Photo-degradation Study for Some 4-aminoantipyrine derivatives by used (Cs-137) as Gamma-Ray Source

دراسة التحطم الضوئي لبعض مشتقات 4ـأمينوأنتي بايرين بأستخدام (Cs-137) كمصدر لأشعة كاما

Thaer Mahdi Madlool

Department of Chemistry/Science College/Kerbala University
E-mail: thr_mahdy@yahoo.com

Abstract

The compounds {4-Benzylidene-amino)-1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-one} , {4-[(4-Hydroxy-benzylidene)-amino]- 1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-one} ,and {4-[(2-Hydroxy-benzylidene)-amino]- 1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-one}were prepared as [4-aminoantipyrine] derivatives by condensation reaction . These compounds were characterized by FTIR spectra , UV.-Vis. Spectra, and melting point measurement .The bond constant (K) for active group that characterized by IR spectrum was calculated .Also ,solvent effect on UV.-Vis. Spectrum was studied which appeared the prepared solution compounds were influenced by solvent polarity .

Photo study for compounds was carry out by used Cs-137 as Gamma Ray source for (24 hour) . IR spectra and UV.-Vis. Spectra after irradiation didn't appear clear bands for compounds spectra that refer all compounds were gotten a photo-degradation because of they absorbed Gamma Ray. The mean external exposure [$X_{(R)}$] , the dose rate in air [D_{aire} (rad)],and the dose rate in compound [$D_{compound}$ (rad)] were calculated .

الخلاصة:

تم تحضير المركبات {(4-بنزيليدين-أمينو)-5,1-داي مثيل-2-فنيل-2,1-داي هايدروبايرزول-3-اون} و { 4](- 4- هايدروكسـي بنزيليدين) ـأمينو] -5,1-داي مثيـل-2-فنيـل-2,1-داي هايـدروبايرزول-3-اون} و { 4] (2-هايدروكسـي بنزيليدين) ـأمينو] -5,1-داي مثيل-2-فنيل-2,1-داي هايدروبايرزول-3-اون} كمشتقات لـ { 4- أمينو أنتي بـايرين} بواسطة تفاعل التكثيف . شخصت هذه المركبات بأستخدام أطياف الأشعة تحت الحمراء والأشعة فوق البنفسجية-المرئية وقياس درجات أنصهارها . تم حساب ثابت الآصرة (K) للمجاميع الفعالة المشخصة بأطياف الأشعة تحت الحمراء وكذلك تم دراسة تـأير المذيب على أطياف الأشعة الفوق البنفسجية – المرئية والتي أظهرت تأثر محاليل المركبات المحضرة بقطبية المذيب .

أجريت الدراسة الضـوئية للمركبـات المحضـرة وذلك بتعريضـها لأشـعة كامـا لمـدة (24 سـاعة) بأسـتخدام السيزيوم-137 كمصدر مشع ، بعدها تمت مقارنة أطياف الأشعة تحت الحمراء وأطياف الأشعة الفوق البنفسجية ـالمرئية للمركبات بعد التشعيع بالأطياف قبل التشعيع حيث لوحظ ان الأطياف بعد التشعيع لم تظهر حزم واضحة لأي من المركبات المدروسة وذلك يدل على أن جميع المركبات حصل لها تحلل ضوئي ناتج عن أمتصاصها لأشعة كاما. كذلك حسبت قيم الجرع الممتصةمن قبل المركبات($D_{compound}$) والجرع الممتصة في الهواء (D_{air}) ومعدل التعرض الخارجي ($X_{(R)}$) لأشعة كاما.

Introduction:

Schiff base of 4-aminoantipyrine and its complexes have a variety of applications in biological, clinical , analytical and pharmacological areas . Studies of a new kind of chemotherapeutic Schiff bases are now attracting the attention of biochemists . Earlier work reported that some drugs showed increased activity , when administered as metal complexes rather than as organic compounds [1,2,3,4,5].

Gamma - Ray irradiation doses do not require catalysts , so there are no catalyst residues in the final product to interfere with physical properties. There is no heat treatment to degrade thermally sensitive components , and the dosage of irradiation can be controlled easily.

The main chains may be degraded or cross linked by the gamma -ray irradiation , with both processes usually taking place concurrently [6].

Two radioactive isotopes of cesium ^{137}Cs and ^{134}Cs came 1986 from Chernobyl in Ukraine and deposited in many countries. These nuclides have the half - lives 30.0 a and 2.06 a , respectively. These isotopes have been used in adsorption, suspension and sedimentation research . Because ^{134}Cs was not with in depositions before 1986 , it has met the particles, with which it has been detected, after the end of April in the year 1986 [7] . The measured ratio ^{134}Cs/^{137}Cs (into the 1986 level corrected) was 57($\pm$ 2) % (Aaltonen, et al., 1990) or 56 % (De Cort, et al. , 1998) [8,9] . If the ratio is smaller, then a part of ^{137}Cs isotopes has deposited before 1986. By this way Froelich and Walling (1994) found the age information for sediment and soil layers and Rafferty et al. (2000) for soil layers[10] . The activity of ^{134}Cs may be measured directly today[11,12,13,14,15] . However , even relatively small cesium -134 amounts are spectrometrically detected by using a coincidence method (Lee and Chung , 1991, Chung et al., 1992)[16].

Today , Ge –detectors with high purity Ge –crystals of 2 kg are readily available and detectors with crystals up to 5 kg can be produced . Use of large area crystals results in high detection efficiencies , which can serve to reduce detection limits or to make faster measurements of low activity samples possible. The second major development that has contributed to lowering detection limits has been research on identifying and reducing the background components in gamma-ray spectrometry systems[17].

In this research, photo stability for 4-aminoantipyrine derivatives were studied , because these compounds are important in medicine manufacture and the field of dyes . These compounds irradiated by gamma ray as type of cosmic ray . For benefit from the results to limitation the keep condition of these materials ,also we can to increase the photo stability by product metal complexes from these compounds.

Experimental:

All of the materials and solvents in table 1 were used as received .

Table(1) : Chemical Materials were used

Chemical material	Purity %	Provided Company
4-aminoantipyrine	95	Fluka A.G.
Benzylaldehyde	98	B.D.H.
Salicylaldehyde	98	B.D.H.
P-Hydroxy benzylaldehyde	98	Fluka A.G.
Ethanol	99	B.D.H.
Methanol	99	B.D.H.
Benzene	97	B.D.H.
Cyclohexane	99	B.D.H.
CCl$_4$	95	B.D.H.

