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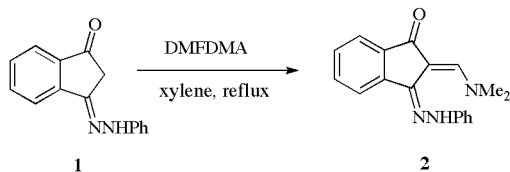
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The hydrazone **1** reacts with DMFDMA to give 2-dimethylaminomethylene-3-(phenylhydrazono)-indan-1-one (**2**) which reacts with hydrazine hydrate and the pyrazole derivative **4** to afford the indenopyrazole derivatives **3** and the indenofluorene **5** respectively. The reaction of **2** with the active methylene compounds, mainly malononitrile, cyanoacetamide and malononitrile dimer was investigated and found to proceed successfully to yield the indenopyran **7**, indenopyridine **8b** and trinitrile **9** respectively. Compound **2** reacted with 1*H*-benzimidazole-2-acetonitrile **10** to give to the diazaindenofluorene derivative **11**. Also, **2** reacted with ω-cyano compounds **12a,b** to afford the indenopyran **14**. On the other hand the hydrazone **1** was allowed to react with the enaminones **15**, **18** and **21** affording the diazabenzozulene derivatives **17**, **20** and the indeno[1,2-*b*]pyridin **23**, respectively.

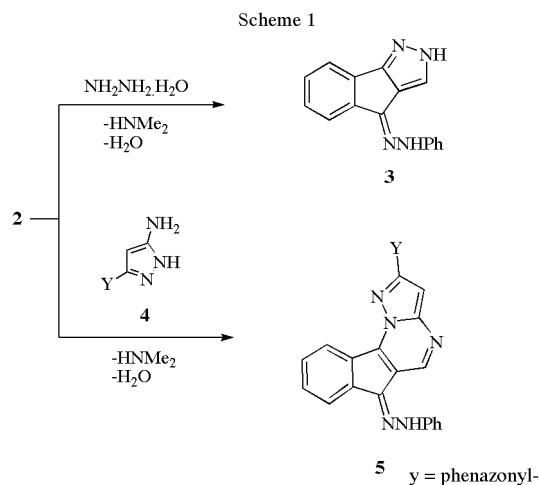
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Enaminones are versatile reagents and their utility in heterocyclic synthesis has recently received considerable attention [1-6]. Our interest in the development of new and simple methods for the synthesis of heterocycles substituted with multiple functional groups with anticipated biological activity using indane-1,3-dione derivatives, that are known as anti-inflammatory agents [7], as blood anticoagulants [8] and miticides [9]. Also, we have recently published new approaches for synthesis of fused indenes as potential biodegradable agrochemicals [10-12]. These interesting biological activities prompted us to synthesize other fused indene derivatives. We report here an efficient synthesis of a variety of target molecules utilizing the readily obtainable 3-(phenylhydrazono)indan-1-one (**1**) and 2-dimethylaminomethylene-3-(phenylhydrazono)-indan-1-one (**2**) as starting materials. Thus, **1** when reacted with dimethylformamide dimethylacetal (DMFDMA) in refluxing xylene, yielded the enaminone **2**. Structure **2** was assigned as a reaction product based on correct spectral and analytical data.



Compound **2** reacted with hydrazine hydrate in acetic acid at reflux temperature to yield the indenopyrazole derivative **3** [13]. It's assumed that compound **3** is formed *via* initial addition of the amino group in hydrazine to the enamine double bond in **2** followed by elimination of dimethylamine and water molecules to give the final isolable product **3**. As previously reported enaminones can be used as potential precursors for fused heterocyclic

systems when reacting with heterocyclic amines. Thus treatment of compound **2** with 5-amino-3-phenazonyl-1*H*-pyrazole (**4**), in refluxing acetic acid gave the indenofluorene derivative **5** (Scheme 1). The formation of compound **5** can be explained on the basis of an initial Michael addition of the exocyclic NH<sub>2</sub> in **4** to the double bond in **2** followed by elimination of dimethylamine and water molecules to give **5**, a product that was found to be in complete agreement with spectral and analytical data.

