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Synthesis and Characterization of New 2-Quinolone Sulfonamide Derivatives

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Abstract:

A series of new 2-quinolone derivatives linked to benzene sulphonyl moieties were performed by many steps: the first step involved preparation of different coumarins (A1,A2) by condensation of different substituted phenols with ethyl acetoacetate. The compound A1 was treated with nitric acid to afford two isomers of nitrocoumarin derivatives (A3) and (A4). The prepared compounds (A2, A3) were treated with hydrazine hydrate to synthesize different 2-quinolone compounds (A5,A6) while the coumarin treated with different amines gave compounds (A7,A8). Then the synthesized 2-quinolone compounds (A5-A8) treated with benzene sulphonyl chloride to afford new sulfonamide derivatives (A9-A12). The synthesized compounds were characterized by FT-IR, ^{1}H-NMR, ^{13}C-NMR spectra and by measurement some of their physical properties.

Key words: 2-quinolones, coumarin, sulfonamide.

Introduction:

Coumarins are the family of lactones [1]. Structurally coumarin is a fused benzene ring with pyron ring while quinolones are molecules structurally derived from the heterocyclic aromatic compound (quinolone), it is classified as a number of the benzopyridone family compounds, all of which compose of a fused benzene moiety with pyridone ring [2]. Natural product of 1-methyl-2-quinolone derivatives have a wide range of biological activities such as antiparasitic [3], antitumor [4], antianemia activity [5], and cytotoxic activities [6], so that lately begun attention has been paid to unnatural 1-methyl-2-quinolone derivatives. Indeed, more than 12,000 compounds having the 1-methyl-2-quinolone framework as a partial structure have been reported. Heterocycles containing sulfonamide moieties have attracted obvious attention due to their significant biological properties and their role as pharmacophores[7–11].

After sulfanilamide discovery, thousands of chemical variations were studied and the best therapeutic results were obtained from the compounds in

which one hydrogen atom of the SO_2NH_2 group was replaced by heterocyclic ring [12]. Therefore, in this work we were so interesting to combine the two of 2-quinolones, and sulfonamide groups together in one molecule

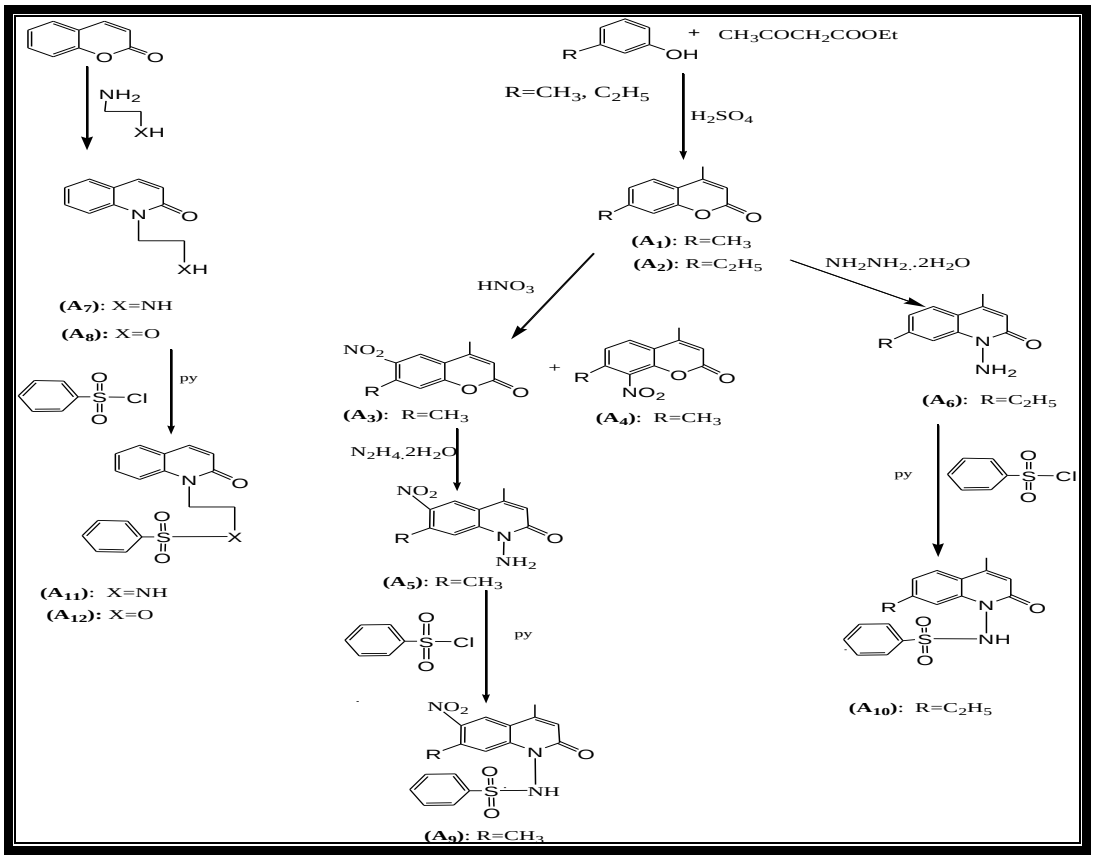
Materials and Methods:

Instruments:

The FT-IR spectra in the range (4000–400) cm^{-1} were recorded on a Shimadzu FT-IR 8300 Spectrophotometer. ^{1}H-NMR and ^{13}C-NMR spectra (solvent DMSO) were recorded on a Bruker-DPX 400 MHz spectrometer with TMS as internal standard at Isfahan University. Melting points were determined on a Gallen-kamp MFB-600 melting point apparatus and are uncorrected. Analytical thin layer chromatography (TLC) in (7:3 ratio of hexane: ethyl acetate) as the mobile phase was performed on plates precoated with silica gel (Merck 60 F254, 0.25 mm) and was visualized with ultraviolet light.

Chemicals:

Starting chemical compounds were obtained from BDH, Sigma Aldrich and Flukaand were used as received without further purification.



Scheme : Synthesis of new 2-quinolone derivatives

Synthesis of 4, 7-dimethyl coumarin and 4 –methyl-7-ethy coumarin (A1,A2)[13]

A mixture of *m*-cresol / *m*-ethyl phenol (0.1 mol) and ethyl acetoacetate (0.1 mol, 12.634ml) was cooled down below (0-4)^{o}C , conc. sulphuric acid 46ml was added drop wise in such away that the temperature does not rise above 8 ^{O}C. After that, the stirring was continued for one hour at room temperature. Then the mixture was heated at (60-70 ^{O}C) for 6 hrs, after cooling, the solution was poured into ice /cold water. The solid product was filtered and washed with cold water, dried at room temperature,

then recrystallized from a suitable solvent. The physical properties are listed in Table (1).

