IJISET - International Journal of Innovative Science, Engineering & Technology, Vol. 3 Issue 8, August 2016
ISSN (Online) 2348 – 7968 | Impact Factor (2015) - 4.332
www.ijiset.com

# Synthesis and Characterization of some Imidazole Derivatives

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**Abstract:**

Three Imidazole derivatives were synthesized by condensation reactions of oxazolone derivative and 2,4- dinitrophenyl hydrazine in the presence of pyridine. The prepared compounds confirmed by using Proton Nuclear Magnetic Resonance (H1 NMR) and Fourier Transform Infra-Red Spectrometer (FT-IR) Techniques. The obtained results agreed well with expected characteristics peaks of the synthesis compounds

**Keywords**: Oxazolone, 2,4-dinitro phenylhydrazine, NMR,FT-IR

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## 1. Introduction:

A five-membered imidazole ring is a structural unit found in many biologically active compounds. The strong therapeutic properties of imidazole containing drugs have encouraged medicinal chemists to synthesize a large number of novel chemotherapeutic agents comprising this entity. Amongst others, imidazole core care structures are found in different carboxypeptidase, hemeoxygenase and lactamase inhibitors, as well as among anti-inflammatory, anticancer, antibacterial, antifungal, antitubercular, antidiabetic and antiviral products(Saudi, Zmurko et al. 2014) The Imidazole compounds exhibited different cytostatic and cytotoxic activities for further developing potential application as anticancer drugs(Chen, Yu et al. 2013, Hossain, Świtalska et al. 2013) the compounds of benzofuran and imidazole moieties and their potential antitumor activities (Liu, Wang et al. 2013, Martins, Jesus et al. 2015) benzimidazole derivatives exhibited moderate tuberculostatic activity (Gobis, Foks et al. 2015) Imidazole is a 5-membered planar ring, which is soluble in water and other polar solvents(Bhatnagar, Sharma et al. 2011). It exists in two equivalent tautomeric forms because the hydrogen atom can be located on either of the two nitrogen atoms. Imidazole is a highly polar compound, Imidazole is amphoteric; that is, it can function as both an acid and a base (Chawla, Sharma et al. 2012) The compound is classified as aromatic due to the presence of a

sextet of - electrons, consisting of a pair of electrons from the protonated nitrogen atom and one from each of the remaining four atoms of the ring(Shalini, Sharma et al. 2010)

The imidazole and derivatives had many Pharmacological Activities like antitubercular , antifungal ,analgesic ,anti-HIV , anticancer and antibacterial (Verma, Joshi et al. 2013)

The objective of the present study is to synthesize three Imidazole derivatives by condensation reactions of oxazolone derivative and 2,4- dinitrophenyl hydrazine in the presence of pyridine.

## 2. Experimental:

All chemicals used in this research were of analytical grade and further purified by recrystallization. The solvents used are laboratory grade type.

## 3. Methods:

### 3.1. Synthesis of benzoyl glycine

g of glycine ( 0.01 mol ) in 150 ml distilled water and added 8 g of sodium hydroxide ( 7.5 0.02 ) and stirring vigorously and till to complete the dissolve of glycine and added 14.17 ml ( 0.012 mol ) of benzoyl chloride in fine portion to solution , after that neutralized the reactant by added concentrated hydrochloric acid slowly and with stirring until the mixture is acid and takes it overnight , after that the mixture wash the precipitate with cold water and . filtered and recrystallize the benzoyl glycine with distilled water 28 g m.p 183 – 184 c

### 1.3.2 Synthesis of oxazolone

The mixture of aldehyde ( 0.025 mole ) V ,VI, VII ,(0.25mole ) of benzoyl glycine ,(0.75mol ) of acetic anhydride and (0.25mol )of sodium acetate in flask ( equipped with reflux condenser ) on hotplate with vigorous stirring for 1hr cooled and left in refrigerator overnight , the solid mass of crystal was stirring with 60ml of cold water ,and filtered and recrystallized .with ethanol

