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Synthesis of some Imidazolidinone compounds under phase transfer conditions and photo cleavages studies of molecular for these compounds

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Abstract

In this paper, two of the new antipyrine heterocyclic derivatives have been synthesized via the formation of Schiff bases. The Schiff bases compounds were generated from the reaction of 4-Ameno-antipyrine (4-AAP) with 3- hydroxybenzaldehyde and 4- nitrobenzaldehyde, which was converted to imidazolidinone compounds by reaction with glycine. The reactions were monitored by Thin Layer Chromatography (TLC). The structure of the prepared compounds was confirmed by physical and available spectroscopic data, i.e., FTIR, ^{1}HNMR, ^{13}C-NMR, GC-Mass and HRMs.

Keywords: Imidazolidinone, phase transfer, photo cleavages, Schiff bases, antipyrine heterocyclic derivatives

Introduction

Schiff bases derived from aromatic amines and aromatic aldehydes have a wide variety of applications in many fields as 4-aminoantipyrine's[1] Schiff bases have been reported to possess antimicrobial activityanti-inflammatory activity[2] antikinetoplastid antimitotic activity, antitumor activity[3] and anticonvulsant activity[4,5]. Imidazolidinones represent an exciting class of compounds concerning biological activity[6]. Through manipulation of the substituent is around the imidazolidinone core molecules with a variety of biological properties have been discovered. Examples include compounds that exhibit antibacterial activity[7]. Imidazolidinone has also been reported to inhibit the binding of vascular cell adhesion molecule (VCAM-1) to very late antigen (VLA-4), which helps treat inflammation associated with chronic inflammatory diseases such as rheumatoid arthritis, multiple sclerosis ,asthma, and inflammatory bowel disease[8,9].

Many valuable reactions cannot be carried out due to insoluble nature of reactants in one solvent. Conventionally a solvent is selected which can dissolve all the reactants in to it but the use of such solvents are not always economical since these solvents are costly. Also, the rate of reaction obtained is too low due to excessive solvation of the nucleophile and has a difficulty of separation of valuable product from reaction mixture[10]. To solve this problem, reactants are allowed to dissolve in their respective aqueous and organic solvents and then the catalyst is added to transfer the reactant from the aqueous or solid phase into the organic phase, where a reaction occurs[11]. The phenomenon is called as phase transfer catalysis (PTC) and catalyst used is called as phase transfer (PT) catalyst. Thus, reaction is made possible by bringing together the reagents initially in different phases[12]. Now a day, PTC is a matured technology used in more than 600 synthesis application covering pharmaceuticals, perfumes, agrochemicals, flavors, dyes, polymer industries, pollution control technologies etc. PTC has proved better than traditional synthesis method because of its mild operating condition, cheaper reagents, high product selectivity in a shorter time and suppression of unwanted side reactions[13].

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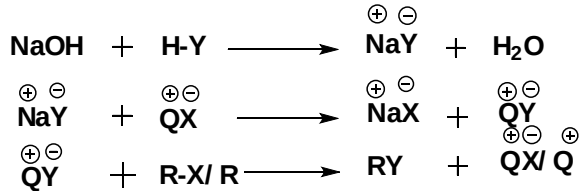
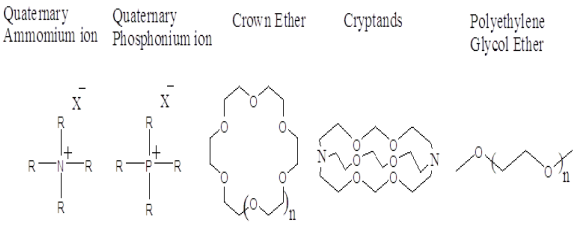
Organic synthesis is still the main way to produce pharmaceuticals. Among the reactions used for trans-formations of substrates into final products, significantare those in which the abstraction of a proton from C, O, N, S, etc. acids, under the action of bases, results in the formation of the corresponding anions. These anions, being nucleophilic agents, enter a variety of reactions with electrophilic partners. properly selecting of the base-solvent system used for reactions induced by bases is crucial[14]. Phase Transfer Catalysis (PTC) seems to be the most general, efficient and environment-friendly methodology of performing reactions in which organic and inorganic anions react with organic substrates. According to this methodology reactions are performed in immiscible two-phase systems[15,16].