Synthesis of 4,7- dimethyl -6-nitrocoumarin and 4,7- dimethyl -8-nitrocoumarin : (A3, A4) [14]

A mixture of 4,7-dimethyl coumarin(A1) (0.0696gm, 0.004mole) and conc. sulphuric acid (75ml) was stirred at 0 ^{O}C for 20 minutes. Then a mixture of conc.sulphuric acid (1.25ml, 98%) and conc. nitric acid (0.4ml.) was added at (0-5) ^{O}C (on ice bath) and the reaction was stirred for 3 hrs. at 5 ^{O}C. Then, it was poured into an ice /cold-water, the solid product was filtered and washed with cold water, dried at room temperature, and the crude mixture was separated in ethanol by using soxhlet apparatus. The precipitate that did not dissolve in hot ethanol was 4,7 dimethy-6-nitro coumarin (A3) which was recrystallized from ethylacetate as pale yellow crystals, yield 60% m.p (259-260) ^{O}C. R_f= 0.5 The filtrate was concentrated, and cooled, yielding a precipitate 4,7-dimethyl -8-nitrocoumarin(A4) which was recrystallized from ethyl acetate m.p (242-243)^{O}C R_f= 0.4 yield 20%. All physical properties are listed in Table (1)

Synthesis of N- amino-4, 7-dimethyl -6-nitro-1H-2-quinolone: (A5)

Compound A3 (1.095gm, 0.005mole) was dissolved in anhydrous pyridine (20ml) and hydrazine hydrate (80%, 0.15mole) was added to the mixture. The reaction mixture was refluxed for (6 h) with stirring at 117°C. The resulting solution was removed by rotary under reduced pressure, and additional ethanol (10ml) was added. The excess solvent was removed by rotary under reduced pressure again. The crude product was recrystallized from ethanol or toluene as yellow crystals, m.p. (265-267)°C, R_f= 0.16:see Table (1)

Synthesis of N- amino -1H—2-quinolone derivatives: (A6)

To the solution of (0.02mole) 4-methyl 7- ethyl coumarin A2 in absolute ethanol 25ml, hydrazine hydrate (80%) (0.2mole ,19 ml) was added then refluxed for (6) days the progress of the reaction was monitored by T.L.C hexane :ethyl acetate (7:3) after completing the period of time, the solvent was concentrated and added another amount of ethanol 25 ml., to the concentrated mixture, after that the mixture was concentrated again. Finally the separated solid product was filtered off and washed with cold ethanol. The formed precipitate was recrystallized from benzene as solvent m.p (179-180)°C R_f=0.298. The physical properties of compound are listed in Table (1)

Synthesis 1-(2-Hydroxy-ethyl)-1H-2-quinolone (A7)

To a mixture of coumarin (0.035 mol., 5g) in absolute ethanol (50ml), ethylene diamine 99% (10ml) was added, then refluxed (24hr.), after complete the time of reaction, the solvent was concentrated, cooled and washed the residue with ether several time and water was added to precipitate the product, recrystallized by MeOH. m.p 140 dec. R_f=0.58 as shown in Table (1)

Synthesis of 1-(2-subsituted-ethyl)-1H-2-quinolon- (A8)

To a mixture of coumarin (0.007mol., 1g) in absolute ethanol (30 ml) ethanol amine (3ml) was added then refluxed (24hr.). After complete the time of reaction the solvent was concentrated by heating then, left the mixture to cool. The residue was translated to separatory funnel and used benzene to wash the desirable product twice, then dried the product at room temperature. R_f=0.2. as shown in Table (1)

Synthesis of N-(substitution-1H-2-quinolon-1-yl)-benzene sulfonamide (A9-A12)

Compounds **(A5-A8)** (0.004 mol.) were dissolved in dry pyridine (10ml), benzene sulfonyl chloride (0.004mole, 0.35g) was added drop wise at (1-5^{o}C). The mixture was stirred at room temperature for (6-10hrs.), then poured on ice /water, filtered, dried, and recrystallized from suitable solvents. All physical properties of compounds (A9-A12) are listed in Table (1)

Table (1): physical properties of compounds (A1-A12)

Comp .No.	m.p.^{0}C	color	Purification solvent	Yield%	R.f inhexane:E.A 7:3
A1	-133 131	white	Ethanol:H_20 (10:2)	75	0.6
A2	70-72	white	Benzene	60	0.64
A3	259-260	Pale yellow	Ethyl acetate	80	0.5
A4	242-243	Yellow	Ethyl acetate	20	0.4
A5	265-267	Pale yellow	Ethyl acetate or toluene	80	0.16
A6	179-180	Off white	Benzene or toluene	63	0.298
A7	140 Dec.	Yellow	MeOH	75	0.58
A8	oily	Yellow	Benzene	68	0.2
A9	135 Dec.	Deep brown	Ethanol :water (10:1)	60	0.2
A10	328 Dec.	Brown	Ethanol :water (10:1)	75	0.23
A11	83-84	Pale yellow	Chloroform: acetone	70	0.229
A12	97-100	Deep red	Acetone	72	0.27

Results and Discussion:

The condensation of ethyl acetoacetate with an equimolar amount of substituted phenol in the presence of conc. sulfuric acid under Pechemann condensation reaction produced coumarin derivatives (A_1 andA_2). The substitution of phenol at the meta position with electron donating groups (-CH_3, -C_2H_5,) cause an increase in the reactivity of the carbon at the ortho position to the hydroxyl group and at para position of the substituent.

