Indian Journal of Chemistry
Vol. 63, May 2024, pp. 518-523
DOI: 10.56042/ijc.v63i5.9561

# Facile synthesis of 3-amino substituted piperidines from L-glutamic acid

Sonam Tashi Khom, Pranjit Saikia & Nagengra Nath Yadav*

Department of Chemistry, North Eastern Regional Institute of Science and Technology, Nirjuli 791 109, Arunachal Pradesh, India

E-mail: nny@nerist.ac.in

*Received 13 March 2024; accepted (revised) 25 April 2024*

Multi-step route toward the synthesis of enantiomerically pure 3-(*N*-Boc amino) piperidine derivatives is described starting from natural *α*-amino acid *i.e.* L-glutamic acid. This route involves the esterification of both carboxylic acid groups in one-pot to give diester followed by $NaBH_4$ reduction to give diol which in turn is converted to various piperidines *via* the reaction of corresponding ditosylate with different amines. The overall yields of substituted piperidine have been found to be 44% to 55% starting from L-glutamic acid.

**Keywords**: 3-(*N*-Boc amino) piperidines, L-Glutamic acid, $NaBH_4$ reduction, Cyclization, Amines

Numerous natural products and biologically active compounds commonly found to contain 3-amino piperidines as a key structural features[1-9]. Optically active 3-amino piperidine is present in many pharmaceutical drugs like alogliptin (**1**)[10], linagliptin[11], tofacitinib[12] and trelagliptin[13] (Fig. 1). In general, the Piperidine ring system proves to be an important constituent core structure of numerous natural products[14-19] and pharmaceutical compounds[20-23]. The 3-(N-Boc amino) piperidine derivatives have gained significant attention due to their versatility and ability to undergo a wide range of selective modifications of the piperidine ring. The N-Boc (*tert*-butoxycarbonyl) group enhances the stability and facilitates the synthesis of various analogs by selective modification. There are several reported methods for the construction of the 3-amino piperidine derivatives, like ring-closing metathesis reaction[24], enantioselective ring expansion of prolinols[25], rhodium-catalyzed asymmetric hydrogenation[26], 1,2-diamination of aldehydes[27], using enzyme cascades[28] and O-alkylation followed by catalytic hydrogenation of lactams[29]. Although many methods have been reported, most experiments suffer from low yield, complex reagents and expensive protocols. We have synthesized various piperidine alkaloids by one-pot multiple steps reaction under the atmospheric hydrogen including the reduction of alkyne, reductive ring-opening of aziridine, debenzylation, and intramolecular reductive amination from suitable chiral aziridines in high yields[30]. We have reported another method for the formation of piperidine ring system from ring expansion of chiral aziridine *via* formation of bicyclic aziridinium ion as active reaction intermediates[31-33]. Thus, herein we described a convenient route for the synthesis of 3-(*N*-Boc amino) piperidine derivatives from natural amino acids called L-glutamic acid in good yields. This route involves five linear steps *i.e.* esterification, Boc-protection, $NaBH_4$ reduction of diester, tosylation of diol and cyclization to give substituted piperidines.

## Experimental Section

All chemicals and materials were of reagent grade as received from commercial outlets. The solvents were used without further purification. Reaction progress was monitored using TLC silica gel 60 $F_{254}$ manufactured by Merck KGaA. Purification of the products was carried out by column chromatography using 60-120 mesh silica gel manufactured by Merck Life Science Pvt. Ltd. The $^1H$ and $^{13}C$ NMR were recorded on a Bruker Avance 400 MHz/Avlll HD-300 MHz spectrometer with TMS as the internal standard and $CDCl_3$ as solvent. High-resolution mass spectrometry (HRMS) was determined on Agilent 6520 (Q-TOF) Mass spectrometer with Agilent 1200 HPLC system. ESI-MS was recorded using Waters Alliance e2695/HPLC-TQD mass spectrometer. Melting point was recorded on digital melting/boiling point apparatus from HOVERLABS model no. HV-115.