In the suggested mechanism, the base pulled a proton from the molecular reactant to convert to anion $(Y^{\ominus})$ replaced by a halide ion in catalyst to form $[Q^{+}Y^{-}]$. This form was adding to the organic reagent molecular to give intermediate compound which was gave the lactam ring added in scheme [17,18].



Scheme (1): Mechanism of Phase transfer catalyst

The commonly used agents for phase transfer catalyst are onium salts (ammonium and phosphonium salts), crown ethers, cryptands and open chain poly ethers like polyethylene glycols (PEG) shown in scheme[19-21].



Scheme (2): Commonly used effective PT Catalyst

Experimental

The melting point was determined on a Stuart melting apparatas SM30. Ultrasound Irradiation was recorded Unisonics PTY. LTD type fxp12.

Infrared spectra were recorded on a Bruker, FT-IR Spectrophotometer Tensor 27, Germany, and a biotech Engineering, FT-IR-600, U.K., using KBr discs. Alpha Bruker/ATR Diamond. Ultra-Violet spectra were recorded on Shimadzu UV – 1650 pc, UV-Visible spectrophotometer, Japan, using chloroform as a solvent. ^{1}HNMR and ^{13}CNMR spectra were recorded on Bruker AC 250, Bruker ARX 300, Bruker ARX 500. For ^{1}H-NMR characterization, ^{13}C- NMR, All ^{1}H-NMR spectra presented in this work were collected in $CDCl_3$ or in DMSO-d6 solution. All chemical shifts are given in ppm. They were using TMS as internal references.

Multiplicities are given as follow: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad signal. Mass spectrometry MS were recorded on AMD MS40, varian MAT CH7, MAT 731 (EI, 70ev), intecta AMD 402 (EI, 70ev and CI), finnigan MAT 95 (CI, 200ev). High Resolution Mass Spectrometry (HRMS). Were recorded on Varian MAT 311, Intecta AMD 402. Elemental Analysis were recorded on LECO CHNS-932, Thermoquest Flash EA 1112. To ensure the purity of the resulting compounds used technique. Thin layer chromatography (TLC) was carried out, the presence of iodine as an aspect of the spot.

General procedure for Synthesis of Schiff bases(Comp 1 and 2):

4-(3-hydroxybenzylideneamino)antipyrine (Comp 1) and 4-(4-nitrobenzylideneamino)antipyrine (Comp 2)[**22**]**.**

A mixture of 4-aminoantipyrine (0.01 mole) and substituted benzaldehydes (0.01 mole) was grinded in a mortar with a pestle made of porcelain for 5-10 min. The mixture turns pastry after few minutes of grinding with the color exchange during grinding process. 15 mL of diethyl ether was added. The solid product filtered, dried and recrystallized from absolute ethanol. The chemical and spectral data of the compounds are given in table (1-7).

General procedure for Synthesis(Comp 3 and 4): 4-(2-(3-hydroxyphenyl)-5-oxoimidazolidin-1-yl)antipyrine (3) and 4-(2-(4-nitrophenyl)-5-oxoimidazolidin-1-yl)antipyrine (4)[14]**.**

To a mixture consist of (0.001 mole) glycine, (3-6 mL) of 30% sodium hydroxide solution, (15 mL) of benzene and (0.1 gm) benzyl tributyl ammonium chloride (BTBAC). (0.001 mole) of compounds (1 or 2) were added. The reaction continues in (25 ^{0}C) with magnetic stirrer. When the reaction was been deep red, after (20 min.) from the time of the reaction.