### 1.3.3 Synthesis of 1-(2.4-dinitrophenyamino),-4-(4-hydroxybenzylidene),-2-phenyl,5-ketone ,imidazole (V)

A 0.01 mol of oxazolone (V)and 2,4 –dinitrophenylhydrazine ( 0.015 mol) reflux for 1ohrs in a present of dry pyridine , after refluxing complete then reaction mass is cooled and the reaction mixture was poured into ice-cold water containing conc .HCL and then filtered and wash with distilled water after that dried and recrystallized with acetone

### 1.3.4 Synthesis of 1-(2,4-dinitrophenylamino),-4-(4-hydroxybenzylidene)-2-phenyl,5-ketone ,imidazole (VI)

A 0.01 mol of oxazolone (VI )and 2,4 –dinitrophenylhydrazine ( 0.015 mol) reflux for 1ohrs in the present of dry pyridine , after refluxing complete then reaction mass is cooled and the reaction mixture was poured into ice-cold water containing conc .HCL and then filtered and wash with distilled water after that dried and recrystallized with acetone

### 1.3.5 Synthesis of 1-(2,4-dinitrophenylamino)-4-(2-furanylmethylene )-2-phenyl,5- . ketone Imidazole (VII)

A 0.01 mol of oxazolone (VII )and 2,4 –dinitrophenylhydrazine ( 0.015 mol) reflux for 1ohrs in the present of dry pyridine , after refluxing complete then reaction mass is cooled and the reaction mixture was poured into ice-cold water containing conc .HCL and then filtered and wash with distilled water after that dried and recrystallized with acetone.

FT-IR spectra were recorded using Agilent Cary 630 FTIR spectrometer, $H^1$ NMR spectra, were also recorded.

### 1.4 Results and discussion

The H1NMR & IR data confirm the assigned structures as follows

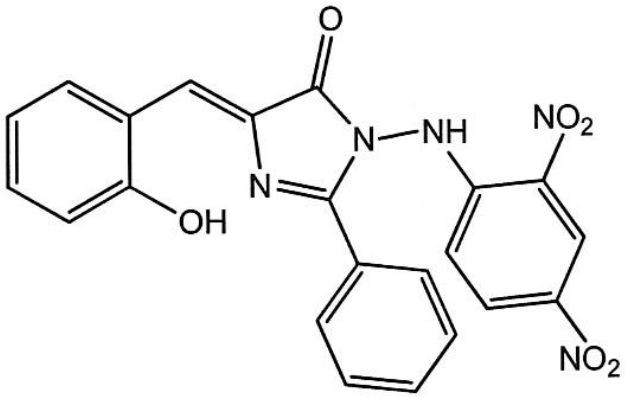
#### 1.4.1. 1-(2.4-dinitrophenyamino),-4-(4-hydroxybenzylidene),-2-phenyl,5-ketone ,imidazole (V)[S]

From the data below it is clearly that the characteristics peaks of the prepared compound confirm the expected peak as shown here:

$H^1$ NMR : 11.0000 ppm(s), 9.0000 ppm (d), 8.2664 ppm (t) , 7.9548 ppm (d), 7.2400 ppm (m) , 6.9687 ppm (d),2.1522 ppm(t) ,1.2725 ppm (m) ,0.8200 ppm (s)

IR : 1700 cm-1 (C=O st. ) , 1350 cm-1 ( CH=C st.) ,1200 cm-1 ( C=C st)

S (salicylaldehyde )