The FT-IR spectrum of compounds (A1and A2) showed the appearance of characteristic absorption very strong band at (1716-1731) cm^{-1} due to (C=O) band of the lactone ring as shown in Table (2) [15][16]. The ^{1}H-NMR spectrum of compound (A1) showed the proton signals due to two groups of CH_3 were recorded at 2.4 and 2.6 ppm also showed three signals appeared in (7.2-7.7) ppm for three aromatic protons and singlet signal at (6.3)ppm for one proton of the lactone ring see Table(2). While the ^{13}C-NMR spectrum for the same compound showed (17.9) and (20.9) ppm for two groups of CH_3, the signal at (116.4, 117,125,125.3, 142.7, 152.9, and 153.2 ppm) belong to aromatic carbons and (159.9) ppm for carbonyl carbon as shown in Table (4)**.** The ^{1}H-NMR spectrum of compound (A2) showed the proton a signal due to two groups of CH_3 were recorded at (1.2) and (2.5) ppm, a signal at 2.8 ppm due to CH_2 and signals at (7.3-7.8) ppm due to for three aromatic protons and singlet signal at (6.3) ppm for one proton of the lactone ring as shown in Table (3). While the ^{13}C-NMR spectrum for the same compound showed (15.1, 18 and 27.9) ppm for two groups of CH_3,and a group of CH_2 the signal at (113.3, 115.2,117.3, 124.2, 125.1, 148.8, 153.2 and 153.9) ppm due to aromatic carbons and (159.9) ppm for carbonyl carbon as shown in Table (4).

The nitration of compound (A1) using concentrated nitric acid in the presence of sulphric acid at (0^{O}C) by electrophilic substitution reaction gave a mixture from (80%) 4,7-dimethyl- 6-nitro coumarin (A3) m.p259-260 ,and (20%) 4,7-dimethyl- 8-nitro coumarin (A4) m.p 242-243.TheFTIR spectrum of compound (A3) showed the appearance of absorption band for C=O of the lactone ring at 1734 cm^{-1} and the appearance of new absorption band at 1527cm^{-1} and 1354cm^{-1} belonging to asymmetric and symmetric to the NO_2 group as shown in Table(2). The ^{1}H-NMR spectrum of (A3) showed signal at 2.4ppm for three protons of the methyl lactone ring, signals at 2.6ppm for three protons of methyl benzene ring; signals at6.525 for proton of the lactone ring,

7.56 and 8.375 ppm for two aromatic protons of the benzene ring as shown in Table (3). While ^{13}C-NMRspecrum showed signals at (17.8 and 19.7 ppm) for carbons of two methyl groups, 154.8 (C-O), 152.3 (C-NO_2), (122.2-115.3ppm) for three aromatic ring carbons and 158.8 ppm for carbonyl carbon of the lactone ring (O-C=O) as shown in Table (4).

Another isomer of nitro coumarin derivatives was 4,7- dimethyl -8-nitro coumarin compound (A4) showed difference solubility in hot ethanol , melting point and $R_f$of TLC from compound (A3) 4,7- dimethyl -6-nitro coumarin. FTIR of compound (A4) showed the strong band at 1737 cm^{-1} for carbonyl compound and two strong bands at 1518 and 1348 cm^{-1} for asymmetric and symmetric of NO_2 group respectively see Table (2).

Table (2): FT-IR Spectral data cm^{-1} of compounds (A1-A4)

Comp. No	υ CH arom	υ CH aliph	υC=O	υC=C arom	υ NO_2
A1	3078	2960, 2869	1716	1620	-
A2	3078, 3059	2960, 2930	1731	1620	-
A3	3124	2960	1734	1622	1354, 1527
A4	3086	2950	1737	-	1348, 1518

Table (3): ^{1}H-NMR spectra of some synthesized compounds (A1, A2, and A3)

Comp.No.	^{1}H-NMR(ppm)
A_1	2.4(s,3H,CH_3), 2.6(s,3H,CH_3), 6.3 (s,1H,H lactone ring), 7.11-7.6 (m,3H,Ar- H)
A_2	1.2(t,3H,CH_3), 2.5(s,3H,CH_3), 2.8(q,2H,CH_2), 6.3(s,1H, H lactone ring), 7.3-7.8(m,3H,Ar-H),
A_3	2.4(s,3H,CH_3), 2.6(s,3H,CH_3), 6.5(s,1H, H-lactam ring), 7.5 (s,1H, Ar-H), 8.3(s,1H, Ar-H),

Table (4): ^{13}C-NMR for some preparing compounds (A1, A2, and A3)

Comp.No.	^{13}C-NMR (ppm)
A1	17.9(7-CH_3), 20.9(4-CH_3), 153.2 (C-O), 113,117,125,125.3,142,153(aromatic ring), 159.9 (C=O) , 116, 152 (for lactone ring)
A2	15.1(4-CH_3), 18.0(CH_2), 27.9($\underline{CH_3}$-CH_2), (C-O), 159.9(C=O)113, 117,1254.125,148,153(aromatic ring), 115,153(for lactone ring)
A3	17.8(7-CH_3),19.7(4-CH_3) , 144.9(C-NO_2), 154.8(C-O), 158.8(C=O),118,120,122,137,154(aromatic ring), 115,152(for lactone ring)

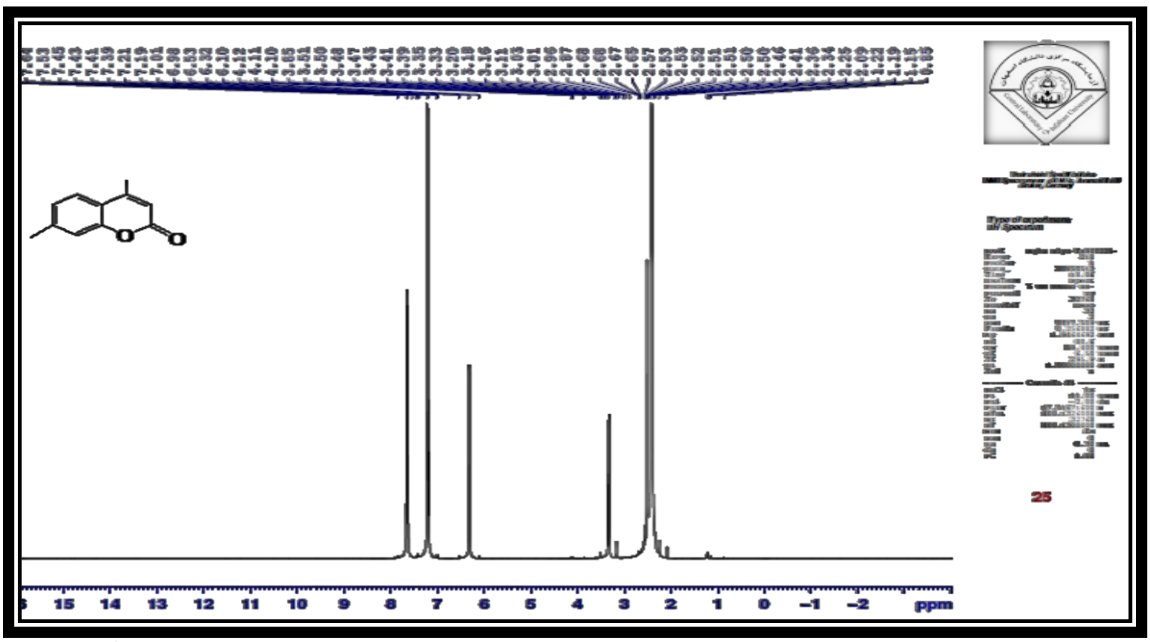


Fig.(1): ^{1}H-NMR spectrum for compound [A1]