IR spectra were recorded as KBr discs using Testscan Shimadzu model 8400 FT-IR spectrophotometer in range (4000-400) cm^{-1} . Electronic spectra of the prepared compounds were measured in the region (200-800)nm for 10^{-3} M solution in ethanol using Shimadzu 160 spectrophotometer, with 1.000 $\pm$ 0.001 cm matched quartz cell. Gallen kamp capillary melting point apparatus was used to measure the melting points of the compounds.

Preparation of (A1):

A solution of 4-aminoantipyrine (0.89 g , 4.4 mmole) in ethanol (20ml) was added to benzylaldehyde (0.466 g , 4.4 mmole) dissolved in ethanol (15ml) adding anhydrous potassium carbonate . The mixture was allowed to reflux with stirred for three hours. The potassium carbonate was filtered off from the reaction mixture and the solvent was evaporated. The yellow solid separated was filtered and recrystallized from ethanol/H_2O.(see Fig. 1.)

Preparation of (A2&A3) :

The preparation of these compounds a similar way which that showed at the (A1) compound preparation ,but the Salicylaldehyde and P-Hydroxy benzylaldehyde used respectively , the physical properties summarized in table 2.(see Fig. 2&3).

Irradiation for Compounds:

Irradiation for compounds was carry out in nuclear physics lab at physics department /college of science/Kerbala university , by used Cs-137 as irradiation source .

The sample was put in photocell after that the photocell was put about 10cm far from the Cs-137 source and the irradiation reaction was left for 24h.

After that the sample was characterized by used FTIR and UV./Vis. Spectra .

Irradiation reaction for (A1) compound was carry out at 4/5/2009 , (A2) compound at 5/5/2009,and (A3) compound at 6/5/2009.

Results and Discussion:

All compounds were characterized (Before & After) irradiation by used FT-IR , UV-Vis spectroscopy , melting point and solubility.

IR Spectra:

The IR spectrum for (A1) before irradiation , Fig.4 appears the bands at (1122,3043,2928 cm-1) ascribed to ν(C-N) ,aromatic ν (C-H) and aliphatic ν (C-H) stretching respectively. The intense strong band at (1638cm-1) assigned to ν(C=N) stretching. The other bands were listed in table 3.

The IR spectrum for (A2) before irradiation , Fig. 5 appears the band at (3454 cm^{-1}) can be attributed to ν(O-H) stretching, the ν(C=N) stretching vibration have been showed at (1610 cm^{-1}), the weak band at (3068 cm^{-1}) assigned to ν(C-H) of aromatic frequencies and appears the band at (1445cm-1) ascribed to δ(C-H) aliphatic frequency .The other bands were listed in table 3 .

While the IR spectrum for (A3) before irradiation, Fig. 6 appears the band at (3464 cm-1) assignable to intramolecular hydrogen bonded –OH group . The spectrum of the compound shows ν(C=N) band in the region (1625cm-1).The other bands were listed in table 3.

After irradiation, the IR spectra of all compounds haven't been showed any clear band, figures (7,8&9). That may be return the bonds destroyed during irradiation of each compound.

Calculation of Bond Constant [K] :

Bond constant of each active group appears in IR spectrum was calculated by using equation 1[18] :

$$ν = (1/2π) (k/μ)^{0.5} ……………(1)$$

ν : vibration frequency
k : bond constant
μ : reduced mass

The reults of (k) data were listed in table 4.

UV-Vis spectra:

The electronic spectrum of (A1) before irradiation , Fig. 10 exhibits the peaks at (306 nm) (32679 cm^{-1}) (ε_{max} =4000 molar^{-1}.cm^{-1}) and (346nm) (2890 cm^{-1}) (ε_{max} =4000 molar^{-1}.cm^{-1}) assigned to ($\pi\rightarrow\pi^*$) and ($n\rightarrow\pi^*$) transitions respectively [19]. Table 5.

The electronic spectrum of (A2) before irradiation, Fig. 11 displays tow peaks (307nm) (32573 cm^{-1}) (ε_{max} =4000 molar^{-1}. cm^{-1}) and (336nm) (29761 cm^{-1}) (ε_{max} =3783 molar^{-1}. cm^{-1}) attributed to ($\pi\rightarrow\pi^*$) and ($n\rightarrow\pi^*$) transitions respectively [19]. Table 5.

Figure 12, showed the electronic spectrum of (A3) before irradiation. The two peaks at (251nm) (39840 cm^{-1}) (ε_{max} = 4000 molar^{-1}.cm^{-1}) and (377nm) (26525 cm^{-1}) (ε_{max} = 4000 molar^{-1}.cm^{-1}) .these peaks were assigned to ($\pi\rightarrow\pi^*$) and ($n\rightarrow\pi^*$) transitions respectively [19]. Table 5.

After irradiation, the electronic spectra of all compounds were disappeared because these compounds destroyed .Fig.(13,14&15).

Solvent effect on UV-Vis. Spectra:

The solvent effect on UV-Vis. Spectra of all compounds were studied by using different solvents polarity. This study appeared the following results:

Compound (A1):
1- Red shift of ($n\rightarrow\pi^*$) transition at (332,346,353,364 nm) by using CCl4,ethanol,methanol and benzene respectively.
2- Blue shift of ($n\rightarrow\pi^*$) transition at (353,346,332,321 nm) by using methanol, ethanol,CCl4 and cyclohexane respectively . Fig.(10,16,17,18&19).

The electronic spectra of (A2 & A3) also effected with polarity as same as compound (A1). See **Fig.(11,20,21,22,23,12,24,25,26&27).**

Effect of Irradiation on Compounds

The study of irradiation for these compounds appeared that all compounds were degradation by Gamma Ray.

