SYNTHESIS OF SOME SUBSTITUTED PYRAZOLO[4,3-c]PYRIDINE-3-OLS AS A NOVEL FUSED HETEROBICYCLES

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The 4-oxo-N,2,6-triphenylpiperidine-3-carboxamides (**1a-f**) were synthesized using arylaldehydes, ammonium formate and acetoacetanilide, which in turn converted into 4,5,6,7-tetrahydro-4,6-diphenyl-2*H*-pyrazolo[4,3-c]pyridine-3-ols (**2a-f**) and 4,5,6,7-tetrahydro-2,4,6-triphenyl-2*H*-pyrazolo[4,3-c]pyridine-3-ols (**3a-f**), by condensing with hydrazine hydrate or phenylhydrazine, respectively. .

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Introduction

Heterocyclic systems with piperdin-4-one nucleus was an interesting subject area of research in the past and recent years due to their various biological properties such as antiviral, anti-inflammatory, local anesthetic, anticancer, antimicrobial activity.[1-5] One of the piperidine derivative, piperidones were also biologically important and act as neurokinin receptor antagonists, analgesic and anti-hypertensive agents.[6-12] The importance of piperdin-4-one as intermediates in the synthesis of a diversity of compounds with potent physiological activity has been reviewed by Prostakov and Gaivoronskaya.[13] The studies undertaken on 4-piperidones have direct relation to the synthesis of drug molecules. The effect of substituent at second, third and sixth position particularly, aryl substituent at second and/or sixth positions with regard to its biological activity have been well documented. Therefore, the institution of general methods for the synthesis of piperidone has been the topic of considerable synthetic effort.[14-19] N-Benzyl-β-chloro propionamide was a well-proven anticonvulsant agent and marketed under the trade name Hibicon and Hydrae.

The aryl piperidine scaffold was a key factor involved in binding to a diverse receptors and therefore can be named as a privileged vast array of piperidine containing compounds, both natural and synthetic, for their biological and medicinal interest and this had led to the development of many synthetic approaches to these scaffolds.[20] However, the biological properties of piperidines are highly dependent on the type and locations of substituent on the heterocyclic ring continue to drive the search for new methodologies. Therefore, keen care has been compensated to the construction of functionalized piperidines and its synthetic methodologies. It is noteworthy that 4-arylpiperidine was an important structural motif in many biologically active compounds, including paroxetine, femoxetine, Roche-haloperidol, and meperidine.[21] Similarly imidazole, oxazole and pyrazole were also exhibiting several activities and the fused bicycles of 4-piperidones with imidazole, pyrazole, and oxazole are not available in the literature and hence this prompted us to carry out the synthesis of fused heterocycles with 4-piperidone moiety with other heterocycles.

Experimental

General Procedure for the synthesis of (1a-f)

Dry ammonium formate (0.01 mol, 0.63 g) was dissolved in ethanol and this solution was mixed with arylaldehyde (0.02 mol, 2.02 g) and acetoacetanilide (0.01 mol, 1.77 g). The mixture was just heated to boil and allowed to stand at room temperature overnight. Then Conc. HCl (5 ml) was added and the precipitated hydrochloride was collected, washed with ethanol-ether mixture (1:5). A suspension of the hydrochloride in acetone was treated with strong liquid ammonia and the free base was obtained by pouring water. The product was twice recrystallized from ethanol. The purity of these compounds was checked by TLC and the melting point recorded by open capillary method.

General Procedure for the synthesis of 4,5,6,7-tetrahydro-4,6-diaryl-2*H*-pyrazolo[4,3-c]pyridin-3-ol (2a-f)

About 0.003246 mol of **1a** and 0.003246 mol of hydrazine hydrate in ethanol was taken in the R. B. flask. The contents were allowed to reflux on a water bath for 4 h. The reaction mixture was concentrated and cooled to room temperature; the obtained solid product was washed with ethanol-ether (1:5) solution, and then recrystallized using ethanol. The reproducibility was checked with **1b-f** to get **(2b-f)**.

General procedure for the synthesis 4,5,6,7-tetrahydro-2,4,6-triaryl-2*H*-pyrrolo[3,2-c]pyridin-3-ol (3a-f)

About 0.003246 mol of **1a** was taken in the R.B. flask in ethanol and about 0.003246 mol. of phenyl hydrazine (0.4g) was added. The contents were allowed to reflux on a water bath for 6 hr. The reaction mixture was cooled to room temperature; the solid product was washed with ethanol-ether (1:5) solution and recrystallized using ethanol. The purity of product was checked through TLC and the melting point was observed.