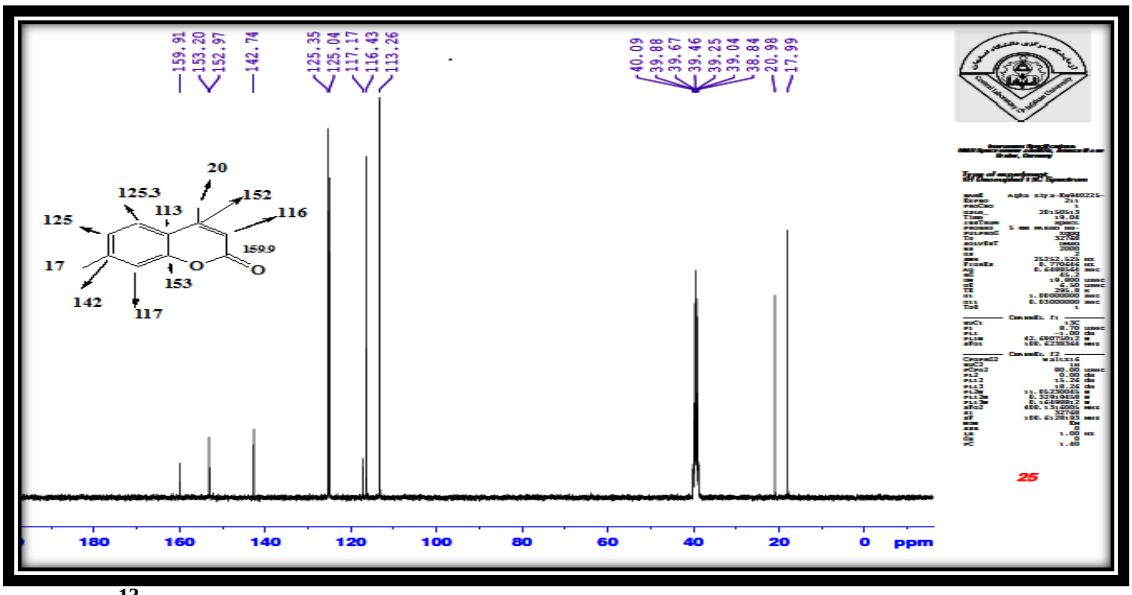


Fig. (2): ^{13}C-NMR spectrum for compound[A1]

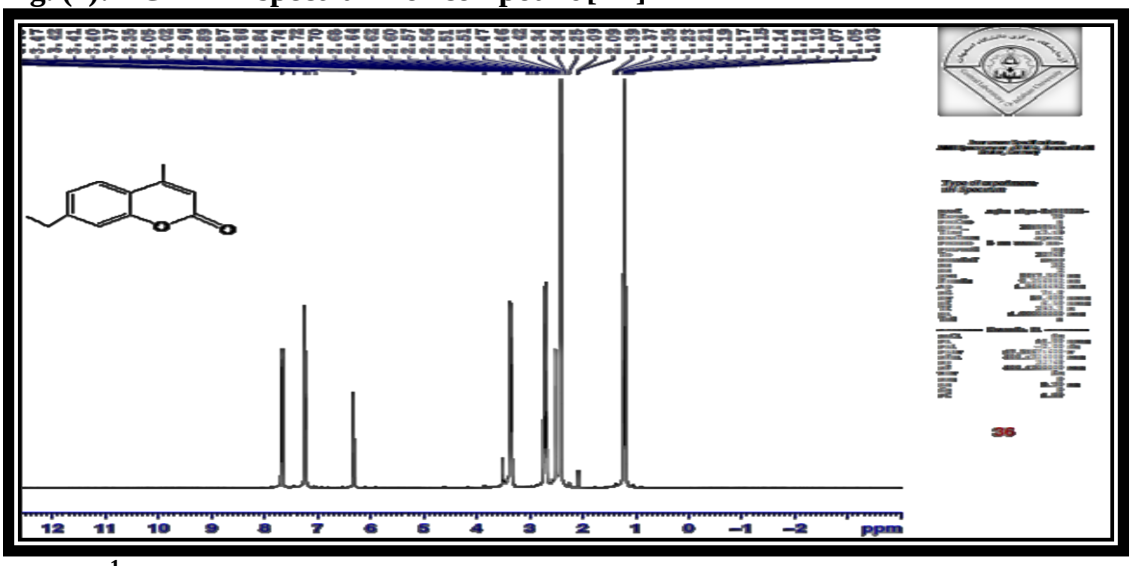


Fig.(3): ^{1}H-NMR spectrum for compound [A2]

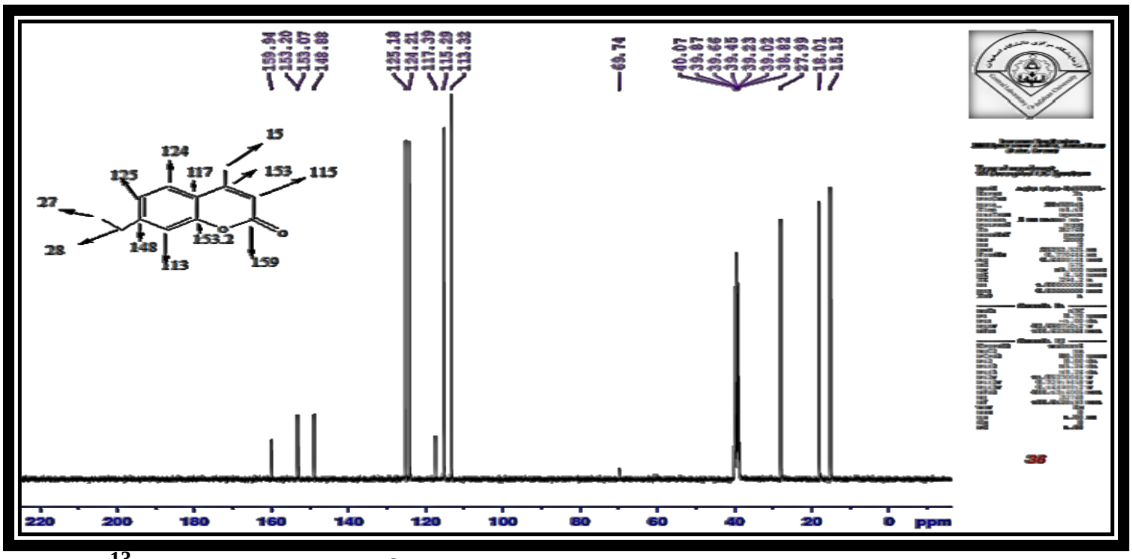


Fig.(4): ^{13}C-NMR spectrum for compound [A2]

