Chapter One

1. Introduction:

1.1. Multicomponent Reaction:

The multi component reactions (MCRs) are highly important reaction for organic chemists especially in medicinal chemistry field (Srivastava, 2013).

In MCRs three or more reactants come together in a single reaction vessel to form a new product that contains portion of all the components (Jalpa et al, 2012).

1.1.1 The Biginelli Reaction:

In 1893, an Italian chemist Pietro Biginelli from University of Florence reported one of the most important multicomponent reactions that allow the synthesis of Dihydropyrimidinones and their sulfur analogues for the first time, which has given birth to this Biginelli reaction. In honor to his novel and excellent discovery this reaction is named as Biginelli reaction after him. To achieve this reaction, he carried out a one-pot, three component, acid catalyzed cyclo condensation reaction of 1, 3-dicarbonyl compounds, aldehyde and urea or thiourea by simply heating a mixture of the three components dissolved in ethanol with a catalytic amount of HCl at reflux temperature. On cooling he got a solid crystalline product of 3,4-dihydropyrimidin-2(1H)-one 6, represented as below.(Jadhav et al, 2012).

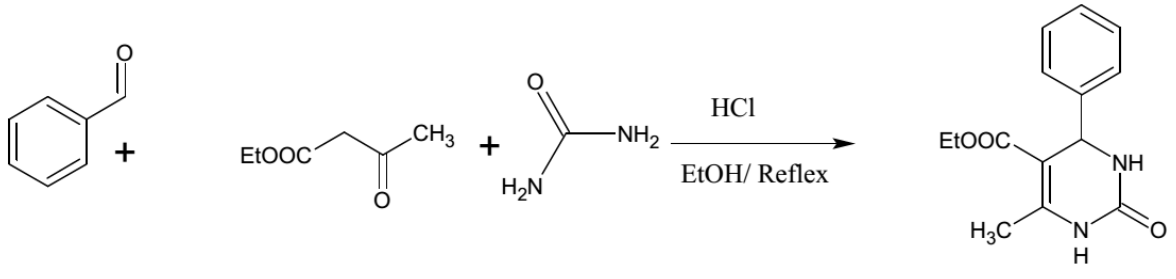


Fig. 1.1.1 Classical synthesis of Biginelli reaction

1.1.2.Mechanistic Studies:

The mechanism of the Biginelli reaction has been the subject of some debate over the past decades. Early work by Folkers and Johnson suggested that bisureide, i.e. the primary bimolecular condensation product of benzaldehyde (2) and urea (3), is the first intermediate in this reaction. (KAPPE, 2000).

Folkers and Johnson based their mechanistic conclusions on reaction yields and visual observation. They proposed that the simultaneous combination of the three reaction components in A was improbable. D was ruled out on the basis of the low reaction yields (2%). In contrast, Band C gave high yields of 6 (80%). The authors note that B may undergo fragmentation of the benzal-bisurea, regenerating the three reaction components, which may then form the product by another pathway. Further, the authors posit that the β-carbamidocrotonate in C hydrolyzes to the original three reaction components. Therefore, they conclude that is likely formed from cyclization, which can be generated from either B or C.(Eric, 2008).

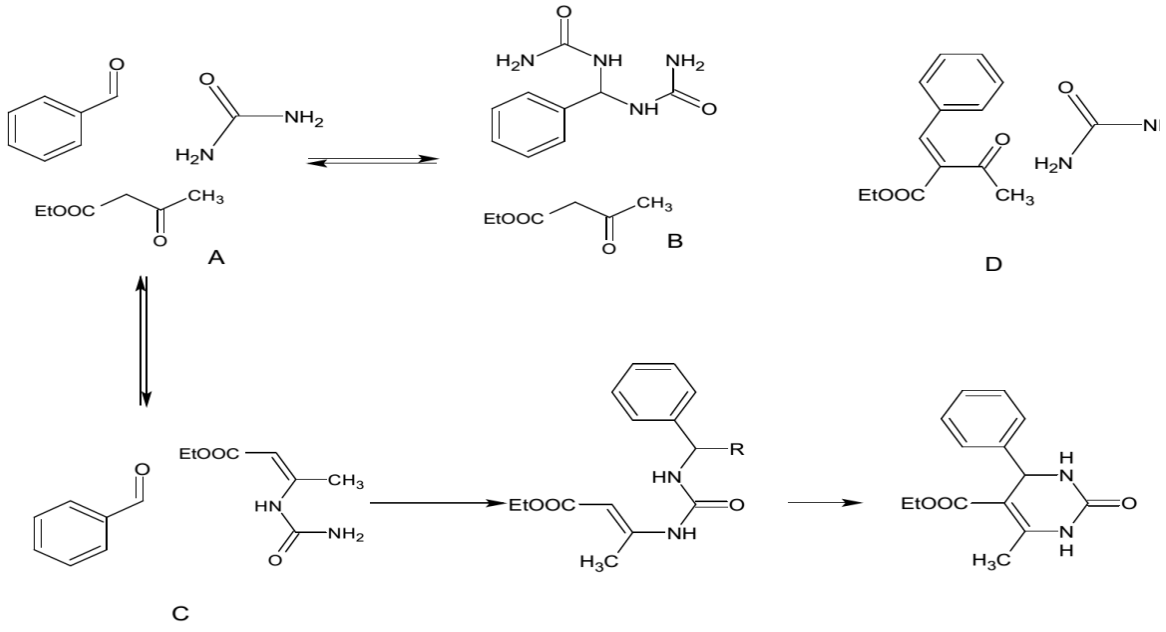


Fig.1.1.2.Folkers and Johnson mechanism

After several decades the reaction was reinvestigated by Sweet and Fissekis who advocated contradictory mechanism to Folkers, proceeding through Aldol reaction (through carbenium ion intermediate) . (Suresh et al ,2012).

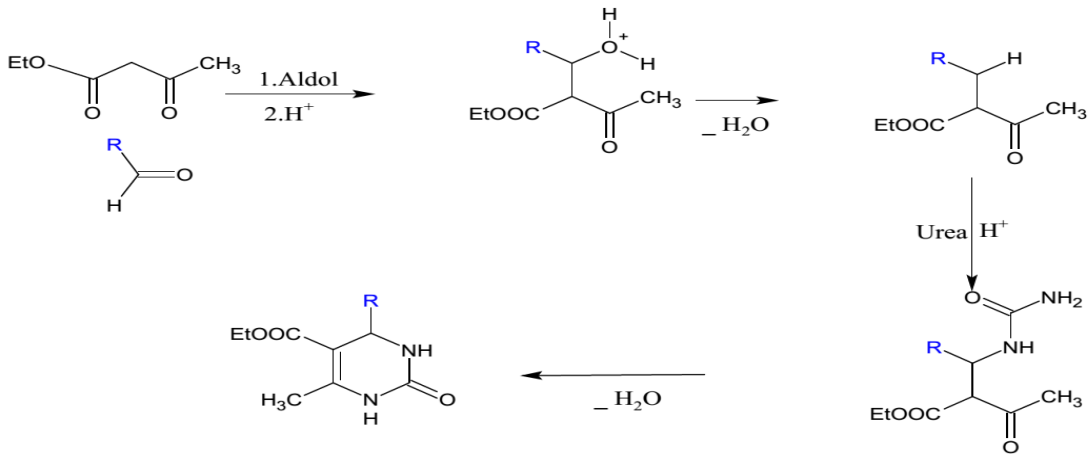


Fig. 1.1.3.Formation of 3,4-dihydropyrimidinon via Aldol condensation

Kappe further explored the mechanism of the Biginelli reaction using NMR spectroscopy and trapping experiments. He proposed the formation of N-acyliminium **2** from benzaldehyde and urea via an unobservable (1H NMR) hemiaminal **1.** Interception of **2** with the enol tautomer of ethyl acetoacetate gave 3, the precursor to dihydropyrimidine. Kappe suggested that the first step, formation of **1**, is rate limiting, thus preventing the observation of intermediates **2** and **3** by NMR. Kappe's proposal is currently the accepted mechanism for the Biginelli reaction. (Phucho et al, 2009).

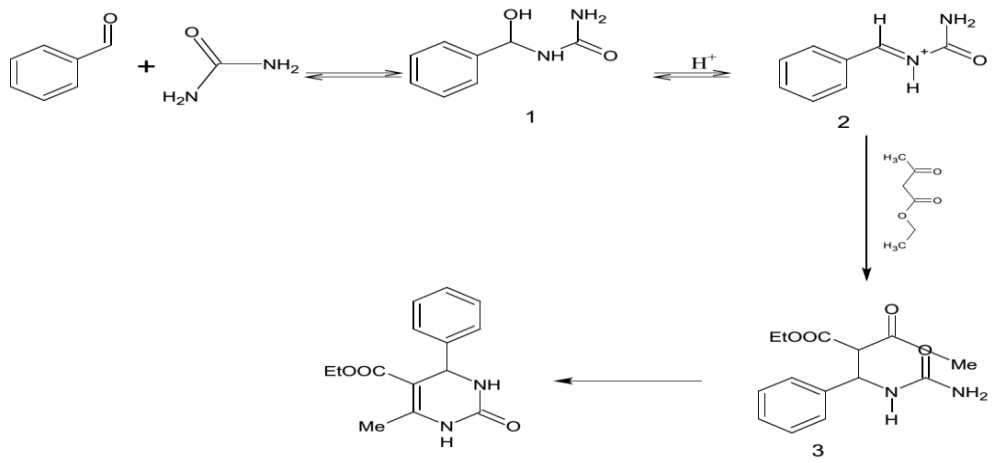


Fig.1.1.4. Kappe mechanism

1.1.3. Modification of Biginelli reaction

Atwal modification of the Biginelli reaction that an enone is first condensed with a suitable protected urea or thiourea derivative21under almost neutral conditions. Deprotection of the resulting 1,4-dihydropyrimidine by HCl or TFA/EtSH (KAPPE,2000).

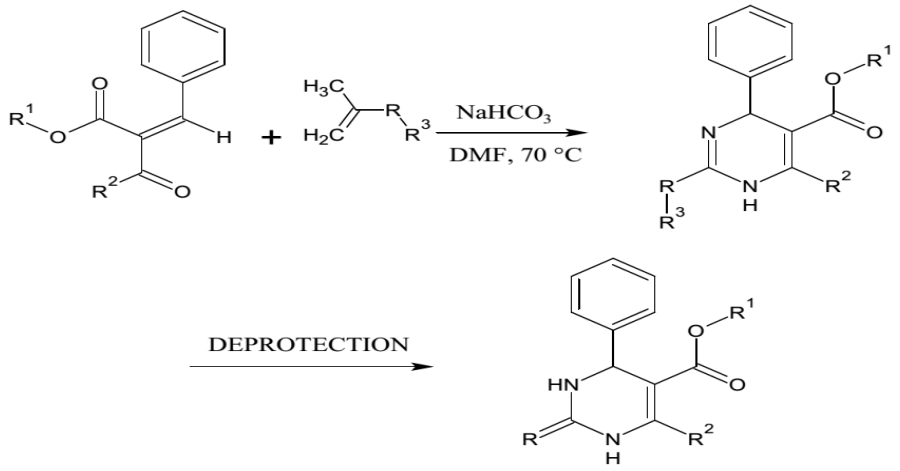


Fig.1.1.5. Atwal modification of Biginelli reaction

Another approach to the Biginelli developed by Shutalev that condensation of α-tosyl-substituted urea's which reacted with acetoacetic ester and aldehydes (Shutalev et al, 1998).

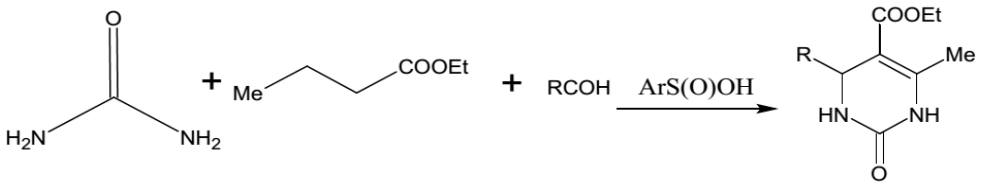


Fig. 1.1.6 Shutalev modification

1.1.4. Reaction Advancements:

Improved Protocol for Biginelli Reaction The utilization of (MCRs) to synthesize novel , drug-like scaffold has permeated in organic transformations from last few decades due to their molecular economy and potential of producing diversified product in single step.(Hegde et al, 2015).