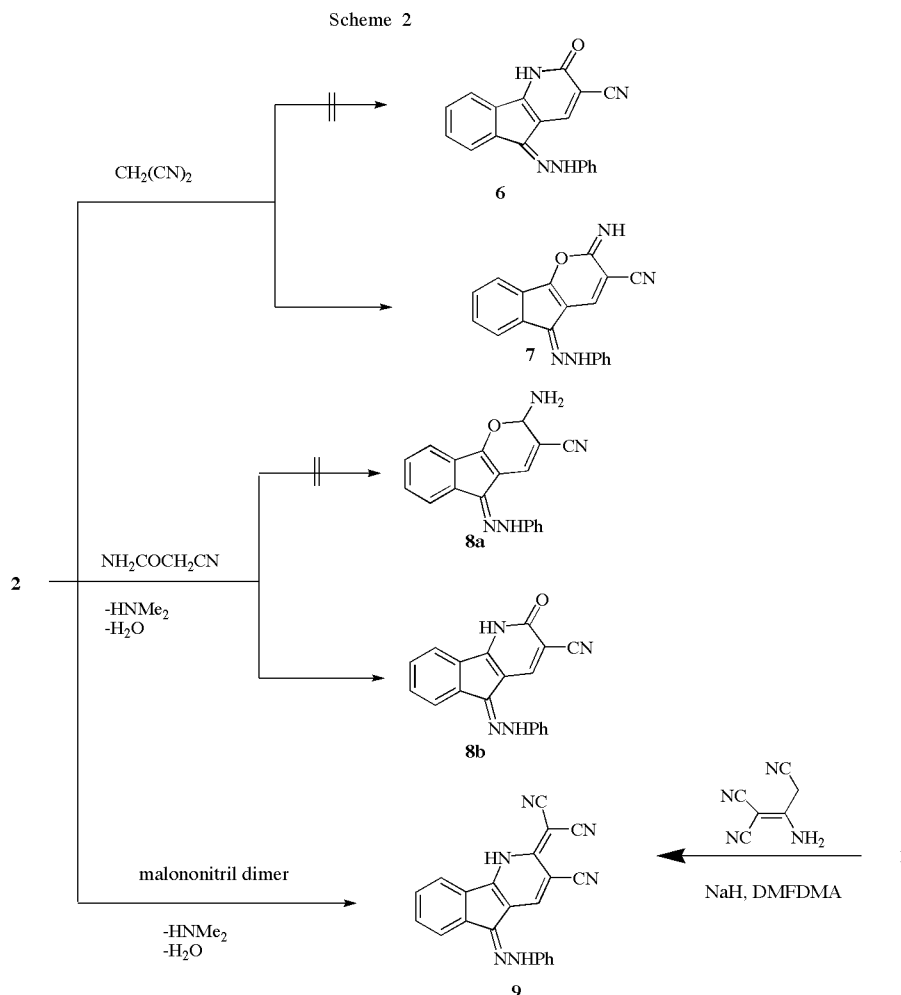


Two possible structures have been proposed for the reaction of compound **2** with malononitrile in refluxing ethanolic sodium ethoxide. Structure **6** was eliminated based on <sup>1</sup>H-NMR spectrum. The presence of the 4-H signal at δ = 8.35 and imine signal at δ = 3.07 are characteristic features of the indenopyran **7**. In contrast to its reactivity with malononitrile, when **2** react with cyanoacetamide under the

same conditions, the indenopyridine derivative **8b** was formed. Compound **8b** was assigned as reaction product based on IR the spectrum which shows the amidic carbonyl absorption at 1685 and  $^1\text{H-NMR}$  spectrum which reveals the absence of two protons relevant to the  $\text{NH}_2$  group. Also, the  $^{13}\text{C-NMR}$  spectrum of compound **8b** shows the presence of the carbonyl carbon absorption characteristic of the pyridine structure. The previous spectral data proves the indenopyridine structure and eliminates the possibility of the isomeric aminopyran **8a**. Compound **2** was also reacted with malononitrile dimer in refluxing ethanolic sodium ethoxide to give the trinitrile **9**. This product was elucidated by independent synthesis from reacting the dimer with the hydrazone **1** and DMFDMA under nitrogen atmosphere [14]. The two reaction products were identical in all spectral data (Scheme 2).