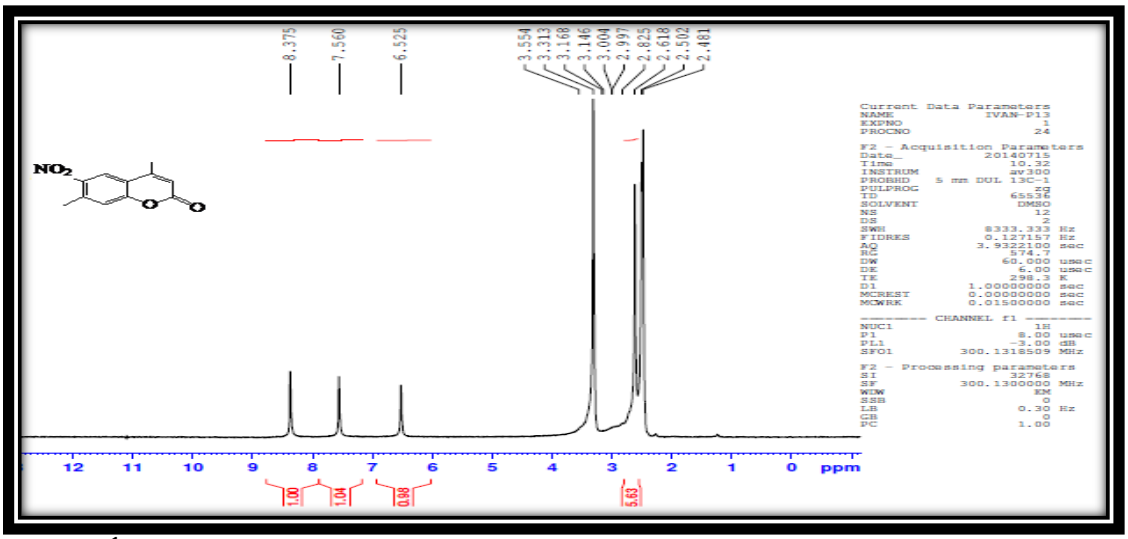


Fig.(5): ^{1}H-NMR spectrum for compound [A3]

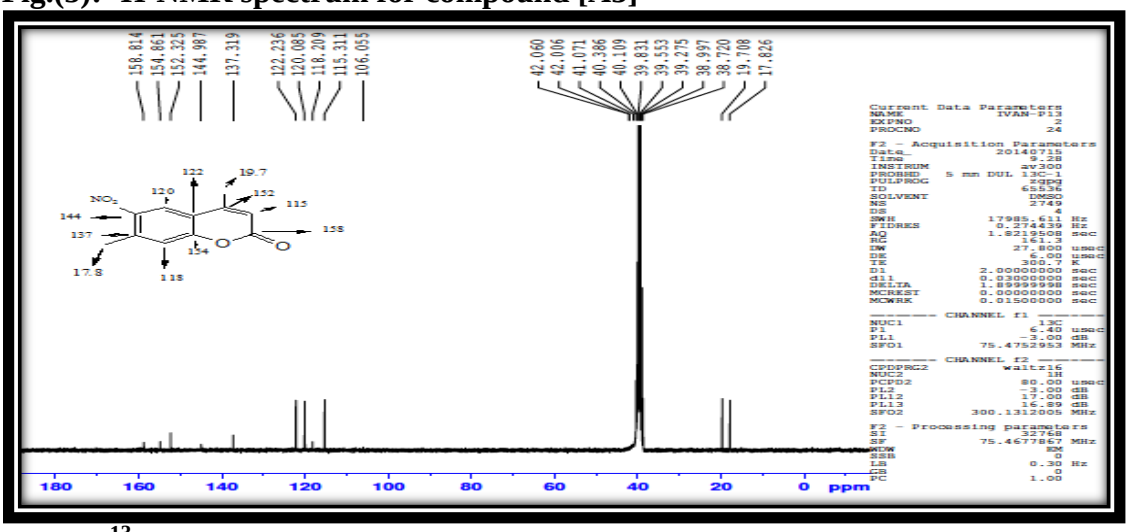


Fig.(6): ^{13}C-NMR spectrum for compound [A3]

The treatment of different coumarins with different amines produced N-amino-2-quinolone derivatives (A5-A8).The reaction was preceded by nucleophilic substitution of amino group with the carbonyl group of the lactone, the ring through the ring opening of the lactone ring and recycling with losing a water molecule giving the corresponding amino compounds.

In these mechanisms nitro as electron withdrawing group plays a very important role to stabilize the transition state and the reaction needs shorter time and a high yield than in case of benzene ring or aryl with releasing group (ethyl) that was noted when 7-ethyl-4-methyl coumarin (A2) was used instead of compound (A3). In other hands the solvent pyridine plays a very important role to reduce the time of reaction may be because it provides the high temperature to react by its high boiling point compared with ethanol.

The FT-IR spectrum of compounds (A5-A7) showed the appearance of characteristic absorption bands at (3300-3267) and (3255-3196) cm^{-1} which belong to the NH_2 and (1697-1652) cm^{-1} due to amid carbonyl cyclic lactam group υ C=O in addition to the appearance of the characteristic absorption band at (3350) due to (OH)

group for compound (A8) as shown in Table (6). The ^{1}H-NMR spectrum of compound (A5) showed signals at (2.4) ppm and (2.6) ppm for two methyl groups (CH_3), a signal at (5.85) ppm for the two protons of NH_2, a signal at (6.71) ppm for the proton of the lactam ring, and signals at (7.91 and 8.41) ppm for two aromatic protons see Table (6) and Figure (8.) ^{13}C-NMR spectrum of compound (A5) showed signals at (18.5 and 20.5) ppm for two methyl groups ($2CH_3$) and showed signals at (117.6, 134.7, 135.4, and 141.8) ppm for aromatic carbons, (142.6) ppm for the amino group carbon C-N-NH_2, (145.6) ppm for C-NO_2 and (159.6) ppm for the carbonyl carbon of the lactam ring see Table (7) Figure (8).

