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Synthesis, Characterization of some new Heterocyclic Compounds Derived from Chalcones Containing Schiff Bases

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ABSTRACT

The present work involves the new chalcones containing Schiff bases, In this study, firstly, reaction of 3-acetylcoumarin with terephthaldehyde to prepared chalcones which contains a free aldehyde group which has been reacted with (4-Aminoantipyrine) to prepare Schiff bases, which was reacted (sodiumazide) as precursor to form (Tetrazole) followed by condensation of these chalcones with some compounds (hydroxylamine, aminophenol, phenylenediamine, hydrazine, semicarbazide, thiosemicarbazide, urea, thiourea and quanidine) as precursor to form Isoxazole, oxazepane, diazepine, pyrazoles and pyrimidine derivatives in basic medium using classical and ultrasonic technique. The comparison of the classical methods with ultrasonic methods. Was achieved. The compositions of the prepared compounds were confirmed by FT- IR and ^{1}H NMR spectroscopy.

Keywords: Tetrazole, Isoxazoles, Chalcones, 3-Acetylcoumarin, ultrasonic technique.

INTRODUCTION

Coumarin A heterocyclic compound formed by the fusion of a benzene ring with an α-pyrone ring, which is called (Benzo-α-Pyrone) (Jain and Joshi, 2012), One of the most important biological effects of coumarin is anti-tumor and cytotoxic (Peng *et al*., 2013), and after many studies and knowledge of the process mechanism through clinical chemistry studies, coumarin and its derivatives were recorded as important phytochemicals, mostly in higher plants to treat a variety of types of cancer (Al-shahwany and Omer, 2021). Chalcones are considered as flavonoid compounds which have therapeutic effects in a range of biological activities as anti-cancer (Elkanzi *et al*., 2022), anti-oxidant (Tiwari *et al*., 2021), and anti-inflammatory (Salehi *et al*., 2021). Schiff bases are prepared from the condensation of aldehydes and ketones with primary amines (R-NH2) and were discovered by the German scientist (Schiff), who first attended them in 1864 and named after him (Alfatlawi *et al*., 2022). The biological efficacy of Schiff bases has been evaluated and demonstrated efficacy, against many cancer diseases, anti-microbial (Venkateswarlu *et al*., 2022), anti-tuberculosis (Jameel and Sheat, 2013), anti-AIDS (Kar *et al*., 2022). most of the heterocyclic organic compounds have biological activity (Devi *et al*., 2022). Some tetrazole compounds have been used as anti-inflammatory and anti-viral agents (Myznikov *et al*., 2022), in addition to being used as herbicides, anti-fungal, and anti-bacterial. (Dalaf *et al*., 2022). Green chemistry It is a technique in chemical preparations that aims to reduce the emitted substances resulting from emissions in chemical manufacturing to the least possible extent (Quispe *et al*., 2022), as it introduces new techniques that can facilitate human life and activities without harming the environment (Chakole *et al*., 2022), as green chemistry includes the use of modern techniques and chemical methods that reduce substances polluting the environment or prohibiting the use of raw materials (Gangrade *et al*., 2015), solvents or catalytic auxiliary agents and materials that may disintegrate and which are hazardous to the environment and humans (Liu *et al*., 2021).

EXPERIMENTAL

Instrumentation

Melting points were measured by Electrothermal Gallen Kamp melting points and were uncorrected. Infrared (FT.IR.) spectra was recorded using (KBr) disk was recorded on Bruker FT.IR. spectrophotometer. ^{1}HNMR spectra were recorded using Inova 500 MHz by using DMSO-d_6 as a solvent, while using TMS as an internal reference in University of Kashan, Iran.