**11** formed *via* initial Michael type addition of the endocyclic NH in **10** to the double bond in **2** followed by elimination of dimethylamine and water molecules to give the final isolable product **11** (Scheme 3). Replacement of malononitrile with the  $\omega$ -cyano compounds **12a,b** in reaction with **2** resulted in an unidentified product. However, carrying out the reaction in ethanol containing a catalytic amount of piperidine afforded the target compound **14**. The indenopyran **14** was assigned as a reaction product based on correct spectral and analytical data.

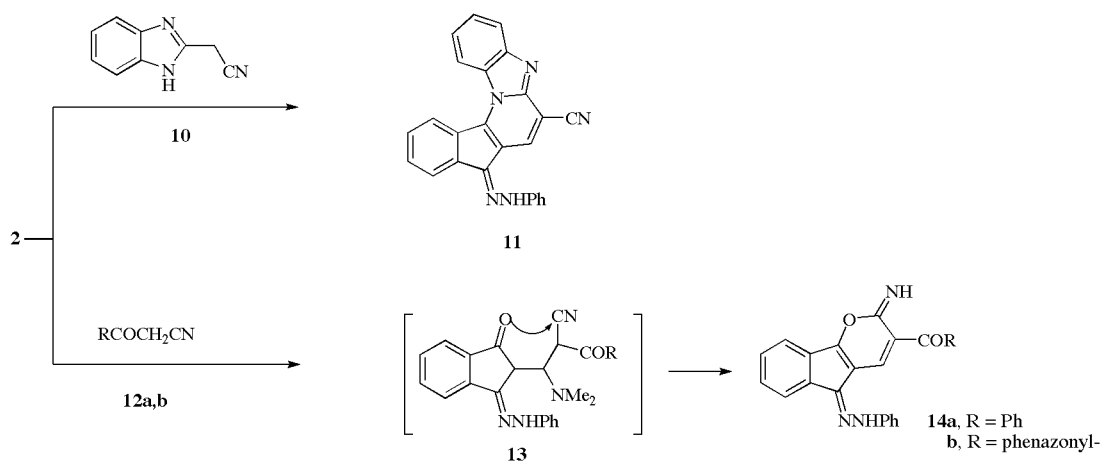
Continuing our work to synthesize a variety of fused indeno systems, compound **1** was allowed to react with the enaminones **15**, **18**, and **21**. Thus **1**, when combined with 2-dimethylaminomethylene-5,5-dimethylcyclohexane-1,3-dione **15**, provided the diazabenzozulene derivative **17**. The spectral and analytical data were found to be in