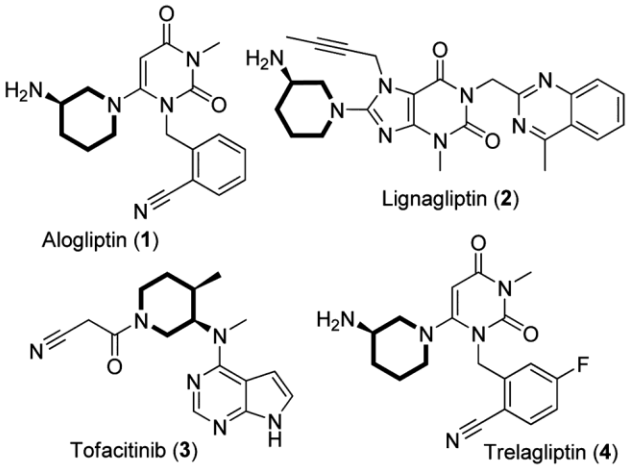



Fig. 1 — Structure of few bio-active compounds having 3-amino piperidine ring system

## Methylation of L-glutamic acid, 5

**(*S*)-1,5-Dimethoxy-1,5-dioxopentan-2-aminium chloride, 6**

To the stirred solution of L-glutamic acid **5** (7.5 g, 51.0 mmol) in methanol (110 mL) at 0°C was added thionyl chloride (5.6 mL, 76.5 mmol) drop-wise. After completion of the addition of thionyl chloride, the ice bath was removed and the reaction was stirred at RT for 12 h. After completion of reaction as confirmed by TLC, the solvent was dried off in a rotary evaporator to give the crude product **6** as HCl salt (10.76 g, quantitative yield) as pale yellow viscous oil. The crude product was used for the next step without further purification. $^1$H NMR (300 MHz, $CDCl_3$): δ 8.74 (br s, 2H), 6.10 (br s, 1H), 4.37 – 4.21 (m, 1H), 3.84 (s, 3H), 3.69 (s, 3H), 2.80 – 2.55 (m, 2H), 2.48 – 2.30 (m, 2H); HRMS (ESI): *m/z* Calcd for $C_7H_{14}NO_4$: $[M+H]^+$ 176.0923. Found: 176.0939.

## N-Boc protection of the amino group of compound, 6

**(*S*)-Dimethyl 2-(*tert*-butoxycarbonyl)amino)pentanedioate, 7**

To a stirred solution of the dimethyl ester **6** (10 g, 57 mmol) in $CH_2Cl_2$ (120 mL) at 0°C was added triethylamine (32 mL, 228 mmol), $(Boc)_2O$ (19.5 mL, 85.5 mmol) and catalytic amount of DMAP (0.1 equiv., 0.7 g). After completion of the addition, the reaction mixture was stirred at RT for 6 h. The reaction mixture was then quenched with distilled water (50 mL) and the reaction mixture was extracted using $CH_2Cl_2$ (3×50 mL). The combined organic layer was further washed with 10% aqueous sodium bicarbonate solution (100 mL) followed by brine (100 mL). The organic layer was finally dried over anhydrous sodium sulphate, filtered and concentrated to obtain crude product, which was purified by column chromatography (silica gel 60-120 mesh; eluent: Ethyl acetate/Hexane, 1:9) to get pure product **7** (5.7g, 92%) as viscous liquid. $^1$H NMR (300 MHz, $CDCl_3$): δ 5.18 (d, *J* = 7.4 Hz, 1H), 4.41 – 4.27 (m, 1H), 3.75 (s, 3H), 3.68 (s, 3H), 2.52 – 2.32 (m, 2H), 2.26 – 2.11 (m, 1H), 2.03 – 1.91 (m, 1H), 1.44 (s, 9H); $^{13}$C NMR (75 MHz, $CDCl_3$): δ 173.2, 172.7, 155.4, 80.1, 52.9, 52.5, 51.8, 30.1, 28.3, 27.8; HRMS (ESI): *m/z* Calcd for $C_{12}H_{21}NNaO_6$: $[M+Na]^+$) 298.1267. Found 298.1281.