Dihydropyrimidinones and their derivatives have attracted increasing interest owing to their therapeutic and pharmaceutical properties, such as antiviral, antibacterial, anti-inflammatory and antitmour activities. Recently, functionalized Dihydropyrimidinones have been successfully used as antihypertensive agents, calcium channel blockers, adrenergic and neuropeptide Y (NPY) antagonists. In addition, some alkaloids containing the dihydropyrimidine core unit which also exhibit interesting biological properties have been isolated from marine sources. Most notably, among these are the batzelladine alkaloids, which were found to be potent HIV gp-120-CD4 inhibitors. The original protocol for the synthesis of Dihydropyrimidinones, reported by Biginelli in 1893, involves a one-pot reaction of benzaldehyde, ethyl acetoacetate and urea in ethanol under strongly acidic conditions. However, this method suffers from drawbacks such as low yields

(20-40%) of the desired products, particularly in case of substituted aldehydes, and loss of acid sensitive functional groups during the reaction. This has led to multi-step synthetic strategies that produce somewhat better yields, but which lack the simplicity of the original one-pot Biginelli protocol. The search for more suitable preparation of Dihydropyrimidinones continues today.

Recently, many synthetic methods for preparing these compounds have been developed to improve and modify this reaction by using Lewis acid catalysts as well as protic acids.(Jetti et al, 2012).

1.1.5.Asymmetric Biginelli Reaction :

Several methods have been developed for the asymmetric synthesis of enantioenriched dihydropyrimidines. The first of these methods to give synthetically useful enantiomeric ratios was reported by Zhu and coworkers in 2005, over one-hundred years after discovery of the Biginelli reaction. Zhu found that the use of chiral ytterbium catalyst allowed for dihydropyrimidines to be Synthesized in high yield and enantioselectivity. The ytterbium catalyst is recoverable and can be recycled several times without diminishing the product. (Woerly, 2008).

1.1.6. Reaction conditions:

The reaction was carried out by simple heating a mixture of three components at reflux temperature in solvent such as ethanol with a catalytic amount of HCl. (Trivedi et al, 2012), THF-benzotrifluoride (BTF) containing HCl (KAPPE, 2000), acid(Aslam et al, 2012), Brønsted acidic ionic liquid [Btto][p-TSA] (Zhang et al, 2015), pineapple juice(Patil et al, 2011), $SnCl_2.2H_2O$(Dennis Russowsky et.al, 2004) potassium tertiary butoxide (Hegde et al, 2015), $H_3PMo_{12}O_{40}$(Salehi et al, 2010), crushed garlic clove(Abd-Elnabi et al, 2013), DBSA (Aswin et al, 2014),

PTC (250 mg of TBAC, 290 mg of TBAB, 0.9 mmol) anions (Cl−and Br-) (Slimi etal,2013), Montmorillonite K-10 (Saeed et al,2010), anhydrous ferrous sulphate (Patil et al, 2011).

1.1.7. Biological Activity of Dihydropyrimidines

Taking into account of biological aspect of Biginelli product i.e. dihydropyrimidines, intensive investigation is carried out because they possess close resemblance of clinically used nicardipine, nifedipine etc. which are analogues of Biginelli product further they had resemblance to marine natural alkaloids batzelladine. Again biologists & chemists synthesized modified Biginelli product scaffolds, which showed activities like antiproliferative, antiviral, antitumor, anti-inflammatory, antibacterial, antifungal and antitubercular activity.

Similarly, the structural core of quinoline is frequently associated with medicinal applications such as anticancer, antimicrobial, HIV-1 integrase inhibition, HIV protease inhibitors, antileishmanial activity, NK-3 receptor antagonists, PLT antagonists and antimalarial activity. In search of more potent and effective medicinally important molecules numerous Biginelli dihydropyrimidine related annulated or multifunctionallized pyrimidine heterocyclic have been investigated & tested against different life threatening diseases. To address its biological utility only selective molecules are presented which are having significant activity and they are examined with clinically used drugs in vivo /in vitroand establishing QSAR studies (Jadhav et al,2012).

1.2. β-diketone:

The importance of 1, 3-diketones in synthetic organic chemistry is difficult to overestimate. Their accessibility, stability, and often unique properties make them promising for use in various fields of human activity. High reactivity of 1, 3-

diketones opens wide prospects for the design of a variety of organic compounds, including those structurally related to natural ones. Continuously growing interest in β-dicarbonyl compounds is observed among researchers working in various fields of medicinal chemistry and chemistry of metal complexes. Sol–gel syntheses with β-diketones afforded organic–inorganic hybrid materials used in gas sensors and molecular thermometers, as well as in the manufacture of optical fiber and light converting materials.

Over the past 10–15 years, not only selectivity parameters and overall yield of reaction products but also such factors as enhanced requirements to starting materials, reaction time, energy consumption, toxicity, etc., have acquired increasing significance in the assessment of the efficiency of chemical syntheses.

1,3-Diketones turned out to be excellent versatile intermediates in multicomponent reactions, in particular regio- and stereo selective, which is especially important in the synthesis of potentially biologically active compounds.

1.2.1. Synthesis of β-diketones

Analysis of published data showed that currently, as well as in the past century, the main method for the synthesis of β-diketones is based on the Claisen condensation which has been known since 1887. It implies acylation of monocarbonyl compounds (most frequently, ketones) in the presence of catalysts favoring their enolization Claisen condensations with dicarbonyl compounds (acetyl acetone, benzoylacetone) are very few in number.

*Syntheses of β-Diketones Based on the Claisen Condensation.

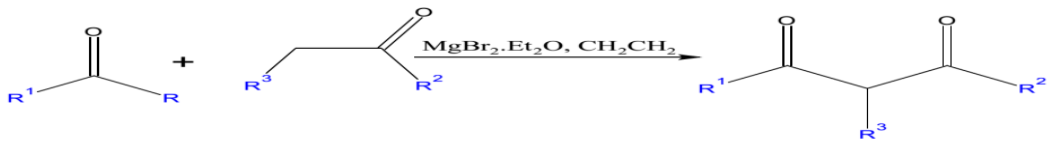


Fig.1.2.1. Acylation of ketones.

1, R= 1H-benzotriazol-1-yl (a), C6F5O (b); R1= Me3CCH2, t-Bu, Ph, PhCH(OSiMe2Bu-t),Bu,CH2=CHCH2CH2,(E)PhCH=CH;

2, R2= H, R3= 2-MeO (HO)C6H4, 4-MeOC6H4, furan-2-yl; R2=Me, R3= Ph, (E)-PhCH=CH, Et; R2= OSiMe2Bu-t, R3= Ph; R2R3=CH(Me)(CH2)2CH2, (CH2)4.

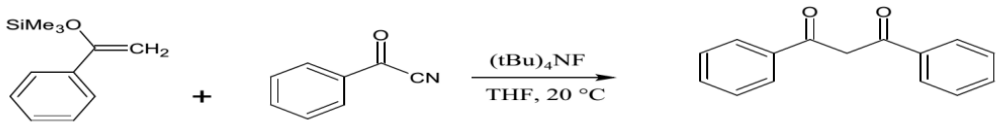


Fig. 1.2.2.Acylation of silyl enol ethers.

* Dicarbonyl compounds in the synthesis of β-diketones.

* Acetylacetone in the synthesis of β-diketones. The simplest β-diketone, acetyl acetone, was converted into a large number of various β-diketones as a result of three-component carbonylation–α-arylation with aryl bromides Carbon (II) oxide was generated ex situ from 9-methylfluorene-9-carbonyl chloride in the course of the process.(Shokova et al ,2015).

* synthesis of benzoylacetone (Furniss et al ,1989).

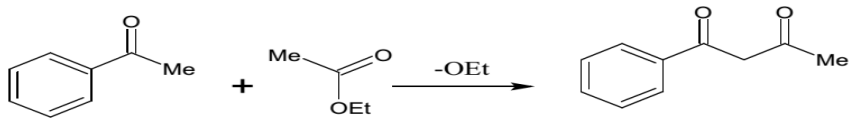


Fig. 1.2.3. synthesis of benzoylacetone

1.2.2. Reaction of β diketones:

1,3-Dicarbonyls, such as 1,3-diketones and 1,3-ketoesters, react directly with elemental fluorine at room temperature to give the corresponding 2-fluoro- and, in some cases, 2,2-difluoro-compounds(Chambers et al ,1995).

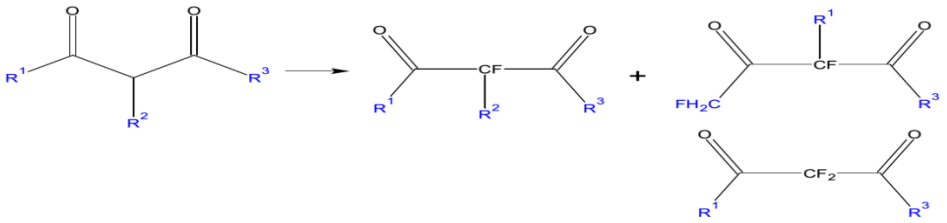


Fig. 1.2.4. formation of 2-fluoro- and 2,2-difluoro-compounds

R^1= Me; R^2= H, Me ,C ; R^3= Me, OEt

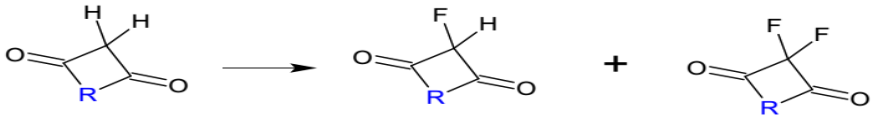


Fig. 1.2.5. Reaction of elemental fluorine with cyclic β-diketone

R= $-CH_2CMeCH_2-$;-NHCONH-

The reaction of acetyl acetone with the reagent prepared from POCl3 and DMF is known to give 2,4-dichlorobenzaldehyde (Nirmala,1995).

Fig. 1.2.6. preparation of 2,4-dichlorobenzaldehyde

Katritzky and Marson have reported the reaction of cyclohexane 1, 3-dione with Vilsmeier reagent lead to the formation of the imino methylene substituted cyclohexadiene dialdehyde (Nirmala, 1995)

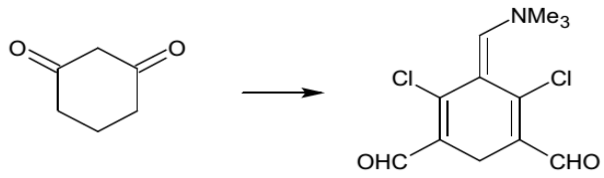
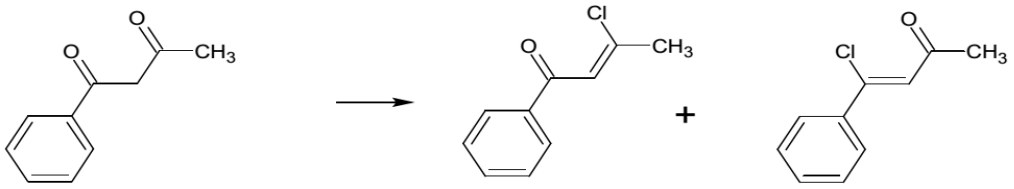


Fig. 1.2.7. formation of the imino methylene substituted cyclohexadiene dialdehyde

The reaction benzoylacetone was also attempted under similar condition but only a mixture of chlorosubstituted enones could be obtained (Nirmala, 1995)



1.3, Objective:

The objective of this research is to synthesis of some dihydropyrimidinone derivatives.

Chapter Tow

2. Materials and Methods

2.1. Materials

2.1.1. solvent

Chloroform (119.38, 1.48 g/L, 99.5% ROMIL LTD).

Ethanol (48.0, 99.7%, Sd fine-chem , India).

Methanol(32.04, (0.790-0.793) g/L, ADWIC ELNASR.

2.1.2. Chemicals

Sodium pellets, xylene, diethylether, Acetophenone (120.15, (1.025-1.027)g/L, 99%, ethylacetate, aceticacid, Benzoylacetone, Benzaldehyde, (1.044-1.047 g/cm^3, 98.5 %), Cinnamaldehyde (1.050-1.052 g/cm^3, 98 %), Salicyaldehyde (1.146 g/cm^3 , 98%), Urea (98%), zincchloride, iodine.