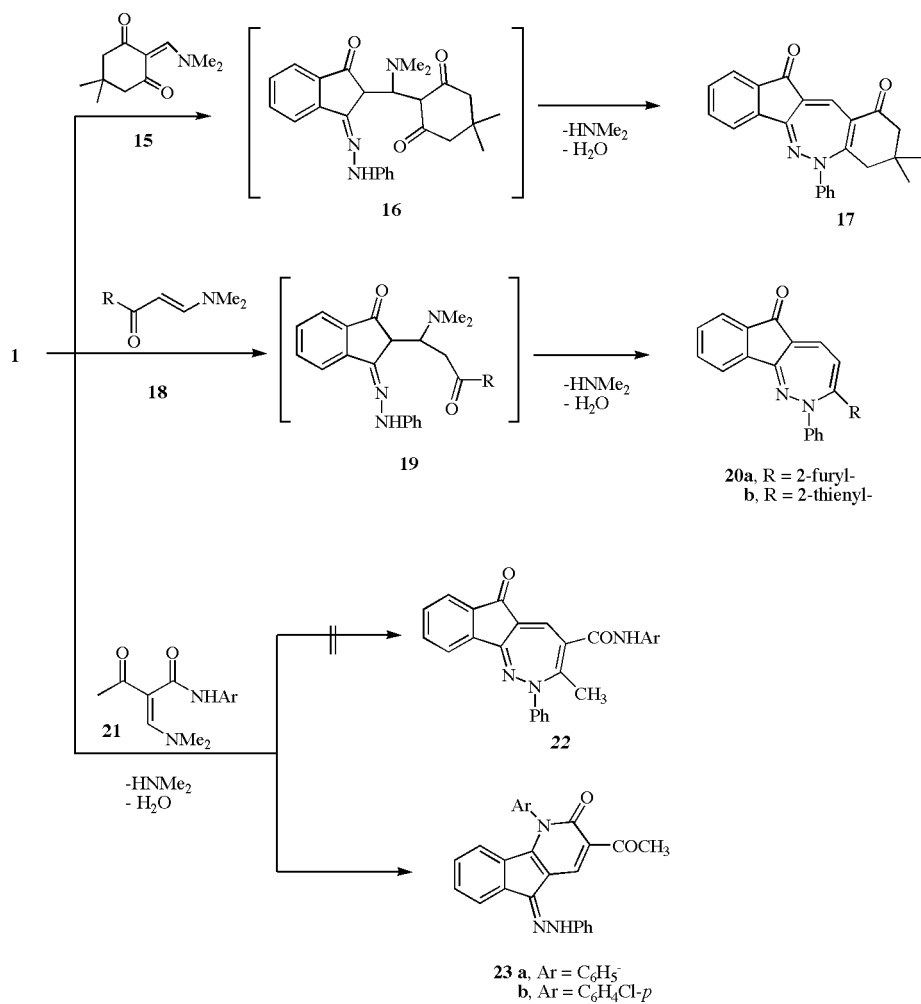
On the other hand, compound **2** reacted with 1*H*-benzimidazol-2-acetonitrile **10** in ethanol containing a catalytic amount of piperidine at reflux temperature to afford the diazaindenofluorene derivative **11**. Presumably, compound

complete agreement with the proposed structure. Similarly, **1** reacted with enaminone **18** to afford the diazabenzozulene **20**. The structure of **20** was elucidated based on  $^1\text{H-NMR}$ , which revealed the presence of

Scheme 3



Scheme 4



doublet-doublet signal characteristic of the H-4 and H-5 of the 1,2-diazepine structure. On the other hand, reacting **1** with the enamionone **21** [15] gave the indenopyridine **23** rather than the diazabenzazulene **22**. Compound **22** was ruled out based on mass spectra and <sup>1</sup>H-NMR spectra, which lacked the H-5 signal characteristic of the diazabenzazulene structure.

### EXPERIMENTAL

All melting points are uncorrected. IR spectra were recorded in KBr disks using a Shimadzu IR-740 spectrophotometer. <sup>1</sup>H- and <sup>13</sup>C-NMR spectra were recorded on a Bruker AC-300 spectrometer with [<sup>2</sup>H<sub>6</sub>]DMSO as solvent and TMS as internal standard; chemical shifts are reported in δ units (ppm). Mass spectra were measured on GC/MS INCOS XL Finnigan MAT. Microanalysis was performed on LECOCHNS-932.

Preparation of 2-Dimethylaminomethylene-3-(phenylhydrazono)-indan-1-one (**2**).

A mixture of compound **1** (0.01 mol, 2.36 g) and DMFDMA (0.01 mol, 1.26 g) in xylene (30 ml) was heated under reflux for 2 hours. The reaction mixture was then evaporated under vacuum. The remaining oil was triturated with ethanol to give a solid product that was collected by filtration and crystallized from ethanol to give compound **2** as deep brown crystals, m.p. 195-197 °C, yield 75%,  $v_{\max}$ : 3320(NH), 1710(CO), 1620(C=N). <sup>1</sup>H-NMR ([<sup>2</sup>H<sub>6</sub>]DMSO): δ 2.47 (s, 6H, 2CH<sub>3</sub>), 6.46-7.80 (m, 10H, aromatic-H and =CHN), 11.1 (s, 1H, NH); <sup>13</sup>C-NMR ([<sup>2</sup>H<sub>6</sub>]DMSO): δ 187.0 (CO-carbon), 155.6 (C=N-carbon), 155.1, 108.7 (vinyl-carbon), 146.7, 137.2, 134.4, 132.4, 131.3, 129.8, 129.5, 129.3, 129.3, 118.5, 115.1, 115.1 (aromatic-carbons), 41.2 (methyl-carbons). MS: m/z 291(M<sup>+</sup>).