## Reduction of N-Boc-dimethyl ester, 7

**(*S*)-*tert*-Butyl(1,5-dihydroxypentan-2-yl)carbamate, 8**

To the stirred solution of diester compound **7** (5 g, 18.18 mmol) in methanol (30 mL) was added sodium borohydride (2.5 equiv.) portion wise at RT. The reaction mixture was allowed to stir for 2 h at RT. After complete conversion of starting material as confirmed by TLC, the reaction mixture was quenched by adding 10% aqueous citric acid solution until the resulting *p*H of the mixture reached in between 5–6. Methanol was removed using a rotary evaporator and the aqueous layer was extracted using $CH_2Cl_2$ (3×30 mL). The combined organic layer was then washed with brine, dried over anhydrous sodium sulphate, filtered and concentrated in vacuum to obtain the crude product, which was purified using column chromatography (silica gel 60-120 mesh; eluent: Ethyl acetate/Hexane, 2:8) to get pure diol **8** (4.7 g, 76%) as major product along with mono-alcohol **9** (0.6 g, 11%) as minor product. $^1$H NMR (500 MHz, $CDCl_3$): δ 4.94 (br s, 1H), 3.74 – 3.53 (m, 4H), 3.19 – 3.09 (m, 1H), 2.72 (br s, 2H), 1.70 – 1.48 (m, 4H), 1.44 (s, 9H); $^{13}$C NMR (101 MHz, $CDCl_3$): δ 156.6, 79.7, 65.0, 62.2, 52.3, 28.7, 28.5, 28.0; MS (ESI): *m/z* 242 $[M+Na]^+$.

**(*S*)-Methy- 4-(tert-butoxycarbonyl)amino)-5-hydroxypentanoate, 9**: $^1$H NMR (400 MHz, $CDCl_3$): δ 4.91 (d, *J* = 5.9 Hz, 1H), 3.69 (s, 3H), 3.65 (s, 1H), 3.63 (s, 1H), 2.45 – 2.39 (m, 2H), 1.95 – 1.84 (m, 2H), 1.83 – 1.72 (m, 2H), 1.44 (s, 9H); $^{13}$C NMR (101 MHz, $CDCl_3$): δ 174.2, 156.2, 79.6, 65.1, 52.2, 51.8, 30.6, 28.3, 26. HRMS (ESI): *m/z* Calcd for $C_{11}H_{21}NNaO_5$: $[M+Na]^+$ 270.1317. Found 270.1338.

## Synthesis of 3-(N-Boc amino) piperidine derivatives, 10-14

To a stirred solution of *N*-Boc-protected diol **8** (1.5 g, 6.85 mmol) in $CH_2Cl_2$ (15 mL) at 0°C, was

added triethylamine (4.8 mL, 34.24 mmol), *p*-toluenesulfonyl chloride (3.9 g, 20.55 mmol) and DMAP (0.5 equiv., 0.42 g). The reaction mixture was warmed to RT and allows stirring for next 1 h. After complete conversion of starting material as confirmed by TLC, the reaction mixture was quenched with 20% aqueous sodium bicarbonate (20 mL). The reaction mixture was extracted with $CH_2Cl_2$ (3×20 mL). The combined organic layer was washed with brine (1×30 mL) and dried over anhydrous sodium sulphate, filtered and concentrated to get crude ditosylate **8a** (3.6 g, quantitative yield) which was used as such for next step without any further purification.