Preparation of (3-AcetylCoumarin)(1)

Compound (1) (3-acetylcoumarin) was prepared by reacting (0.01 mol) of salicylaldehyde with (0.01mol) of methyl acetoacetate with the addition of (1 ml) of piperidine. Piperidine with stirring in the ethanol solvent with continuous stirring for 15 minutes where a dark yellow precipitate is formed and washed with ethanol until the color turns and is recrystallized using hot Methanol , (Table 1) shows the physical data of the synthesized compound. (Al-shahwany and Omer, 2021)

Preparation 4-(3-oxo-3-(Coumarin-3-yl) prop-1-en-1-yl) benzaldehyde(2)

Chalcones are prepared by dissolving (0.01mol) of the 3-acetylcoumarin compound in (25 mL) of ethanol and then adding (0.01 mol) of piperidine while stirring for 15 minutes, then adding to the reaction mixture (0.01 mol) of terephthaldehyde dissolved in (10ml) of ethanol over Form batches with stirring at room temperature for 10 minutes and then the mixture rises for 6 hours. The mixture was cooled, then crushed ice is added to it where a solid precipitate is formed, filtered and washed with cold water until the solution is equalized, dried and recrystallized like (methanol) as in the equation below, The physical properties of the prepared compounds as shown in (Table 1). (Al-Hayali *et al*., 2018).

Preparation 1, 5-dimethyl-4-((-4(-3-oxo-3-(Coumarin-3-yl) prop-1-en-1-yl) benzylidene) amino)-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (3)

The compound was prepared by reacting (0.01 mol) of the compound (3) with (4-Aminoantipyrine), where the amine compound is dissolved in (10 ml) of ethanol and the aldehyde compound in (10 ml) from ethanol, when mixing the two materials, we notice the formation of a yellow crystalline precipitate immediately and it is recrystallized in hot Methanol (Deshmukh *et al*., 2021). Table 1 shows the physical properties for the prepared compounds.

Preparation 1, 5-dimethyl-4-(5-(4-(3-oxo-3-(coumarin-3-yl) prop-1-en-1-yl) phenyl)-4, 5- dihydro-1H-tetrazol-1-yl)-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (4)

The Schiff base (3) (0.01mole) was dissolved in (25ml) of (tetrahydrofuran [THF]) then sodiumazide (0.01 mol) was. added after it had been heated in a water bath for 7 hours at 60-70 degrees Celsius. The reaction mixture was crystalline precipitate immediately and it is recrystallized from Methanol, (Goshisht *et al*., 2021). The physical properties of the synthesized compound as shown in Table 1.

Preparation 4-(5-(4-(1-acetyl-3-(coumarin-3-yl)-4, 5-dihydro-1H-pyrazol-5-yl) phenyl)-4, 5-dihydro- 1H-tetrazol-1-yl)-1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (4b)

(0.001 mol, 0.474 g) of compound (4) is dissolved in (25 mL) ethanol, (0.004 mol) (hydrazine hydrate (80%) and 0.01 mol phenylhydrazine hydrate) is added and the reaction is ramped up for (12 hrs.). Cooled and poured into crushed ice, the resulting precipitate is separated by filtration, and recrystallized using methanol. (Table 1) gives the physical properties of the prepared compounds. (Ibarra *et al*., 2021) .

Preparation 1,5-dimethyl-4-(5-(4-(3-(coumarin-3-yl)-1-phenyl-4,5-dihydro-1H-pyrazol-5-yl) phenyl)-4,5-dihydro-1H-tetrazol-1-yl)-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (4c)

A mixture of the compound (4) (0.001 moles, 0.474 gm) in glacial acetic acid (25mL), (0.004 moles) hydrazine hydrate (80%). The mixture was refluxed for (12 hrs.). The cooled and poured into crushed ice water, the product was filtered then dried, recrystallized from ethanol, the physical properties of the prepared compounds as shown in (Table 1). (Abdel-Latif, 2005).

Preparation 5-(4-(1-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-4,5-dihydro-1H-tetrazol-5-yl) phenyl)-3-(coumarin-3-yl)-4,5-dihydro-1H-pyrazole-1-carboxamide (5yl)-4,5-dihydro-1H-tetrazol-5-yl) phenyl)-3-(coumarin-3-yl)-4,5-dihydro-1H-pyrazole-1-carbothioamide (4e)

A mixture of the Compound (4) (0.001 mol) in (10 mL) ethanol, and (0.001 mol) (semicarbazide, thiosemicarbazide) dissolved in ethanol (10 mL), 10 mL of 10 % KOH was added dropwise. The contents were reflux for 9 hrs. The reaction mixture was then poured into ice cold- water, to give the solid product, filtered then recrystallized from ethanol/water, (Table 1) shows the physical properties of the prepared compound (Abdel-Latif, 2005).