*Anal.* Calcd. for C<sub>18</sub>H<sub>17</sub>N<sub>3</sub>O (291.35): C, 74.20; H, 5.88; N, 14.42 %. Found: C, 74.5; H, 6.00; N, 14.42.

Reaction of **2** with Hydrazine Hydrate and 3-Amino-5-phenazonylpyrazole: Formation of Compounds **3** and **5**.

To a solution of **2** (0.01 mol, 2.91 g) in acetic acid (30 ml) was added (0.01 mol) of hydrazine hydrate or 3-amino-5-phenazonylpyrazole. The mixture was then heated under reflux for 6 hours. The solid deposited after cooling was then collected by filtration and crystallized from the appropriate solvent.

*N*-(2*H*-indino[1,2-*c*]pyrazol-4-ylidene)-*N'*-phenylhydrazine (**3**).

Compound **3** was obtained as yellow crystals from dioxane, m.p. 297-299 °C, yield 70%;  $v_{\max}$ : 3390-3300(NH), 1620(C=N). <sup>1</sup>H-NMR ([<sup>2</sup>H<sub>6</sub>]DMSO) δ: 6.46-7.55 (m, 10H, arom-H + vinyl-H), 11.1 (s, 1H, NH), 13.1 (s, 1H, NH). <sup>13</sup>C-NMR ([<sup>2</sup>H<sub>6</sub>]DMSO) δ: 155.6 (C=N-carbon), 150.0, 134.0, 104.0 (pyrazol-carbons), 146.7, 137.0, 131.3, 129.7, 129.5, 129.3, 129.3, 128.6, 127.1, 118.5, 115.1, 115.1 (aromatic-carbons). MS: m/z 260(M<sup>+</sup>).

*Anal.* Calcd. for C<sub>16</sub>H<sub>12</sub>N<sub>4</sub> (260.29): C, 73.83; H, 4.65; N, 21.52 %. Found: C, 74.00; H, 4.61; N, 21.43.

*N*-Phenyl-*N'*-(2-phenazonyl-1,4,10c-triazacyclopenta[*c*]fluoren-6-ylidene)hydrazine (**5**).

Compound **5** was obtained as yellow crystals from ethanol, m.p. 243-245 °C, yield 67%.  $v_{\max}$ : 3330(NH), 1665(CO-phenazonyl). <sup>1</sup>H-NMR ([<sup>2</sup>H<sub>6</sub>]DMSO) δ: 1.71 (s, 3H, CH<sub>3</sub>), 2.47

(s, 3H, CH<sub>3</sub>), 6.2 (s, 1H, pyrazole-CH), 6.5-7.7 (m, 14H, arom-H), 9.14 (s, 1H, CH) 11.1 (s, 1H, NH).

*Anal.* Calcd. for C<sub>30</sub>H<sub>23</sub>N<sub>7</sub>O (497.55): C, 72.42; H, 4.66; N, 19.71 %. Found: C, 72.00; H, 4.61; N, 19.43.

Reaction of **2** with Active Methylene Compounds: Formation of compounds **7**, **8b** and **9**.

Method A.

To a solution of **2** (0.01 mol, 2.91 g) in ethanolic sodium ethoxide solution (0.23 g sodium metal in 30 ml absolute ethanol) was added (0.01 mol) of each of malononitrile, cyanoacetamide and malononitrile dimer. The reaction mixture was heated under reflux for 3 hours, and then poured into ice-cold water. The solution was acidified with dilute HCl and the solid deposited was collected by filtration and crystallized from the proper solvent.

Method B: for Compound (**9**).

A mixture of hydrazone **1** (0.01 mol), dry DMF (10 mL) and dimethylformamide dimethyl acetal (1.19 g, 0.01 mol) was stirred under an inert atmosphere at room temperature for 24 hours. In a second flask, a mixture of dry DMF (10 mL), sodium hydride (0.48 g, 0.02 mol) and malononitrile dimer (1.32 g, 0.01 mol) was stirred at an inert atmosphere for 10 minutes. The contents of the second flask were transferred by syringe into the first flask and the resulting mixture was stirred for 24 hours. A mixture of EtOH (25 mL) and water (25 ml) was added, the mixture was acidified with HCl (36%) to pH 4, and the stirring was continued for 24 hours. The product was recovered by filtration and recrystallised to give **9**.