The mean external exposure [$X_{(R)}$] , the dose rate in air [D_{aire} (rad)],and the dose rate in compound [$D_{compound}$ (rad)] were calculated by following equations [20] :

$$D_{air} (rad) = 0.87 * X_{(R)} \ldots\ldots\ldots(2)$$

$$D_{compound} (rad) = 0.87 * [\mu_{mm}/\mu_{ma}] * X_{(R)} \ldots\ldots(3)$$

μ_{ma} : the mass absorption coefficient for Cs-137 in air.(1.79×10^{-2} m^2/Kg)
μ_{mm}: the mass absorption coefficient for compound .

$$\mu_{mm}(m^2/Kg) = \Sigma\ w_i\ \mu_i \ \ldots..(4)$$

w_i : weight fraction of element in the compound.
μ_i : mass absorption coefficient of element.

$$X_{(R)} = \Gamma * [A/d^2]\ldots\ldots\ldots..(5)$$

$$A = A_0 * e^{-\lambda t}\ldots\ldots\ldots(6)$$

Γ : exposure constant.(0.333 R.m^2/h.ci).
A : radio active source at experimented time.
A_0 : radio active source at industrial time. (100 μci).
λ : decay constant = [ln2/$T_{1/2}$]. $T_{1/2}$:half time.(30 year).

$$t = t_1 - t_0 \ldots\ldots\ldots.(7)$$

t_1 : experimental time .(4,5,and 6/5/2009 respectively).

t_0 :industrial time. (1/12/2006).

d : the distance between the sample and Cs-137 source.(10cm).

The results of The mean external exposure [$X_{(R)}$] , the dose rate in air [D_{aire} (rad)] ,and the dose rate in compound [$D_{compound}$ (rad)] data were listed in table 6.

Table (2): Physical properties of compound prepared

Comp.	Formula	Color	m.p.	Yield %	Solubility					
					H$_2$O	Et-OH	Meth-OH	Benzene	Cyclo hexane	CCl$_4$
A1	C$_{18}$H$_{17}$N$_3$O	Yellow-Crystal	216	75	X	+	+	±	±	±
A2	C$_{18}$H$_{17}$N$_3$O$_2$	Brown-Powder	248	89	X	+	+	±	±	±
A3	C$_{18}$H$_{17}$N$_3$O$_2$	Yellow-Needles	262	65	X	+	+	±	±	±

Soluble = + , Insoluble = x , Slightly soluble = ±

Table (3): IR spectral data(wave number)cm-1 of compounds [A1],[A2] and[A3]

Comp.	ν(C-H) aromatic	(C-H) aliphatic	ν(O-H)	ν(C=C) aromatic	ν(C=N)	Additional bands
A1	3043	ν (2928)	-----	1564	1638	1446w δ(C-N) 1720 ν(C=O) 1564s,1047m,838s(Ring vibration)
A2	3068	δ (1445)	3454	1600	1610	1440m δ(C-N) 1718 ν(C=O) 1508s,1030m,831s(Ring vibration) 1147s δ(C-O) 1369m (O-H)in plane 761s (O-H)out plane
A3	3047	δ (1490)	3464	1590	1625	1485w δ(C-N) 1748 ν(C=O) 1578s,1040m,831s(Ring vibration) 1130s δ(C-O) 1400m (O-H)in plane 758s (O-H)out plane

Table (4): Bond Constant data(dyne.cm^{-1}) of active groups

Comp.	K(C-H) aromatic	K(C-H) aliphatic	K(C=C)	K(C=N)	K(C-O)	K(C-N)	K(O-H)
A1	5.04x10^5	4.67x10^5	8.66x10^5	10.22x10^5	------	7.97x10^5	-----
A2	5.12x10^5	1.13x10^5	9.06x10^5	9.88x10^5	5.32x10^5	7.90x10^5	6.62x10^5
A3	5.05x10^5	1.21x10^5	8.95x10^5	10.06x10^5	5.16x10^5	8.40x10^5	6.66x10^5

Table (5): UV-Visible data of Compounds

Comp.	λ (nm)	Wave number (cm^{-1})	$\varepsilon_{max.}$ (molar^{-1}. cm^{-1})	Assignments
A1	306	32679	4000	$\pi \rightarrow \pi^*$
	346	2890	4000	$n \rightarrow \pi^*$
A2	307	32573	4000	$\pi \rightarrow \pi^*$
	336	29761	3783	$n \rightarrow \pi^*$
A3	251	39840	4000	$\pi \rightarrow \pi^*$
	377	26525	4000	$n \rightarrow \pi^*$

Table (6): Data of absorption doses for Gamma Ray

Comp.	A (µci)	$X_{(R)}$ (R.h^{-1})	D_{air} (rad)	$D_{compound}$ (rad)
A1	94.552	31.486×10^{-4}	27.392×10^{-4}	25.189×10^{-3}
A2	94.546	31.483×10^{-4}	27.390×10^{-4}	25.630×10^{-3}
A3	94.540	31.481×10^{-4}	27.388×10^{-4}	25.628×10^{-3}

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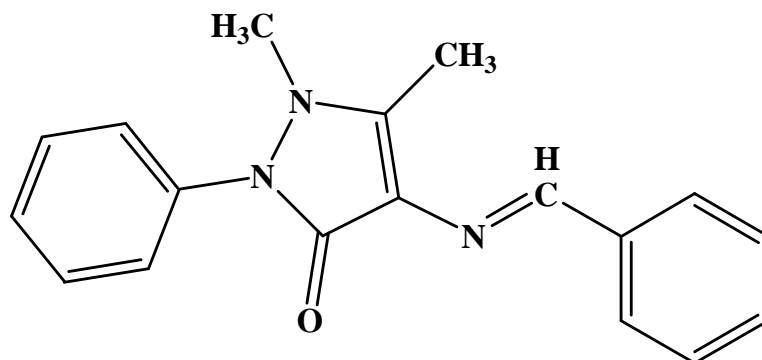
4-(Benzylidene-amino)-1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-one