Preparation 1,5-dimethyl-4-(5-(4-(3-(coumarin-3-yl)-4,5-dihydroisoxazol-5-yl) phenyl)-4,5-dihydro-1H-tetrazol-1-yl)-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (4f)

To a mixture of the Compound (4) (0.001 mol), dissolved in (10 mL) of ethanol, and (0.002mol) hydroxylamine hydrochloride dissolved in ethanol (10 mL), 10 mL of (10 % NaOH) was added dropwise. The reaction mixture was refluxed for (8 hrs.). The mixture was then poured into ice- water, the product was filtrate, recrystallized from methanol, the physical properties of the prepared compound illustrated in (Table 1) (Abdel-Latif, 2005).

Preparation 1,5-dimethyl-4-(5-(4-(4-(coumarin-3-yl)-2,3-dihydro-1H-benzo[b] [1,4] diazepin-2-yl) phenyl)-4,5-dihydro -1H-tetrazol-1-yl)-2 -phenyl-1,2-dihydro-3H-pyrazol-3-one (4g)

Preparation 1,5-dimethyl-4-(5-(4-(4-(coumarin-3-yl)-2,3-dihydrobenzo[b] [1,4] oxazepin-2-yl) phenyl)-4,5-dihydro-1H-tetrazol-1-yl)-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (4h)

A mixture of the compound 4 (0.001 mol) with (0.001mol) (2-aminophenol and ortho phenylenediamine) dissolved in ethanol (25 mL) and few drops of glacial acetic acid. The mixture was refluxed for (10 hrs.). The mixture was then poured into crushed-ice to give the solid product, filtered, recrystallized from ethanol/water (Abdel-Latif, 2005). (Table 1) shows the physical properties of the prepared compound.

Preparation 6-(4-(1-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-4,5-dihydro-1H-tetrazol-5-yl) phenyl)-4-(coumarin-3-yl) pyrimidin-2(1H)-one (4i)

Preparation 1,5-dimethyl-4-(5-(4-(6-(coumarin-3-yl)-2-thioxo-2,3-dihydropyrimidin-4-yl) phenyl)-4,5-dihydro-1H-tetrazol-1-yl)-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (4j)

A mixture of the compound 4 (0.001 mol), and urea and thiourea (0.001 mol) were dissolved in ethanol (0.002 mol) piperidine were refluxed for (12 hrs.), cool and poured into an ice-cold water, the formed product was recrystallized from methanol; (Table 1) shows the physical data of the prepared compound. (Abdel-Latif, 2005).

Preparation 4-(5-(4-(2-amino-6-(coumarin-3-yl)-4,5-dihydropyrimidin-4-yl) phenyl)-4,5-dihydro-1H-tetrazol-1-yl)-1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (4k)

A mixture of the compound 4 (0.001 mol), and guanidine nitrate (0.001 mol) were dissolved in ethanol (0.001moles) sodium ethoxide were added. Then refluxed for (12 hrs.). Cooled then poured into ice-cold water, filtrated and recrystallized from ethanol, (Table 1) shows the physical properties of the prepared compound. (Abdel-Latif, 2005).

Green methods (ultrasonic technique)

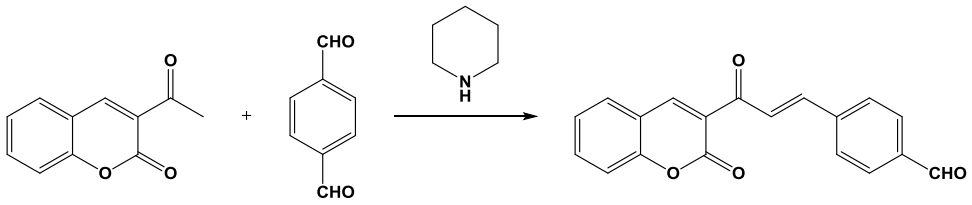
The above compounds were prepared using ultrasonic technology and zirconium chloride octahedrate was used as catalyst and irradiated using ultrasonic technology in a water bath at 45-65°C for 40 minutes to give the products shown in (Table 1) (Safai-Ghoumi, 2005).