V

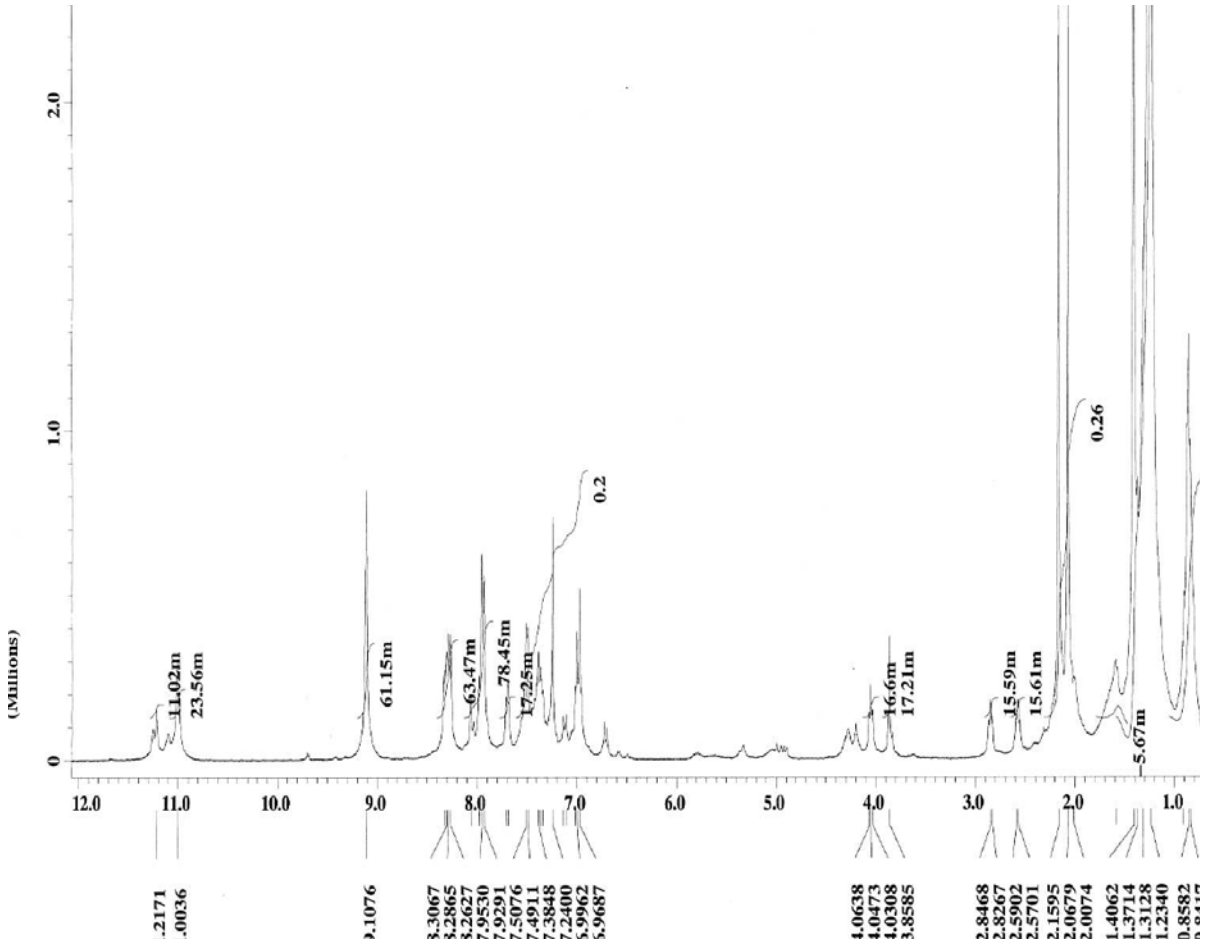



Fig (1) $H^1$-NMR of 1-(2.4-dinitrophenyamino),-4-(4-hydroxybenzylidene),-2-phenyl,5-ketone ,imidazole (V)

.

### 1.4.2. 1- (2,4 dinitrophenyl amino ) ,-4 – (4-hydroxybenzylidene )-2- phenyl , 5- ketone ,imidazole (VI) [A]

No doubt that the NMR and IR information below represented the preparation of the above compound.