The organic layer was separated and wash with water at any times. When the wash water to be equal the organic layer was taken and dried by ($MgSO_4$). The mixture was filtered and evaporated and the formed precipitate was recrystallized from appropriate solvent to give the products comp 3 and comp 4. Table 1 included chemical and physical properties of these compounds.

Results and Discussion

Synthesis of Schiff base compounds

Schiff base compounds were prepared by condensation of 4-aminoantipyrine (4-AAP) with aldehyde derivatives (3-hydroxy benzaldehyde, 4-nitrobenzaldeyde) Schiff base, which has been prepared in the following scheme (1). Table 1 included chemical and physical properties of these compounds with a melting point.

In the FT-IR spectrum table (3) there are four significant peaks; which are depending upon the different substitution groups that appeared in the comp 1 and comp 2. And its relative to azomethen groups[23] (-N=CH-), (=CHAr), (C=O Ap) and (C=C) groups. These compounds contain (-N=CH-) and (=CHAr.); there are four different peaks which are appeared at (1605, 1596 cm^{-1}), (3095,3051 cm^{-1}) respectively, (C=O Ap) at (1622,1641 cm^{-1}) and (C=C) at (1580,1572 cm^{-1}) beside that compound (1) appeared peak at (3156 cm^{-1}) for (C-OH) group[24].

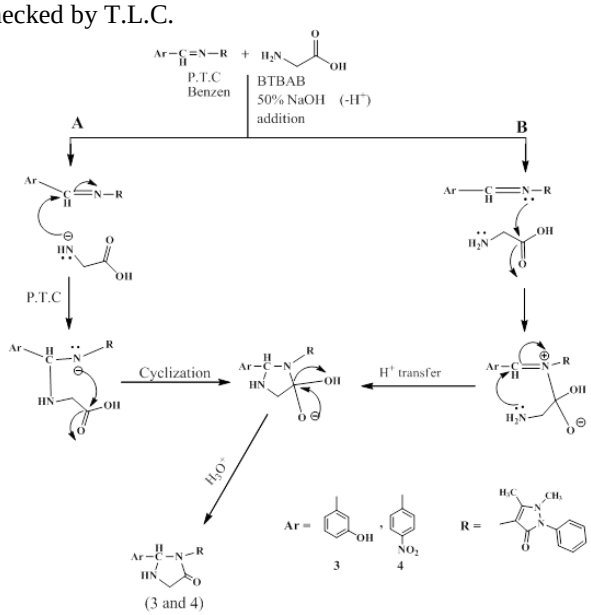
^{1}H NMR spectrum table (4) appeared that (δ9.48-9.52ppm, s, CH for-N=CH), (δ7.13-8.43ppm, m, C-HAr.), (δ 9.46ppm,s,b, OH) and (δ2.12-2.43ppm, s, CH for C-CH_3), (δ3.13-3.15ppm, s, CH for N-CH_3) the (C-CH_3 and N-CH_3) peaks are nearly constant in the all of compounds also in ^{13}C-NMR. ^{13}C-NMR spectrum appeared[25] that a major peaks at (δ157.7-159.8ppm,C for-N=C),(δ116-152ppm, Ar-C.) and (153.21ppm, C-OH) the other peaks in table (5); at (38-41ppm,solvent DMSO). GC-Mass, EI and HRMS spectrum table (6) appeared the exactly molecular weight and the elemental analysis (CHNS) gives acceptable results in table (7).

Synthesis of imidazolidinone comp 3 and comp 4:

Comp 3 and comp 4 were synthesized from reaction Comp 1 and comp 2 with glycine by P.T.C technique.

According to the Synthetic scheme (3). The reaction was divided into two divisions (A and B). Division (A) consists of a nucleophile (after loss of hydrogen by base) attack the carbon of schiff base and the reaction continue to give the correct compound after cyclization. This correct compound also formed in division (B) by attacking the lone pair of nitrogen in schiff base to carbon of carboxylic acid and then was cyclized after loss a hydrate molecule[26].

