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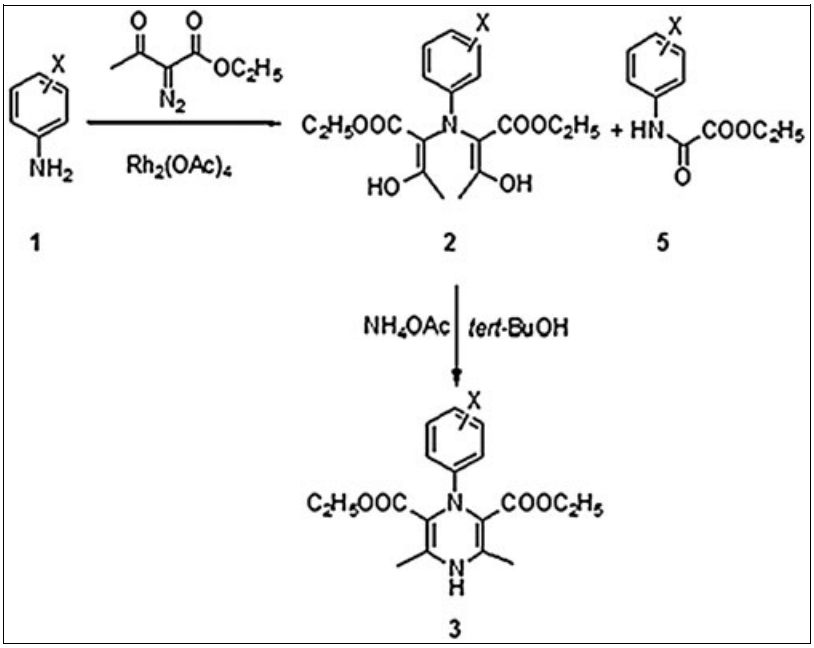

The 4-aryl-1,4-dihydropyrazines were prepared via the cyclization of *N*,*N*-bisalkylated anilines with ammonium acetate. These reactions were aided by improvements in the synthesis of *N*,*N*-bisalkylated anilines which were alkylated with anilines using ethyl 2-diazo acetoacetate in a reaction catalyzed by rhodium acetate in the absence of oxygen. A possible mechanistic route is postulated on the basis of the isolation of the *N*-alkylation intermediates, which were determined to be *N*-aryloxamates by ^{1}H NMR data and X-ray diffraction.

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INTRODUCTION

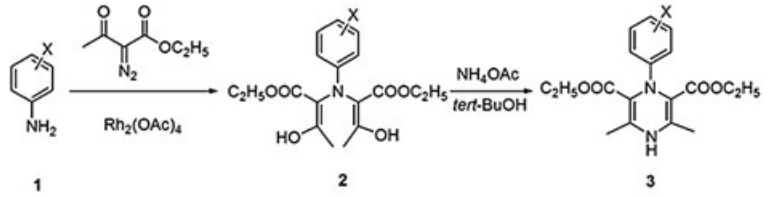
1,4-Dihydropyrazine is an important component of flavin coenzymes and of several marine luciferins [1]. This compound is especially useful as a potent Neuropeptide Y antagonist and as a surrogate of dihydropyridine in the study of biologically active compounds [2, 3]. The 1,4-dihydropyrazine ring itself is unknown, various substitutions of the ring system are required to produce stable, isolable molecules. This has been achieved with the placement of electron withdrawing or organometallic groups on the nitrogen in the structure and with the addition of bulky or conjugated substituents in other locations [4]. In the course of our investigations on the synthesis of nitrogen heterocyclic derivatives, we succeed in the synthesis of 1,4-dihydropyridines as precursors of 3,9-diazatetraasteranes [5]. The 1,4-dihydropyrazines are useful structural modifications of the 1,4-dihydropyridine system, and these structures are substantially different from the parent molecules in terms of molecular polarization and electron distribution. These altered electronic properties should therefore provide us with additional information on the synthesis and insight into the mechanism of this process.