2-Imino-5-(phenylhydrazono)-2,5-dihydroindeno[1,2-*b*]pyran-3-carbonitrile (**7**).

Compound **7** was obtained as orange crystals from ethanol/dioxane, m.p. 233-235 °C, yield 65%;  $v_{\max}$ : 3350-3275(NH), 2210(CN). <sup>1</sup>H-NMR ([<sup>2</sup>H<sub>6</sub>]DMSO) δ: 3.07 (s, 1H, NH), 6.46-7.6 (m, 9H, arom-H), 8.35 (s, 1H, pyran-4H), 11.1 (s, 1H, NH). <sup>13</sup>C-NMR ([<sup>2</sup>H<sub>6</sub>]DMSO) δ: 164.0, 155.6 (imine-carbons), 157.3, 151.0, 99.0, 97.0 (pyran-carbons), 117.2 (nitrile-carbon), 146.7, 135.0, 130.7, 129.3, 129.3, 128.9, 128.9, 127.8, 126.3, 118.5, 115.1, 115.1 (aromatic-carbons). MS: m/z 312(M<sup>+</sup>).

*Anal.* Calcd. for C<sub>19</sub>H<sub>12</sub>N<sub>4</sub>O (312.32): C, 73.07; H, 3.87; N, 17.94 %. Found: C, 73.00; H, 3.61; N, 17.43.

2-Oxo-5-(phenylhydrazono)-2,5-dihydro-1*H*-indeno[1,2-*b*]pyridine-3-carbonitrile (**8b**).

Compound **8b** was obtained as yellow crystals from ethanol/dioxane, m.p. 285 °C, yield 70%;  $v_{\max}$ : 3385, 3350(NH), 2210(CN), 1685(CO-amide). <sup>1</sup>H-NMR ([<sup>2</sup>H<sub>6</sub>]DMSO) δ: 6.46-7.6 (m, 9H, arom-H), 8.11 (s, 1H, H-4), 11.1 (s, 1H, NH), 12.4 (s, 1H, NH).

*Anal.* Calcd. for C<sub>19</sub>H<sub>12</sub>N<sub>4</sub>O (312.32): C, 73.07; H, 3.87; N, 17.94 %. Found: C, 73.20; H, 3.66; N, 17.63.

2-[3-Cyano-5-(phenylhydrazono)-1,5-dihydroindeno[1,2-*b*]pyridin-2-ylidene]malononitrile (**9**).

Compound **9** was obtained as reddish brown powder from ethanol/dioxane, m.p. 252-254 °C, yield 74%;  $v_{\max}$ : 3350, 3300(NH), 2220, 2210(CN), 1590(C=N). <sup>1</sup>H-NMR

( $^2\text{H}_6$ )DMSO)  $\delta$ : 6.46-7.6(m, 9H, arom-H), 8.43(s, 1H, H-4), 11.1(s, 1H, NH), 12.4(s, 1H, NH). MS: m/z 360( $\text{M}^+$ ).

Anal. Calcd. for  $\text{C}_{22}\text{H}_{12}\text{N}_6$  (360.37): C, 73.32; H, 3.36; N, 23.32 %. Found: C, 73.20; H, 3.66; N, 23.63.

Reactions of **2** with Benzimidazol-2-acetonitrile (**10**) and  $\omega$ -Cyano Compounds **12a,b**: Formation of Compounds **11** and **14a,b**.

To a solution of **2** (2.91 g, 0.01 mol) in ethanol (30 mL) containing piperidine (0.5 mL) as a catalyst, was added (0.01 mol) of compound **10** or compound **12**. The reaction mixture was heated under reflux for several hours. And the progress was monitored by tlc. The solid deposited after cooling was collected by filtration and recrystallized from the proper solvent to give **11**, **14a,b** respectively.

8-(Phenylhydrazono)-8H-5,12c-diazaindeno[2,1-c]fluorene-6-carbonitrile (**11**).

