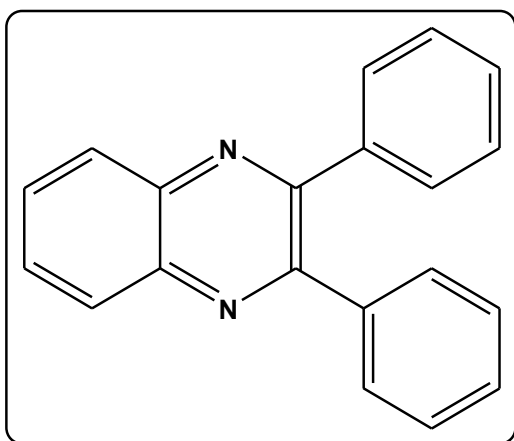


### Scheme: 1

A mixture of 1,2-diamino-benzene (2mmol), benzil (2mmol) and catalytic amount of H-ZSM-5 (Si/Al ratio 30) were taken in methanol and stirred at room temperature. The reaction was monitored by TLC till its completion. After 10 minutes, the mixture was then poured into ice-water. Solid reaction product was filtered out. The quinoxaline product thus formed was purified by solvent recrystallization using ethanol as solvent. The product thus obtained was confirmed by measuring its melting point first.  $^1\text{H}$  NMR spectrum on Varian's Avance 400 MHz and Fourier Transform - Infra Red (FT-IR) spectrum on Shimadzu IR Spectrophotometer were recorded to confirm the structural aspects of the product (*cf.* Figure 7 & 8 in this paper). In future works, *Isak R. Shaikh et al.* might try synthesizing other quinoxaline derivatives over this catalyst in high purity and quantitative yields.



Compound Name: 2,3-diphenylquinoxaline

Molecular Formula:  $\text{C}_{20}\text{H}_{14}\text{N}_2$

Yield : 88%

Solvent system: n-hexane/ethyl acetate(7:3)

M.P.: 125-127  $^{\circ}\text{C}$

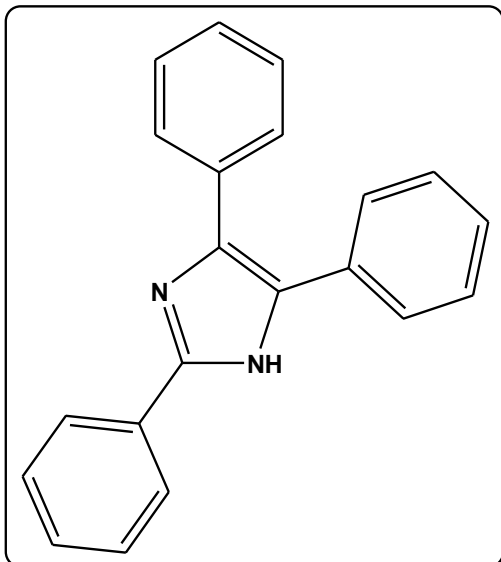
$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) in **Figure 7**:  $\delta$  8.18 (d, 2H), 7.78 (d, 2H), 7.48 (m, 4H), 7.34 (m, 4H), 7.22 (s, 2H).

IR in **Figure 8** (KBr)  $\nu_{\text{max}}/\text{cm}^{-1}$ : 3057, 1602, 1442, 1346, 1246, 852, 767, 698

### Scheme: 2

A mixture of benzaldehyde (2mmol), benzil (2mmol), ammonium acetate (2mmol) and H-ZSM-5 (with Si/Al 30) catalyst (10mg) in ethanol refluxed in ethanol and the reaction was monitored by TLC ( $\text{CHCl}_3/\text{MeOH}$  (8:2)) until the completion. Reaction mixture was then cooled and the product was precipitated by pouring the reaction mixture into ice-water. The product is collected after

filtration, washed with water and dried. The melting point was measured and compared it with that available in scientific literature.