Table 1: The physical data for the prepared compound

Compound No.	M.P. (°C)	Colour	Ultrasonic (min.)	Classic method yield%	Ultrasonic yield%
1	120-122	light Yellow	5	85	80
2	150-152	Yellow	1	85	90
3	167-169	Orange	30	75	80
4	185-187	Brown	60	60	64
4a	200-203	Red	60	60	70
4b	210-112	Yellow red	50	68	71
4c	232-235	Green	60	50	55
4d	225-228	Deep orange	45	65	73
4e	240-242	Brown	50	62	65
4f	265-268	Deep yellow	60	79	80
4g	266-270	Yellow	60	54	62
4h	270-272	Deep brown	60	60	60
4i	240-242	Brown	60	60	68
4j	250-252	Yellow	60	50	55
4k	256-259	Green	60	65	65

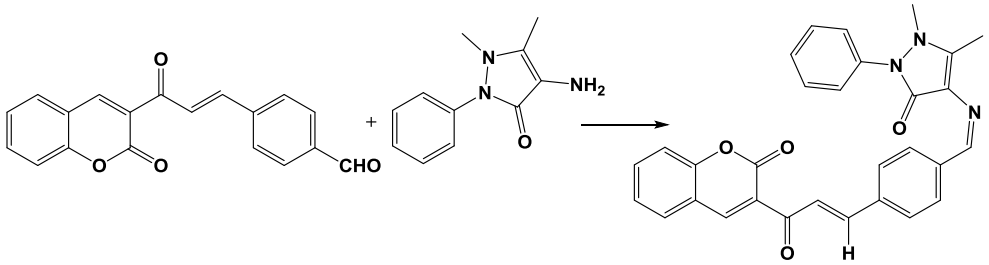
RESULTS AND DISCUSSION

The key of this work is the chalcone intermediate was obtained by Clasian –Schmidt condensation of corresponding 3-acetylcoumarin (1) with aldehyde (terephthaldehyde), in basic condition by addition of piperidine, the structural formula of the compounds (2) was established by physical and spectral data of FT.IR which show two band position of carbonyl groups at (1650 cm^{-1} and at 1750 cm^{-1}), as shown in scheme 1. The prepared chalcone was identified by studying the spectral properties of the compounds (I.R, 1H-NMR) by infrared spectrum and nuclear magnetic resonance spectrum. The infrared spectra of these compounds showed the appearance of absorption bands in the frequency range (1700 cm^{-1}). It goes back to the stretching frequency of the carbonyl group (C = O), where the stretching frequency of the carbonyl group in chalcones is less than its natural frequency due to the resonance of the double bond, and it also gave absorption bands in the range of frequencies (υ 1508-1585 cm^{-1}) that go back to the frequency of Stretch the double-joint (C=C).



Scheme 1: The synthetic route of compound (2)

The reaction of chalcone (2) with one moles 4-aminoantipyrine and gave Schiff bases compound (3), the scheme (2) showed these reactions, Schiff bases were diagnosed by studying the spectral properties of compounds (FT- IR and ^1H NMR spectroscopy). The infrared spectra of these compounds showed the appearance of absorption bands in the frequency range (1650 υ cm-1) dating to the stretching frequency of the Schiff base group (C=N), also showed in situ absorption bands (1210-1280 υ cm-1) due to the band frequency (C-N).



Scheme 2: the synthetic route of compound (3)

The reaction of compound 3 with one mole sodium azide gave Tetrazol compound (4), the scheme (3) showed these reactions, Schiff bases were diagnosed by studying the spectral properties of compounds (FT- IR and ^1H NMR spectroscopy). The infrared spectra of these compounds showed the appearance of absorption bands in the frequency range (1410-1460 υ cm-1). It refers to the stretching frequency of the Schiff bases group (N=N), and the in-situ absorption bands (1210-1280 υ cm-1) are due to the band stretching frequency (C-N). It also showed in situ absorption bands (3250-3350 υ cm-1) due to the band stretching frequency (H-N),