The synthesis of 4-aryl-2,6-dimethyl-1,4-dihydropyrazine-3,5-dicarboxyl esters reported by Wehinger and Chorvat, and improved by Sit was involved the cyclization of *N*,*N*-bisalkylated anilines with ammonium acetate [2, 3]. However, two 4-aryl-2,6-dimethyl-1,4-dihydropyrazines (Aryl = 3-nitrophenyl and 4-fluoro-3-nitrophenyl) were prepared by Sit with about 18% aggregate yield. The purpose of this article is to synthesize a series of 4-aryl-1,4-dihydropyrazines (**3**) and elucidate the reason for the lower yields (Scheme 1).

RESULTS AND DISCUSSION

The 4-aryl-1,4-dihydropyrazine-3,5-dicarboxyl esters (**3**) was synthesized by the cyclization of bis-alkylated aniline (**2**) with ammonium acetate; **2** was prepared by the alkylation of aniline (**1**) with diazo compound in the presence of catalytic rhodium acetate dimmer (Table 1).

Scheme 1



X = a: H; b: 4-CH_3; c: 3-CH_3; d: 4-NO_2; e: 3-NO_2; f: 4-OCH_3; g: 4-Cl; h: 3-Cl; i: 4-$COOC_2H_5$; j: 2,5-diF; k: 3,5-diCF_3

It is worth mentioning out that the yields of compounds **2** seriously affected the synthesis of **3**. Compounds **2a-k** were synthesized by the *N*-alkylation of anilines with 2-diazo acetoacetate catalyzed by rhodium acetate, and the end of the reaction was identified by the conversion of mono-alkylated anilines (**4**) to *N*,*N*-bisalkylated anilines (**2**) by TLC. The resulting mixtures were filtered over silica gel, eluting with ethyl acetate-petroleum ether, and **2a-k** were obtained in approximately 30% yields (Table 2). A significant amount of by-product (**5**) was always generated in these reactions, sometimes equaling the amount of **2** (Scheme 2). The structures of **5** were *N*-aryl-oxamates, which were confirmed by ^{1}H NMR spectroscopy and X-ray diffraction analysis (Fig. 1).

Based on the experimental results, **5** may be produced by the oxidation of **4** in the presence of rhodium acetate dimer. At the beginning of reaction, only a mono-alkylated anilines (**4**) was obtained after approximately 15 min, and then **5** began to emerge after approximately 40 min. To confirm the transformation of **4** to *N*-aryl-oxamates (**5**), a catalytic amount of cerium (IV) ammonium nitrate (CAN) was added to a solution of **4**. The results indicated that **4** was completely transformed to **5**. Without CAN as catalyst, no product **5** was observed. The mechanism of the formation of **5** could thus be similar to that of acetoacetamides forming oxamates when exposed to cerium (IV) ammonium nitrate under oxygenated conditions [7]. Moverover, the rhodium acetate dimer was found to play an important role in accelerating the oxidation from **4** to **5** besides *N*-alkylation of anilines with 2-diazo acetoacetate, while **5** was not obtained from **4** in the absence of rhodium acetate dimer after refluxing for 24 hours. Additional evidence is the fact that oxygen takes part in the reaction leading to the **5**. When the conversion of **4** to **5** was repeated in an atmosphere of oxygen with the catalytic amount of rhodium acetate dimer (0.2–0.4 mol %), the yield of the **5** increased significantly. But there was no **5** observed in the absence of oxygen under the same catalytic condition (Table 2). For example, **5j** was obtained in 24% yield under refluxing conditions, 45% yield when refluxed in the presence of oxygen, and none was obtained under an atmosphere of nitrogen. From this data, it can be seen that it is very important to avoid oxygen in the preparation of **2**. Under an atmosphere of nitrogen, **2** was obtained in acceptable yields (40–69%).

Furthermore, the electronic effects of substituents on the anilines visibly affected the yields of **2** under an atmosphere

Table 1

The yield of 4-aryl-1,4-dihydropyrazin-3,5-dicarboxyl esters (**3**).

