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Directed Aminomethylation of Pyrrole, Indole, and Carbazole with *N*,*N*,*N*′,*N*′-Tetramethylmethanediamine

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Abstract—Catalytic aminomethylation of pyrrole and indole with *N*,*N*,*N*′,*N*′-tetramethylmethanediamine in the presence of 5 mol % of $ZrOCl_2 \cdot 8H_2O$ proceeds selectively at the positions *2*, *5* of pyrrole and *1*, *3* of indole. Carbazole under the same conditions affords 3-formyl-9-aminomethyl derivative. The reaction in the presence of 5 mol % of K_2CO_3 occurs as monoaminomethylation: for pyrrole at the position *2*, for indole at the position *3*, and for carbazole at the nitrogen atom of the substrate. Water-soluble 1,1'-(1*H*-pyrrole-2,5-diyl)bis(*N*,*N*-dimethylmethanamine) exhibits a fungistatic activity with respect to phytopathogenic fungi *Rhizoctonia solani*.

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Aminomethylation of aromatic CH-acids with formaldehyde and secondary amines or with *N*,*N*,*N*′,*N*′-tetramethylmethanediamine is used in the synthesis of antifriction and antimicrobial additives, corrosion inhibitors [1–3]. The aminomethylation of pyrrole occurs in the positions *2* and *N*-, and of indole, in the position *3* and *N*- at the promotion with excess SO_2 or with a system K_2CO_3–succinic anhydride [4, 5]. The aminomethylation of pyrrole in the presence of equimolar amount of activators Me_3SiCl or $MeSiCl_3$ proceeds with the formation of a mixture of 2- and 2,5-aminomethyl derivatives [6].

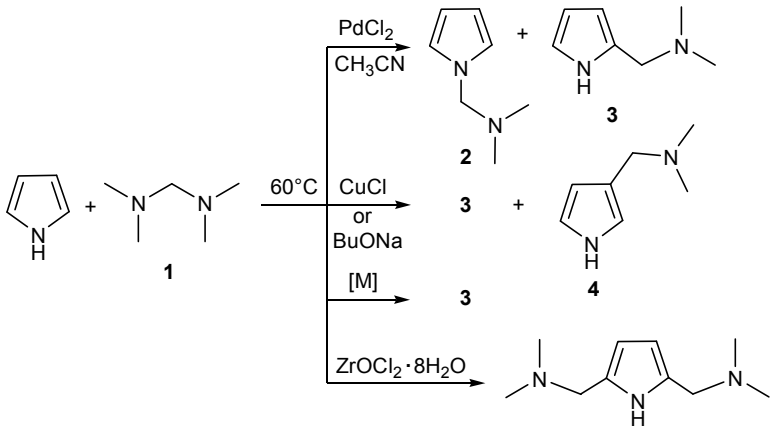
We lately established that Lewis acids were efficient catalysts of CH-activation of 1,3-dicarbonyl compounds in thiomethylation [7–9]. Considering this result we planned in this study to investigate the catalytic activity of Lewis acids capable of activation of *C*,*N*- and *C*,*C*-aminomethylation of pyrrole and its benzo-fused systems, indole and carbazole, in reaction with accessible *N*,*N*,*N*′,*N*′-tetramethylmethanediamine **1** [10].

We tested as catalysts "hard" ($AlCl_3$, $ZrOCl_2 \cdot 8H_2O$, $FeCl_3 \cdot 6H_2O$, Cp_2TiCl_2, HCl, K_2CO_3, BuONa), "borderline" [$Ni(acac)_2$, $Sm(NO_3)_3 \cdot 6H_2O$, $CoCl_2 \cdot 6H_2O$], and "soft" (CuCl, $PdCl_2$) acids as estimated by Pearson concept [11]. The reaction was carried out with excess of *N*,*N*,*N*′,*N*′-tetramethylmethanediamine **1** that acted as both a solvent and an efficient *C*-electrophilic reagent.

The reaction of pyrrole with reagent **1** at 60°C in the presence of "borderline" Lewis acids gives a mixture of *N*- (**2**) and *C*- (**3**, **5**) aminomethyl derivatives whose content was measured by GC and GC-MS methods (Scheme 1, Table 1, runs nos. *2*, *3*, *5*).