Figure(1) : Structural Formula of (A1) Compound
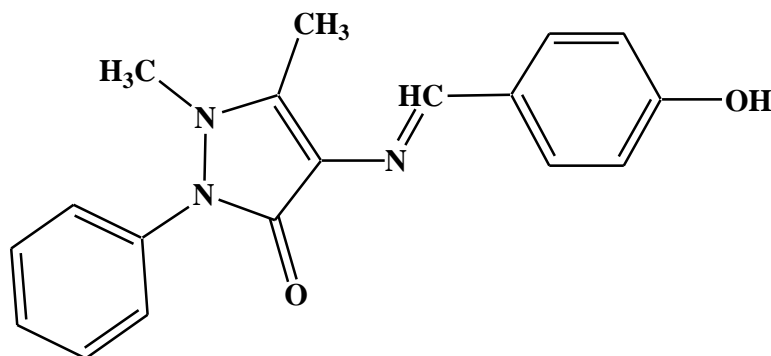
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Figure(2) : Structural Formula of (A2) Compound
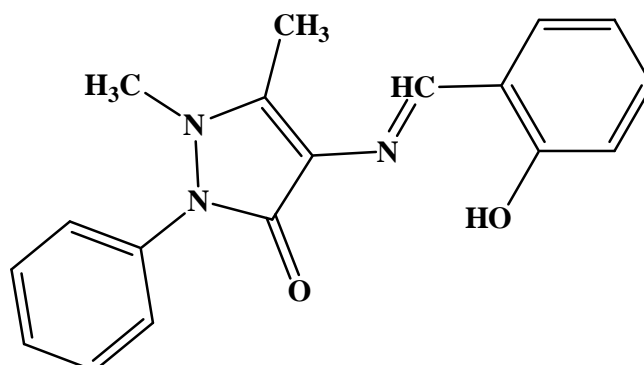
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Figure(3) : Structural Formula of (A3) Compound
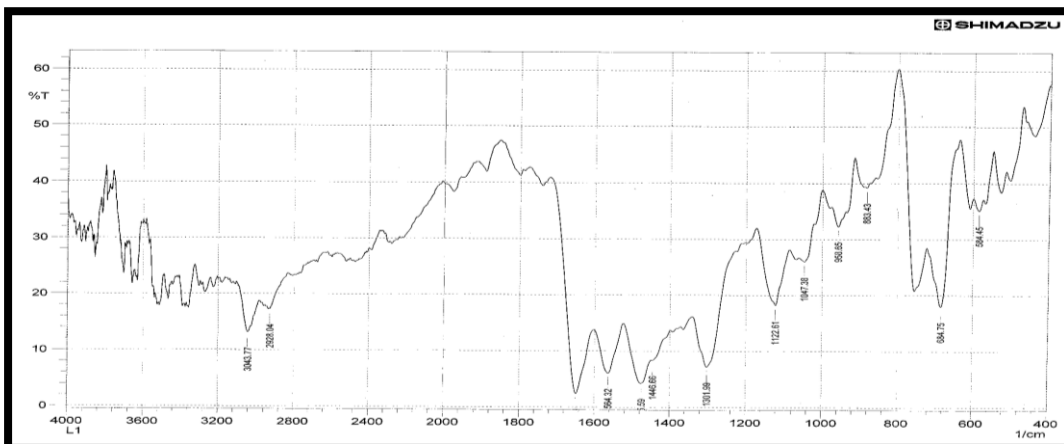
Figure(4):FT-IR Spectrum of (A1)Compound before irradiation
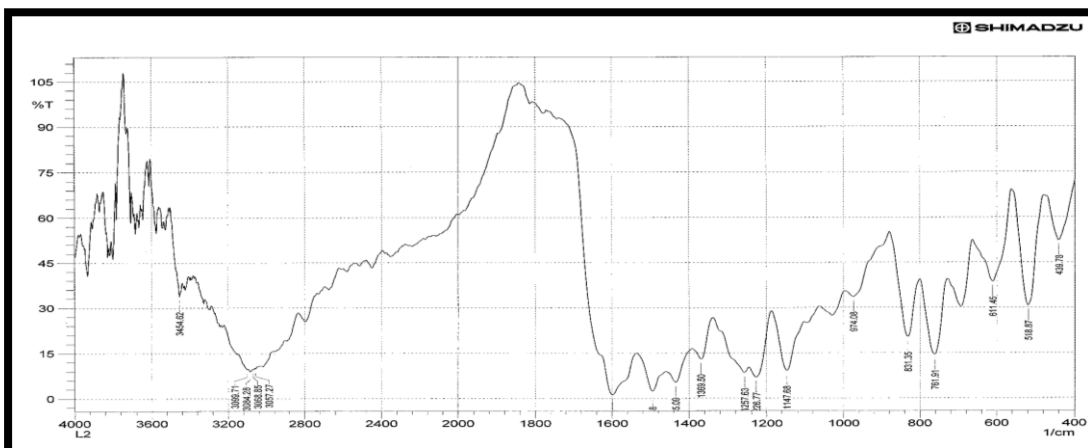
Figure(5):FT-IR Spectrum of (A2) Compound before irradiation
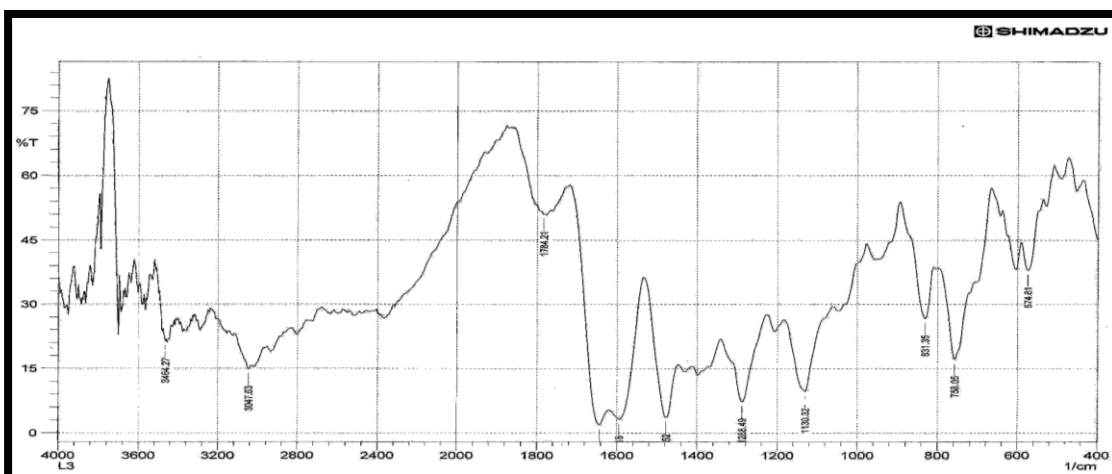
Figure(6):FT-IR Spectrum of (A3) Compound before irradiation
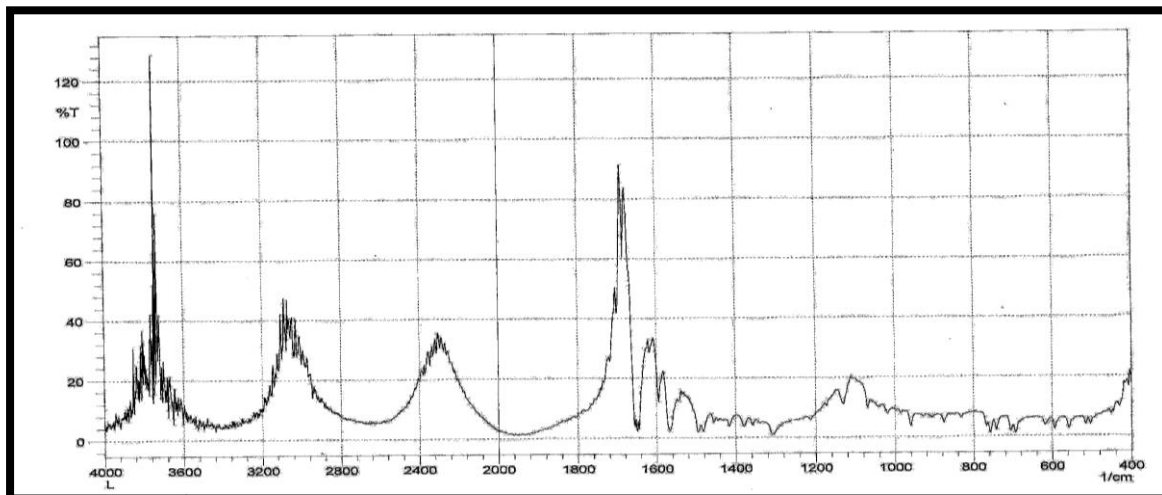
Figure(7):FT-IR Spectrum of(A1)Compound after irradiation
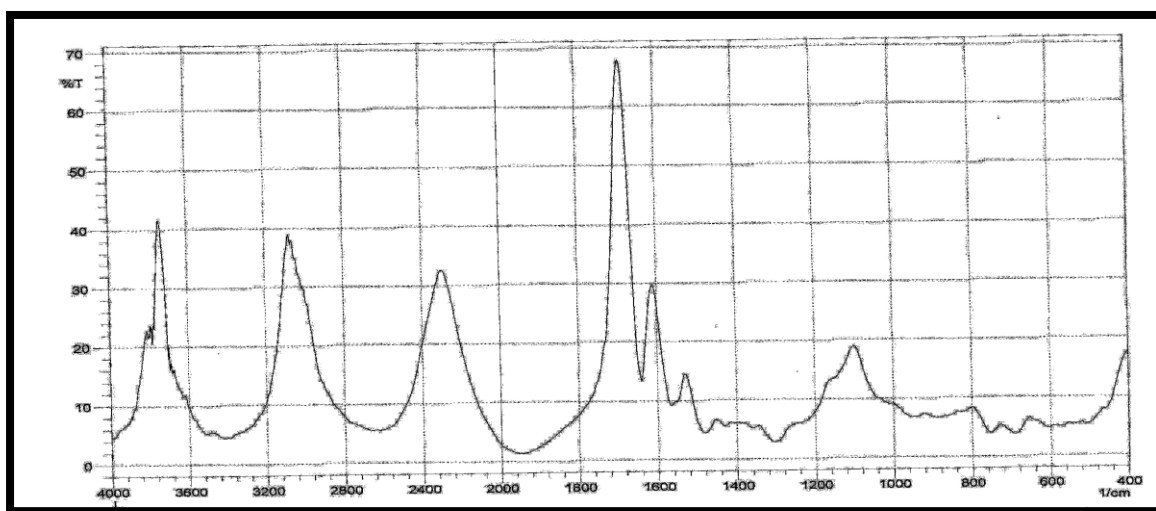