11.2171ppm (s) , 9.1076 ppm (s) ,8.0700 ppm (d),8.3000 ppm (dd), 7.9600 ppm (d),7.5200 .ppm (d), 7.7200 ppm (d) ,7.3900 ppm (dd), 7.2500 ppm (s)

IR : 1700 cm-1 (C=O st. ) , 1350 cm-1 ( CH=C st.) ,1200 cm-1 ( C=C st)

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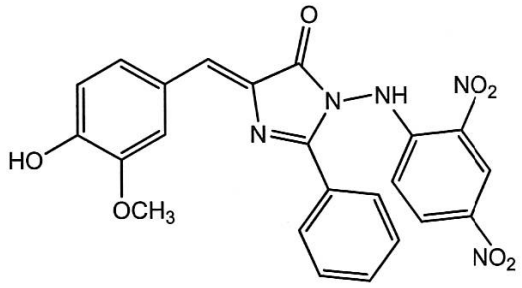
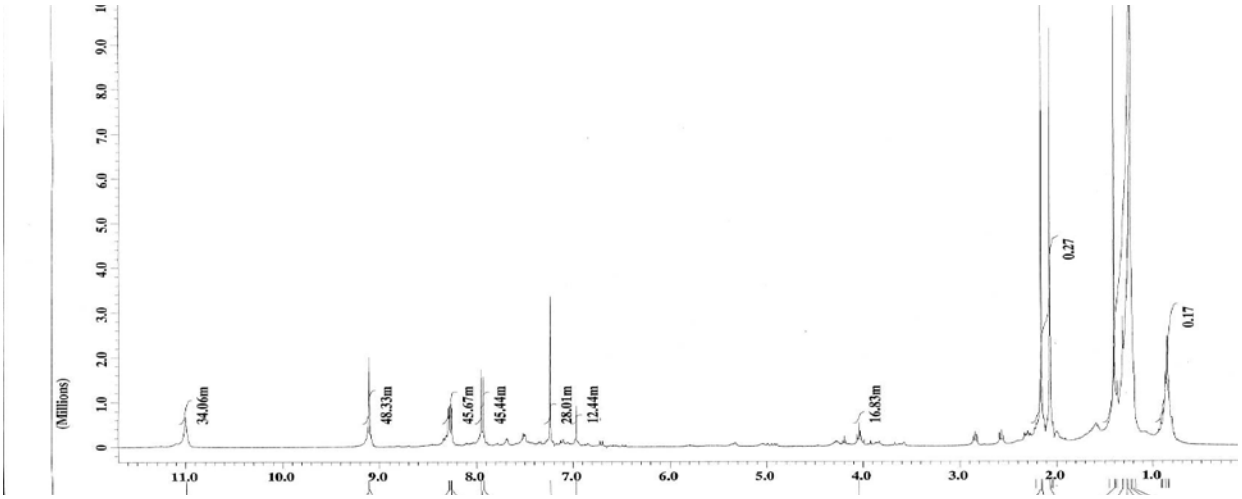
3- A(vanillin)




VI




Fig(2) 1- (2,4 dinitrophenyl amino ) ,-4 – (4-hydroxybenzylidene )-2- phenyl , 5- ketone ,imidazole (VI)

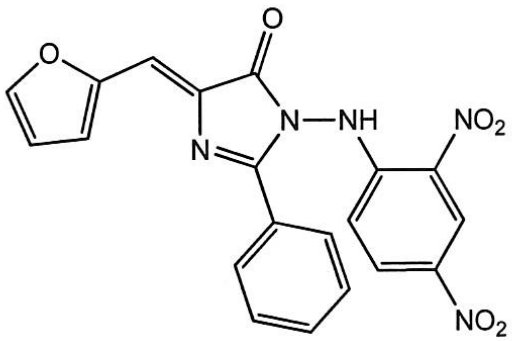
.