The^{1}H-NMR spectrum of compound (A6) showed triplet signal at (1.1) ppm for three protons of methyl groups (CH_2- $C\underline{H_3}$), singlet signal at (2.7) ppm for methyl groups of lactam ring (Ar-CH_3),quartet signal at (2.5) ppm for methylene group (CH_2), singlet signal at (5.9) ppm for two protons of (NH_2), and signals at (6.6-9.3) ppm for four aromatic protons see Table (6) Figure (9). The ^{13}C-NMR spectrum of compound (A6) showed signals at (15, 24.7 and 27.65) ppm for aliphatic protons and showed signals at (116, 116, 118, 126, 126.8, 127, 144 and 155.3) ppm for aromatic carbons, and (155.5) ppm for carbonyl group see Table (7) and Figure (10).

Table (5): FT-IR Spectral data cm^{-1} of compounds (A5-A8)

Comp. No	υ NH	υ CHar	υ CH aliph	υ C=O	υ C=Carom	other bands
A5	3293,3196	3086	2983, 2929	1672	1637	1527, 1354 NO_2
A6	3267, 3198	3053	2991, 2961	1697	1622	
A7	3300, 3255	3033	2925, 2858	1652	1647, 1635	-
A8			2939	1653	1634,1587	3350 (OH)

Table (6): ^{1}H-NMR spectra of some synthesized compounds (A5 and A6)

Comp.No.	^{1}H-NMR (ppm)
A5	2.4(s,3H,CH_3),2.6(s,3H,CH_3),5.8(s,2H,$N\underline{H_2}$),6.7(s,1H,H-lactam ring), 7.9(s, 1H, Ar-H), 8.4(s,1H,Ar-H)
A6	1.1(t,3H,CH_3), 2.5(q,2H,CH_2), 2.7(s,3H, Ar-CH_3), 5.9(s, 2H, NH_2), 6.6-9.3(m, 4H, Ar-H)

Table (7): ^{13}C-NMR for some preparing compounds (A5 and A6)

Comp.No.	^{13}C-NMR(ppm)
A5	19.1(4-CH_3),21.1(4-CH_3), (C-NO_2) 145.8,159.6(C=O) and, 117,119,134,135,141 (aromatic ring), 121,142(lactone ring)
A6	15.4(4-CH_3), 24.7($CH_3\underline{CH_2}$), 27.6($\underline{CH_3}CH_2$) ,155.5(C=O)and,115,118,125,126,127, 143-155(aromatic ring), 115,155(lactone ring)

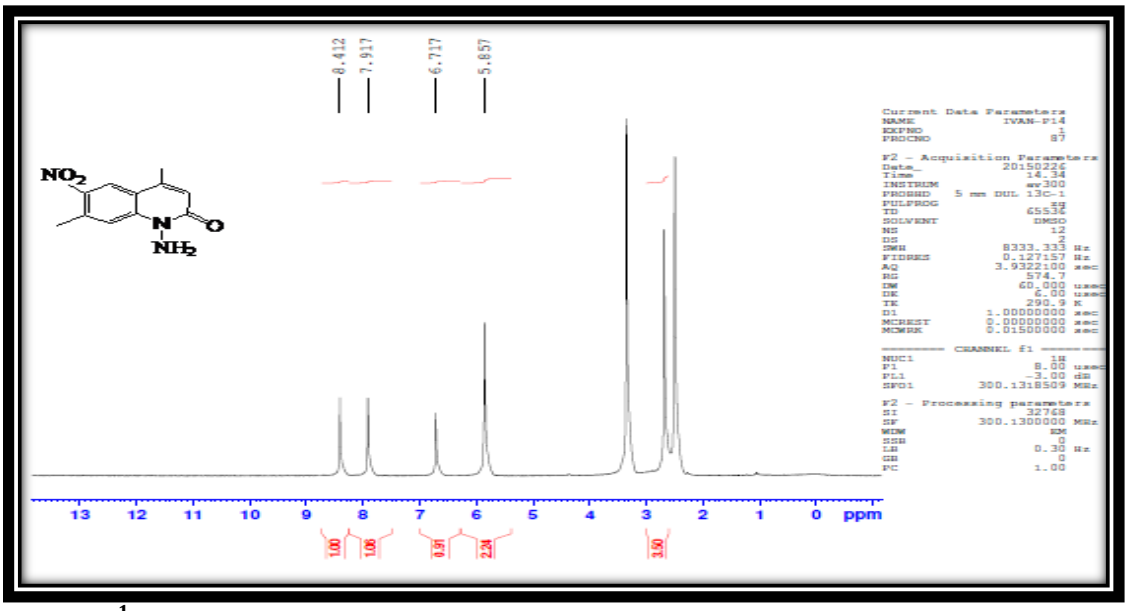


Fig. (7): ^{1}H-NMR spectrum for compound [A5]