Figure(8):FT-IR Spectrum of (A2)Compound after irradiation
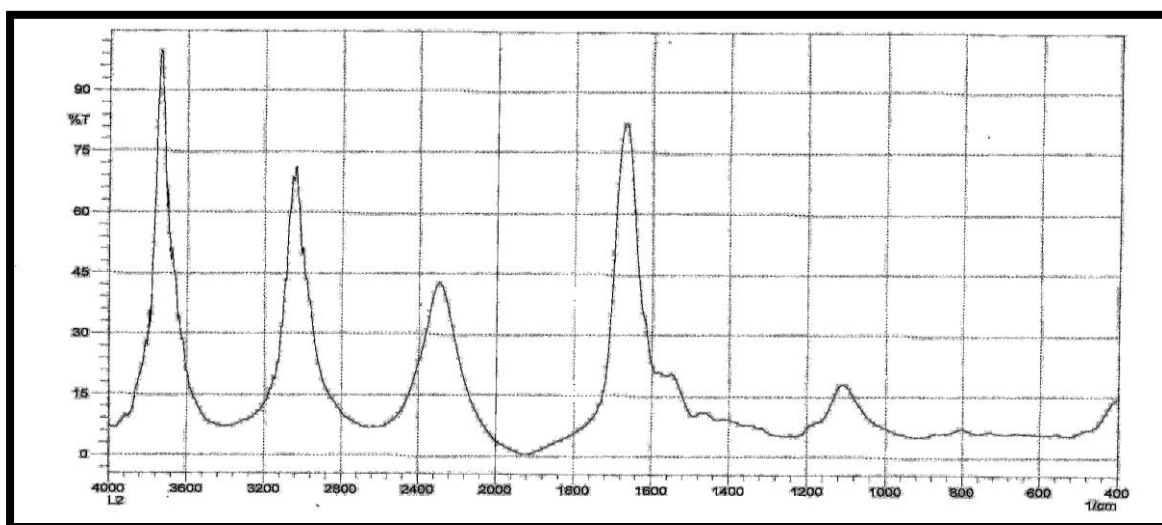
Figure(9):FT-IR Spectrum of(A3) Compound after irradiation
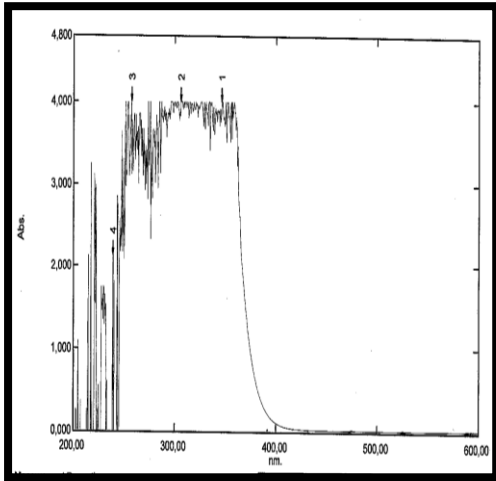
Figure(10):UV-Vis.Spectrum of(A1)
Before irradiation
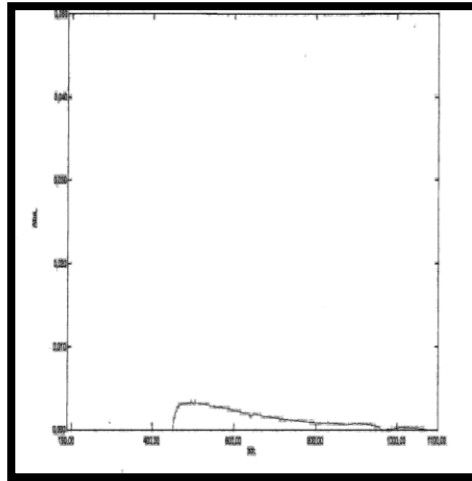
Figure(13):UV-Vis.Spectrum of(A1)
After irradiation
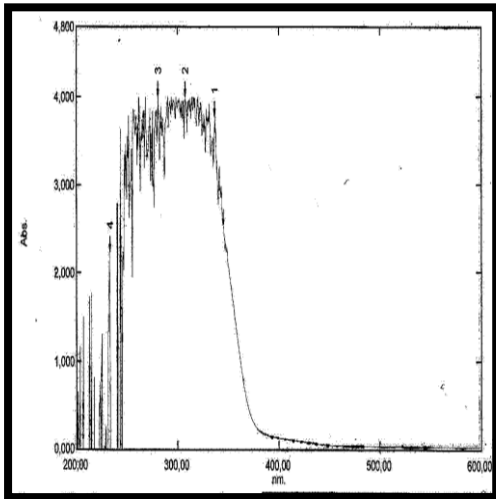
Figure(11):UV-Vis.Spectrum of(A2)
Before irradiation
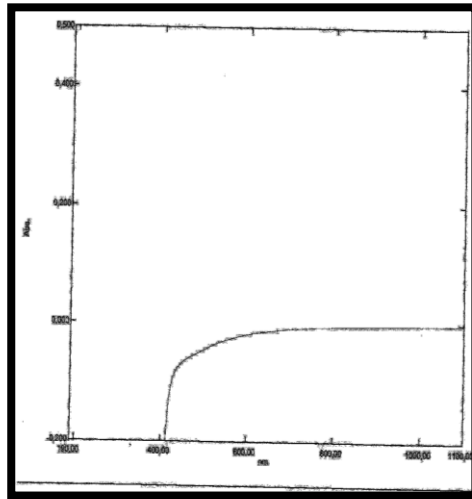
Figure(14):UV-Vis.Spectrum of(A2)
After irradiation
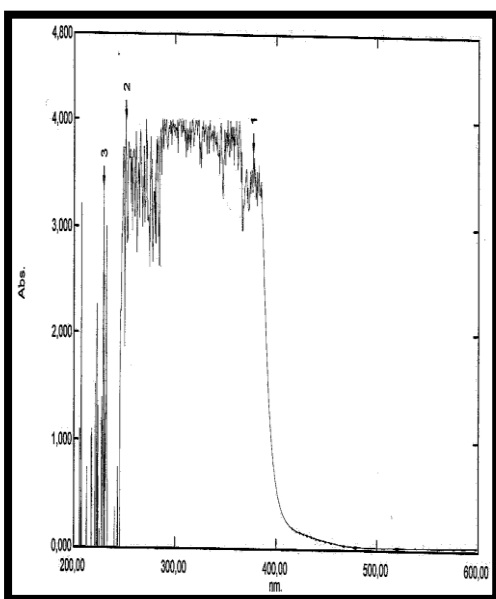
Figure(12):UV-Vis.Spectrum of(A3)
Before irradiation
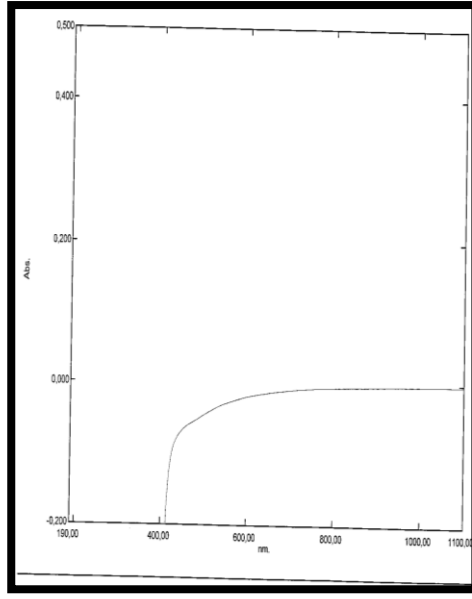
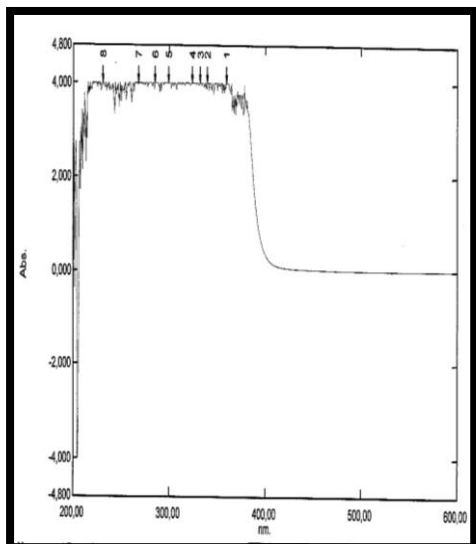Figure(15):UV-Vis.Spectrum of(A3)
After irradiation
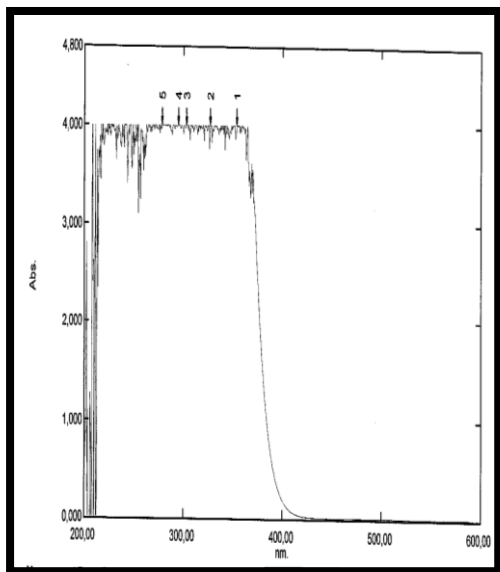