### 1.4.3. 1-(2,4-dinitrophenylamino)- 4 – (2-furanyl)- 2-phenyl,5- ketone, imidazole (VII)[F]

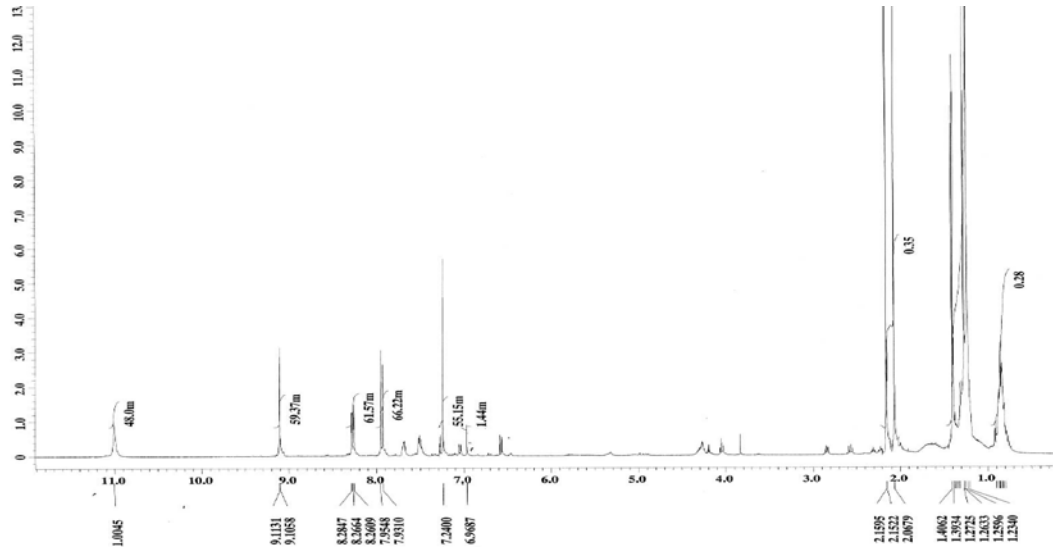
11.0009ppm (s) , 9.1076 ppm (d) ,8.2627 ppm (t) ,7.9273 ppm (d) , 7.2400 ppm (s) ,6.9678 ppm (s), 4.045 ppm (t) ,2.1504 ppm (dd) ,1.600 ppm (s), 1.2706 ppm (m)

IR : 1700 cm-1 (C=O st. ) , 1350 cm-1 ( CH=C st.) ,1200 cm-1 ( C=C st)

F(fural)