The aminomethylation in the presence of "soft" ($PdCl_2$, $CuCl/SiO_2$) or "hard" ($AlCl_3$, $FeCl_3 \cdot 6H_2O$) acids, as well as in the presence of K_2CO_3 base led to a selective formation of monoaminomethyl derivative **3** (Table 1, runs nos. *7*, *9–11*, *14*), and in the presence of 5 mol % of $ZrOCl_2 \cdot 8H_2O$ or HCl, of 2,5-bis(aminomethyl) derivative **5** in a high yield (Table 1, runs nos. *12*, *13*). The *C*-aminomethylation under the effect of 5 mol % of CuCl and BuONa occurred nonselectively with the formation of a mixture of 2- and 3-(dimethylaminomethyl)pyrroles **3** and **4** in a ratio 1 : 1 (Table 1, runs nos. *8*, *15*). In acetonitrile in the presence of 5 mol % of $PdCl_2$ a mixture formed of aminomethylpyrroles **2** and **3**, 6 : 1. The reaction

Scheme 1.



[M] = $PdCl_2$, $CuCl/SiO_2$, $AlCl_3$, $FeCl_3·6H_2O$, K_2CO_3.

without catalyst proceeded with the formation of a mixture of products of *N*- and *C*-aminomethylation **2–4** in an overall yield 30% (Table 1, run no. *1*).

Hence at heating pyrrole with *N*,*N*,*N*′,*N*′-tetramethylmethanediamine **1** the following reactions proceed selectively: 2-aminomethylation in the presence of Lewis acids (5 mol % $CuCl/SiO_2$, $PdCl_2$, $AlCl_3$ and $FeCl_3·6H_2O$ or K_2CO_3), 2,5-diaminomethylation in the presence of 5 mol % of $ZrOCl_2·8H_2O$ or HCl; prevailing *N*-aminomethylation in acetonitrile in the presence of 5 mol % of $PdCl_2$.

Table 1. Conditions of pyrrole aminomethylation, reaction products ratio, and overall yield

Run no.	Catalyst, 5 mol %	Reaction products ratio	Overall yield, %
1	–	**2** : **3** : **4**, 1 : 20 : 10	30
2	$Sm(NO_3)_3 · 6H_2O$	**2** : **3** : **5**, 1 : 10 : 9	81
3	$Ni(acac)_2$	**2** : **3** : **5**, 2 : 1.5 : 1	90
4	Cp_2TiCl_2	**2** : **3**, 1 : 1.2	95
5	$CoCl_2 · 6H_2O$	**2** : **3**, 1 : 6	95
6[a]	$PdCl_2$	**2** : **3**, 6 : 1	40
7	$PdCl_2$	**3**	96
8	CuCl	**3** : **4**, 1 : 1	28
9	$CuCl/SiO_2$	**3**	60
10	$AlCl_3$	**3**	78
11	$FeCl_3 · 6H_2O$	**3**	30
12	$ZrOCl_2 · 8H_2O$	**5**	92
13	HCl	**5**	77
14	K_2CO_3	**3**	83
15	BuONa	**3** : **4**, 1 : 1	75

[a] Solvent CH_3CN.

The aminomethylation of indole with bisamine **1** similarly depends on the catalyst type. The reaction takes two paths: *C*-aminomethylation gives *N*-(1*H*-indol-3-yl-methyl)-*N*,*N*-dimethylamine **6** in the presence of K_2CO_3 at the indole conversion as low as 10% (Table 2, run no. *8*). Yet in the presence of

Scheme 2.