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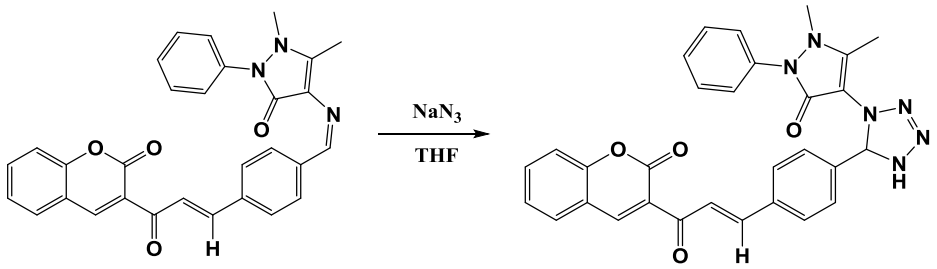
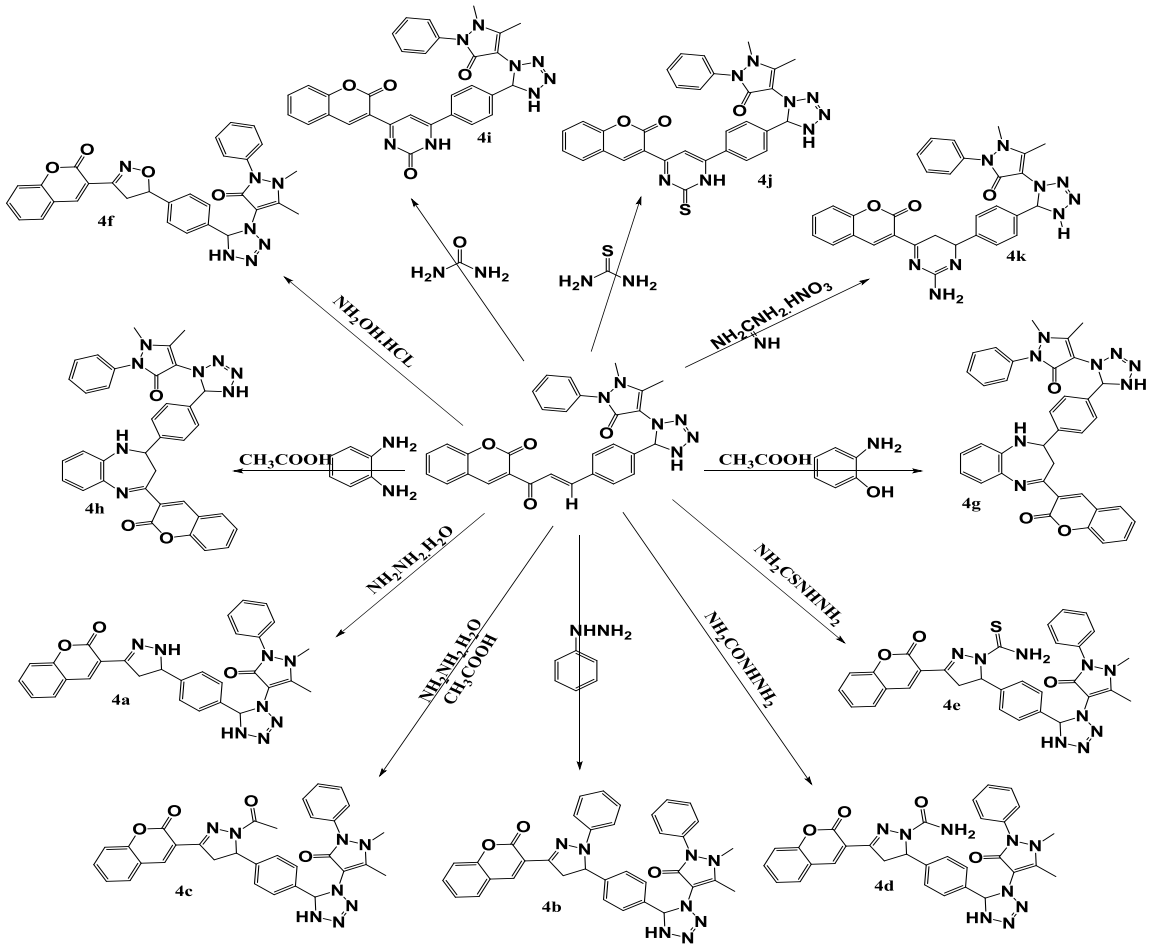


Scheme 3: the synthetic route of compound 4.