Entry	Products	X	Time (min)	Yield (%) [Lit.]
1	**3a**	H	60	55 [3a]
2	**3b**	4-CH_3	50	48 [3a]
3	**3c**	3-CH_3	50	51 [3a]
4	**3d**	4-NO_2	30	69
5	**3e**	3-NO_2	30	67
6	**3f**	4-OCH_3	50	33
7	**3g**	4-Cl	40	63
8	**3h**	3-Cl	30	60 [3a]
9	**3i**	4-$COOC_2H_5$	30	62
10	**3j**	2,5-diF	30	76
11	**3k**	3,5-diCF_3	30	73

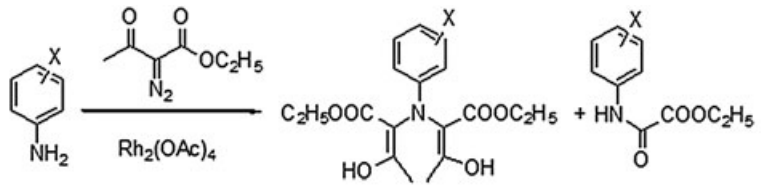
Table 2

Yields of **2** and **5** under different condition.[a]

		Yield[b] (%)		Yield[c] (%)		Yield[d] (%)	
Entry	X	**2**	**5** [Lit.][e]	**2**	**5**	**2**	**5**
a	H	28	8 [6]	10	15	49	0
b	4-CH_3	21	8 [6]	8	14	45	0
c	3-CH_3	23	7 [6]	7	16	47	0
d	4-NO_2	31	23 [6]	14	35	66	0
e	3-NO_2	36	25 [6]	12	37	64	0
f	4-OCH_3	19	6 [6]	5	14	40	0
g	4-Cl	37	23 [6]	15	31	67	0
h	3-Cl	35	22 [6]	11	38	60	0
i	4-$COOC_2H_5$	30	29 [6]	13	41	69	0
j	2,5-diF	31	24	11	45	63	0
k	3,5-diCF_3	39	31 [6]	18	43	68	0

[a]**2** not characterized; yields refer to partially purified material.
[b]Yields under the reflux.
[c]Yields in the presence of air.
[d]Yields under an atmosphere of nitrogen.
[e]Isolated yields and their ^{1}H NMR data were verified and compared with those reported in the literature.

Scheme 2



X = **a**: H; **b**: 4-CH_3; **c**: 3-CH_3; **d**: 4-NO_2; **e**: 3-NO_2; **f**: 4-OCH_3; **g**: 4-Cl; **h**: 3-Cl; **i**: 4-$COOC_2H_5$; **j**: 2,5-diF; **k**: 3,5-diCF_3

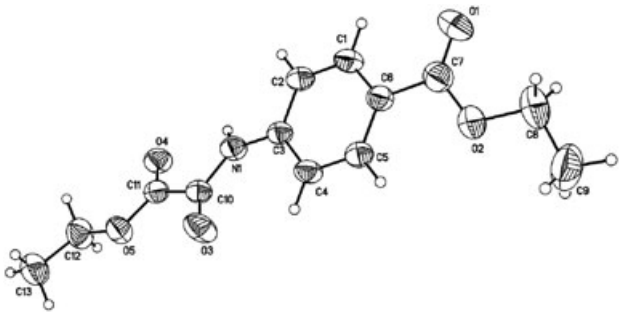


Figure 1. ORTEP diagram of the crystal structure of compound **5i** (Drawn at the 50% thermal ellipsoids).

of nitrogen. The anilines with electron-donating substituents, the yields of **2** were low (40–47%). However, anilines with electron-withdrawing groups, **2** were obtained in the higher yields (60–69%). For example, when X was 4-CH_3, **2b** was obtained in 45% yield, but when X was 4-NO_2, **2d** was obtained in 66% yield.

A speculated mechanism for the formation of **5** is described in Scheme 3.