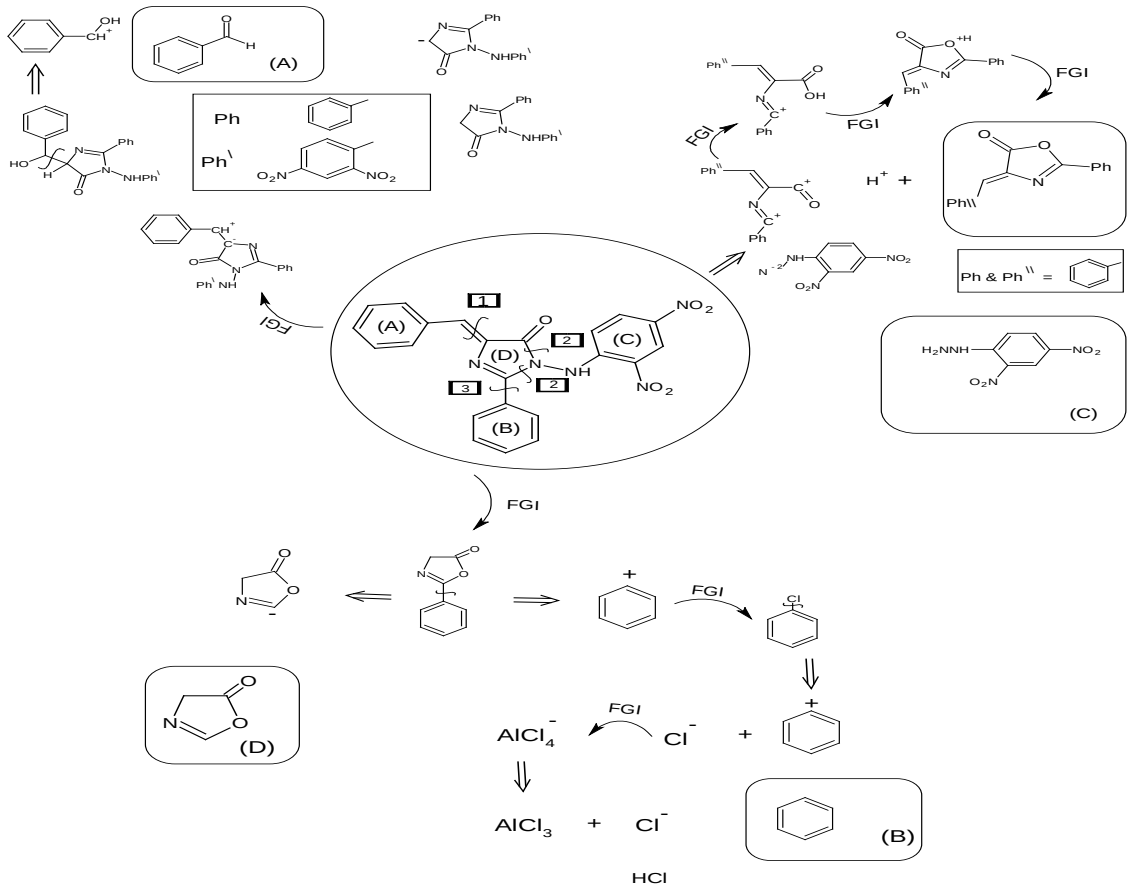

VII



Fig(3) 1-(2,4-dinitrophenylamino)- 4 – (2-furanyl)- 2-phenyl,5- ketone, imidazole (VII)

Base on the $H^1$NMR &FT-IR data have found to be exact functional groups were confirm for the three synthesized compounds V,VI and VII, The details were monitored in the figures for both of them ($H^1$NMR&FT-IR) as discussed before.