All chemicals were used without further purification.

2.1.3. Instruments

2.1.3.1.TLC 20×20 cm plates of aluminum precoating silica gel 60 F_{254} (magnesium activated zinc silicate)

2.1.3.2. Infra-red spectroscopy

IR spectra (ν, cm-1) were recorded on FTIR spectrophotometer obtained

from SHIMADZU in KBr pellets.

2.1.3.3. Ultraviolet Spectroscopy UV-Vis spectrophotometer were recorded on UV-1800 obtained from Shimadzu, Japan.

2.1.3.4. Mass spectrometer

Mass spectrophotometer ionization source of energy electron- impact (Shimadzu .japan).

2.1.4. General Equipment

Sensitive Balance, A&D-GR-120, japan, Hot plate with magnetic stirrer, Bibby sterilin LTD, UK, Oven, Round bottom flask (capacity 250,50 ml) and fitted with condenser, Electro thermal melting point, Gallen Kamp ,England, cylinders, Beakers, funnel and Conical flask .

2.2. Methods:

2.2.1. general procedure for synthesis of Benzoyl acetone

Sodium ethoxide 11.5 g of granulated sodium was prepared in 75 ml of xylene and transfared to round bottom flask. sodium was washed by decantation with two 20ml portion of diethyl ether and covered with 200 ml of diethyl ether. the flask was set on water bath and fit it with sealed stirrer unit and reflux condenser. The stirrer was started and run (29ml,0.5 mol)of absolute ethanol from the dropper during 1–2 hours ,and continue to reflux the mixture with stirring unit until nearly all of sodium has reacted (up to 6 hours)stirrer was stopped and condenser set downward to distil the ether as completely possible. the residual sodium ethoxide was white .

Benzoyl acetone Condenser was return to the reflux position and 200ml of dry ethyl acetate (2mol) was added and was set the ice bath, then was stirrer. (60g) (58ml 0.5mol) of acetophenone from the dropper was added. Continue stirring for 2 hours and then allow to stand overnight the solid was filtered with aid of addition of little ether. the dried solid was dissolved in cold water and acidify with acetic acid . crude benzoyl acetone was filtered and dried in air .

2.2.2. General Procedure for synthesis of 5-methyl-6-phenyl-4aryl-1,3-dihydropyrimidin-2-one (I,II,III,IV)

In 50ml round bottom flask equipped with reflux condenser were placed.

a mixture of aromatic aldehyde (0.01mol) , benzoyl acetone (0.01mol), urea (0.01mol 0.6g), zinc chloride (0.01mol 1.36g) as a catalyst and absolute ethanol (5ml) . mixture was heated with stirring under reflux for 7 hours. The progress of reaction was monitored by thin-layer chromatography, after cooling the reaction

mixture was poured onto crushed ice with stirring. The crude product was filtered, washed with cold water, dried and recrystallized from That by used different aromatic aldehyde in each reaction.

2.5-Synthetic method

Synthesis of 5-benzoyl-6-methyl-4-aryl-3,4-dihydropyrimidin-2-one(I,II,III,IV) from benzoylacetone with urea and different aromatic aldehydes.(Fig. 2.5.1)

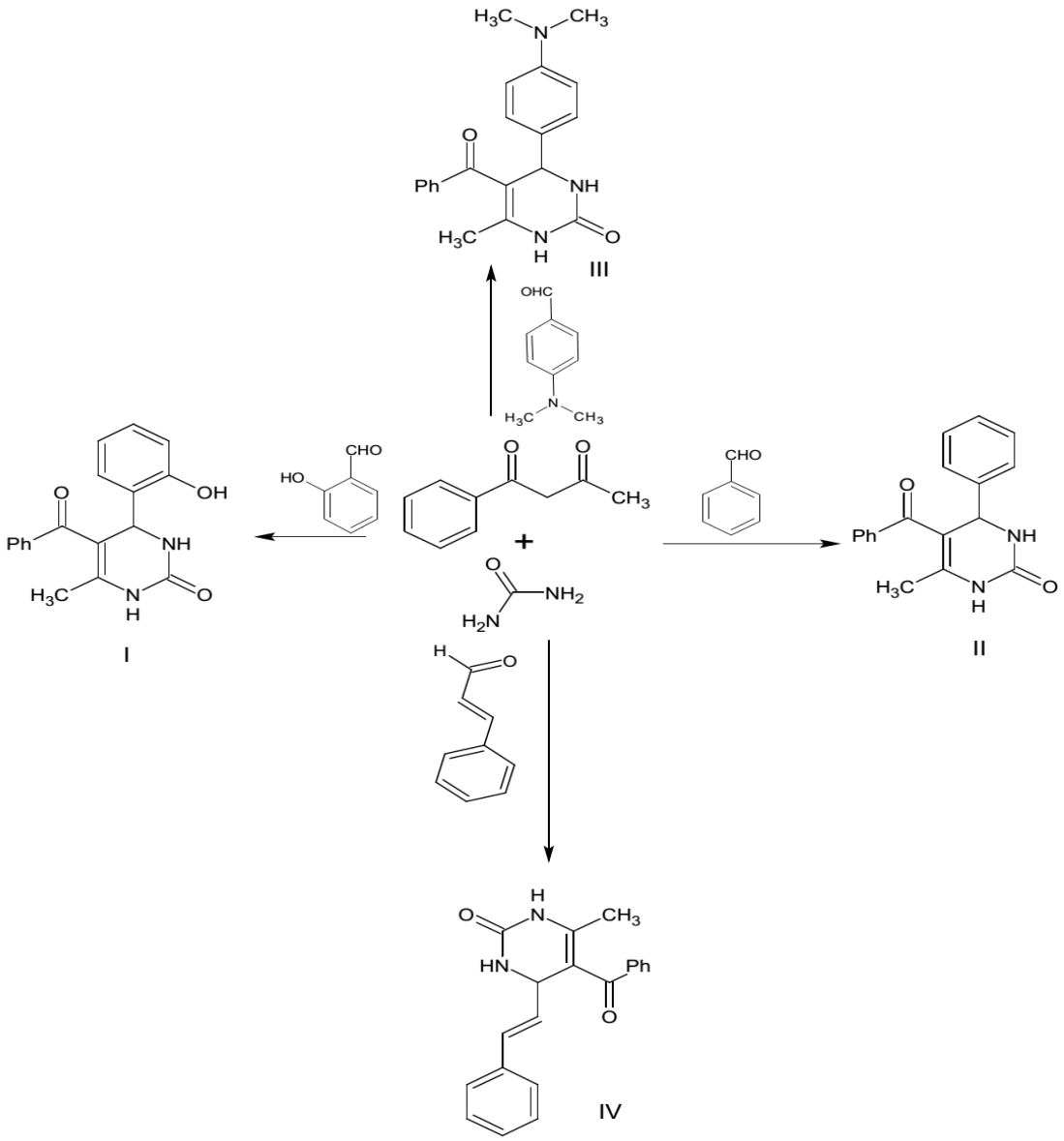
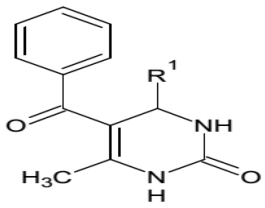
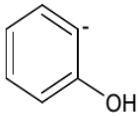
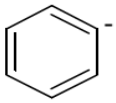
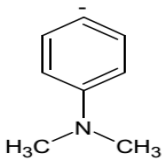
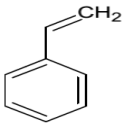


Fig. 2.5.1. chemical reaction of benzoylacetone with urea and different aldehydes .

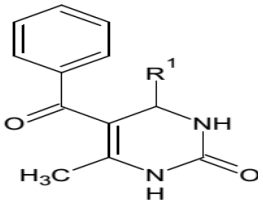
Table (2.5.1) Chemical name of the prepared compound:

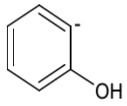
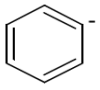
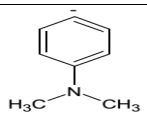
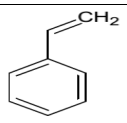


Compound No.	**R^1**	**Chemical name**
i.		5-benzoyl-4-(2-hydroxyphenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one
ii.		5-benzoyl-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1*H*)-one
iii.		5-benzoyl-4-[4-(dimethylamino)phenyl]-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one
IV.		5-benzoyl-6-methyl-4-[(*E*)-2-phenylethenyl]-3,4-dihydropyrimidin-2(1*H*)-one

2.6. Reaction conditions

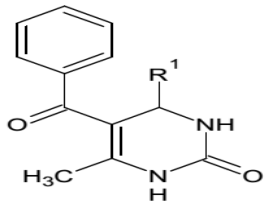
Table (2.6.1) Reaction condition of 5-benzoyl-6-methyl-4-aryl-3,4-dihydropyrimidine-2-one (I,II,III,IV)

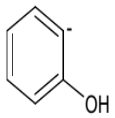
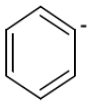
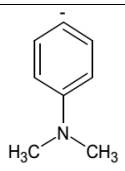
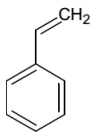


Comp. No	R^1	X	Reaction Temp C°	Time h	Yield %	Yield gram	Color	m.p C°
I		O	Reflux temperature	7	49.85	1.68	Brown	Viscous
II		O	Reflux temperature	7	35.17	1.027	Pale yellow	270-272
III		O	Reflux temperature	7	84.03	2.815	Pale yellow	72-74
IV		O	Reflux temperature	7	47.23	1.502	Pale yellow	200-204

2.7. IR of synthesized compound

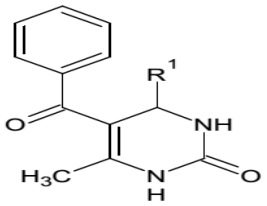
Table (2.7.1) Infra-red spectral data of the 5-benzoyl-6-methyl-4-aryl-3,4-dihydropyrimidine-2-one (I,II,III,IV) IR spectra ,wave number cm^{-1} ,of synthesized compounds were obtained by preparing KBr pellet

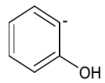
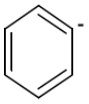
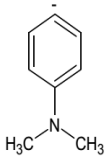
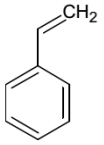


Comp. No	R^1	N-H st.vib	C=O st.vib	C-H st.vib	Other
I		3357.84	1652.88	3357.48	3456.20 (-OH) 1577.66 (C=C)
II		3452.34	1562.23	2927.74	715.54 ben (ph-) 1598.88 st. (C=C)
III		3089	1660	3047	1232 (N-C)
IV		3230.54	1687.60	3085.89	1525.59 (C=C)

2.8.UV of synthesized compound

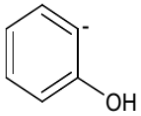
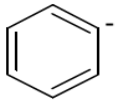
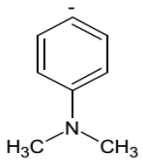
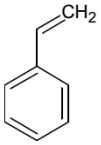
Table (2.8.1) Ultra violet spectroscopy data of the 5-benzoyl-6-methyl-4-aryl-3,4-dihydropyrimidine-2-one (I,II,III,IV)



Comp. No	R^1	λ_{Max}
I		296
II		284
III		346
IV		296

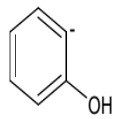
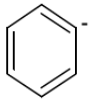
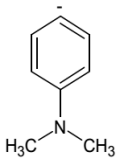
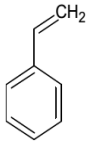
2.9.Mass of synthesized compound

Table (2.9.1) mass spectral data of the 5-benzoyl-6-methyl-4-aryl-3,4 dihydropyrimidine-2-one (I,II,III,IV)

Comp. No	R^1	Base peak (m/z)	Fragments (m/z)
I		281	77, 183,139, 295
II		129	55,85,142,
III		254	85,118,134,227
IV		270	86,104,226, 244,313

2.10. Retention factor for synthesized compounds

Table (2.10.1)TLC data of 5-benzoyl-6-methyl-4-aryl-3,4-dihydropyrimidine-2-one (I,II,III,IV) by thin layer chromatography 20x20 plates of aluminum precoating silica gel G F_{254} (magnesium activated zinc silicate)

No.	R^1	Solved system	Retention factor
I		Chloroform: methanol (4:1)	0.7
II		Chloroform: methanol (4:1)	0.5
III		Chloroform: methanol (4:1)	0.6
IV		Chloroform: methanol (4:1)	0.8

Chapter There

3. Discussion

3.1.Retro synthetic analysis (RSA):

Retro synthetic analysis (RSA), aid in the establishment of good synthetic scheme in (RSA), key steps are developed by examine important structural element in the final product and figuring out how specific reaction could lead to the product. The procedure is performed it relatively so that a complex final molecule is reduced to simpler intermediates. The advantage of such an approach is that greatly simplifies planning the synthesis of a complex product and readily leads to convergent synthesis. (Golan, 2008).