**(*S*)-*tert*-Butyl(1-cyclohexylpiperidin-3-yl)carbamate, 10**

To the above crude di-tosylate **8a** (0.53 g, 1.0 mmol) was added cyclohexylamine (1.7 mL, 15 mmol) and reaction mixture was allowed to stir for 12 h. After the completion of the reaction, the reaction mixture was quenched using saturated aqueous ammonium chloride (5 mL) and extraction was done with $CH_2Cl_2$ (3×10 mL). The combined organic layer was then washed with brine solution (20 mL) and dried over anhydrous sodium sulphate, filtered and concentrated in vacuum to obtain product which was further subjected to purification using column chromatography (silica gel 60-120 mesh; eluent: Ethyl acetate/Hexane, 2:8) to get pure piperidine compound **10** (208 mg, 74%) as off-white solid. m.p.79–81°C. $[\alpha]^{20}_D$ = –18.4 (*c* = 0.15, EtOH); $^1$H NMR (300 MHz, $CDCl_3$): δ 5.05 (br s, 1H), 3.65 (br s, 1H), 2.58 (d, *J* = 9.5 Hz, 1H), 2.51 – 2.30 (m, 3H), 2.28 – 2.17 (m, 1H), 1.83 – 1.45 (m, 9H), 1.38 (s, 9H), 1.29 – 0.94 (m, 5H); $^{13}$C NMR (75 MHz, $CDCl_3$): δ 155.3, 79.0, 64.0, 54.4, 49.8, 46.7, 30.2, 28.8, 28.5, 26.4, 26.1, 26.1; MS (ESI): *m/z* 283 $[M+H]^+$.

**(*S*)-*tert*-Butyl (1-(tert-butyl)piperidin-3-yl) carbamate, 11**: The procedure was analogous to that used for the preparation of compound **10**. From the crude di-tosylate **8a** (0.53 g, 1.0 mmol) and *tert*-butylamine (1.6 mL, 15 mmol) was obtained *N*-*tert*-butylpiperidine **11** (200 mg, 71%) as colorless viscous oil. $[\alpha]^{25}_D$ = –6.9 (*c* = 0.15, $CH_2Cl_2$); $^1$H NMR (500 MHz, $CDCl_3$): δ 5.25 (br s, 1H), 3.76 (br s, 1H), 2.81 – 2.29 (m, 4H), 1.69 – 1.36 (m, 4H), 1.42 (s, 9H), 1.07 (s, 9H); $^{13}$C NMR (101 MHz, $CDCl_3$): δ 155.2, 78.9, 51.3, 46.3, 41.1, 29.6, 28.3, 25.8, 22.6; HRMS (ESI): *m/z* Calcd for $C_{14}H_{29}N_2O_2$: $[M+H]^+$ 257.2229. Found: 257.2227.

***tert*-Butyl ((*S*)-1-(*S*)-1-phenylethyl)piperidin-3-yl)carbamate, 12**: The procedure was analogous to that used for the preparation of compound **10**. From the crude di-tosylate **8a** (0.53 g, 1.0 mmol) and *(S)*-1-phenylethanamine (1.9 mL, 15 mmol) was obtained piperidine derivative **12** (194 mg, 64%) as off-white solid. m.p.76–78°C. $[\alpha]^{25}_D$ = –9.6 (*c* = 0.16, $CH_2Cl_2$); $^1$H NMR (500 MHz, $CDCl_3$): δ 7.31 – 7.09 (m, 5H), 4.97 (br s, 1H), 3.64 (br s, 1H), 3.37 (q, *J* = 6.5 Hz, 1H), 2.47 – 2.11 (m, 4H), 1.66 – 1.39 (m, 4H), 1.36 (s, 9H), 1.27 (d, *J* = 6.7 Hz, 3H); $^{13}$C NMR (101 MHz, $CDCl_3$): δ 155.1, 143.0, 128.0, 127.5, 126.7, 78.8, 64.1, 55.4, 50.3, 46.3, 29.6, 28.3, 22.3, 18.7; HRMS (ESI): *m/z* Calcd for $C_{18}H_{29}N_2O_2$: $[M+H]^+$ 305.2229. Found: 305.2227.