Spectral data of newly prepared compounds

1a: Yield (85 %), m.p =110°C. FT-IR data in cm^{-1}: 1655 (Ring C=O str), 1722 (NH C=O str), 3458 (NH str), 3353 (Aromatic str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.89 (s, 1H), 2.68-2.90 (t, 2H), 3.87 (d, 1H), 4.06 (t, 1H), 4.40 (d, 1H), 7.19-7.29 (m, 7H), 7.40-7.43 (m, 6H), 7.61 (d, 2H). $C_{24}H_{22}N_2O_2$ (Calculated) C = 77.81, H = 5.99, N = 7.56; (Found) C = 77.54, H = 6.09, N = 7.76

1b: Yield (79 %), m.p =108°C. IR data (cm^{-1}): 1634 (Ring C=O str), 1727 (NH C=O str), 3425 (NH str), 3285, 3218 (Aromatic str).^{1}H-NMR (400MHz, $CDCl_3$): δ 1.91 (s, 1H), 2.70-2.73 (t, 2H), 3.84 (s, 6H), 3.93 (d, 1H), 4.10 (t, 1H), 4.37 (d, 1H),6.94 (d, 4H), 7.15-7.23 (m, 6H), 7.43 (t, 2H), 7.60 (d, 2H). $C_{26}H_{26}N_2O_4$ (Calculated) C = 72.54, H = 6.09, N = 6.51. Found; C = 72.58, H = 5.99, N = 6.54.

1c: Yield (93 %), m.p =110°C. IR data in cm^{-1}: 1609 (Ring C=O str), 1738 (NH C=O str), 3429 (NH str), 3382 (Aromatic str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.89 (s, 1H), 2.68-2.74 (t, 2H), 3.87 (d, 1H), 4.21 (t, 1H), 4.50 (d, 1H), 6.43-6.46 (m, 4H), 7.19-7.20 (m, 2H), 7.40 (t, 2H), 7.60-7.64 (m, 4H). $C_{20}H_{18}N_2O_4$ (Calculated): C = 68.56; H= 5.18; N = 8.00 Found: C = 68.44; H = 4.08; N = 8.10.

1d: Yield (75 %), m.p =105°C. IR data in cm^{-1}: 1646 (Ring C=O str), 1749 (NH C=O str), 3417 (NH str), 3172, 3139 (Aromatic str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.88 (s, 1H), 2.74-2.78 (t, 2H), 3.83 (s, 6H), 3.89 (d, 1H), 4.13 (t,3H), 4.30 (d, 1H), 6.75-6.80 (m, 4H), 7.07 (s, 2H), 7.19-25 (m, 4H), 7.46 (t, 2H), 7.61 (t, 2H). $C_{26}H_{26}N_2O_4$ (Calculated) C = 72.54, H = 6.09, N = 6.51; Found: C = 72.54, H = 5.13, N= 6.12.

1e: Yield (91 %), m.p =109°C. IR data in cm^{-1}: 1634 (ring C=O str), 1727 (NH C=O str), 3425 (NH str), 3285, 3218 (Aromatic str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.90 (s, 1H), 2.70-2.74 (t, 2H), 3.83 (s, 6H), 3.91 (d,1H), 4.10 (t, 1H), 4.40 (d, 1H), 6.80 (t, 2H),6.90-94 (m,4H), 7.16-7.20 (m, 4H), 7.43 (t, 2H), 7.61 (d, 2H). $C_{26}H_{26}N_2O_4$ (Calculated), C = 72.54, H = 6.09; N = 6.51; Found: C = 72.34, H = 4.99; N = 6.43.

1f: Yield (79 %), m.p =107°C. IR data in cm^{-1}: 1645 (Ring C=O str), 1716 (NH C=O str), 3434 (NH str), 3300, 3201(Aromatic str). GC-Mass Base Peak = 298, ^{1}H-NMR (400MHz, $CDCl_3$): ä 1.92 (s, 1H), 2.70-2.72 (m, 2H), 3.90 (d, 1H), 4.15 (t, 1H), 4.30 (d, 1H), 7.19-7.20 (m, 2H), 7.45-7.48 (m, 10H), 7.61 (d, 2H). $C_{24}H_{20}N_2Cl_2O_2$,(Calculated): C = 65.61; H = 4.59; Cl = 16.14; N = 6.38**;** Found: C = 65.51; H = 5.53; N = 6.33.

