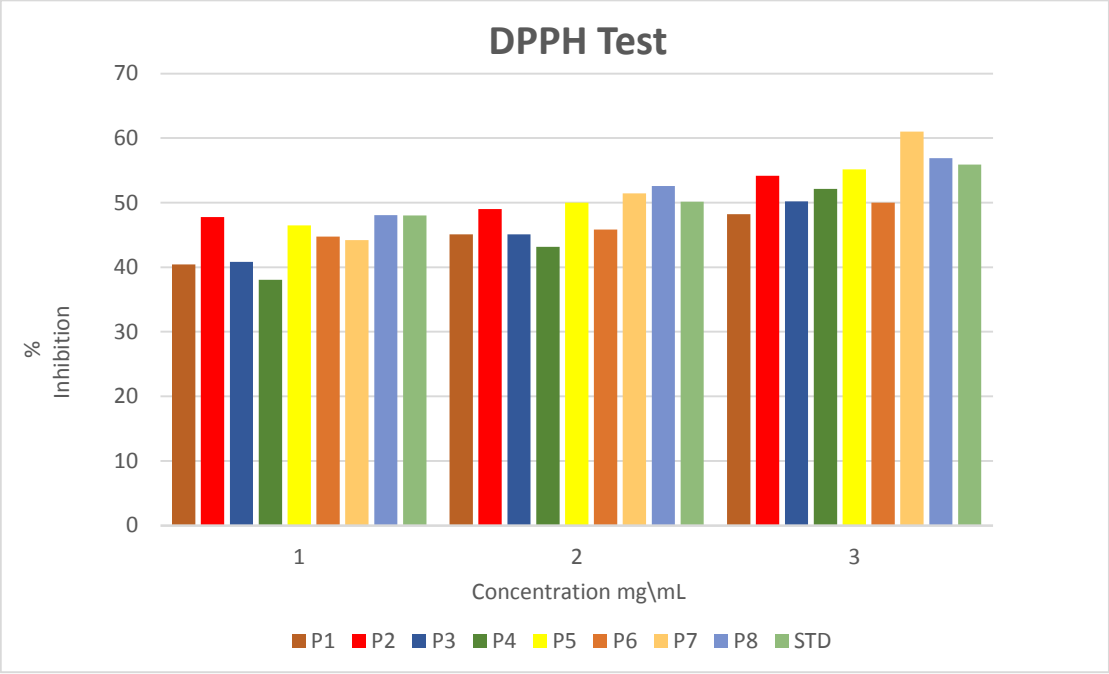


FIGURE 20 Standard DPPH method

Conclusion

This study of new preparation compounds (P1-P8) containing 8-HQ moieties of triazoles and oxirane their biological uses. The compounds were identified by 1HNMR, 13CNMR and FTIR. The compounds were found to have antioxidant potential and high efficacy to inhibit various types of bacteria. It was found that many of these compounds have good and high antioxidant activity compared the control.

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Conflict of Interest

The authors declare that there is no conflict of interest.

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