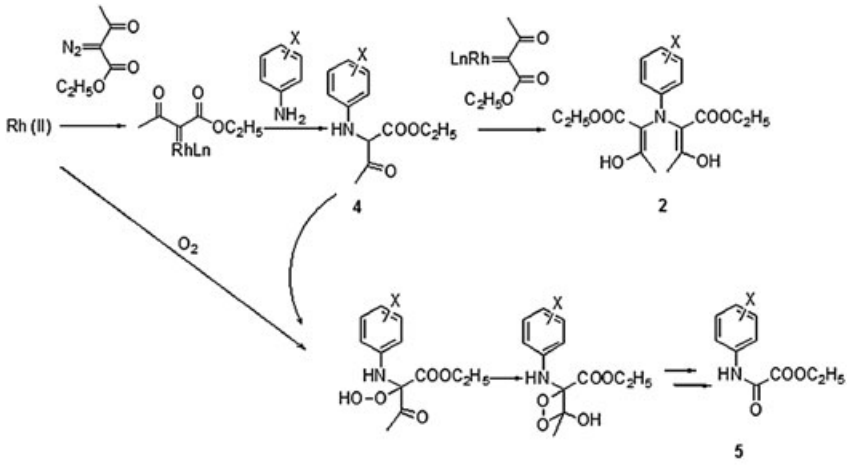
CONCLUSION

A series of 4-aryl-3,5-bis(ethoxycarbonyl)-2,6-dimethyl-1,4-dihydropyrazines (**3**) were synthesized from the reaction of *N,N*-bisalkylated aniline (**2**) with ammonium acetate (**1**), producing the final products in fair to good yields by the improvement on the synthesis of **2**. Ethyl *N*-aryloxamates (**5**) were by-products of the *N*-alkylation of anilines, which were produced by the oxidation of mono-alkylated anilines (**4**). The yields of **2** were markedly improved in the absence of oxygen, which is supported by the postulated mechanism.

EXPERIMENTAL

General. All chemicals and reagents were purchased from commercial sources and used without further purification. M. p.: X-5 melting point apparatus (uncorrected). IR spectra: Vertex-70-FTIR Spectrophotometer; KBr pellets. NMR spectra: Bruker-Avance-II-400 instrument (400 MHz) in $CDCl_3$. MS spectra: ZAB-HS & ESQUIRE 6000 mass spectrometer. Elemental analyses: CARLO ERBA 1106 elemental analysis instrument. X-ray diffractions were recorded on a Siemens P4 or Simart-1000 diffractometer. Flash chromatography was carried out on silica gel

Scheme 3



X = **a**: H; **b**: 4-CH_3; **c**: 3-CH_3; **d**: 4-NO_2; **e**: 3-NO_2; **f**: 4-OCH_3; **g**: 4-Cl; **h**: 3-Cl; **i**: 4-$COOC_2H_5$; **j**: 2,5-diF; **k**: 3,5-diCF_3

(Merck 230-400 mesh). All reactions were monitored using thin layer chromatography (TLC) on silica gel plates (Merck $60F_{254}$).

General procedure for the synthesis of compounds (2). A mixture of aniline (2.0 mmol), rhodium acetate dimmer (2.6 mg, 0.3 mol %) and 10 mL of dry benzene was warmed to reflux under an atmosphere of nitrogen. A solution of ethyl 2-diazo acetoacetate [8] (5.0 mmol) in 10 mL benzene was added dropwise to the refluxing solution over a period of 30 min. The resulting mixture was heated to reflux until all of the mono-alkylated anilines (**4**) was converted into compound **2** within 60–100 min, as monitored by TLC. After cooling the solution to room temperature, the volatile components were removed in vacuo and the residue was purified by flash chromatography using petroleum ether and EtOAc as eluents. **2** was instable and used without further purification for the synthesis of **3**.

General procedure for the synthesis of compounds (3). Compound **2** (1.0 mmol) was heated to reflux with ammonium acetate (1.1 mmol, 84.7 mg) in *tert*-butanol (10 mL). The reaction progress was monitored by TLC. The solvent was removed in vacuo and the residue was recrystallized from ethyl acetate and hexane to give pure compound **3**.