Compound Name: *2,4,5-triphenyl-1H-imidazole*

Molecular Formula:  $C_{21}H_{16}N_2$

Yield : 80%(dull white solid)

Solvent system:  $CHCl_3/MeOH(8:2)$

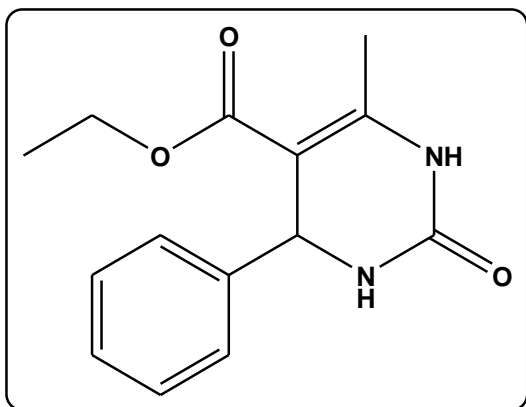
**M.P.:** 274-275<sup>0</sup>C

**IR (KBr)**  $\nu_{max}/cm^{-1}$  : 3434,2993, 2470, 1638, 1216;

**<sup>1</sup>H NMR** (DMSO; 300 MHz): 12.61 (1H, s), 7.42-8.12 (15H, m)

### Scheme: 3

A reaction mixture containing benzaldehyde (2mmol), ethyl acetoacetate (2mmol), urea (2.2mmol) and H-ZSM-5 (with Si/Al 30) catalyst (20mg) was refluxed. After the completion of the reaction, the chemical mixture was poured into crushed ice (10g) and stirred for 5-10 minutes and a solid was separated by filtration. The product was many times with cold ethanol and recrystallized from ethanol. The measurement of melting point, TLC purity spot and solubility study were good enough in identifying this known product.



Compound Name: *ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-phenylpyrimidine-5-carboxylate*

Molecular Formula:  $C_{14}H_{16}O_3N_2$

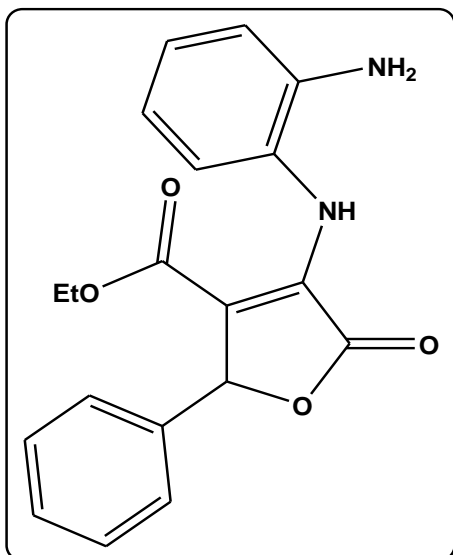
Yield : 80% (white solid)

Solvent system: pet ether/Ethyl acetate (8:2)

**M.P.:** 203-205 °C

#### Scheme: 4

A mixture of 1,2-diamino benzene(2mmol), aldehyde (2mmol) and dialkylacetylenedicarboxylate were subjected to catalyzed reaction over H-ZSM-5 with Si/Al 30 (10mg) in methanol at room temperature. The reaction was monitored by TLC (70% EtOAc/n-hexane). The reaction was worked up by pouring mixture into ice water and a solid product is obtained. The solid is filtered out, washed and purified by solvent recrystallization. Similar reaction with a methyl substituent was also catalyzed successfully. The details of products and their characterization are given below.



Compound Name: *ethyl 4-(2-aminophenylamino)-2,5-dihydro-5-oxo-2-phenylfuran-3-carboxylate*

Molecular Formula:  $C_{19}H_{18}N_2O_4$

Yield: 85% (yellow solid)