These compounds were studied and characterized by their melting points table (2). FT-IR,^{1}HNMR, ^{13}CNMRspectra, GC-Mass, EI, HRMS, CHNS and checked by T.L.C.



Scheme (3): preparation of compounds (3, 4)

The FT-IR spectrum of these compounds table (3) ,(Fig. 1) and (Fig. 2), shown that disappearance of (-N=CH-) group at (1605-1596 cm^{-1}) and its appeared new peak at (1663-1677 cm^{-1}) which is relative to the (C=O) lactam group and its appeared peak at (1643,1652 cm^{-1}) which is relative to (C=O) (Ap)[27,28]. (C=C) appeared at (1604-1597 cm^{-1}) and OH group appeared at (3156cm^{-1}) But (NH) was appeared at (368, 3173 cm^{-1}).

^{1}H-NMR spectrum chart table (4) ,(Fig. 3) and (Fig. 4) for compounds (3 and 4) respectively was appeared that: (δ6.31-6.03ppm, s, CH), at (δ3.99-4.24, δ3.76-3.93ppm, q, CH_2), at (δ5.07, 4.90ppm, s, N-H) at (δ7.10-8.51ppm, m, Ar-H.); and (δ9.46 ppm, s, OH)[29,30]. ^{13}CNMR spectrum table (5) ,(Fig. 5) and (Fig. 6)appeared that (δ75.78-81.20ppm, C for CH); (δ204.82-197.32ppm, C for C=O Lactam); (δ161.17-161.29ppm, C for C=O Ap); (δ113-150ppm, Ar-C.) (δ31.70-34.00ppm, C, for CH_2) and at (δ38-41ppm, solvent DMSO). GC-Mass, EI and HRMS spectrum table (6) ,(Fig. 7) and (Fig. 8) appeared the exactly molecular weight and the elemental analysis (CHNS) gives acceptable results in table (7).

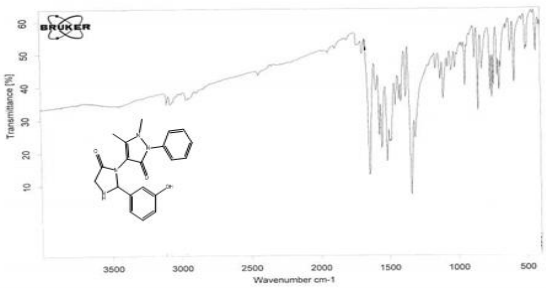


Fig. 1. FTIR Spectra of Comp 3.

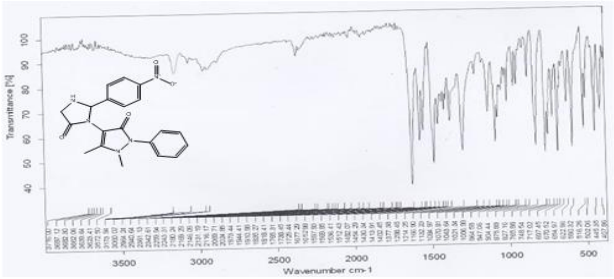


Fig. 2. FTIR Spectra of Comp **4**.

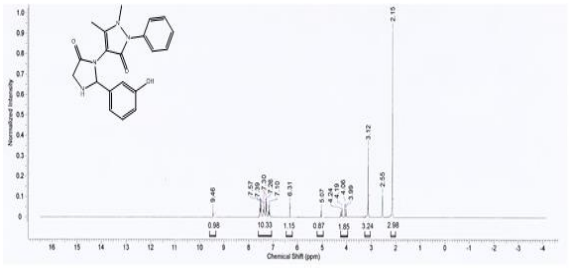


Fig. 3. ^{1}H NMR spectrum of Comp 3.