3,5-Bis(ethoxycarbonyl)-2,6-dimethyl-4-phenyl-1,4-dihydropyrazine (3a). Pale yellow solid, mp. = 226.5–227.7°C (lit [3a], 228°C), yield (0.18 g, 55%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.23–1.26 (*m*, 6H, CH_3), 2.40 (*s*, 6H, CH_3), 4.21–4.26 (*m*, 4H, CH_2), 5.80 (*s*, 1H, NH), 6.70 (*d*, 2H, *J* = 8.0 Hz, Ar-H), 6.70 (*d*, 2H, *J* = 9.2 Hz, Ar-H), 6.82–6.85 (m, 1H, Ar-H), 7.13–7.17 (*m*, 2H, Ar-H).

3,5-Bis(ethoxycarbonyl)-2,6-dimethyl-4-(4-methylphenyl)-1,4-dihydropyrazine (3b). Yellow solid, mp. = 151.9–153.1°C (lit [3a], 211°C), yield (0.16 g, 48%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.23–1.27 (*m*, 6H, CH_3), 2.23 (*s*, 3H, CH_3, Ar-CH_3), 2.38 (*s*, 6H, CH_3), 4.20–4.25 (*m*, 4H, CH_2), 5.76 (*s*, 1H, NH), 6.61 (*d*, 2H, *J* = 8.4 Hz, Ar-H), 6.98 (*d*, 2H, *J* = 8.4 Hz, Ar-H).

3,5-Bis(ethoxycarbonyl)-2,6-dimethyl-4-(3-methylphenyl)-1,4-dihydropyrazine (3c). Pale yellow solid, mp. = 160.8–161.4°C (lit [3a], 176°C), yield (0.17 g, 51%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.24–1.27 (*m*, 6H, CH_3), 2.25 (*s*, 3H, CH_3, Ar-CH_3), 2.38 (*s*, 6H, CH_3), 4.21–4.26 (*m*, 4H, CH_2), 5.29 (*s*, 1H, NH), 6.49 (*s*, 1H, Ar-H), 6.64 (*d*, 1H, *J* = 7.2 Hz, Ar-H), 7.05 (*m*, 2H, Ar-H).

3,5-Bis(ethoxycarbonyl)-2,6-dimethyl-4-(4-nitrophenyl)-1,4-dihydropyrazine (3d). Yellow solid, mp. = 212.8–214.3°C, yield 69%. IR (KBr): 2983, 1699, 1672, 1638, 1596, 1560, 1543, 1500, 1373, 1317, 1280, 1194, 1107, 1105, 1063 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.23–1.26 (*m*, 6H, CH_3), 2.45 (*s*, 6H, CH_3), 4.21–4.27 (*m*, 4H, CH_2), 6.52 (*s*, 1H, NH), 6.63 (*d*, 2H, *J* = 9.2 Hz, Ar-H), 8.04 (*d*, 2H, *J* = 9.2 Hz, Ar-H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 14.3, 17.5, 60.6, 107.6, 111.7, 125.1, 139.8, 149.0, 154.8, 164.7. MS (ESI): *m/z* (%) = 375.0 $[M]^+$. Anal. Calcd. for $C_{18}H_{21}N_3O_6$: C 57.59, H 5.64, N 11.19; Found C 57.65, H 5.65, N 11.17.

3,5-Bis(ethoxycarbonyl)-2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyrazine (3e). Yellow solid, mp. = 212–214°C, yield 67%. IR (KBr): 3293, 1696, 1668, 1531, 1332, 1193, 1109, 779, 734 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.25–1.28 (*m*, 6H, CH_3), 2.46 (*s*, 6H, CH_3), 4.24–4.31 (*m*, 4H, CH_2), 5.91 (*s*, 1H, NH), 6.95–7.68 (*m*, 4H, Ar-H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 14.3, 17.8, 60.5, 107.8, 108.6, 114.0, 118.8, 129.0, 148.7, 149.1, 151.1, 165.1. MS (ESI): *m/z* (%) = 375.0 $[M]^+$. Anal. Calcd. for $C_{18}H_{21}N_3O_6$: C 57.59, H 5.64, N 11.19; Found C 57.60, H 5.66, N 11.21.