**(*S*)-*tert*-Butyl (1-butylpiperidin-3-yl)carbamate, 13**: The procedure was analogous to that used for the preparation of compound **10**. From the crude di-tosylate **8a** (0.53 g, 1.0 mmol) and *n*-butylamine (1.5 mL, 15 mmol) was obtained *N*-butylpiperidine **13** (204 mg, 80%) as brownish viscous liquid. $[\alpha]^{25}_D$ = –1.8 (*c* = 0.11, $CH_2Cl_2$); $^1$H NMR (500 MHz, $CDCl_3$): δ 5.10 (br s, 1H), 3.68 (br s, 1H), 2.59 – 2.18 (m, 6H), 1.71 – 1.46 (m, 4H), 1.38 (s, 9H), 1.30 – 1.21 (m, 4H), 0.85 (t, *J* = 7.3 Hz, 3H); $^{13}$C NMR (101 MHz, $CDCl_3$): δ 155.1, 78.8, 58.3, 58.2, 53.6, 46.1, 29.5, 28.6, 28.3, 22.0, 20.5, 13.8; HRMS (ESI): *m/z* Calcd for $C_{14}H_{29}N_2O_2$: $[M+H]^+$ 257.2229. Found 257.2225.

**(*S*)-*tert*-Butyl (1-benzylpiperidin-3-yl) carbamate, 14**: The procedure was analogous to that used for the preparation of compound **10**. From the crude di-tosylate **8a** (0.53 g, 1.0 mmol) and benzylamine (1.6 mL, 15 mmol) was obtained *N*-benzylpiperidine **14** (197 mg, 68%) as off-white solid. m.p.78–80°C. $[\alpha]^{25}_D$ = –5.9 (*c* = 1.0, EtOH); $^1$H NMR (500 MHz, $CDCl_3$): δ 7.30 – 7.11 (m, 5H), 4.97 (br s, 1H), 3.67 (br s, 1H), 3.38 (s, 2H), 2.53 – 2.11 (m, 4H), 1.70 – 1.40 (m, 4H), 1.36 (s, 9H); $^{13}$C NMR (101 MHz, $CDCl_3$): δ 155.0, 138.0, 128.8, 128.0, 126.8, 78.8, 62.9, 58.5, 53.2, 46.2, 29.5, 28.3, 22.1; HRMS (ESI): *m/z* Calcd for $C_{17}H_{27}N_2O_2$: $[M+H]^+$ 291.2073. Found 291.2068.

## Results and Discussion

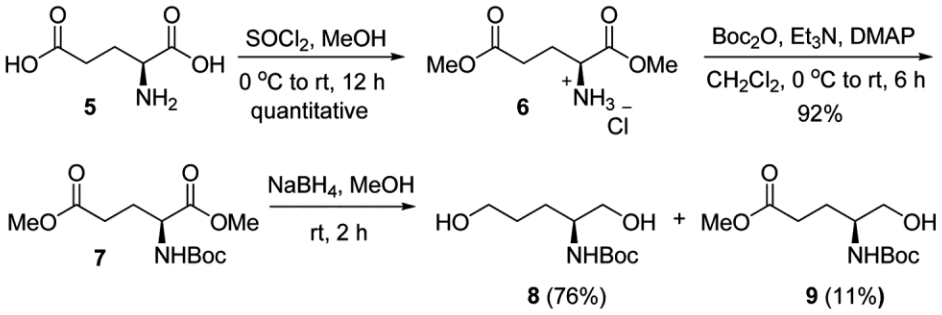
In continuation of our interest for the construction of piperidine ring system using chiral aziridine[30-33], we decided that L-glutamic acid would be the alternate synthetic equivalent for generation of chiral piperidines as it provides five carbon atoms in the

chain, which can be integrated into the six-membered aza-heterocyles. We herein report the synthesis of piperidine ring system from L-glutamic acid in five linear steps. Accordingly, naturally occurring L-glutamic acid (**5**) was transformed to *N*-Boc derivative **7** using slightly modified procedure as reported in literature[34-36]. First we converted compound **5** to the corresponding diester compound **6** in quantitative yield by using thionyl chloride in methanol at 0°C to RT for 12 h. *N*-Boc protection of compound **6** using $(Boc)_2O$ and triethylamine along with catalytic amount of 4-dimethylaminopyridine (DMAP) in $CH_2Cl_2$ furnished compound 7 in 92% yields. The reduction of diester compound 7 with sodium borohydride was found to be quite interesting because when reduction reaction was carried out at 0°C to RT then we observed mono hydroxyl compound **9** as the major product (45%) along with dihydroxyl compound **8** as minor product (19%). However the reduction of diester compound **7** with sodium borohydride in methanol at RT furnished (*S*)-*tert*-butyl (1,5-dihydroxypentan-2-yl)carbamate (**8**) as the major product with 76% yield along with (*S*)-methyl 4-(*tert*-butoxycarbonyl)amino)-5-hydroxypentanoate (**9**) as minor product with 11% yield (Scheme 1).