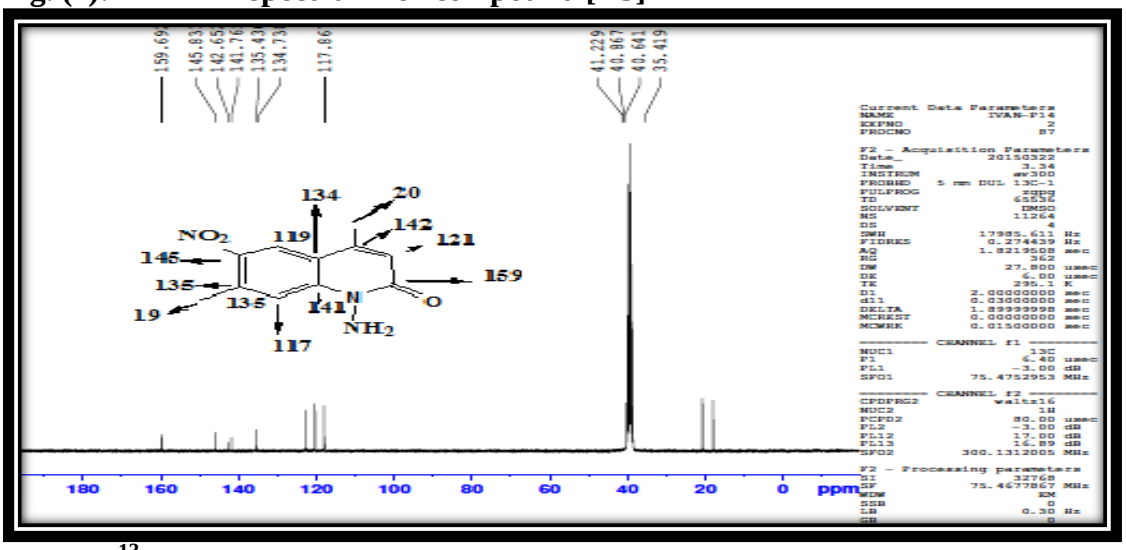


Fig. (8): ^{13}C-NMR spectrum for compound[A5]

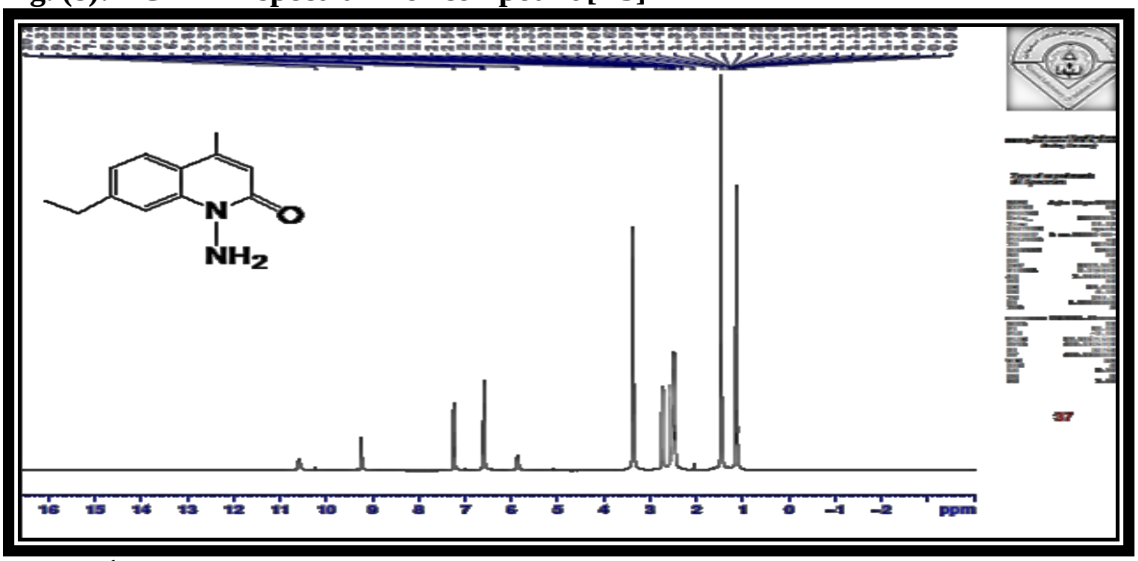


Fig.(9): ^{1}H-NMR spectrum for compound [A6]

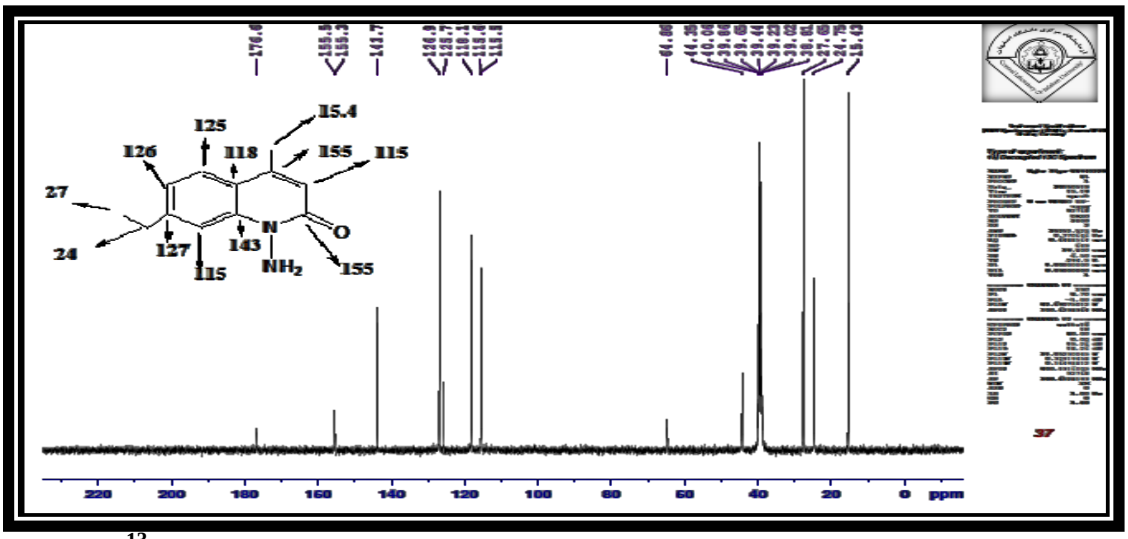


Fig.(10): ^{13}C-NMR spectrum for compound [A6]

The synthesized compounds (A9-A12) were prepared by reaction of benzene sulfonyl chloride with (substituted-2-oxo-2H-quinolin-1-yl)-amino derivatives (A5-A7) or 1-(2-hydroxy-ethyl)-1H-quinolin-2-one A8 in pyridine as a solvent under cooling conditions then refluxing conditions. The FT-IR spectra of compounds (A9-A12) were characterized by disappearance of symmetric and asymmetric absorption bands for υ(NH_2) and appearance of new absorption band in the synthesized compounds between (3354-3185) cm^{-1} belong to υ(NH) group,(1327-1346) cm^{-1} and at (1196-1165) cm^{-1} of asymmetric and symmetric absorption bands of the υ(SO_2) group. Other FT-IR spectrum data of the prepared compounds were pointed in Table (8).

The ^{1}H-NMR spectrum of compound (A11) showed signal at (3.2) ppm due to aliphatic four protons and signals at (7.2-9) ppm due to aromatic protons and the proton of NH as shown in Table (9) Figure (11).