3,5-Bis(ethoxycarbonyl)-2,6-dimethyl-4-(4-methoxyphenyl)-1,4-dihydropyrazine (3f). Pale yellow solid, mp. = 171.4–171.8°C, yield 33%. IR (KBr): 2975, 1672, 1634, 1505, 1473, 1370, 1330, 1303, 1243, 1181, 1105, 1058, 1039 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.24–1.28 (*m*, 6H, CH_3), 2.38 (*s*, 6H, CH_3), 3.73 (*s*, 3H, OCH_3), 4.20–4.26 (*m*, 4H, CH_2), 5.74 (*s*, 1H, NH), 6.73 (*s*, 4H, Ar-H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 14.4, 17.8, 55.1, 60.3, 99.7, 104.6, 109.6, 129.2, 148.1, 152.1, 160.1, 166.0. MS (ESI): *m/z* (%) = 361.1 $[M-1]^+$. Anal. Calcd. for $C_{19}H_{24}N_2O_5$: C 63.32, H 6.71, N 7.71; Found C 63.31, H 6.70, N 7.75.

3,5-Bis(ethoxycarbonyl)-4-(4-chlorophenyl)-2,6-dimethyl-1,4-dihydropyrazine (3g). Pale yellow solid, mp. = 212.8–214.3° C, yield 63%. IR (KBr): 3434, 3291, 1694, 1668, 1635, 1592, 1329, 1300, 1193, 1108, 814, 776 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.24–1.27 (*m*, 6H, CH_3), 2.41 (*s*, 6H, CH_3), 4.21–4.26 (*m*, 4H, CH_2), 5.79 (*s*, 1H, NH), 6.61 (*d*, 2H, *J* = 9.2 Hz, Ar-H), 7.08 (*d*, 2H, *J* = 9.2 Hz, Ar-H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 14.3, 17.8, 60.3, 109.4, 114.3, 124.3, 128.3, 148.4, 149.0, 165.8. MS (ESI): *m/z* (%) = 364.1 $[M]^+$. Anal. Calcd. for $C_{18}H_{21}ClN_2O_4$: C 59.26, H 5.80, N 7.68; Found C 59.21, H 5.81, N 7.70.

3,5-Bis(ethoxycarbonyl)-4-(3-chlorophenyl)-2,6-dimethyl-1,4-dihydropyrazine (3h). Pale yellow solid, mp. = 181.2–183.0°C (lit [3a], 214°C), yield (0.22g, 60%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.26–1.30 (*m*, 6H, CH_3), 2.43 (*s*, 6H, CH_3), 4.25–4.26 (*m*, 4H, CH_2), 6.02 (*s*, 1H, NH), 6.56–7.08 (*m*, 4H, Ar-H).

3,5-Bis(ethoxycarbonyl)-2,6-dimethyl-4-(4-(ethoxycarbonyl) phenyl)-1,4-dihydropyrazine (3i). Pale yellow solid, mp. = 198.6–200.3°C, yield 62%. IR (KBr): 3290, 2975, 1710, 1671, 1635, 1603, 1509, 1423, 1375, 1266, 1105, 1024, 768 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.23–1.27 (*m*, 6H, CH_3), 1.34–1.38 (*m*, 3H, CH_3), 2.44 (*s*, 6H, CH_3), 4.23–4.26 (*m*, 4H, CH_2), 4.29–4.35 (*m*, 2H, CH_2), 6.11 (*s*, 1H, NH), 6.65 (*d*, 2H, *J* = 9.2 Hz, Ar-H), 7.84 (*d*, 2H, *J* = 9.2 Hz, Ar-H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 14.3, 14.4, 17.5, 60.3, 60.4, 108.3, 111.9, 120.9, 130.4, 148.7, 153.8, 165.4, 167.0. MS (ESI): *m/z* (%) = 402.0 $[M]^+$. Anal. Calcd. for $C_{21}H_{26}N_2O_6$: C 62.67, H 6.51, N 6.96; Found C 62.69, H 6.52, N 6.98.