**Retro synthetic analysis of Imidazole**



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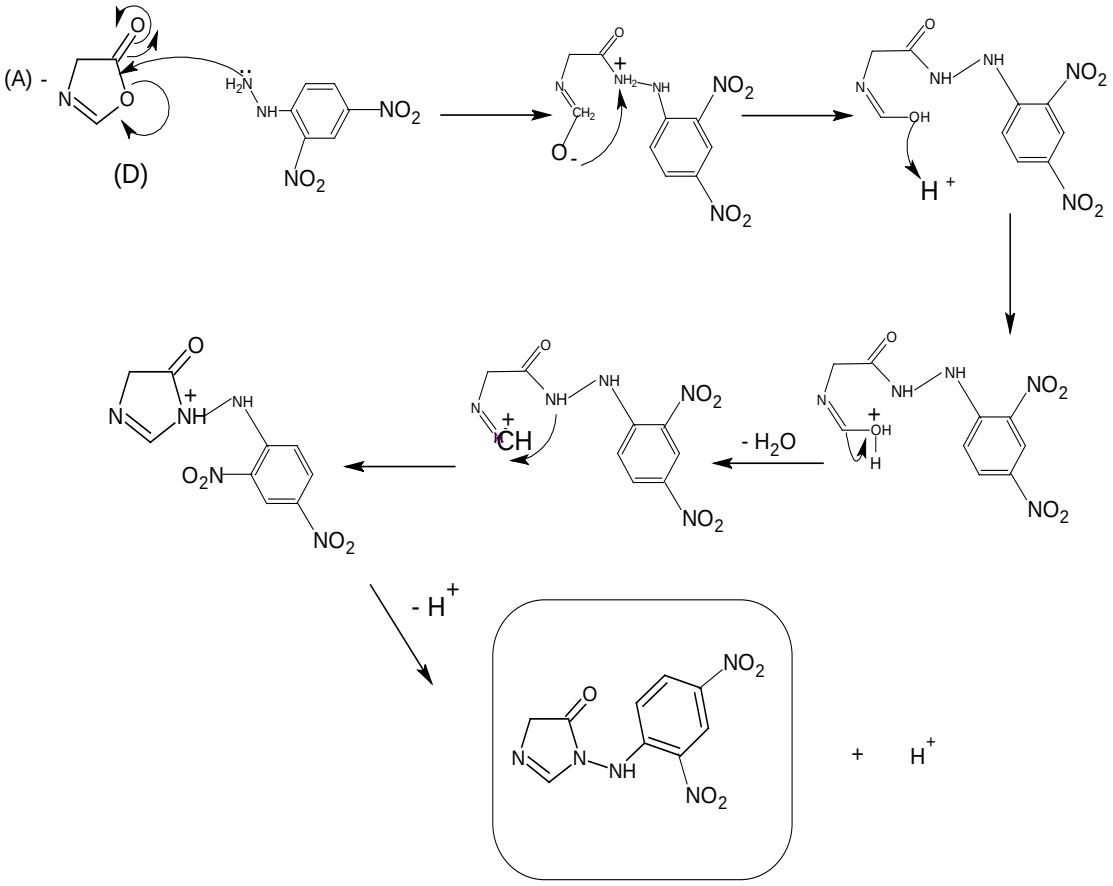
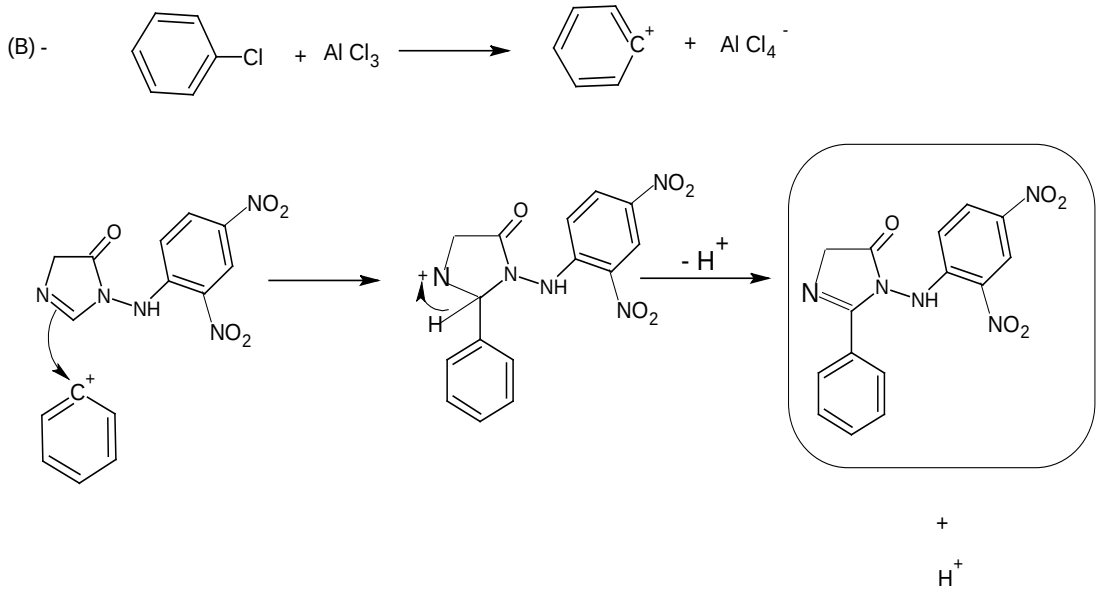
And the expected mechanism for these reactions can be assigned here