Compound **11** was obtained as brown crystals from ethanol/dioxane, m.p. 238-240 °C, yield 80 %  $v_{\text{max}}$ : 3330(NH), 2220(CN), 1590 (C=N).  $^1\text{H-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 6.46-7.9 (m, 13H, arom-H), 8.23 (s, 1H, H-4), 11.1 (s, 1H, NH). MS: m/z 385( $\text{M}^+$ ).

Anal. Calcd. for  $\text{C}_{25}\text{H}_{15}\text{N}_5$  (385.42): C, 77.91; H, 3.92; N, 18.17 %. Found: C, 77.90; H, 3.66; N, 18.63.

[2-Imino-5-(phenylhydrazono)-2,5-dihydroindeno[1,2-b]pyran-3-yl]-phenylmethanon (**14a**).

Compound **14a** was obtained as orange crystals from ethanol, m.p. 261 °C, yield 68%.  $v_{\text{max}}$ : 3360, 3335(NH), 1710(CO), 1595(C=N).  $^1\text{H-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 3.1 (s, 1H, NH), 6.62-7.84 (m, 14H, arom-H), 8.23 (s, 1H, H-4), 11.1 (s, 1H, NH).

Anal. Calcd. for  $\text{C}_{25}\text{H}_{17}\text{N}_3\text{O}_2$  (391.42): C, 76.71; H, 4.38; N, 10.74 %. Found: C, 76.20; H, 4.66; N, 10.63.

4-[2-Imino-5-(phenylhydrazono)-2,5-dihydroindeno[1,2-b]pyran-3-carbonyl]-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (**14b**).

Compound **14b** was obtained as yellow crystals from ethanol/dioxane, m.p. 270-272 °C, yield 78%.  $v_{\text{max}}$ : 3350, 3335(NH), 1710(CO), 1635 (CO), 1595(C=N).  $^1\text{H-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 1.71 (s, 3H,  $\text{CH}_3$ ), 2.47 (s, 3H,  $\text{CH}_3$ ), 3.1 (s, 1H, NH), 6.62-7.84 (m, 14H, arom-H), 8.40 (s, 1H, H-4), 11.1 (s, 1H, NH).

Anal. Calcd. for  $\text{C}_{30}\text{H}_{23}\text{N}_5\text{O}_3$  (501.54): C, 71.84; H, 4.62; N, 13.96 %. Found: C, 71.70; H, 4.66; N, 13.63.

Reaction of **1** with Enaminones **15**, **18** & **21**: Formation of Compounds **17**, **20** and **23**.

A mixture of compound **1** (0.01 mol) and each of the enaminones **15**, **18**, **21** (0.01 mol) in acetic acid (30 mL) was heated under reflux for several hours. The reaction was followed by tlc. The volatile components were evaporated *in vacuo* and the residue was recrystallized from an appropriate solvent to give **17**, **20** and **23** respectively.

8,8-Dimethyl-6-phenyl-6,7,8,9-tetrahydro-diazabenzofluorene-10,12-dione (**17**).

Compound **17** was obtained as brown crystals from benzene/ethanol, m.p. 148-150 °C, yield 60%.  $v_{\text{max}}$ : 1710(CO), 1700(CO).  $^1\text{H-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 1.11 (s, 6H,  $\text{diCH}_3$ ), 2.45

(s, 2H,  $\text{CH}_2$ ), 2.86 (s, 2H,  $\text{CH}_2$ ), 6.62-7.84 (m, 9H, arom-H), 8.40 (s, 1H, H-5).  $^{13}\text{C-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 197.6, 187.0 (CO-carbons), 155.6 (imine-carbon), 152.1, 144.7, 133.7, 112.4 (ethylene-carbons), 146.7, 137.2, 134.4, 134.4, 132.4, 129.8, 129.5, 129.3, 118.5, 115.1, 115.1 (aromatic-carbons), 53.7, 43.3 ( $\text{CH}_2$ -aliphatic), 17.0 (CH-aliphatic), 26.8, 26.8 (methyl-carbons).

Anal. Calcd. for  $\text{C}_{24}\text{H}_{20}\text{N}_2\text{O}_2$  (368.43): C, 78.24; H, 5.47; N, 7.60 %. Found: C, 78.20; H, 5.66; N, 7.63.