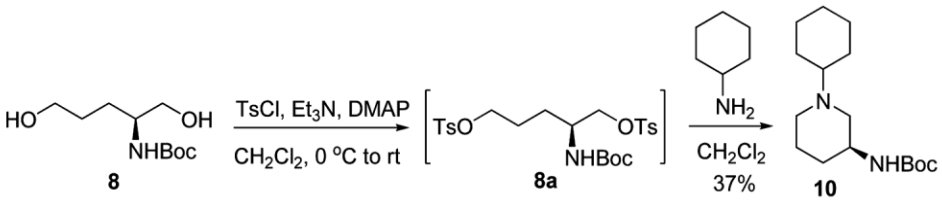
After successful synthesis of diol compound **8**, next we tried the cyclization reaction of diol **8** to get piperidine derivative **10**. Accordingly, di-tosylation of diol **8** with *p*-toluenesulphonyl chloride in $CH_2Cl_2$ in presence of catalytic amount of DMAP furnished the crude di-tosylate **8a** in quantitative yields. The crude di-tosylate was further treated with cyclohexylamine in $CH_2Cl_2$ for 24 hrs to give (*S*)-*tert*-butyl-1-cyclohexylpiperidin-3-ylcarbamate (**10**) in 37% yields (Scheme 2).

The structure of compound **10** was confirmed by NMR and mass spectrometry. Once we confirmed the structure of compound **10**, next we optimized the reaction condition for the synthesis of compound **10**. Use of other solvents such as 1,2-dichloroethane, THF and ether gave the similar results and no improvements were observed either for reaction time and yields. Increasing the molar ratio of cyclohexylamine under neat condition gave the desired product **10** in good yields with shorter reaction time. Use of 15 equivalent of cyclohexylamine under neat condition resulted in the formation of desired compound **10** in 74% yields in 12 hrs (Table 1).

After optimizing the reaction condition for the synthesis of piperidine derivative **10**, we applied the optimized reaction condition for several other piperidine derivatives (Table 2). Reaction of di-tosylate **8a** with least hindered amine such as *n*-butylamine and benzylamine furnished the piperidine derivative **13** and **14** in 80% and 68% yields, respectively. However amine with bulky alkyl substituents such as *t*-butylamine and *(S)*-1-phenylethanamine gave corresponding piperidines **11** and **12** in 71% and 64% yields, respectively. It was observed that amine with less hindered alkyl




Scheme 1 — Step-wise synthesis of alcohol derivatives **8** and **9** from L-glutamic acid



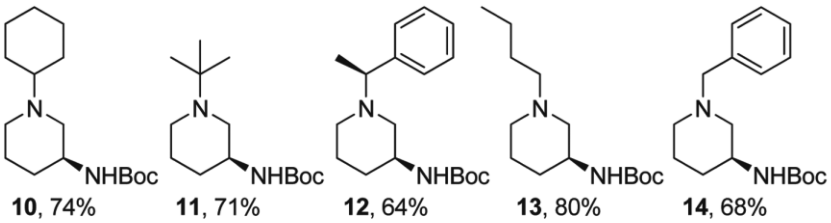

Scheme 2 — Synthesis of (*S*)-*tert*-butyl-1-cyclohexylpiperidin-3-ylcarbamate (**10**) from diol **8**