Fig.(16):UV-Vis.Spectrum of (A1)
In Methanol
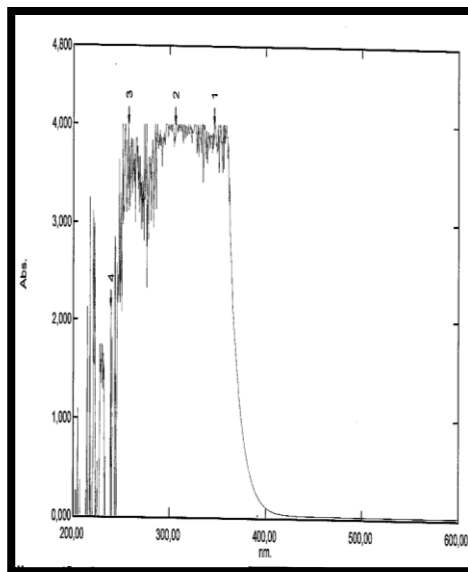
Fig.(10):UV-Vis.Spectrum of(A1)
In Ethanol
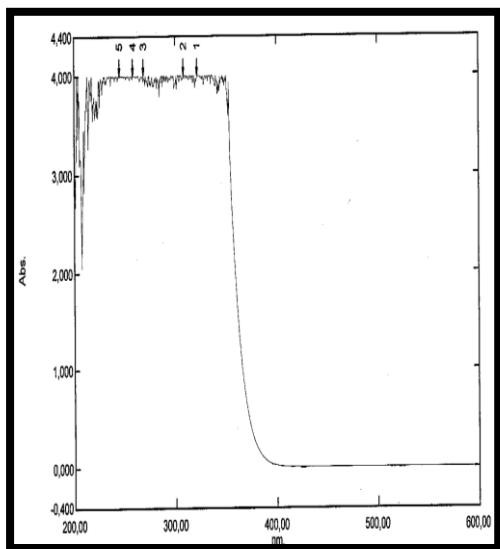
Fig.(17):UV-Vis.Spectrum of (A1)
In Cyclohexane
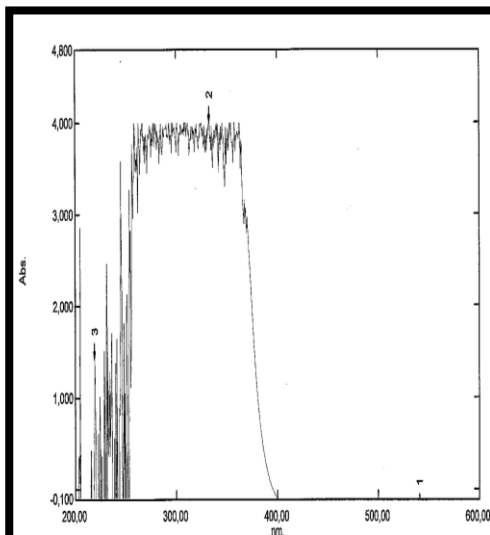
Fig.(18):UV-Vis.Spectrum of (A1)
In CCl_4
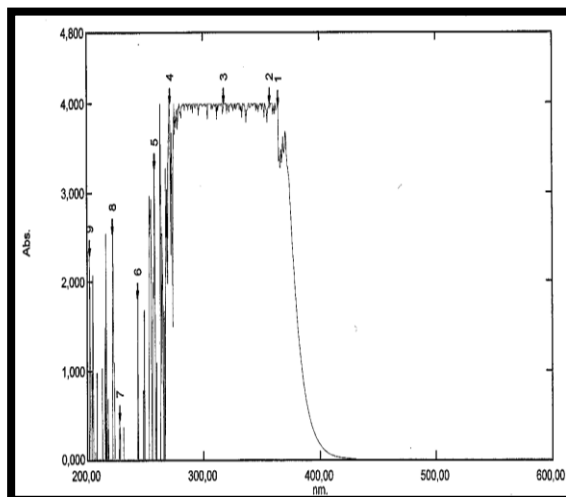
Fig.(19):UV-Vis.Spectrum of(A1) In Benzene
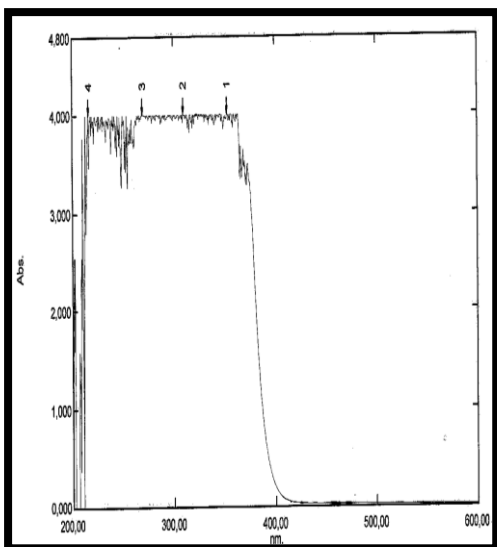
Fig.(20):UV-Vis.Spectrum of (A2)
In Methanol
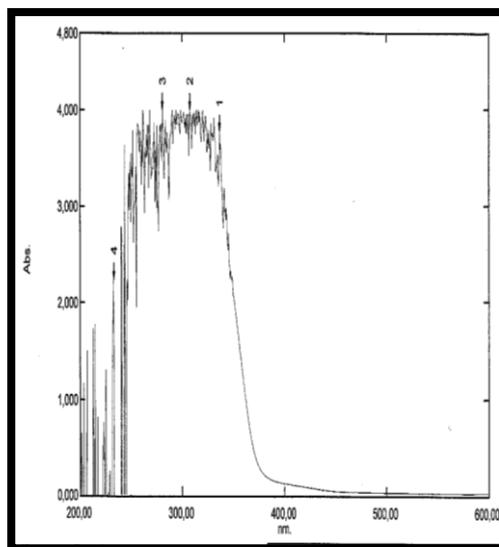
Fig.(11):UV-Vis.Spectrum of(A2)
In Ethanol
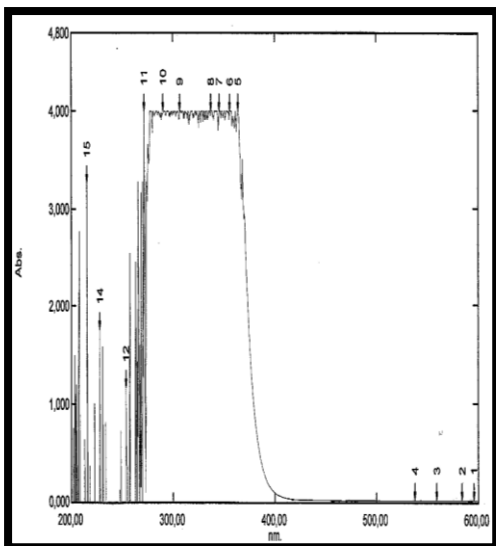
Fig.(22):UV-Vis.Spectrum of(A2)
In Cyclohexane
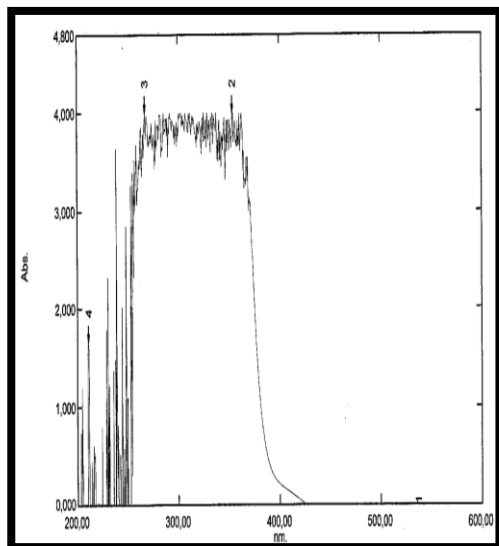
Fig.(21):UV-Vis.Spectrum of (A2)
In CCl$_4$
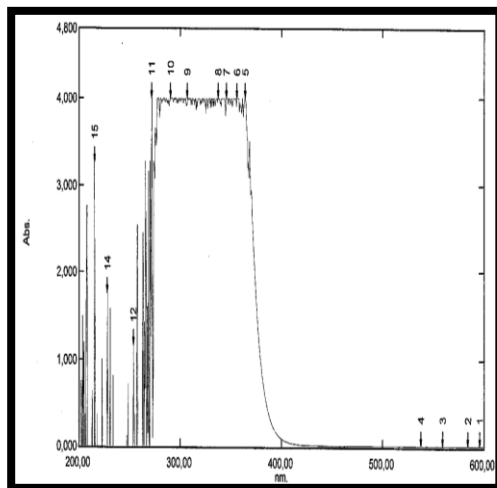
Fig.(23):UV-Vis.Spectrum of(A2)In Benzene