In performing (RSA), it may also be useful to disconnect a bond showing the fragment not as real compound but only as an electrophile and nucleophile (synthons) his may help bring to mind other reaction that can be used to reassemble the fragment.(Hornback, 2005).

The dihydropyrimidinones is a heterocyclic with tow hetero atoms, it is useful to look for recognizable fragments containing both the ring of (DHPM) can be disconnected to urea and suitable electrophilic fragment.

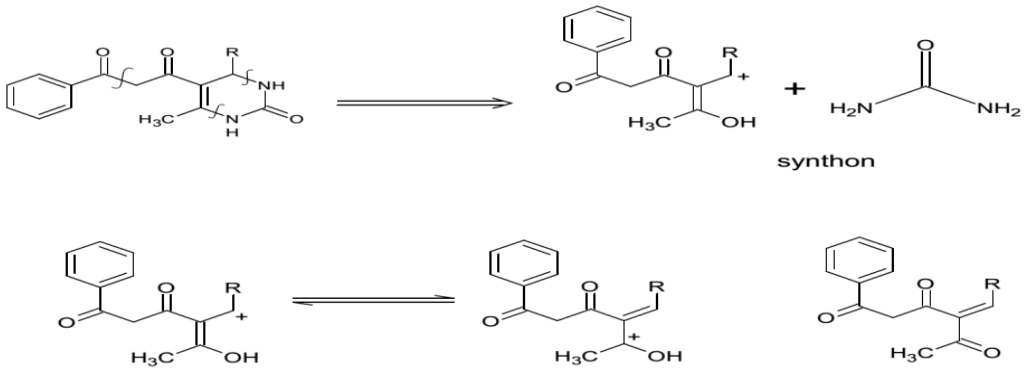


Fig (3.1.1.) retro synthetic analysis of 5-benzoyl-6-methyl-4-aryl-3,4-dihydropyrimidine-2-one (I,II,III,IV)

The full analysis of electrophilic fragment which containing an enone could be obtained by FGI followed by C-C disconnection.

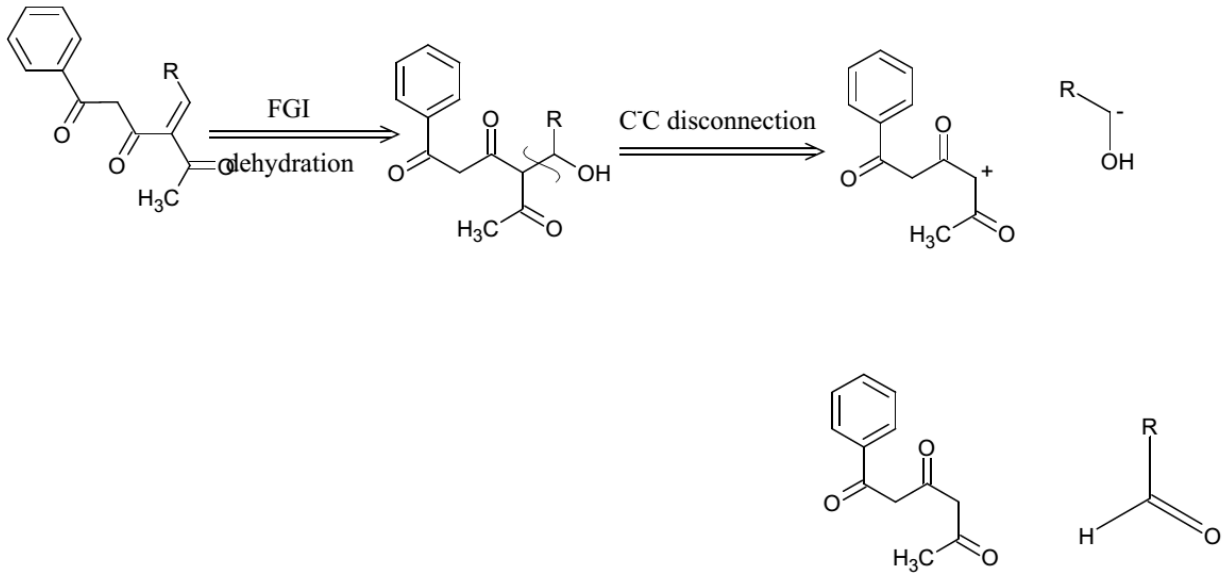


Fig (3.1.2.) full analysis of 5-benzoyl-6-methyl-4-aryl-3,4-dihydropyrimidine-2-one (I,II,III,IV)

3.2. Reaction mechanism:

The reaction started with Aldol addition and followed by dehydration to form the α, β unsaturated compound which react as electrophile with nucleophilic site of $–NH_2$ group in urea . the product will cyclize by direct addition to second $–NH_2$ group of urea to the carbonyl group of ketone to close the ring of target molecule.

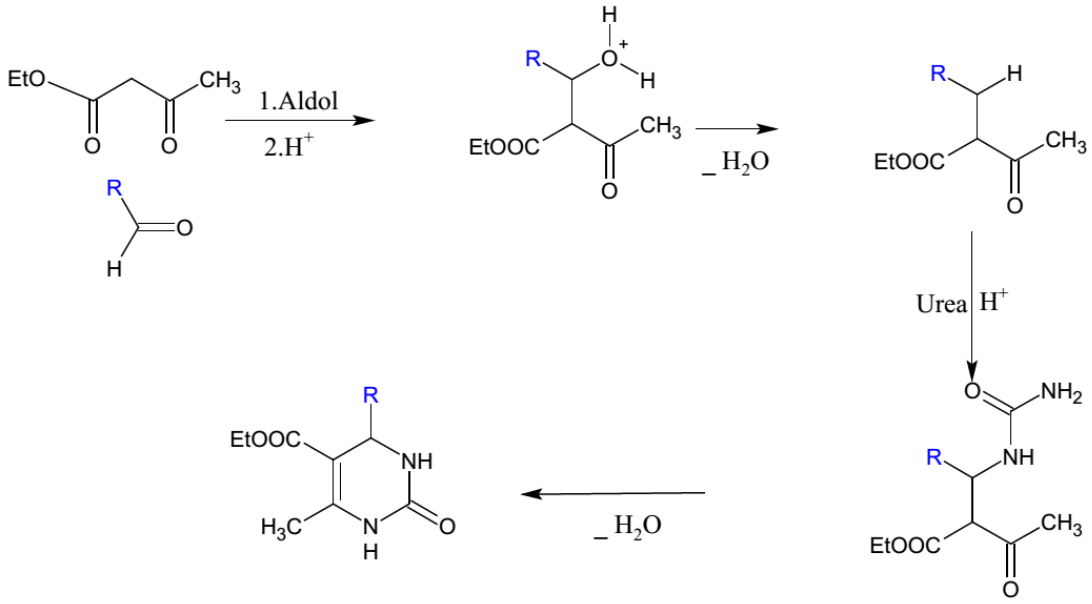


Fig. 3.2.1. reaction mechanism of dihydropyrimidinone derivatives

3.3 Spectral Characterization:

According to the general procedure DHPM KT-01 was obtained from benzoylacetone , benzaldehyde and urea.

The structure of DHPMs are confirmed by FT-IR, and Mass spectroscopy.

The most synthesized dihydropyrimidinone derivatives gave higher melting point between 200-272 but (iii) show less melting point.

The present product was determined by analysis of functional group with IR spectra and recorded with FT-IR 8400s using KBr disk .its showed sharp peaks in (3456.20-3230.54cm^{-1}) for N-H st.vib in pyrimidinone ring except compound (III) show weak peak in 2916.17. the C=O appear at (1687–1562 cm^{-1}), the of all compound show absorption at (1625 -1525 cm^{-1}) due to C=N group. the compound 5-benzoyl-4-(2-hydroxy phenyl)-6-methyl-3,4-dihydropyrimidin-2(1)-one show broad peak for O-H group at(3456 cm^{-1}) ,the compounds show peaks at (3504-2860) due to combination bands and showed peak at (900-702) indicated to that substitution on the Aromatic ring are out of plane or pyrimidine ring are perpendicular to the aromatic ring .

Mass spectroscopy, ionization source of energy electron- impact, mass spectrum relative abundance in vertical axis Vs mass/ charge ratio m/z, most compound synthesized give no molecular ions in their spectrum this indicates that most of them are not stable and fragmented to small molecular ion

The mass spectral analysis compound (I, II, III, IV) showed retention time (8.373, 8.021, 9.260, 8.651) respectively. and basic peaks at (281, 129, 254, 270).

The UV spectroscopy explain the possible transition in molecule, such as n to π* or π to π* transition, all product shows λ_{max} between (284 to 346) this high wavelength mean the transition from n to π*

3.4. Conclusion

In conclusion, new DHPMs was synthesized using classical Biginelli approach. describe here an efficient method for the synthesis of 3,4-dihydropyrimidinones reaction of aromatic aldehydes, benzoylacetone and urea under in ethanol solvent catalyzed by zinc chloride under reflux.

Chapter Four

4. References:

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Salehi1 , H.; Kakaei , S.; Ahmadi , S.J.; Zareh M.A. F.; Kiai , S.M.S.; Pakoyan H.R.; Ahmadi, H.T.; (2010). Green procedure for synthesis of 3,4 Dihydropyrimidinones using 12-molybdophosphoric acid, as a catalyst and solvent free condition under microwave irradiation. *Journal of applied chemical researches* .vol. 4, 14,

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Brønsted Acidic Ionic Liquid under Solvent-Free Conditions. *Molecules* .**20,** 3811-3820.

Appendixes

Spectrum Peak Pick Report

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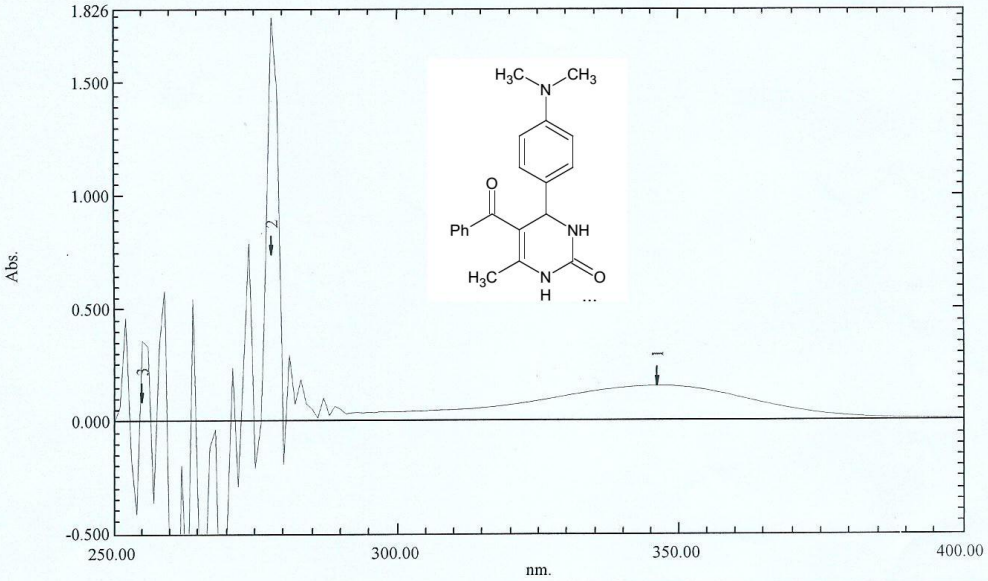