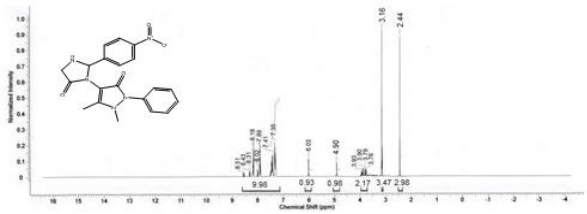


Fig. 4. ^{1}H NMR spectrum of Comp 4.

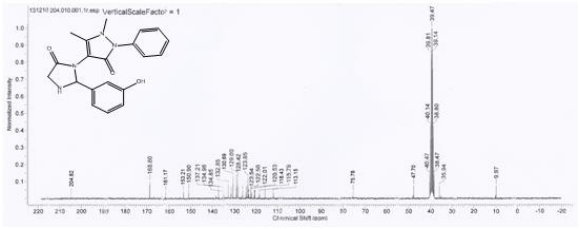


Fig. 5. ^{13}CNMR spectrum of Comp 3.

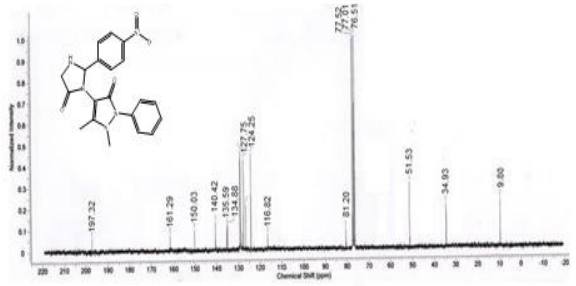


Fig. 6. ^{13}CNMR spectrum of Comp 4.

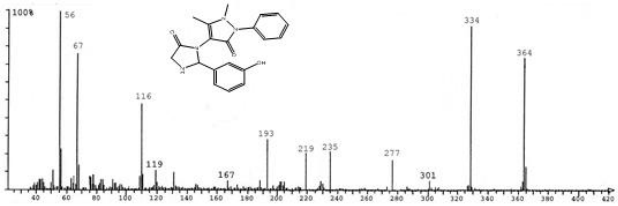


Fig. 7. GC-Mass spectrum of Comp 3

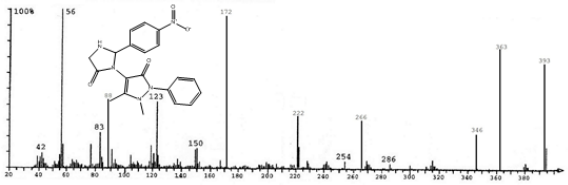


Fig. 8. GC-Mass spectrum of Comp 4.

Table 1. physical properties and other characteristics for the synthesis Schiff base derivatives (1,2)

Comp.	m.p ^{0}C	color	Solvent	Rf	Time	M.F	M.W	Yield %
1	280-282	White crystals	Abs.EtoH	0.6	10 min.	$C_{18}H_{17}N_3O_2$	307	96
2	260-261	Orange	Abs.EtoH	0.7	5 min.	$C_{18}H_{16}N_4O_3$	336	98

Table 2. physical properties and other characteristics for the synthesis Schiff base derivatives (3,4)

Comp.	m.p ^{0}C	color	Solvent	Rf	Time	M.F	M.W	Yield %
3	245-247	White	Abs.EtoH	0.5	5 min.	$C_{20}H_{20}N_4O_3$	364	85
4	225-228	Pale yellow	Abs.EtoH	0.6	3 min.	$C20H_{19}N_5O_4$	393	87

Table 3. IR Spectral of compounds (1 - 4)

Comp.	IR (KBr), ν (cm^{-1})						U.V ($CHCl_3$) λmax (nm)
	C=O lactam	C=O AP	C=C	N-H	C=N	OH	
1	—	1622	1580	—	1605	3156	342
2	—	1641	1572	—	1596	—	398
3	1663	1643	1604	3168	—	3156	362
4	1677	1652	1597	3173	—	—	320

Table 4. 1H-NMR data of compounds (1 - 4)