Table 1 — Screening of different reaction conditions for the synthesis of piperidine derivative **10**

| Entry | Cyclohexylamine (eq.) | Solvent[a] | Time (hrs) | Yield (%)[b] |
|---|---|---|---|---|
| 1 | 3.0 | $CH_2Cl_2$ | 24 | 37 |
| 2 | 3.0 | $(CH_2)_2Cl_2$ | 24 | 31 |
| 3 | 3.0 | THF | 24 | 24 |
| 4 | 3.0 | Ether | 24 | 41 |
| 5 | 5.0 | $CH_2Cl_2$ | 24 | 47 |
| 6 | 10.0 | Neat | 15 | 67 |
| **7** | **15.0** | **Neat** | **12** | **74** |
| 8 | 20.0 | Neat | 12 | 73 |

[a] Anhydrous solvents were used.
[b] Isolated yields after purification by column chromatography

Table 2 — Synthesis of 3-(*N*-Boc amino) piperidine from diol compound **8** and amines


substituents gave good yields of product as compare to amine with bulky alkyl substituents. This difference in reactivity may be attributed by the fact that the reaction proceeds *via* bimolecular nucleophylic substitution reaction ($S_N2$).

Reaction with other bifunctional amines such as ethylenediamine and ethanolamine did not give the desired product. Aromatic amines such as aniline and 2-nitroaniline were found unreactive and unable to produce desired piperidine compounds because of least nucleophilicity of anilines as compare to 1° amines.

## Conclusions

In conclusion, we report a facile synthesis of 3-(*N*-Boc amino) piperidine derivatives in five linear steps starting from naturally occurring L-glutamic acid with 44% to 55% overall yields. This synthesis offers several notable advantages, including mild reaction conditions, uses of non-toxic catalysts and good yields. Furthermore, the structural diversity inherent in the 3-*N*-Boc amino piperidine scaffold presents opportunities for further derivatization and exploration of structure-activity relationships.

## Acknowledgements

STK thanks the Ministry of Tribal Affairs and PS thanks National Fellowship for Other Backward Classes (NFOBC) from University Grants Commission (UGC), India for the award of fellowships. For analytical support, SAIF-CSIR-Central Drugs Research Institute (CDRI), Lucknow (India) was very helpful and provided all the necessary data.

## Supplementary Information

Supplementary information is available in the website http://nopr.niscpr.res.in/handle/123456789/58776.