**The Mechanism of Imidazole**



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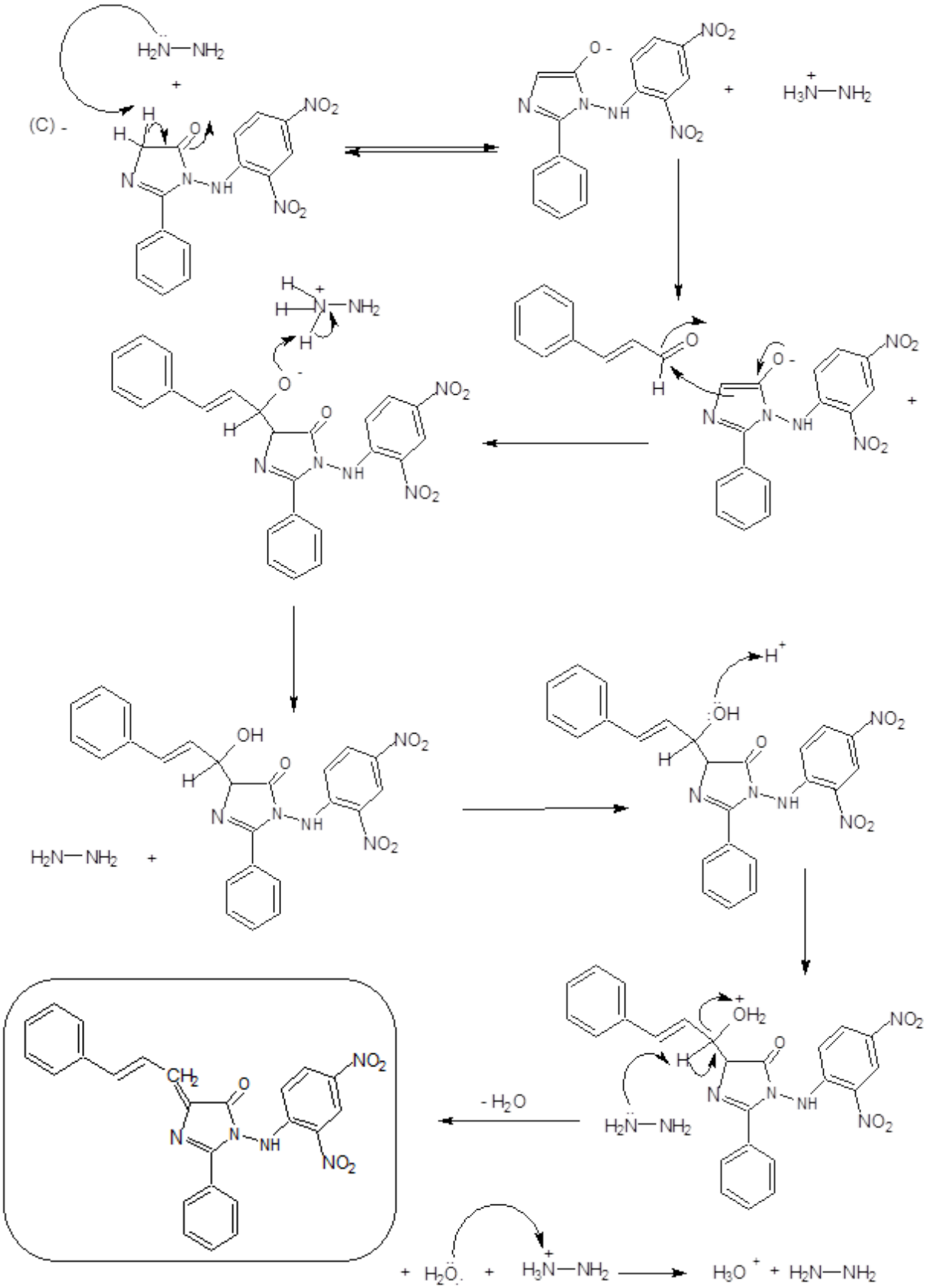

## 1.5 Conclusion

From results and discussion, the confirm compounds were well synthesized and characterized

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