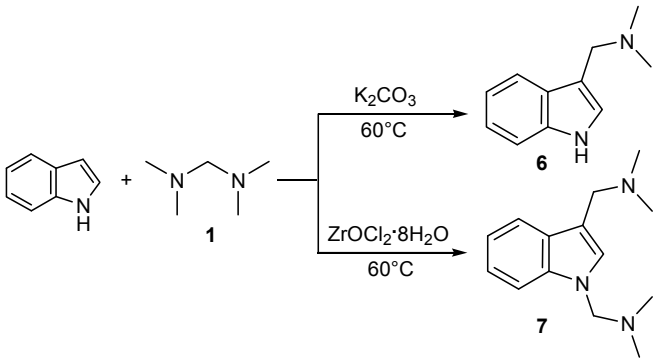
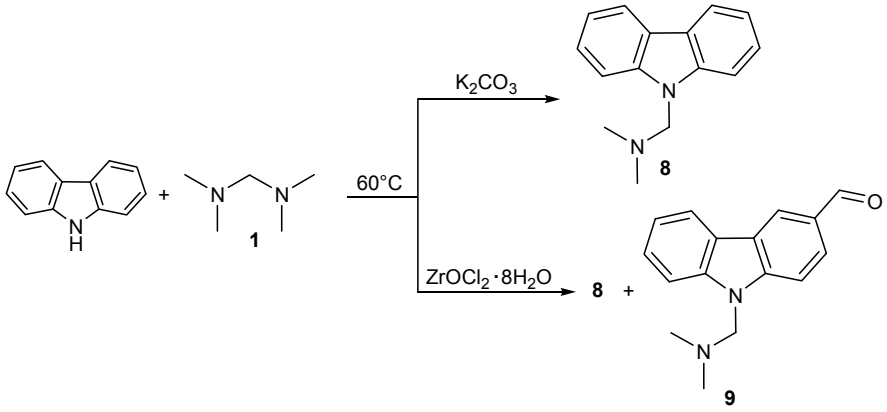


Table 2. Conditions of indole aminomethylation, reaction products ratio, and overall yield

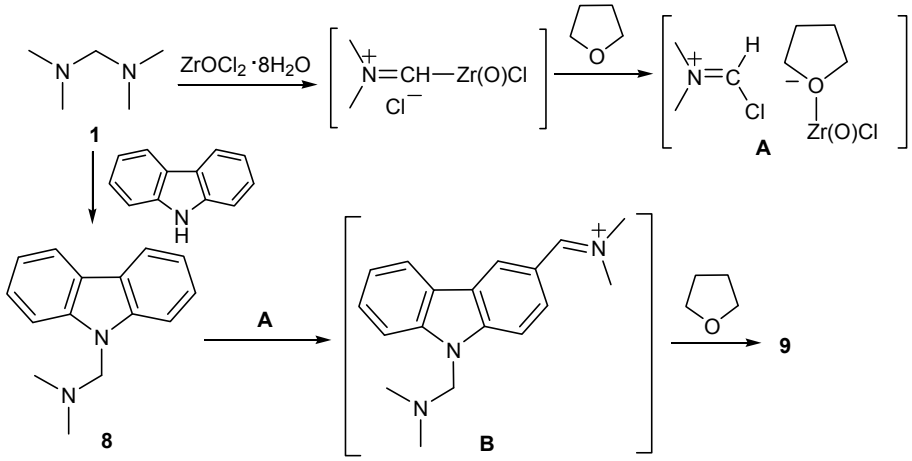
Run. No.	Catalyst, 5 mol %	Reaction products ratio	Overall yield, %
1	–	**7**	10
2	$Sm(NO_3)_3·6H_2O$	**7**	43
3[a]	$Sm(NO_3)_3·6H_2O$	**7**	40
4[b]	$PdCl_2$	**7**	52
5	$AlCl_3$	**6** : **7**, 1 : 1	48
6	$ZrOCl_2·8H_2O$	**7**	79
7	HCl	**6** : **7**, 1 : 5	77
8	K_2CO_3	**6**	10

Solvents:
[a] MeOH or
[b] CH_3CN.

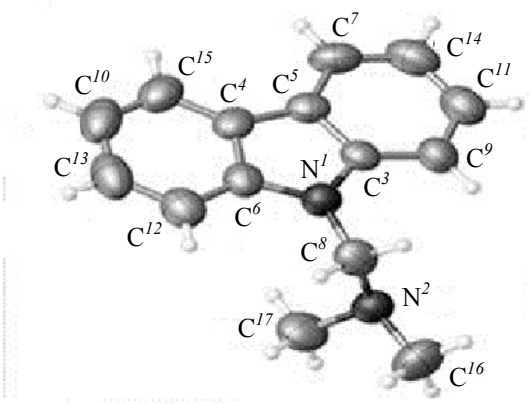
Scheme 3.