The reaction of compound 4 with one mole hydroxylamine, aminophenol, phenylenediamine, hydrazine, semicarbazide, thiosemicarbazide, urea, thiourea and quinidine to gave Isoxazole, oxazepane, diazepine, pyrazoles and pyrimidine derivatives (4a- 4k), the scheme (4) showed these reactions, These prepared compounds [4a-4k] were diagnosed by studying the spectral properties of the compounds (FT- IR and ^{1}H NMR spectroscopy), as they showed the infrared spectra of these compounds as shown in the (Table 2).

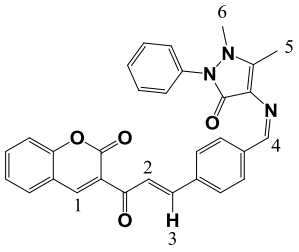
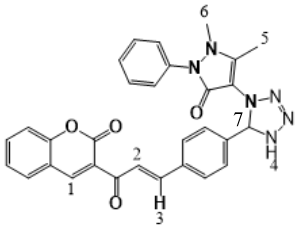
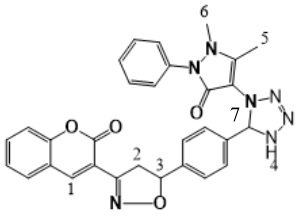
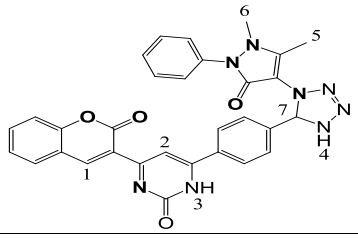


Scheme 4: The synthetic route of compound (4a, 4b, 4c, 4d, 4e, 4f, 4g, 4h, 4i, 4j, 4k).

Table 2: I.R. spectrum of synthesized compounds.

Comp. No.	**IR υ(cm-1), KBr**						
	N-H	**C-H (Ar.)**	**C-H (Aliph.) Sym. Asym**	**C=O Cyclic Ester**	**C=O**	**C=N (Schiff bases) Cyclic**	**C-O-C Ether Sym. Asym**
1	-----	3090	2955 2950	1745	1700	-----	1205 1150
2	-----	3069	2924 2862	1743	1630	-----	1240 1170
3	-----	3070	2964 2840	1740	−−−−−	1650	1205 1185
4	3350	3054	2920 2830	1741	-----	-----	1209 1190
4a	3380	3097	2935 2890	1730	-----	1640 1637	1201 1156
4b	3390 3364	3071	2901 2834	1741	1687	1630	1208 1193
4c	3319	3069	2900 2860	1730	1700	1630	1209 1183
4d	3369 3259	3040	2922 2869	1730	-----	1630	1199 1165
4e	3240 3443	3080	2970 2856	1721	1694	1630	1204 1168
4f	3390	3090	2939 2870	1745	−−−−−	1604	1190 1149
4g	3389	3074	2954 2845	1709	1663	1605	1200 1184
4h	3354	3089	2930 2841	1741	------	1627	1208 1182
4i	3340	3065	2947 2837	1729	1670	1620	1197 1183
4j	3300	3059	2983 2914	1703	1616	1600	1226 1120
4k	3400 3350	3089	2850 2812	1742	−−−−−	1615	1201 1178

Table 3: The ^{1}HNMR spectrum of synthesized compounds.

Comp. No.	Structure	^{1}HNMR, DMSO-d6, δ (ppm)
3		6.9 (1H, s, H1), 6.34 (1H, d, H2), 7.67(1H, d, H3),8.60 (1H, s H4),-7.34 7.95(H, m, Ar-H), 3.18-2.30 (H, S, H5, H6), 7.4(7.71-8H, m, Benzylic ring)
4		8.55 (1H, s, H1) 6.34 (1H, d, H2) 7.34(1H, d, H2)7.66 (1H, s, H3) 3.90 (1H, d, H4)7.33-7.63 (8H, m, Ar-H) 2.20-3.01(6H, S, H5, H6) 7.71-7.48(4H, m, Benzylic ring)
4f		8.33 (1H, s, H1) 3.02-3.14 (2H, d, H2) 5.66(1H, t, 3H) 5.21 (1H, s, H4) 2.30-3.01(6H, m, 5H,6H) 7.31-7.50(8H, m, Ar-H) 7.48-7.61(4H, m, Benzylic ring) 6.67(2H, d, H7)
4i		8.59 (1H, s, H1) 7.34 (2H, s, H2) 5.66 (1H, t, H2) 11.29 (1H, s, H3) 9.84(1H, d, H4) 2.30-3.01(6H, m, 5H,6H) 7.31-7.50(8H, m, Ar-H) 7.48-7.61(4H, m, Benzylic ring) 6.67(2H, d, H7)