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Sampling Interval:	1.0
Auto Sampling Interval:	Disabled
Scan Mode:	Single
Instrument Properties	
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Measuring Mode:	Absorbance
Slit Width:	1.0 nm
Light Source Change Wavelength:	340.8 nm
S/R Exchange:	Normal
Attachment Properties	
Attachment:	None
Sample Preparation Properties	
Weight:	0.01 g
Volume:	5
Dilution:	100 ml
Path Length:	1
Additional Information:	o.1 HCl

No.	P/V	Wavelength	Abs.	Description
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2	↑	278.00	0.736	
3	↑	255.00	0.085	
4	↑	248.00	0.200	
5	↑	241.00	0.246	
6	↑	229.00	-0.028	
7	↑	217.00	0.091	
8	↑	208.00	-0.141	

Spectrum Point Pick Report

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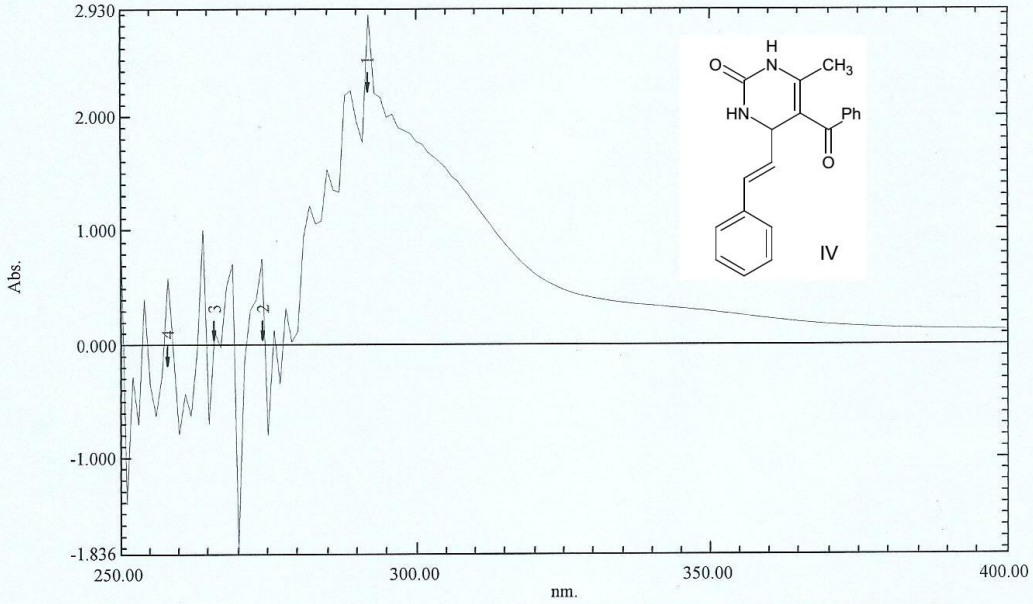

No.	Wavelength	Absorbance	Description
1	290.00	1.949	
2	288.00	2.180	
3	292.00	2.883	
4	294.00	2.158	
5	296.00	2.011	
6			

Measurement Properties

Wavelength Range (nm.):	200.00 to 800.00
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Sampling Interval:	1.0
Auto Sampling Interval:	Disabled
Scan Mode:	Single

Instrument Properties

Instrument Type:	UV-1800 Series
Measuring Mode:	Absorbance
Slit Width:	1.0 nm
Light Source Change Wavelength:	340.8 nm
S/R Exchange:	Normal

Attachment Properties

Attachment:	None

Sample Preparation Properties

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Volume:	5
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Path Length:	1
Additional Information:	o.1 HCl

Spectrum Peak Pick Report

03/28/2017 12:45:04 PM

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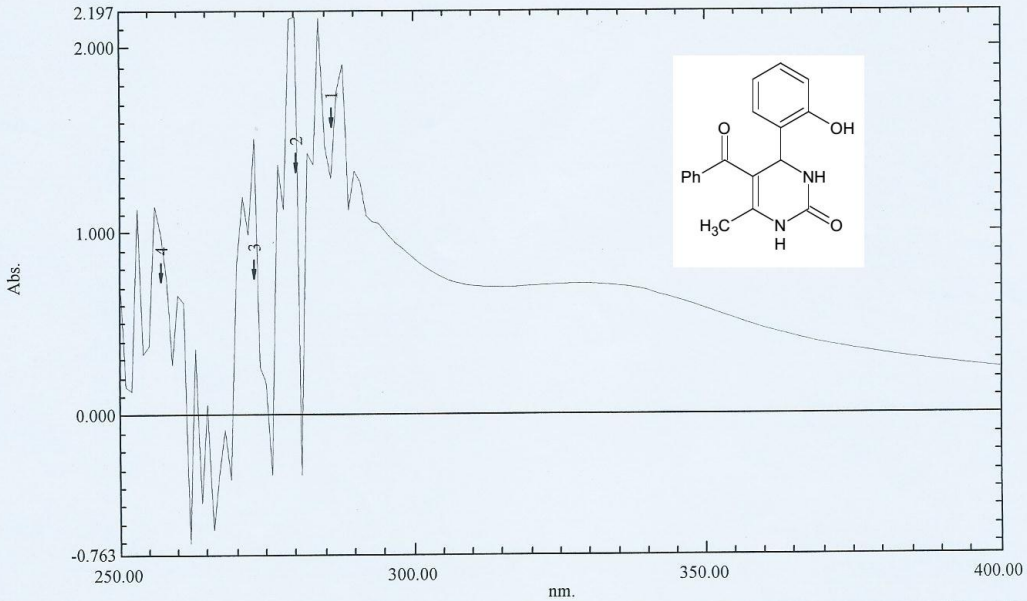

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Auto Sampling Interval:	Disabled
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Measuring Mode:	Absorbance
Slit Width:	1.0 nm
Light Source Change Wavelength:	340.8 nm
S/R Exchange:	Normal
Attachment Properties	
Attachment:	None
Sample Preparation Properties	
Weight:	0.01 g
Volume:	5
Dilution:	100 ml
Path Length:	1
Additional Information:	o.1 HCl

No.	P/V	Wavelength	Abs.	Description
1		286.00	1.570	
2		280.00	1.322	
3		273.00	0.742	
4		257.00	0.724	
5		243.00	0.117	
6		233.00	0.415	
7		229.00	0.383	
8		217.00	0.404	
9		206.00	0.152	

Spectrum Peak Pick Report

03/28/2017 12:28:49 PM

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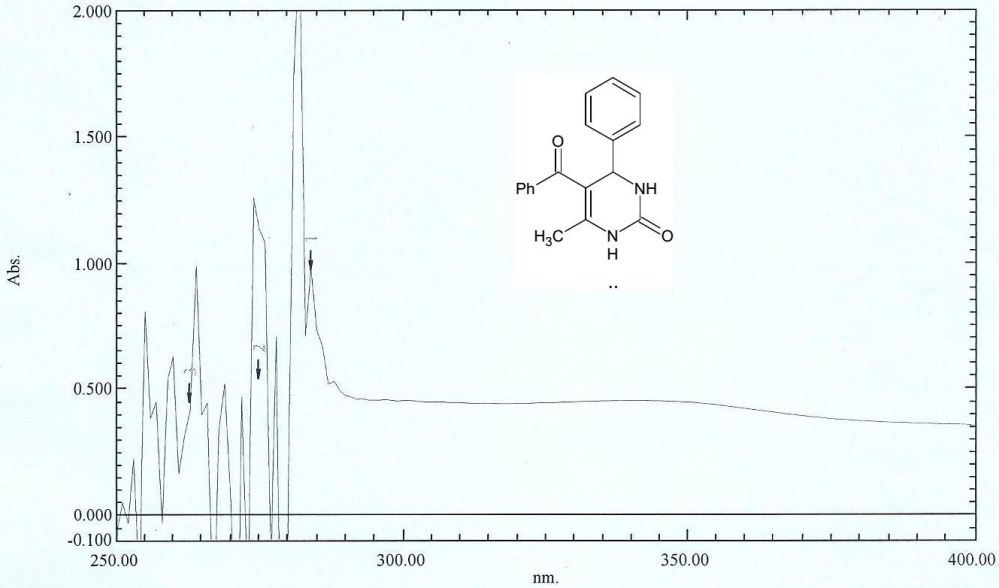

Measurement Properties	
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Scan Speed:	Medium
Sampling Interval:	1.0
Auto Sampling Interval:	Disabled
Scan Mode:	Single
Instrument Properties	
Instrument Type:	UV-1800 Series
Measuring Mode:	Absorbance
Slit Width:	1.0 nm
Light Source Change Wavelength:	340.8 nm
S/R Exchange:	Normal
Attachment Properties	
Attachment:	None
Sample Preparation Properties	
Weight:	0.01 g
Volume:	5
Dilution:	100 ml
Path Length:	1
Additional Information:	o.1 HCl

No.	P/V	Wavelength	Abs.	Description
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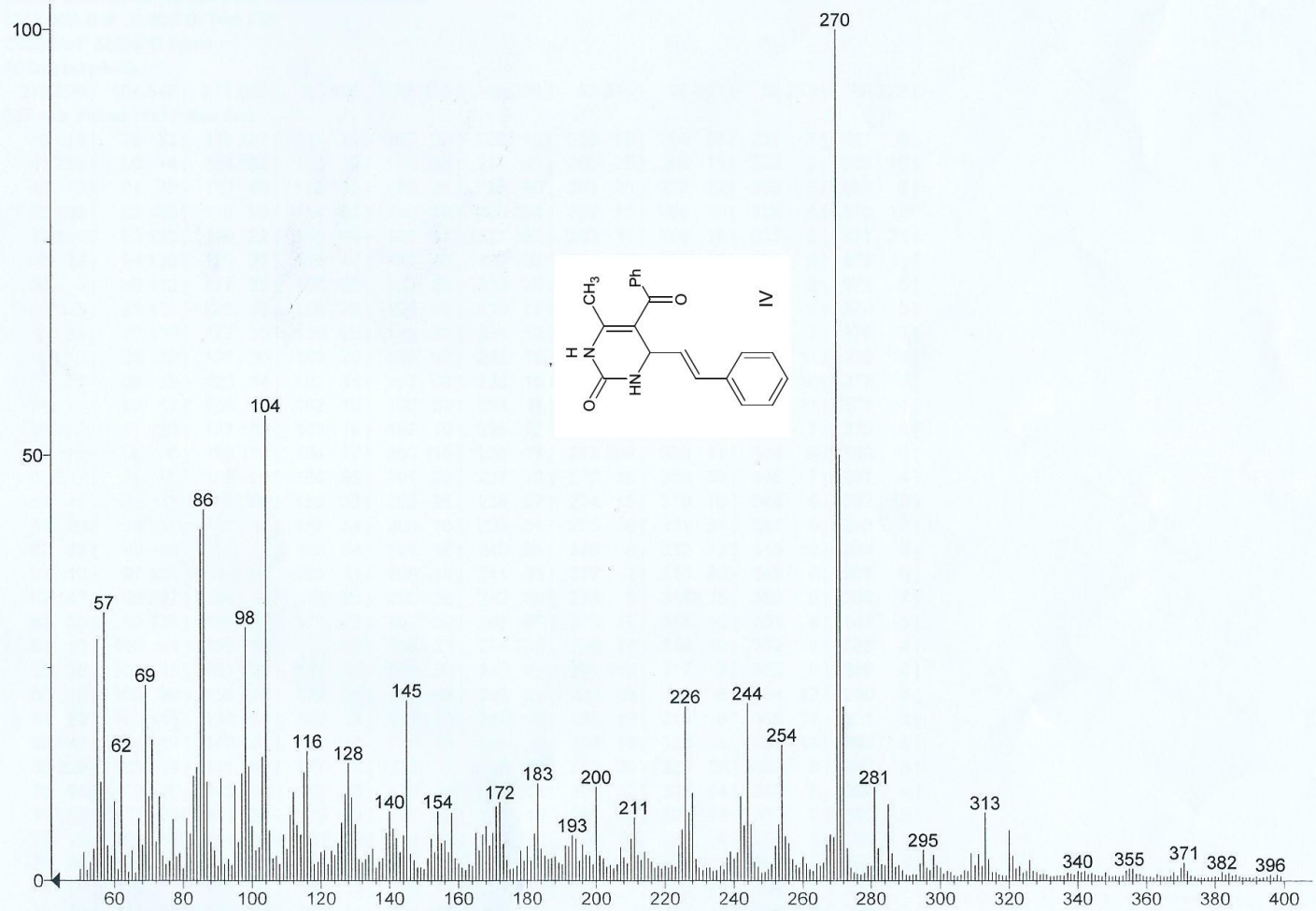
2	↑	275.00	0.542	
3	↑	263.00	0.448	
4	↑	246.00	0.366	
5	↑	231.00	0.336	
6	↑	207.00	0.316	
7	↑	203.00	0.360	