8-Furan-3-yl-6-phenyl-6H-5,6-diazabenzofluorene-10-one (**20a**).

Compound **20a** was obtained as deep brown crystals from ethanol/DMF, m.p. 203-205 °C, yield 80%.  $v_{\text{max}}$ : 1700(CO), 1590(C=N).  $^1\text{H-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 4.82 (d, 1H, J = 5.1, H-4), 6.06-7.8 (m, 12H, arom-H), 8.1 (d, 1H, J = 8.3, H-5).  $^{13}\text{C-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 187.0 (CO-carbon), 155.6 (imine-carbon), 144.7, 133.7, 127.7, 119.4 (ethylene-carbons), 146.7, 143.2, 139.3, 137.2, 134.4, 132.4, 131.3, 129.8, 129.5, 129.3, 129.3, 124.5, 118.5, 115.1, 115.1, 107.7 (aromatic-carbons).

Anal. Calcd. for  $\text{C}_{22}\text{H}_{14}\text{N}_2\text{O}_2$  (338.36): C, 78.09; H, 4.17; N, 8.28 %. Found: C, 78.29; H, 4.32; N, 8.38.

6-Phenyl-8-thiophene-3-yl-6H-5,6-diazabenzofluorene-10-one (**20b**).

Compound **20b** was obtained as deep brown crystals from ethanol/DMF, m.p. 210-212 °C, yield 70%.  $v_{\text{max}}$ : 1700(CO), 1585(C=N).  $^1\text{H-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 4.50 (d, 1H, J = 5.1, H-4), 6.20-7.65 (m, 12H, arom-H), 8.22 (d, 1H, J = 8.3, H-5).

Anal. Calcd. for  $\text{C}_{22}\text{H}_{14}\text{N}_2\text{OS}$  (354.43): C, 74.66; H, 4.12; N, 8.00 %. Found: C, 74.55; H, 3.98; N, 7.90.

3-Acetyl-1-phenyl-5-(phenylhydrazono)-1,5-dihydroindeno[1,2-b]pyridin-2-one (**23a**).

Compound **23a** was obtained as brown crystals from ethanol/DMF, m.p. 258-260 °C, yield 65 %.  $v_{\text{max}}$ : 3360(NH), 1710(CO), 1700(CO), 1590 (C=N).  $^1\text{H-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 2.3 (s, 3H,  $\text{CH}_3$ ), 6.62-7.60 (m, 14H, arom-H), 8.14 (s, 1H, CH), 11.1 (s, 1H, NH).  $^{13}\text{C-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 196.5, 158.4 (CO-carbons), 155.6 (imine-carbon), 147.4, 146.0, 138.0, 102.3 (ethylene-carbons), 146.7, 138.2, 134.5, 130.7, 129.3, 129.3, 128.9, 128.9, 128.7, 128.4, 127.8, 126.3, 124.4, 120.8, 120.4, 118.5, 115.1, 115.1 (aromatic-carbons), 22.2 (methyl-carbon).

Anal. Calcd. for  $\text{C}_{26}\text{H}_{19}\text{N}_3\text{O}_2$  (405.45): C, 77.02; H, 4.72; N, 10.36 %. Found: C, 77.11; H, 4.66; N, 10.42.

3-Acetyl-1-(4-chlorophenyl)-5-(phenylhydrazono)-1,5-dihydroindeno[1,2-b]pyridin-2-one (**23b**).

Compound **23b** was obtained as deep brown crystals from ethanol/DMF, m.p. 241 °C, yield 70%.  $v_{\text{max}}$ : 3360(NH), 1710(CO), 1700(CO), 1590 (C=N).  $^1\text{H-NMR}$  ( $^2\text{H}_6$ )DMSO)  $\delta$ : 2.3 (s, 3H,  $\text{CH}_3$ ), 6.62-7.60 (m, 13H, arom-H), 8.14 (s, 1H, CH), 11.1 (s, 1H, NH). MS: m/z 439( $\text{M}^+$ ).

Anal. Calcd. for  $\text{C}_{26}\text{H}_{18}\text{ClN}_3\text{O}_2$  (439.89): C, 70.79; H, 4.36; N, 9.38 %. Found: C, 70.99; H, 4.12; N, 9.55.

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