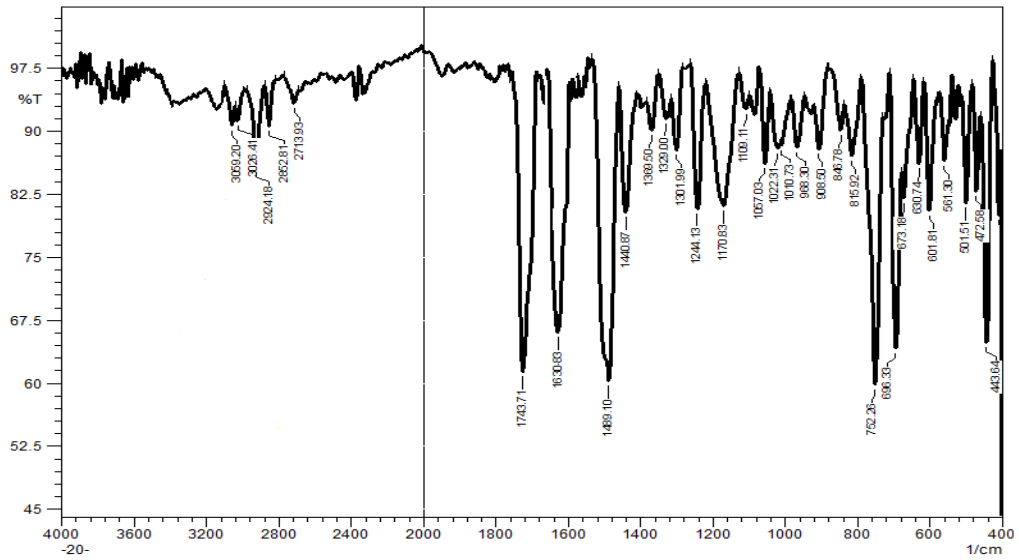


Fig. 1: The infrared (I.R) spectrum of the compound (2).