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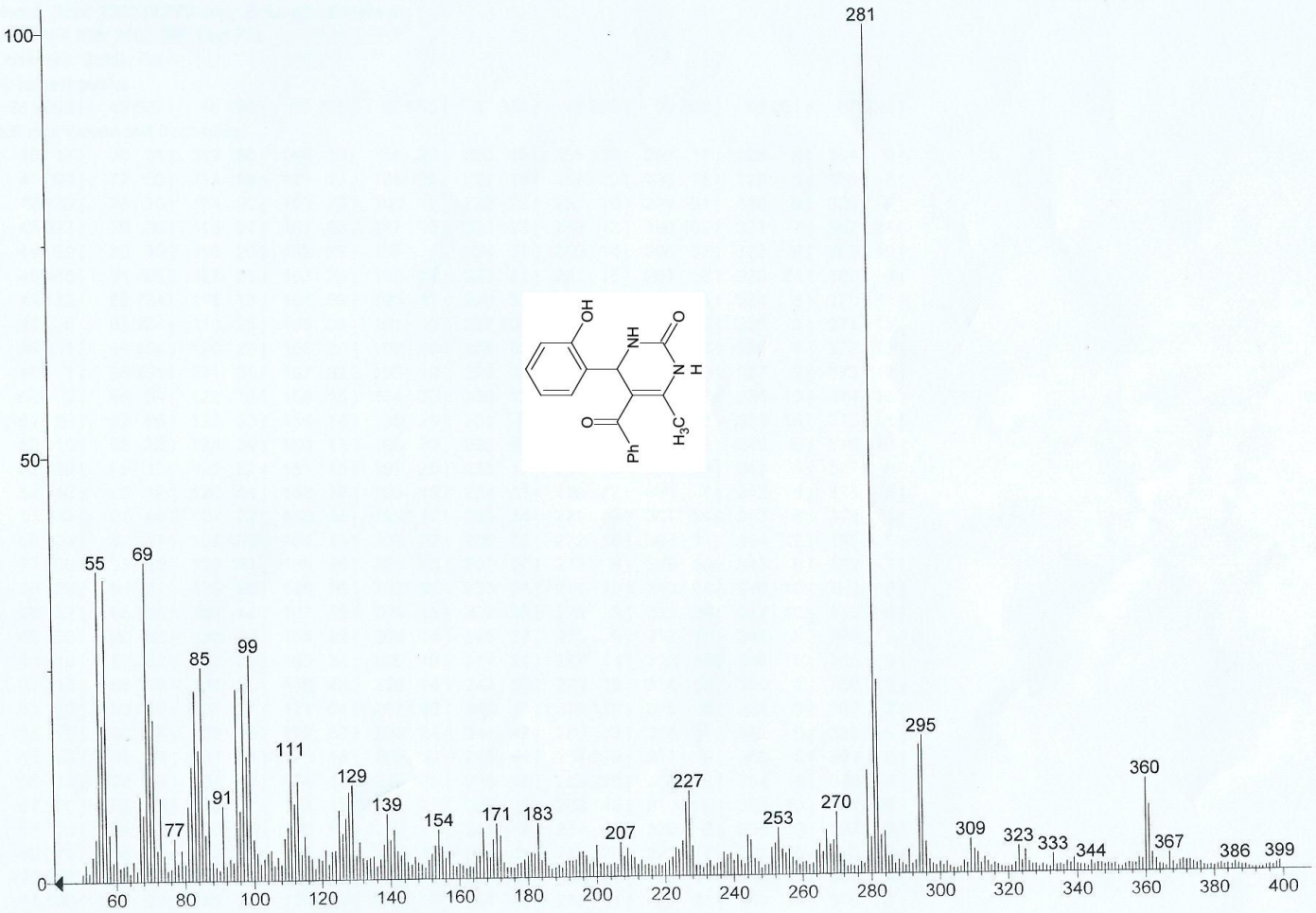

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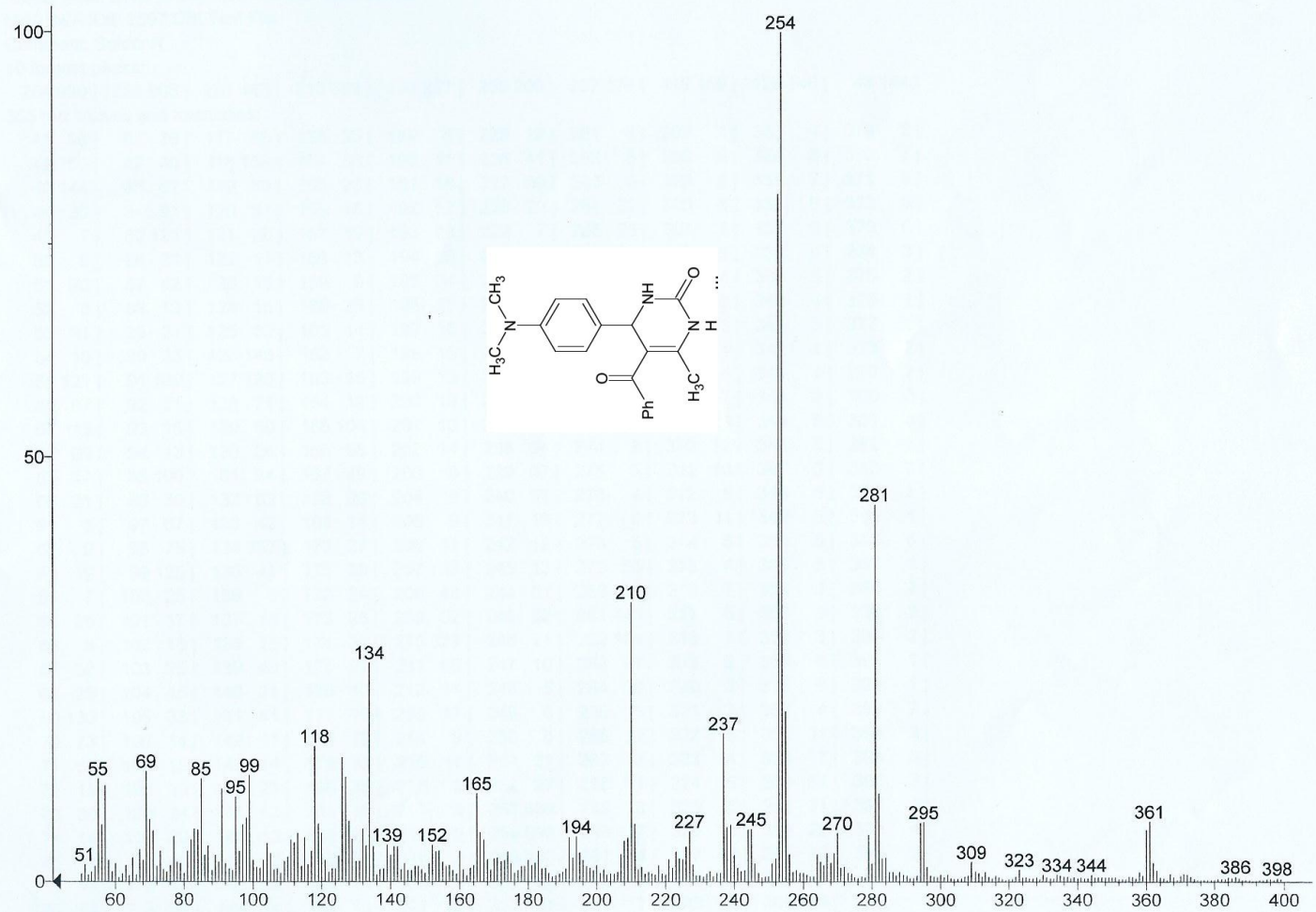

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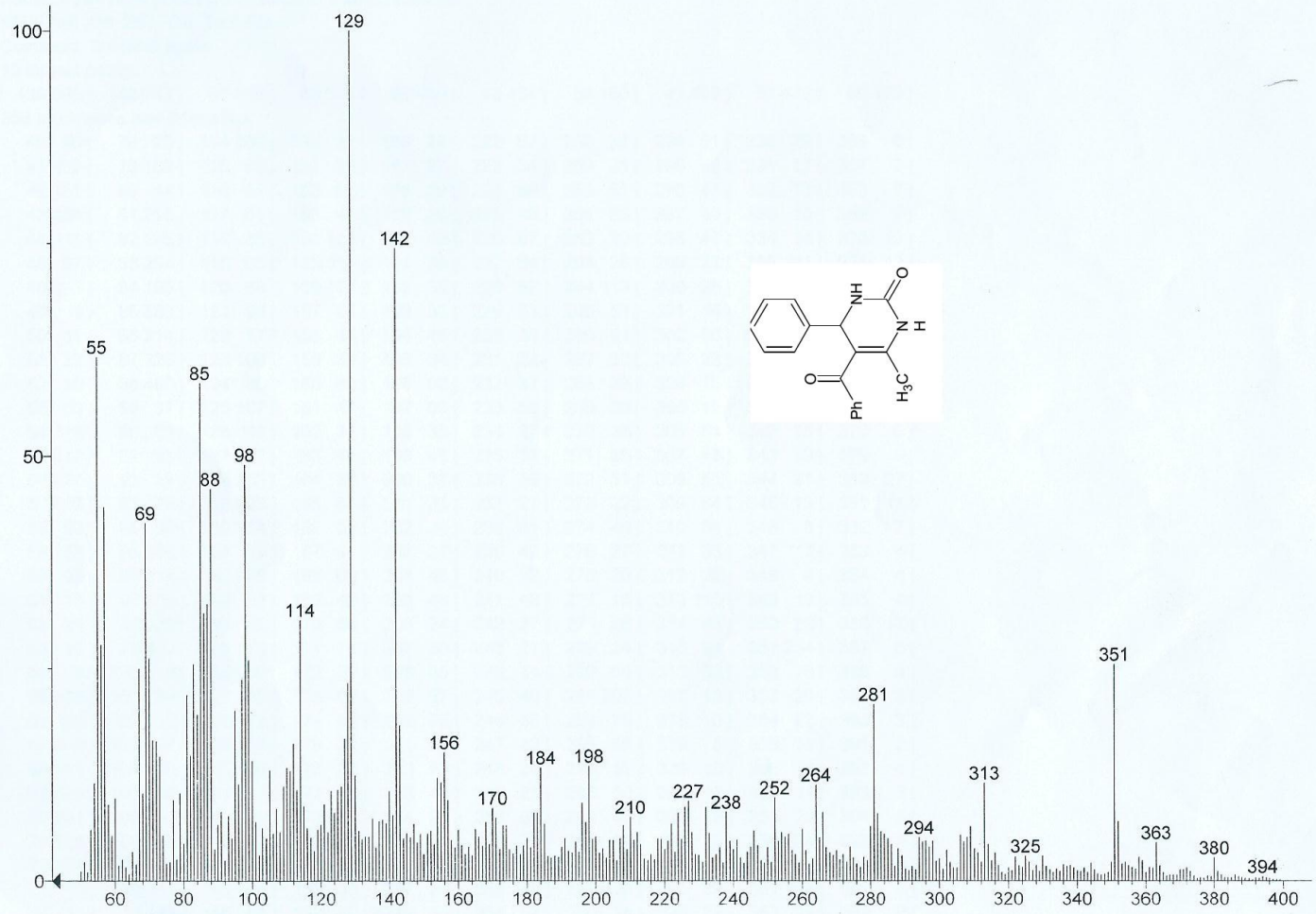