2a: Yield (81 %), m.p =175°C. FT-IR data in cm^{-1}: 3318 (NH str), 3198 (Aromatic str), 3076 (Aromatic str), 3435 (OH str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.90 (s, 1H), 1.5-1.60 (t, 2H), 2.0 (s, 1H), 2.1 (d, 1H), 3.9 (t,1H), 5.19 (s,1H), 7.26-7.30 (m,10H). GC-Mass Base Peak = 272. $C_{18}H_{17}N_3O$ (Calculated) C = 74.20; H = 5.88; N = 14.42; O = 5.49; Found: C = 73.80; H = 4.98; N = 14.56.

2b: Yield (78 %), m.p =197 °C. IR data in cm^{-1}: 3436 (NH str), 3267, 3096 (Aromatic str), 3436 (Aromatic OH str), 1690 (C-O-C str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.85 (s, 1H), 1.56-1.60 (t, 2H), 2.04 (s, 1H), 2.02 (d, 1H), 3.83 (s,6H), 3.89 (t, 2H), 6.90 (d, 4H), 7.20 (d, 4H). $C_{20}H_{21}N_3O_3$ (Calculated): C = 68.36; H = 6.02; N = 11.96; Found: C = 68.52; H = 6.58; N = 12.03.

2c: Yield (73 %), m. p =207 °C. IR data in cm^{-1}: 3454 (NH str), 3271, 3102 (Aromatic str), 3445 (Aromatic OH str), 1592 (C-O-C furyl str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.88 (s, 2H), 1.64-1.70 (t, 2H), 2.12 (d, 1H), 4.1 (t, 2H), 6.43-6.46 (m, 4H), 7.70 (d, 2H). $C_{14}H_{13}N_3O_3$ (Calculated) C = 61.99; H = 4.83; N = 15.49; Found: C = 62.77; H = 3.71; N = 14.42.

2d: Yield (78 %), m.p =199 °C. IR data in cm^{-1}: 3435 (NH str), 3267, 3097 (Aromatic str), 3436 (Aromatic OH str), 1690 (C-O-C str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.90 (s, 2H), 1.60-1.64 (t, 2H), 2.09 (d, 1H), 3.83 (s, 6H), 3.85 (t, 2H), 6.86 (t, 2H), 6.94-6.98 (m, 4H), 7.15 (d, 2H). $C_{20}H_{21}N_3O_3$ (Calculated): C = 68.36; H = 6.02; N = 11.96; Found: C = 68.52; H = 6.52; N = 12.03.

2e: Yield (80 %), m.p =197 °C. IR data in cm^{-1}: 3436 (NH str), 3267, 3096 (Aromatic str), 3434 (Aromatic OH str), 1693 (C-O-C str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.92 (s, 2H), 1.61-1.63 (t, 2H), 2.10 (d, 1H), 3.83 (s,6H), 3.90 (t, 2H), 6.85 (t, 2H), 6.94-6.98 (m, 4H), 7.15 (d, 2H). $C_{20}H_{21}N_3O_3$ (Calculated): C = 68.36; H = 6.02; N = 11.96; Found: C = 69.02; H = 6.48; N = 11.83.

2f: Yield (85 %), m.p =217 °C. IR data in cm^{-1}: 3359 (NH str), 3187, 3087 (Aromatic str), 3432 (OH str), 684 C-Cl str. ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.85 (s, 1H), 1.55-1.60 (t, 2H), 2.10 (s, 1H), 3.95 (t, 2H), 7.50-7.60 (m, 8H). $C_{18}H_{15}N_3Cl_2O$ (Calculated): C = 60.01; H = 4.20; Cl = 19.68; N = 11.66; Found; C = 59.77; H = 3.23; N = 11.23.

3a: Yield (74 %), m.p =170 °C. IR data in cm^{-1}: 3251 (NH str), 3182, 3058 (Aromatic str), 3409 (Aromatic OH str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.90 (s, 1H), 2.70-2.90 (t, 2H), 4.27 (t, 1H), 5.20 (s, 1H, 7.29-7.45 (m, 11H), 7.62-7.70 (m, 4H), 10.90 (s, 1H). GC-Mass Base Peak = 208. ES-Ms: 367; $C_{24}H_{21}N_3O$ (calculated): C = 78.45; H = 5.76; N = 11.44; Found: C = 78.85; H = 5.47; N = 11.24.