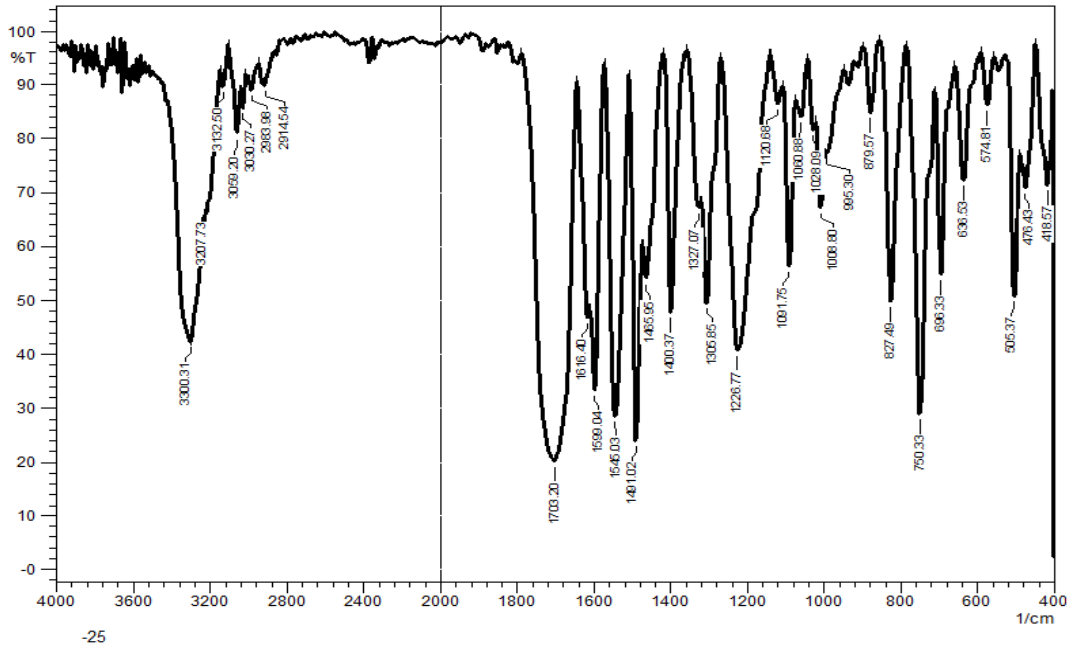


Fig. 2: The infrared (I.R) spectrum of the compound (4j).

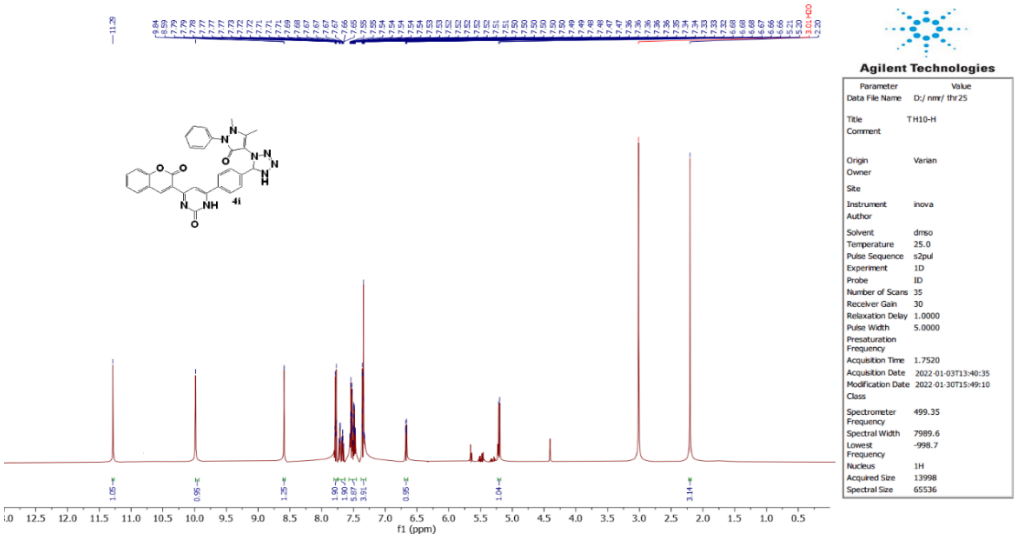


Fig. 3: The nuclear magnetic resonance (1H-NMR) spectrum of the compound (4i)

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تحضير وتشخيص بعض المركبات الحلقية غير المتجانسة الجديدة المشتقة من الجالكونات المحتوية على قواعد شف

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الملخص

يتضمن العمل الحالي تحضير الجالكونات التي تحتوي على قواعد شف، في هذه الدراسة أولاً تم مفاعلة (3–اسيتايل كومارين) مع (تريفثالدهيد) لتحضير الجالكون الذي يحتوي على مجموعة الألديهيد الحرة التي تم مفاعلتها مع (4–أمينو أنتيبيرين) لتحضير قواعد شيف والتي فوعلت مع (صوديوم أزيد) لتحضير (تيترازول)، وتم مفاعلة الجالكونات مع بعض المركبات (هيدروكسيل امين هيدروكاوريد وامنيو فينول وفنيلين ثنائي الأمين وهيدرازين وسيميكاربازيد وثيوسيميكاربازيد ويوريا وثيوريا وكوانيدين) لتحضير المركبات الحلقية غير المتجانسة اوكسازول واوكسازبين ودايازبين وبايرزول ومشتقات البيريميدين باستخدام الطريقة التقليدية وطريقة الأمواج فوق الصوتية وتم مقارنة النتائج بين الطريقة التقليدية والامواج الفوق الصوتية، تم تأكيد تراكيب المركبات المحضرة باستخدام التحليل الطيفي FT-IR و H NMR1.

الكلمات الدالة: تيترازول، اوكسازول، جالكونات، 3–اسيتايل كومارين، الأمواج الفوق الصوتية