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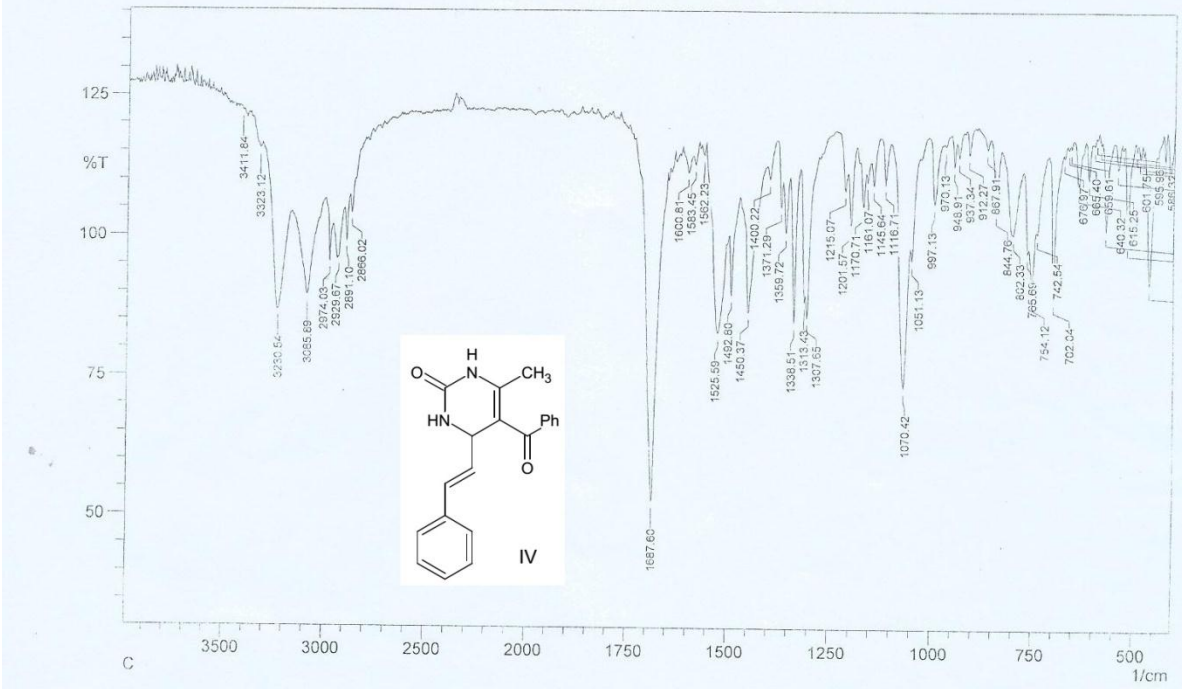

No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	416.6	109.81	5.992	424.31	412.74	-0.685	0.087
2	428.17	113.961	4.981	433.95	424.31	-0.646	0.079
3	464.81	92.153	23.263	478.31	449.38	-0.542	1.255
4	480.24	114.958	1.204	484.1	478.31	-0.365	0.014
5	489.89	114.503	2.192	493.74	484.1	-0.603	0.044
6	497.6	115.827	1.148	501.46	493.74	-0.513	0.013
7	518.82	99.641	17.145	528.46	501.46	-1.181	0.649
8	530.39	116.032	0.644	534.25	528.46	-0.379	0.01
9	543.89	112.524	4.541	553.53	534.25	-1.182	0.138
10	572.82	101.529	15.723	584.39	553.53	-1.528	0.603
11	586.32	116.916	0.789	592.11	584.39	-0.547	0.01
12	595.96	117.812	0.549	597.89	592.11	-0.418	0.009
13	601.75	116.667	0.986	607.54	597.89	-0.661	0.016
14	615.25	110.678	6.492	624.89	607.54	-1.024	0.172
15	640.32	109.272	8.194	653.82	624.89	-1.655	0.367
16	659.61	116.185	0.736	661.54	653.82	-0.522	0.008
17	665.4	116.012	0.64	669.25	661.54	-0.507	0.009
18	676.97	114.47	0.848	678.9	669.25	-0.598	0.016
19	702.04	89.447	25.408	715.54	678.9	-1.075	1.133
20	742.54	100.998	1.305	744.47	719.4	-0.853	-0.047
21	754.12	89.924	9.892	763.76	744.47	0.389	0.373
22	765.69	97.617	2.666	777.26	763.76	-0.286	-0.005
23	802.33	100.782	12.812	833.19	777.26	-1.892	1.242
24	844.76	105.171	11.379	860.19	833.19	-1.336	0.474
25	867.91	116.007	2.263	891.05	860.19	-2.221	0.088
26	912.27	114.978	4.403	918.05	896.84	-1.519	0.122
27	937.34	114.503	3.139	943.13	925.77	-1.169	0.076
28	948.91	112.952	4.695	956.63	943.13	-0.857	0.101
29	970.13	116.421	0.873	973.99	962.41	-0.791	0.02
30	997.13	106.227	11.227	1012.56	977.84	-1.867	0.549
31	1051.13	96.116	5.029	1054.99	1033.77	-0.502	0.032
32	1070.42	73.511	30.464	1105.14	1054.99	1.178	2.685
33	1116.71	109.703	7.662	1130.21	1105.14	-1.5	0.247

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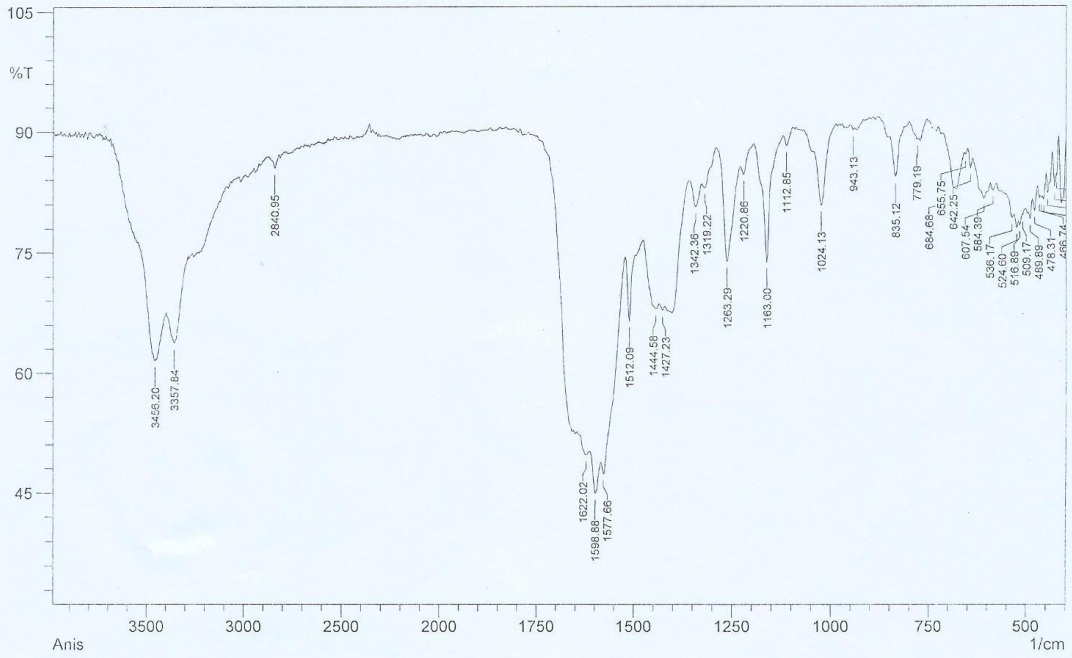

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	422.38	84.561	1.314	424.31	416.6	0.472	0.007
2	428.17	83.208	2.598	433.95	424.31	0.684	0.056
3	445.53	82.453	3.042	449.38	433.95	1.165	0.166
4	455.17	81.608	1.53	459.03	449.38	0.798	0.038
5	460.96	81.789	0.263	464.81	459.03	0.5	0.003
6	466.74	81.814	0.691	470.6	464.81	0.491	0.013
7	478.31	80.207	2.143	484.1	470.6	1.201	0.07
8	489.89	79.206	1.436	493.74	484.1	0.925	0.033
9	509.17	79.625	0.185	511.1	501.46	0.931	-0.001
10	516.89	78.451	0.722	520.74	511.1	0.991	0.017
11	524.6	78.109	1.022	532.32	520.74	1.196	0.029
12	536.17	79.34	0.828	553.53	532.32	1.953	0.011
13	584.39	82.717	0.905	592.11	576.68	1.238	0.04
14	607.54	81.733	2.961	636.47	592.11	3.505	0.406
15	642.25	85.519	1.694	649.97	636.47	0.845	0.049
16	655.75	87.214	0.405	659.61	649.97	0.561	0.01
17	684.68	83.021	0.458	713.61	682.75	1.935	-0.024
18	779.19	89.077	0.444	800.4	775.33	1.153	0.034
19	835.12	84.453	5.551	850.55	800.4	2.646	0.436
20	943.13	90.242	0.354	952.77	939.27	0.582	0.008
21	1024.13	80.928	7.913	1045.35	987.49	3.683	0.833
22	1112.85	88.321	1.589	1122.49	1095.49	1.318	0.091
23	1163	73.875	15.115	1195.78	1122.49	5.456	1.761
24	1220.86	84.762	1.74	1230.5	1195.78	2.159	0.08
25	1263.29	73.921	13.024	1292.22	1230.5	5.5	1.725
26	1319.22	83.075	1.48	1326.93	1292.22	2.45	0.135
27	1342.36	80.706	3.055	1357.79	1326.93	2.619	0.244
28	1427.23	67.771	0.64	1436.87	1421.44	2.565	0.025
29	1444.58	67.974	2.228	1475.44	1436.87	5.859	0.469
30	1512.09	66.508	8.047	1521.73	1496.66	3.686	0.493
31	1577.66	47.373	3.815	1583.45	1521.73	14.462	0.89
32	1598.88	44.943	4.579	1612.38	1583.45	9.42	0.577
33	1622.02	49.677	1.176	1647.1	1612.38	10.187	0.148

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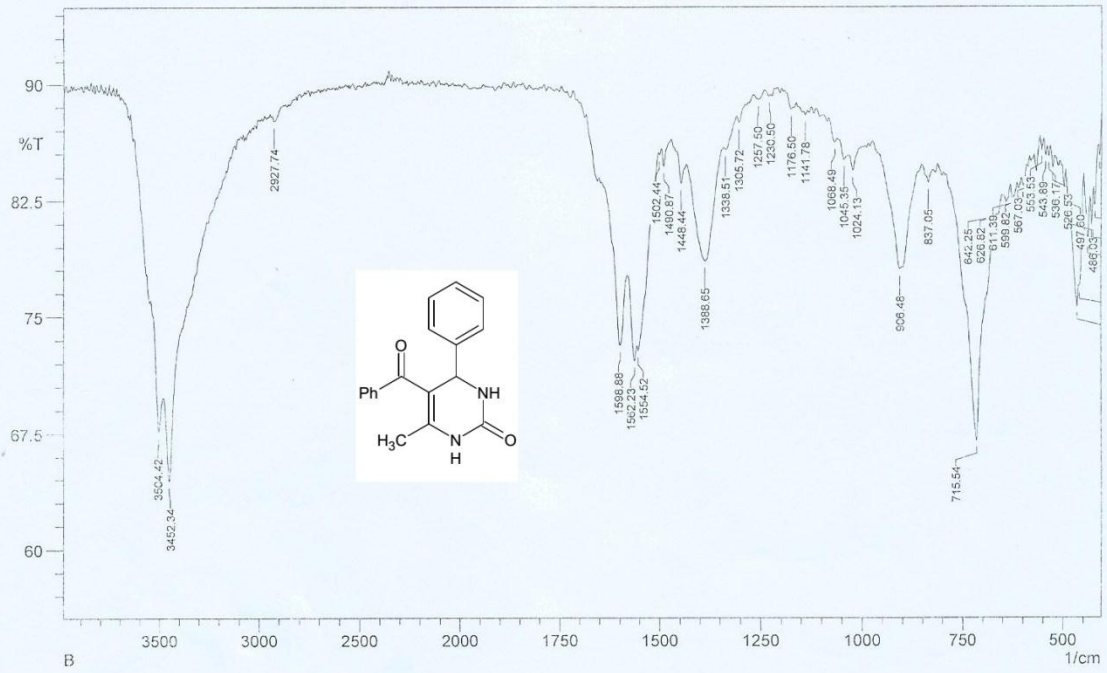
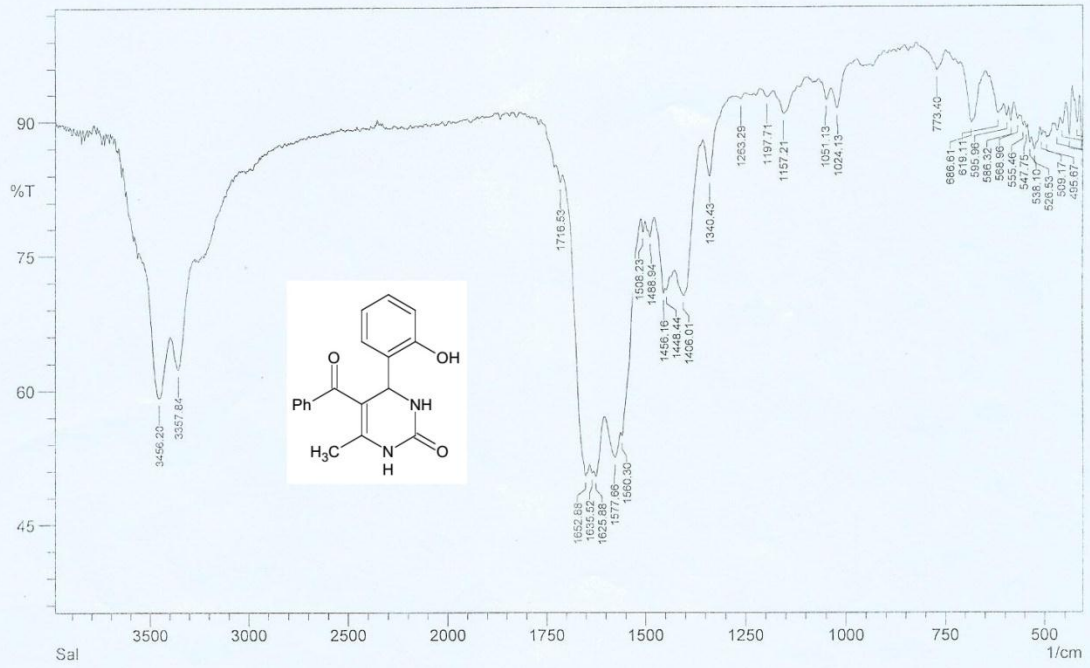