The ^{1}H-NMR spectrum of compound (A12) showed signals at (3.2-4) ppm due to aliphatic protons and signals at (7.2-9) ppm due to aromatic protons as shown in Table (9) Figure (12).

Table (8): FT-IR data of synthesized compounds (A9-A12)

Comp.No	υ(NH)	υ(C-H) Ar	υ (C-H) Aliph	υ(C=O) amide	υ(C=C) Ar	υ(SO_2)asym.	υ(SO2)sym.	Other bands
A9	3354	3055	2920	1683	1583	1341	1192	1525, 1340 NO2
A10	3207	3002	2878	1686	1616, 1578	1346	1166	-
A11	3185	3005	2803	1641	1603	1327	1196	-
A12	-	3090,3064	2953	1658	1616, 1635	1327	1165	-

Table (9): ^{1}H-NMR of synthesized compounds (A11 and A12)

Comp. No.	^{1}HNMR/ ppm
A11	3.2(m,4H,2CH_2), 7.2-8.6(m,11H, Ar-H and N<u>H</u>)
A12	3.2-4(m,4H,N-CH_2-CH_2-O), 7.2-9(m,11H, Ar-H)

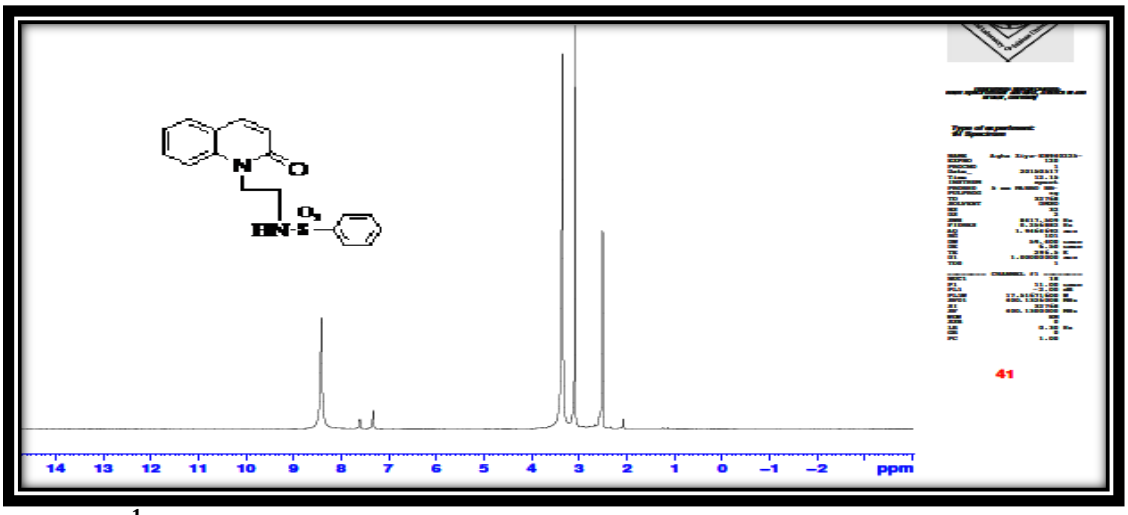


Fig. (11): ^{1}H-NMR spectrum for compound [A11]

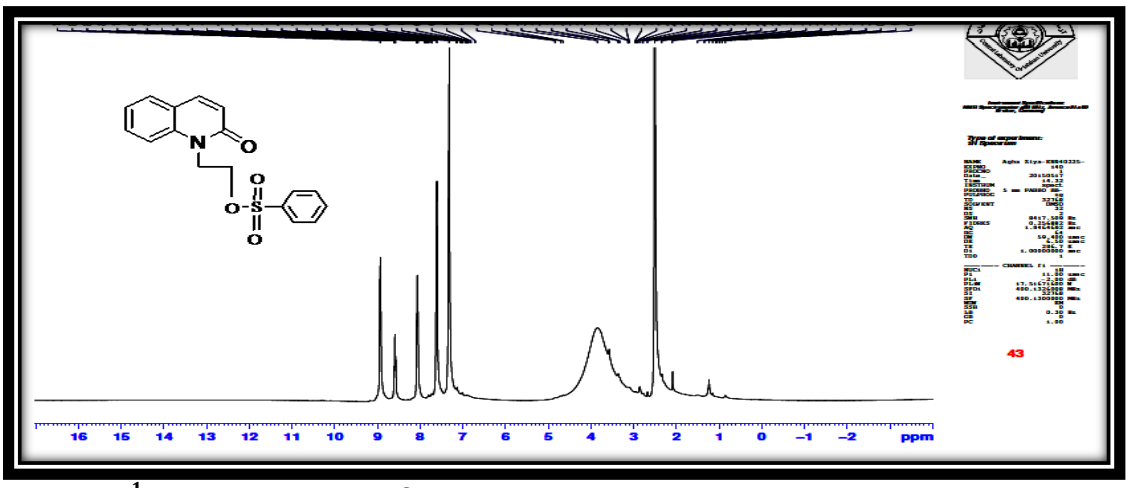


Fig. (12): ^{1}H-NMR spectrum for compound [A12]

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تحضير وتشخيص مشتقات جديدة من -2-كوينولون سلفونأمايد

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الخلاصه:

يشتمل هذا البحث تحضير مشتقات جديدة من 2-كوينولون مرتبطه مع جزيئة بنزين سلفونأمايد من عدة خطوات. الخطوة الاولى تضمنت تحضير كومارينات مختلفة A2وA1 عن طريق تكاثف فينولات معوضة مع مركب الاسيتواسيتيت. مركب رقم A1 تم معاملتة مع حامض النتريك المركز لينتج ايزومرين مختلفين من النايترو كومارين A3و A4. المركبان المحضران A2وA3 تم معاملتهم مع الهيدرازين المائي لينتج المركبان -2-الكوينولون مختلفة (A6,A5)، بينما مركب الكومارين تم مفاعلته مع امينات مختلفه لينتج مركبات 2-الكوينولون (A8,A7) بعدها تمت مفاعلة الكوينولونات المحضرة (A8-A5) مع البنزين سلفونيل كلورايد لينتج مشتقات السلفونأمايد (A12-A9).تم تشخيص المركبات المحضرة بالطرق الطيفية والفيزياوية المتيسرة FTIRوطيف النووي المغناطيسي ^{1}HNMR و . ^{13}CNMR

الكلمات المفتاحية: 2-الكوينولون ، الكومارين، السلفونامأيد