## References


1 Ran Y, Pei H, Shao M & Chen L, *Chem Biol Drug Des*, 87 (2016) 290.
2 Li Q, Han L, Zhang B, Zhou J & Zhang H, *Organic Biomol Chem*, 14 (2016) 9598.
3 Fromont C, Atzori A, Kaur D, Hashmi L, Greco G, Cabanillas, A, Nguyen H V, Jones D H, Garzon M, Varela A, Stevenson B, Lacobini G P, Lenoir M, Rajesh S, Box C, Kumar J, Grant P, Novitskaya V, Morgan J, Sorrell F J, Redondo C, Kramer A, Harris C J, Leighton B, Vickers S P, Cheetham S C, Kenyon C, Grabowska A M, Overduin M, Berditchevski F, Weston C J, Knapp S, Fisher P M & Butterworth S, *J Med Chem*, 63 (2020) 6784.
4 Evison B J, Palmer J T, Lambert G, Treutlein H, Zeng J, Nativel B, Chemello K, Zhu Q, Wang J, Teng Y, Tang W, Xu Y, Rathi A K, Kumar S, Suchowerska A K, Parmar J, Dixon I, Kelly G E & Bonnar J, *Bioorg Med Chem*, 28 (2020) 115344.
5 Oza V, Ashwell S, Almeida L, Brassil P, Breed J, Deng C, Gero T, Grondine M, Horn C, Ioannidis S, Liu D, Lyne P, Newcombe N, Pass M, Read J, Ready S, Rowsell S, Su M, Toader D, Vasbinder M, Yu D, Yu Y, Xue Y, Zabludoff S & Janetka J, *J Med Chem*, 55 (2012) 5130.
6 Berggren K, Vindebro R, Bergström C, Spoerry C, Persson H, Fex T, Kihlberg J, Rammingen U V P & Luthman K, *J Med Chem*, 55 (2012) 2549.
7 Dax S L & Wei C C, *J Org Chem*, 57 (1992) 744.
8 Eckhardt M, Langkopf E, Mark M, Tadayyon M, Thomas L, Nar H, Pfrengle W, Guth B, Lotz R, Sieger P, Fuchs H & Himmelsbach F, *J Med Chem*, 50 (2007) 6450.
9 Atobe M, Serizawa T, Yamakawa N, Takaba K, Nagano Y, Yamaura T, Tanaka E, Tazumi A, Bito S, Ishiguro M & Kawanishi M, *J Med Chem*, 63 (2020) 7143.
10 Feng J, Zhang Z, Wallace M B, Stafford J A, Kaldor S W, Kassel D B, Navre M, Shi L, Skene R J, Asakawa T, Takeuchi K, Xu R, Webb D R & Gwaltney S L, *J Med Chem*, 50 (2007) 2297.
11 Lajara R, *Exp Opin Pharmacotherap*, 13 (2012) 2663.
12 Dhillon S, *Drugs*, 77 (2017) 1987.
13 McKeage K, *Drugs*, 75 (2015) 1161.
14 Buffat M G, *Tetrahedron*, 60 (2004) 1701.
15 Laschat S & Dickner T, *Synthesis*, 2000 (2000) 1781.
16 Weintraub P M, Sabol J S, Kane J M & Borcherding D R, *Tetrahedron*, 59 (2003) 2953.
17 Bari A, Iqbal A, Khan Z A, Shahzad S A & Yar M, *Synth Commun*, 50 (2020) 2572.
18 Felpin F X & Lebreton J, *Eur J Org Chem*, 2003 (2003) 3693.
19 O'Hagan D, *Natural Product Reports*, 17 (2000) 435.
20 Vitaku E, Smith D T & Njardarson J T, *J Med Chem*, 57 (2014) 10257.
21 Lennox A J, Goes S L, Webster M P, Koolman H F, Djuric S W & Stahl S S, *J Am Chem Soc*, 140 (2018) 11227.
22 Watson P S, Jiang B & Scott B, *Organic Lett*, 2 (2000) 3679.
23 Haider S, Saify Z S, Begum N, Ashraf S, Zarreen T & Saeed S G, *World J Pharm Res*, 3 (2014) 2277.
24 Hu X E, Kim N K & Ledoussal B, *Organic Lett*, 4 (2002) 4499.
25 Cochi A, Gomez Pardo D & Cossy J, *Organic Lett*, 13 (2011) 4442.
26 Royal T, Dudognon Y, Berhal F, Bastard Y, Boudet B, Ayad T & Ratovelomanana-Vidal V, *Synlett*, 27 (2016) 2009.
27 Ansari A & Ramapanicker R, *J Org Chem*, 83 (2018) 8161.
28 Ford G J, Kress N, Mattey A P, Hepworth L J, Baldwin C R, Marshall J R, Seibt L, Huang M, Birmingham W, Turner N J & Flitsch S L, *Chem Commun*, 56 (2020) 7949.
29 Kadyrov R & Tok O L, *Synthesis*, 53 (2021) 3573.
30 Yadav N N, Choi J & Ha H-J, *Org Biomol Chem*, 14 (2016) 6426.
31 Dolfen J, Yadav N N, Kimpe N D, D'hooghe M & Ha H-J, *Adv Synth Catal*, 358 (2016) 3485.
32 Choi J, Yadav N N & Ha H-J, *Asian J Org Chem*, 6 (2017) 1292.
33 Yadav N N, Lee Y-G, Srivastava N & Ha H-J, *Frontiers Chem*, 7 (2019) 460.
34 Hosangadi B D & Dave R H, *Tetrahedron Lett*, 37 (1996) 6375.
35 Ragnarsson U & Grehn L, *RSC Adv*, 3 (2013) 18691.
36 Soai K, Oyamada H & Ookawa A, *Synth Commun*, 12 (1982) 463.