	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	410.81	85.375	1.499	412.74	401.17	0.658	0.045
2	420.45	82.199	2.009	424.31	412.74	0.875	0.038
3	430.1	80.944	1.992	433.95	424.31	0.826	0.045
4	437.81	79.907	3.179	449.38	433.95	1.343	0.133
5	459.03	76.992	1.235	460.96	449.38	1.159	0.073
6	464.81	75.677	2.253	482.17	460.96	2.266	0.15
7	486.03	81.829	0.979	493.74	482.17	0.957	0.033
8	497.6	83.272	1.267	509.17	493.74	1.141	0.027
9	526.53	84.593	1.076	532.32	520.74	0.807	0.029
10	536.17	85.331	0.623	540.03	532.32	0.519	0.012
11	543.89	85.385	0.751	549.67	540.03	0.639	0.017
12	553.53	85.735	0.784	557.39	549.67	0.502	0.016
13	567.03	84.378	1.486	572.82	557.39	1.058	0.048
14	599.82	83.427	0.722	603.68	590.18	1.027	0.028
15	611.39	83.259	0.428	615.25	603.68	0.904	0.013
16	626.82	82.448	1.06	632.61	615.25	1.408	0.051
17	642.25	82.381	0.767	651.89	632.61	1.591	0.045
18	715.54	67.063	15.533	792.69	665.4	14.589	4.194
19	837.05	83.654	0.934	852.48	825.48	2.019	0.06
20	906.48	78.084	7.347	975.91	852.48	10.351	1.966
21	1024.13	84.424	1.143	1033.77	1000.99	2.263	0.061
22	1045.35	85.125	0.763	1056.92	1033.77	1.577	0.048
23	1068.49	86.222	0.619	1083.92	1062.7	1.307	0.038
24	1141.78	87.984	0.251	1145.64	1135.99	0.529	0.005
25	1176.5	88.371	0.743	1193.85	1164.92	1.487	0.05
26	1230.5	89.212	0.165	1242.07	1226.64	0.753	0.006
27	1257.5	88.993	0.431	1269.07	1242.07	1.341	0.033
28	1305.72	87.505	0.551	1309.58	1288.36	1.168	0.021
29	1338.51	85.772	0.36	1342.36	1309.58	2.032	0.033
30	1388.65	78.587	6.774	1434.94	1342.36	8.054	1.69
31	1448.44	83.591	1.711	1458.08	1434.94	1.695	0.089
32	1490.87	84.679	1.301	1496.66	1473.51	1.553	0.052
33	1502.44	85.431	0.211	1504.37	1496.66	0.521	0.004



	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	408.88	86.352	7.327	414.67	403.09	0.515	0.186
2	420.45	89.638	2.809	426.24	414.67	0.485	0.09
3	439.74	88.279	4.625	449.38	432.03	0.723	0.16
4	457.1	89.535	1.376	462.88	449.38	0.603	0.051
5	470.6	88.754	1.08	476.38	462.88	0.664	0.038
6	495.67	87.94	0.39	499.53	493.74	0.316	0.007
7	509.17	88.233	0.731	513.03	505.31	0.405	0.013
8	526.53	86.671	1.763	534.25	513.03	1.227	0.111
9	538.1	87.406	1.175	543.89	534.25	0.533	0.032
10	547.75	89.059	0.549	551.6	543.89	0.377	0.01
11	555.46	89.337	0.672	565.1	551.6	0.618	0.011
12	568.96	90.056	0.876	578.6	565.1	0.567	0.029
13	586.32	89.844	1.85	592.11	578.6	0.558	0.051
14	595.96	90.436	1.161	603.68	592.11	0.475	0.037
15	619.11	90.717	2.384	640.32	603.68	1.29	0.193
16	686.61	89.711	5.443	713.61	665.4	1.594	0.584
17	773.4	95.597	1.094	779.19	750.26	0.452	0.085
18	1024.13	91.38	3.037	1037.63	991.34	1.263	0.23
19	1051.13	92.344	1.65	1060.78	1037.63	0.713	0.084
20	1157.21	90.754	2.837	1182.28	1122.49	2.097	0.388
21	1197.71	92.658	0.855	1215.07	1182.28	1.019	0.066
22	1263.29	92.433	0.371	1269.07	1251.72	0.568	0.011
23	1340.43	83.811	5.213	1357.79	1284.5	3.677	0.421
24	1406.01	70.532	7.588	1427.23	1365.51	7.435	1.452
25	1448.44	71.148	0.543	1452.3	1438.8	1.944	0.016
26	1456.16	70.777	1.664	1479.3	1452.3	3.355	0.008
27	1488.94	77.028	1.296	1494.73	1479.3	1.662	0.04
28	1508.23	77.605	1.358	1512.09	1502.44	1.031	0.04
29	1560.3	54.928	2.172	1564.16	1512.09	9.272	-0.071
30	1577.66	52.525	3.417	1604.66	1564.16	10.714	0.589
31	1625.88	50.397	1.926	1631.67	1604.66	7.431	0.203
32	1635.52	50.764	0.534	1641.31	1631.67	2.813	0.023
33	1652.88	50.445	2.463	1710.74	1649.02	10.971	-0.432

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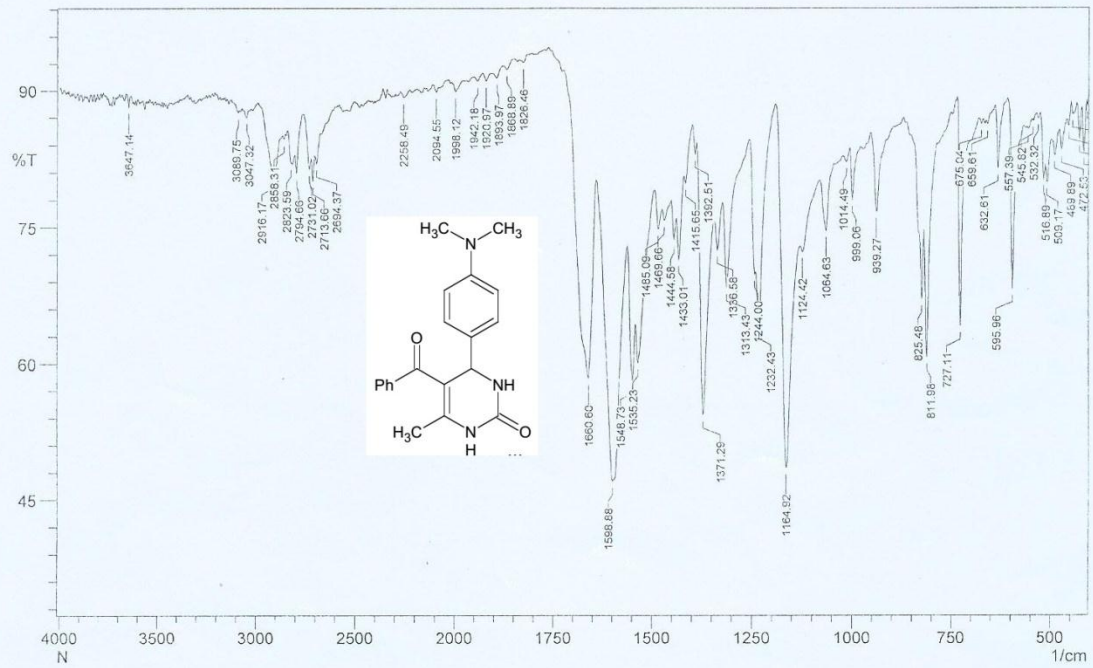

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	408.88	88.341	0.901	410.81	405.02	0.293	0.012
2	416.6	83.348	5.155	422.38	410.81	0.767	0.153
3	428.17	84.676	3.679	435.88	422.38	0.822	0.099
4	445.53	87.416	1.27	449.38	435.88	0.761	0.057
5	455.17	86.162	1.616	460.96	449.38	0.698	0.043
6	472.53	83.558	2.519	478.31	460.96	1.215	0.101
7	489.89	82.957	2.156	499.53	478.31	1.569	0.089
8	509.17	80.013	2.65	513.03	499.53	1.167	0.091
9	516.89	80.372	3.121	526.53	513.03	1.076	0.099
10	532.32	86.993	0.5	536.17	526.53	0.571	0.011
11	545.82	86.707	0.248	547.75	536.17	0.696	0.005
12	557.39	85.796	0.34	559.32	547.75	0.743	0.01
13	595.96	68.273	19.893	609.46	568.96	3.578	1.257
14	632.61	81.568	6.987	640.32	609.46	1.933	0.336
15	659.61	86.32	0.682	663.47	653.82	0.595	0.015
16	675.04	86.381	0.53	680.83	671.18	0.6	0.013
17	727.11	64.173	24.726	736.76	680.83	4.923	1.851
18	811.98	60.766	16.222	819.69	786.9	4.227	0.933
19	825.48	67.145	9.15	864.05	819.69	4.431	0.236
20	939.27	76.813	8.744	954.7	896.84	4.696	0.823
21	999.06	78.137	5.606	1006.77	979.77	2.342	0.266
22	1014.49	82.254	1.066	1024.13	1006.77	1.427	0.047
23	1064.63	74.743	6.265	1080.06	1045.35	3.637	0.479
24	1124.42	72.396	1.436	1130.21	1080.06	5.815	-0.014
25	1164.92	48.535	33.202	1191.93	1130.21	11.231	5.381
26	1232.43	64.296	9.391	1242.07	1191.93	5.5	0.314
27	1244	69.894	2.066	1257.5	1242.07	1.699	-0.048
28	1313.43	68.397	10.371	1323.08	1257.5	6.922	0.962
29	1336.58	72.547	4.23	1344.29	1323.08	2.642	0.232
30	1371.29	54.383	26.691	1388.65	1344.29	7.546	3.273
31	1392.51	83.059	1.82	1400.22	1388.65	0.853	0.048
32	1415.65	79.882	1.896	1419.51	1400.22	1.566	0.046
33	1433.01	71.541	5.857	1438.8	1419.51	2.326	0.276