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Fig.(24):UV-Vis.Spectrum of (A3)
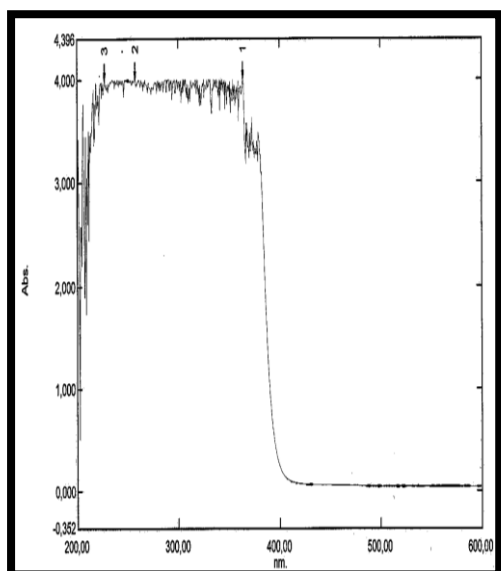In Methanol
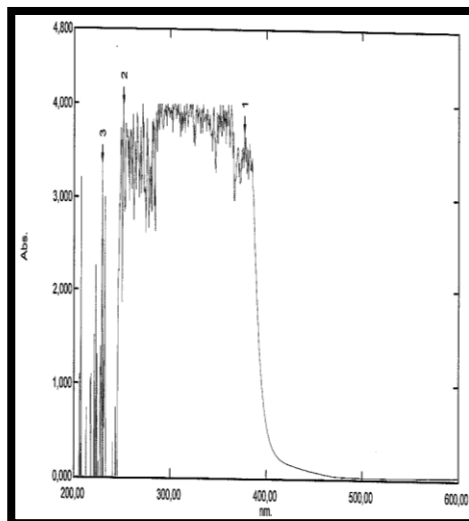
Fig.(12):UV-Vis.Spectrum of(A3)
In Ethanol