3,5-Bis(ethoxycarbonyl)-2,6-dimethyl-4-(2,5-bis(fluorophenyl))-1,4-dihydropyrazine (3j). Pale yellow solid, mp. = 143.2–145.4°C, yield 76%. IR (KBr): 3316, 2982, 2932, 1696, 1624, 1503, 1470, 1370, 1309, 1188, 1108, 769 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.26-1.29 (*m*, 6H, CH_3), 2.39 (*s*, 6H, CH_3), 4.20-4.25 (*m*, 4H, CH_2), 5.99 (*s*, 1H, NH), 6.45-6.91 (*m*, 3H, Ar-H). MS (ESI): *m/z* (%) = 366.1 $[M]^+$. Anal. Calcd. for $C_{18}H_{20}F_2N_2O_4$: C 59.01, H 5.50, N 7.65; Found C 59.15, H 5.51, N 7.67.

3,5-Bis(ethoxycarbonyl)-4-(3,5-bis(trifluoromethyl)phenyl)-2,6-dimethyl-1,4-dihydropyrazine (3k). Pale yellow solid, mp. = 187.8–189.2°C, yield 73%. IR (KBr): 3287, 2988, 2918, 1704, 1669, 1615, 1378, 1278, 1182, 1124, 1108, 865, 778, 699 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.25–1.28 (*m*, 6H, CH_3), 2.49 (*s*, 6H, CH_3), 4.20–4.23 (*m*, 4H, CH_2), 5.98 (*s*, 1H, NH), 7.02 (s, 2H, Ar-H), 7.31 (*s*, 1H, Ar-H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 14.2, 17.9, 60.6, 108.2, 112.3, 112.5, 122.2, 124.9, 131.4, 131.7, 149.2, 150.6, 164.7. MS (ESI): *m/z* (%) = 466.0 $[M]^+$. Anal. Calcd. for $C_{20}H_{20}F_6N_2O_4$: C 51.51, H 4.32, N 6.01; Found C 51.40, H 4.33, N 6.02.

General procedure for the synthesis of compound (5). Compound **5** was synthesized by the alkylation of aniline with ethyl ethyl 2-diazo acetoacetate, as in the procedure used to synthesize compound **2** in the presence of air, which was bubbled into the reaction mixture at the speed of 2 L/h. The resulting residue was purified by flash chromatography to give compound **5**, using petroleum ether and EtOAc as eluents.

Crystallographic data of 5i. Crystallographic data for the structures of **5i** reported in this article have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 753366. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax.: (internet) + 44 1223/ 336-033; e-mail: deposit@ccdc.cam.ac.uk].

Ethyl 2-(2,5-difluorophenylamino)-2-oxoacetate (5j). Colorless solid, mp. = 44.5–45.3°C, yield 45%. IR (KBr): 3273, 3028, 2934, 1726, 1695, 1618, 1503, 1025 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.44-1.48 (*m*, 3H, CH_3), 4.43–4.49 (*m*, 2H, CH_2), 6.82–6.88 (*m*, 1H, Ar-H), 7.09–7.15 (*m*, 1H, Ar-H), 8.20–8.25 (*m*, 1H, Ar-H), 9.15 (*s*, 1H, NH). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 13.9, 64.0, 108.6, 111.8, 115.7, 125.8, 148.6, 153.9, 155.7, 160.1. MS (ESI): *m/z*(%) = 229.1 $[M]^+$. Anal. Calcd. for $C_{10}H_9F_2NO_3$: C 52.41, H 3.96, N 6.11; Found C 52.50, H 3.95, N 6.09.

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