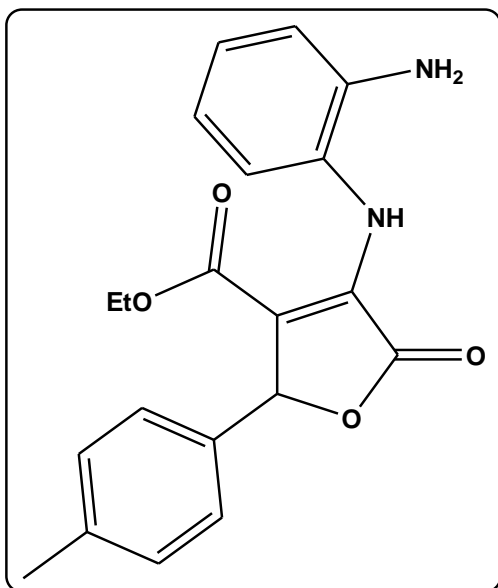
Solvent system: 70% EtOAc/n-hexane

**M.P.:** 198-199 °C

**IR (KBr)**

$\nu_{max}/cm^{-1}$ : 3292, 1716, 1684, 1655  $cm^{-1}$

**$^1H$  NMR** ( $CDCl_3$ , 300 MHz): 1.19 (t, 3H,  $J_{\frac{1}{4}}$  6.6 Hz) - $CH_3$ . 4.16 (q, 2H,  $J_{\frac{1}{4}}$  7.3 Hz), 5.69 (s, 1H) -benzylic proton, 7.03–7.32 (m, 5H) –aromatic proton, 7.45 (m, 5H) –aromatic, 4.90(s, 1H) -NH, 5.04 (s, 2H) - $NH_2$  proton



Compound Name: *ethyl 4-(2-aminophenylamino)-2,5-dihydro-5-oxo-2-p-tolylfuran-3-carboxylate*

Molecular Formula:  $C_{20}H_{20}N_2O_4$

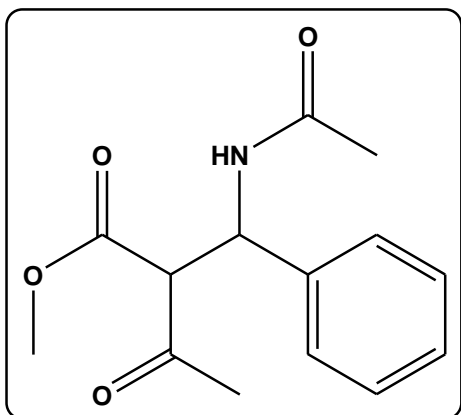
Yield: 81%

M.P.: 190-192 °C

**IR (KBr)**  $\nu_{max}/cm^{-1}$ : 3390 (corresponding to N-H stretching), 2930, 1715, 1680, 1586, 1488, 1248  $cm^{-1}$   
 **$^1H$  NMR** ( $CDCl_3$ , 300 MHz):  $\delta$  1.29 (t, 3H,  $J=8.0$  Hz,  $-CH_2CH_3$ ), 2.22 (s, 3H,  $CH_3$ ), 4.14 (q, 2H,  $J=7.3$  Hz,  $-OCH_2$ ), 5.67 (s, 1H, benzylic), 6.95–7.30 (m, 9H, aromatic), 4.96 (s, 1H, amine), 5.02 (s, 2H, amine)

#### Scheme: 5

Ethyl acetoacetate (2mmol), benzaldehyde (2mmol), H-ZSM-5 (15mg) were charged in round-bottom flask containing 7ml acetonitrile. Acetyl chloride (3mmol) was added and the reaction mixture was stirred at 40°C till the completion as evident by TLC reaction monitoring technique (EtOAc/pet ether = 1:9). The reaction mixture was then allowed to settle down for about 5 days. Upper layer of liquid from the reaction mixture was carefully decanted leaving H-ZSM-5 catalyst at the bottom. The liquid was poured into 10gm ice-water mixture and a product was precipitated out. This was further separated by careful filtration, dried and used as it was for measuring its melting point.



Compound Name: *methyl 2-(acetamido(phenyl)methyl)-3-*

*oxobutanoate*      Molecular      Formula:      C<sub>14</sub>H<sub>17</sub>O<sub>4</sub>N

Yield:                  60%                  (white                  solid)

Solvent      system:                  EtOAc/pet      ether      (1:9)

**M.P.:** 127-129 °C