Fig.(25):UV-Vis.Spectrum of (A3)
In Cyclohexane

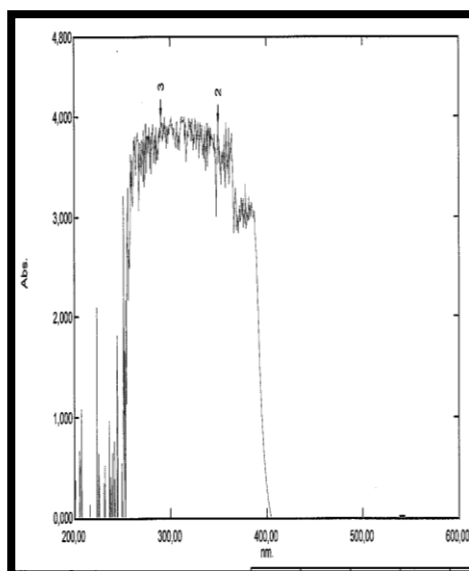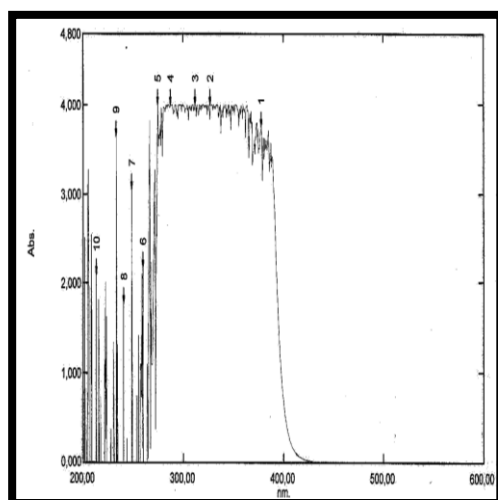Fig.(26):UV-Vis.Spectrum of(A3)
In CCl$_4$

Fig.(27):UV-Vis.Spectrum of(A3) In Benzene