3b: Yield (85 %), m.p =263 °C. IR data in cm^{-1}: 3249 (NH str), 3194, 3078 (Aromatic str), 3421 (Aromatic OH str), 1625 (C-O-C str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.93 (s, 1H), 2.75-2.87 (t, 2H), 3.80 (s, 6H), 4.30 (t, 1H), 5.20 (s, 1H, 6.87-6.98 (m, 4H), 7.15-7.20 (m, 4H), 7.45 (t, 1H), 7.60-7.65 (m, 4H), 11.02 (s, 1H). ES-Ms: 427. $C_{26}H_{25}N_3O_3$ (Calculated): C = 73.05; H = 5.89; N = 9.83; Found: C = 73.23; H = 4.78; N = 8.93.

3c: Yield (70 %), m.p =212 °C. IR in cm^{-1}: 3287 (NH str), 3136 (Aromatic str), 3470 (Aromatic OH str), 3060 (C-C str furan). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.93 (s, 1H), 2.80-2.85 (t, 2H), 4.60 (t, 1H), 5.51 (s, 1H), 6.30 (d, 1H), 6.58-6.70 (m, 4H), 7.52 (d, 1H), 7.64-7.67 (m, 5H), 11.0 (s, 1H). EI-MS= 347[M+]; $C_{20}H_{17}N_3O_3$ (Calculated): C = 69.15; H = 4.93; N = 12.10; Found: C = 68.95; H = 3.98; N = 12.02.

3d :Yield (78 %), m.p =204 °C. IR in cm^{-1}: 3245 (NH str), 3198, 3082 (Aromatic str), 3421 (Aromatic OH str), 1626 (C-O-C str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.93 (s, 1H),

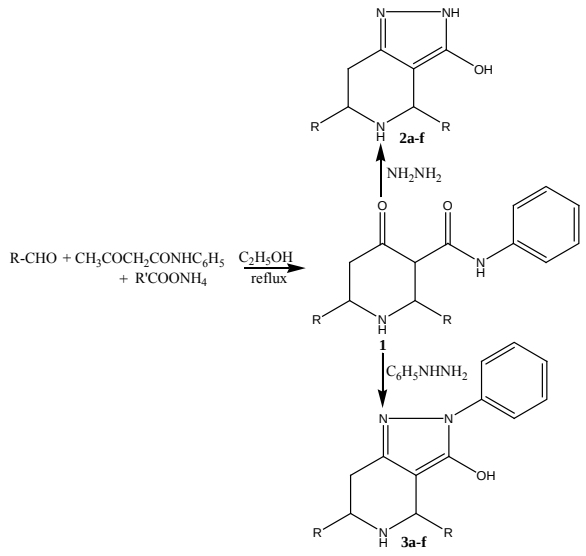
2.75-2.87 (t, 2H), 3.80 (s, 6H), 4.30 (t, 1H), 5.20 (s, 1H), 6.87-6.98 (m, 4H), 7.15-7.20 (m, 4H), 7.45 (t, 1H), 7.60-7.65 (m, 4H), 11.02 (s, 1H). ES-Ms: 427; $C_{26}H_{25}N_3O_3$ (Calculated): C = 76.03; H = 6.14; N = 6.57; Found: C = 73.10; H = 5.89; N = 6.87.

3e:Yield (78 %), m.p =198 °C. IR in cm^{-1}: 3249 (NH str), 3194, 3078 (Aromatic str), 3421 (Aromatic OH str), 1625 (C-O-C str). ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.93 (s, 1H), 2.75-2.87 (t, 2H), 3.80 (s, 6H), 4.30 (t, 1H), 5.20 (s, 1H, 6.87-6.98 (m, 4H), 7.15-7.20 (m, 4H), 7.45 (t, 1H), 7.60-7.65 (m, 4H), 11.02 (s, 1H). ES-Ms: 427; $C_{26}H_{25}N_3O_3$ (Calculated): C = 76.03; H = 6.14; N = 6.57; Found: C = 73.08; H = 5.79; N = 6.86.

3f:Yield (83 %), m.p =206 °C. IR data in cm^{-1}: 3276 (NH str), 3176, 3082 (Aromatic str), 3423 (Aromatic OH str), 691 C-Cl str. ^{1}H-NMR (400MHz, $CDCl_3$): δ 1.90 (s, 1H), 2.80-3.0 (t, 2H), 4.28 (t, 1H), 5.20 (s, 1H), 7.20 (d, 2H), 7.45-7.60 (m, 11H), 11.02 (s, 1H). ES-MS: 435 [M+]; $C_{24}H_{19}N_3Cl_2O$ (Calculated): C = 66.06; H = 4.39; Cl = 16.25; N = 9.63; Found: C = 65.97; H = 3.43; N = 9.73.