Comp	$C-CH_3$	$N-CH_3$	CH=N-	CH_2	NH_2	OH	C-H	Ar-H
1	s, 2.12	s, 3.13	s, 9.48			s.br, 9.46		7.13-7.51 (m, 9H)
2	s, 2.43	s, 3.15	s, 9.52					7.21-8.43 (m, 10H)
3	s, 2.15	s, 3.12		q,3.99-4.24	s.br5.07	s.br, 9.46	S6.31	7.10-7.57 (m, 9H)
4	s, 2.44	s, 3.16		q,3.76-3.93	s.br, 4.90		s, 6.03	7.35-8.51(m, 9H)

Table 5. ^{13}C-NMR data of compounds (1- 4)

Comp.	$C-^*CH_3$	$N-^*CH_3$	$CH_3-^*C=^*C-N$	CH_2, C-OH	$^*CH$, CH=N	Ar-C	C=O AP	C=O lactam
1	9.97	35.33	105.23-136.73	,— 153.21	,— 157.7	116-147	160.33	—
2	10.12	34.87	109.83-135.97	—	,— 159.8	122-152	161.47	—
3	9.80	35.55	113.75-135.15	47.70 153.21	75.78 —	113-138	161.17	204.82
4	9.87	34.93	116.82-136.00	51.53	81.20	120-150	161.29	197.32

Table 6. GC-Mass, EI and HRMS spectrum of compounds (1-4)

Comp	m/z	%	m/z	%	m/z	%	m/z	%	m/z	%	HRMS Calc.	HRMS Meas.
1	308	16	307	75	199	23	188	25	171	19	307.13228	308.14353
	121	33	89	15	77	17	56	100				
2	337	6	336	34	188	20	121	33	91	22	336.12172	337.12952
	78	14	44	13								
3	365	13	364	75	334	90	277	17	235	20	364.153541	365.160817*
	219	21	193	35	116	50	67	78	56	100		
4	394	14	393	64	363	77	346	20	266	30	393.143704	394.150981*
	222	35	172	96	88	45	56	100				

* = $[M+H]^+$ = Calculated Molecular Ion Mass or Measured Molecular Ion Mass for some compounds.

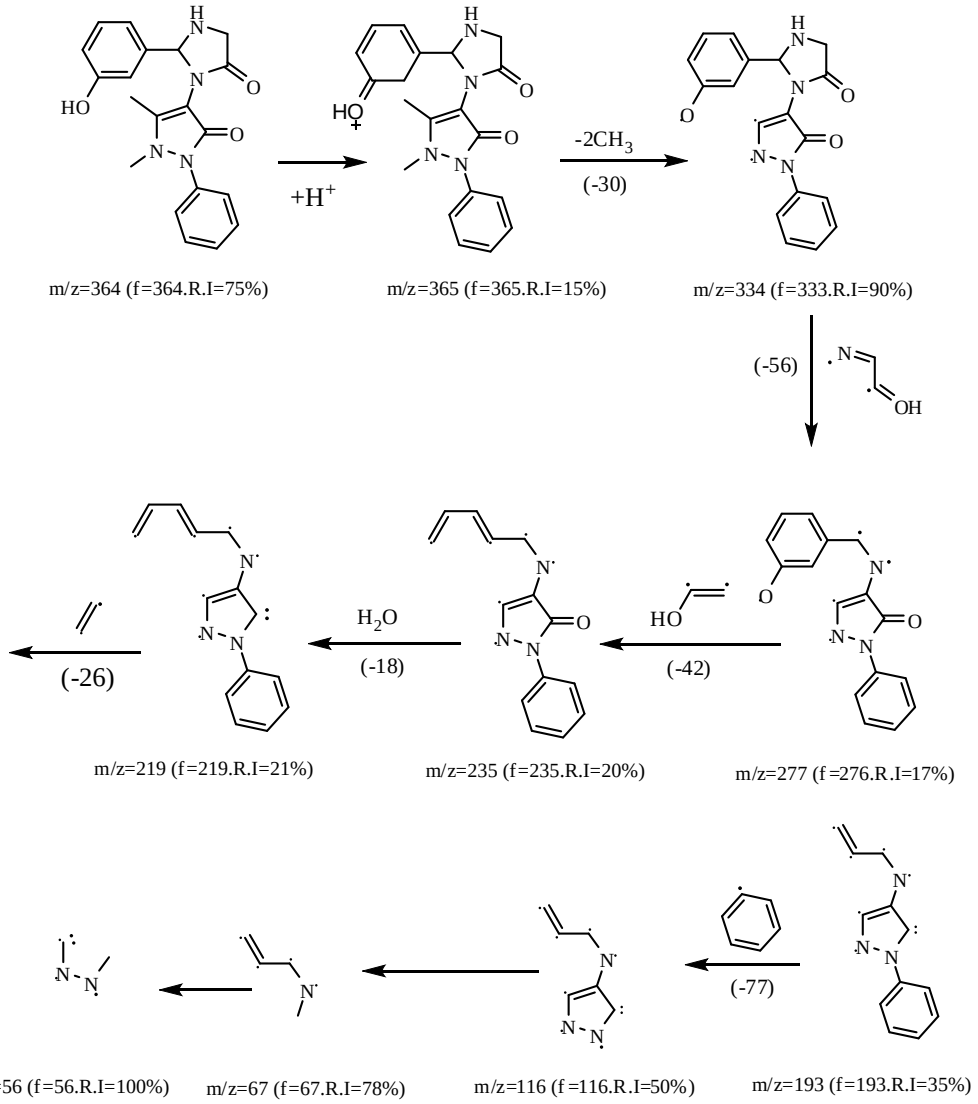
.

Table 7. Element analysis (CHNS) of compounds (1-4)

Comp.	$C_{theo.}$	$C_{prac.}$	$H_{theo.}$	$H_{prac.}$	$N_{theo.}$	$N_{prac.}$	M.F	Mol. Mass
1	70.13	70.232	5.58	5.682	13.67	13.875	$C_{18}H_{17}N_3O_2$	307.13
2	64.28	64.473	4.79	4.811	16.66	16.753	$C_{18}H_{16}N_4O_3$	336.12
3	65.92	65.913	5.53	5.487	15.38	15.376	$C_{20}H_{20}N_4O_3$	364.15
4	61.06	61.054	4.87	4.791	17.80	17.864	$C_{20}H_{19}N_5O_4$	393.14

Mass spectra *(photocleavage reactions)* :

The electron impact mass spectrum of (3) Imidazolidinone shows a molecular ion peak m/z at 364 (M+) with a relative intensity 13% which is equivalent to its molecular weight[20]. The different pathways of the fragments of the parent molecular ion peaks are given in Scheme4[31,32] . The other molecular ion peaks appeared in the mass spectrum (abundance range from 1% to 100%) is attributed to the fragmentation of 3 molecule obtained from the rupture of different bonds inside the molecule as shown in Scheme 4[32]. The compound 3 gives a molecular ion peak at m/z 364 (M+) and m/z 365 (M+1) with a relative intensity 75% and 13% respectively. The intensities of these peaks give the idea of the stability and abundance of the fragments. The mass spectra of other compounds confirm this type of stoichiometry(3). This is in good agreement with the microanalytical data[33,34] .



Scheme (4): The different pathways of the fragmentation of the parent molecular ion peaks (Comp 3).

Conclusions

The grinding and ptc- techniques has been used in this work to reduce the energy consumption and chemical additives and, more importantly, the reduction of environmental impacts relative to prepare imidazolidinone compounds. The ptc - mediated synthesis for two imidazolidinone derivatives exhibits high yields. Characteristics of the final product including FTIR ^{1}HNMR, ^{13}C-NMR, GC-Mass, HRMs, CHNS indicate the configuration of the required compounds. Further, photo cleavages Studies give to how molecular fractions, down to the real wieght of the molecules.

Acknowledgment

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