Results and Discussion

The 4-oxo-N,2,6-triphenylpiperidine-3-carboxamides (**1a-f**) were synthesized by adopting the procedure available in literature[1]. In this method about 0.01 mol of dry ammonium formate (0.63 g) was dissolved in ethanol and the solution was mixed with 0.02 mol of arylaldehyde (2.02 g) and 0.01 mol of acetoacteanilide (1.77 g). The mixture was just heated to boil and kept at room temperature over night. Then Conc. HCl (5 ml) was added and the precipitated hydrochloride was collected, washed with ethanol-ether mixture (1:5). A suspension of the hydrochloride in acetone was treated with liquid ammonia and the free base was obtained by pouring on to water. Product was purified by repeated recrystallization from ethanol.



Scheme 1. Synthesis of substituted pyrazolo[4,3-c]pyridine-3-ols

Table 1. Physical data of the compounds (**1a-f**), (**2a-f**) and (**3a-f**)

S. No	R	M.P. ºC	Yield, %
1a	Phenyl	110-12	90
1b	4-Methoxyphenyl	108-10	78
1c	Furfural	110-12	98
1d	3-Methoxyphenyl	105-08	75
1e	2-Methoxyphenyl	107-09	91
1f	4-Chlorophenyl	109-13	70
2a	Phenyl	175-78	81
2b	4-Methoxyphenyl	197-201	78
2c	Furfural	207-08	73
2d	3-Methoxyphenyl	195-99	80
2e	2-Methoxyphenyl	198-202	79
2f	4-Chlorophenyl	217-19	85
3a	Phenyl	170-74	74
3b	4-Methoxyphenyl	194-96	78
3c	Furfural	212-15	70
3d	3-Methoxyphenyl	195-98	76
3e	2-Methoxyphenyl	198-203	78
3f	4-Chlorophenyl	263-64	85

The compounds were characterized by IR and ^{1}H-NMR. The formation of piperidones confirmed by the appearance of two signals for the carbonyl function in IR spectra around 1610cm^{-1} and 1710-1750 cm^{-1}. ^{1}H-NMR for the compound **1f** shows the following peaks. A doublet appeared at 4.51 ppm due to the methylene proton at C5. The other aliphatic signals may be merged with aromatic protons due to the anisotropic effect of amide group. δ7.24 (d, *J* = 7.88 Hz, 2H), δ 7.48-7.50 multiplet (12H), δ 7.68 (d *J*=8.44Hz, 2H), δ 8.40 (s 2H). The physical parameters synthesized 4-piperidones **1a-f** were given in Table 1.

The 4,5,6,7-tetrahydro-4,6-diphenyl-2*H*-pyrazolo[4,3-c] pyridine-3-ols (**2a-f**) were synthesized by dissolving a mixture of about of 4-piperidone (0.003246 mol, 1.20 g) (**1a-f**) and 0.003246 mol of hydrazine hydrate in ethanol. The contents were refluxed on a water bath for 4 h. The reaction mixture was cooled to room temperature; the obtained solid was washed with ethanol-ether (1:5) and recrystallized using ethanol. The purity of the product was checked through TLC and the melting point. The formation of the product was confirmed by the absence of carbonyl peaks which appeared in the piperidones. The yield and physical parameters of **2a-f** are given in the Table 1.

Similarly the 4,5,6,7-tetrahydro-2,4,6-triphenyl-2*H*-pyrazolo[4,3-c]pyridine-3-ols (**3a-f**) was synthesized by refluxing the mixture of dissolving about 0.003246 mol of 4-piperidones (**1a-f**) in ethanol and 0.003246 mol of phenyl hydrazine for about 6 hrs. Then reaction mixture was cooled to room temperature; the solid obtained was washed with ethanol-ether (1:5) and recrystallized using ethanol. The purity of the product was checked through TLC and the melting point was recorded. The formation of the product was confirmed by the absence of carbonyl stretching frequency in IR spectrum which was present in piperidones.

The yield and other physical parameters of **3a-f** are given in the Table 1.

Conclusion

In conclusion, synthesis of some fused heterocycles with 4-piperidone moiety and 4,5,6,7-tetrahydro-4,6-diaryl-2*H*-pyrazolo[4,3-c]pyridin-3-ols, 4,5,6,7-tetrahydro-4,6-diaryl-2*H*-pyrazolo[4,3-c]pyridin-3-ols, were attempted successfully, the compounds with fused pyrazole were proved as biologically important molecules. In the present work molecules with pyrazole moiety were obtained using a simple reaction protocol with moderate yield. The synthesized compounds were characterized by IR, Mass, ^{1}H NMR data along with elemental analysis.

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