Scheme 4.



5 mol % of $Sm(NO_3)_3 \cdot 6H_2O$, $PdCl_2$, $ZrOCl_2 \cdot 8H_2O$ selectively formed 1,3-bis-(aminomethyl) derivative **7** in yields of 43, 52, and 79% respectively (Table 2, runs nos. *2–4*, *6*). At the use of equimolar quantity of K_2CO_3 amine **6** formed in the yield up to 70% [4] (Scheme 2).



Molecular structure of *N*,*N*-dimethyl-9*H*-carbazole-9-methanamine **8** according to XRD data.

The carbazole reaction with bisamine **1** under the chosen conditions occurred less effectively. We succeeded to perform the *N*-aminomethylation in THF in the presence of K_2CO_3 as a base with the formation of *N*,*N*-dimethyl-9*H*-carbazole-9-methanamine **8**, whose structure was reliably proved by X-ray diffraction (XRD) (see the figure). The reaction in the presence of 5 mol % of $ZrOCl_2 \cdot 8H_2O$ proceeded with the formation of a mixture of amine **8** and 3-formyl

Table 3. Conditions of carbazole aminomethylation, reaction products ratio, and overall yield

Run no.	Catalyst, 5 mol %	Solvent	Reaction products ratio	Overall yield, %
1	K_2CO_3	THF	**8**	32
2	$ZrOCl_2 \cdot 8H_2O$	THF	**8** : **9**, 1.6 : 1	50
3	$ZrOCl \cdot 8H_2O$	C_6H_6–MeOH 1 : 1	**8** : **9**, 1 : 1	63
4	$ZrOCl_2 \cdot 8H_2O$	CH_3CN	**8** : **9**, 2 : 1	81

derivative **9**, and their ratio depended on the solvent nature (Table 3, runs nos. *2–4*) (Scheme 3).

Amine **8** was formerly prepared from carbazole and reagent **1** in two stages through intermediate formation of an organomagnesium derivative involving a Grignard reagent [12], and the *C*-formylation of the *N*-substituted carbazole occurred readily in the Duff hexamine reaction yielding compounds possessing antitumor activity [13].

It is presumable that the carbazole *C*-formylation with the use of reagent **1** proceeds similarly to Duff [14] or Vilsmeier-Haack reaction with the formation of intermediate complex **A** and compound **B** (Scheme 4).

We presume that the carbazole formylation in the position *3* occurs in polar solvents, e.g., THF, through the formation of salt **A** related by the activity to Vilsmeier-Haack reagent (chloroiminium salt). Apparently the *N*-(aminomethyl) derivative **8** first forms and under the action of chloroiminium salt **A** in polar solvents converts into formyl derivative **9** via intermediate **B**.

Thus the catalyst $ZrOCl_2 \cdot 8H_2O$ is effective for the directed *C*,*C*-aminomethylation of pyrrole, *C*,*N*-aminomethylation of indole applying bisamine **1** without solvent, and also for *C*-formylation and *N*-aminomethylation of carbazole in polar solvents. Catalysis with 5 mol % of K_2CO_3 affords *C*-monoaminomethylated derivatives of pyrrole and indole, and with carbazole the reaction in THF proceeds as *N*-aminomethylation.

Among the synthesized compounds diamine **5** is water-soluble. The evaluation of its biologic activity was performed by diffusion in agar method [15], using as test objects phytopathogenic fungi *Fusarium oxysporum*, *Bipolaris sorokiniana*, and *Rhizoctonia solani*, which are causing diseases of various agricultural plants.

Compound **5** at the concentration of 0.5% showed a fungistatic effect with respect to *Rhizoctonia solani,* hampering its development and causing the formation of untypical mycelium.

EXPERIMENTAL

GC of reaction products was carried out on a chromatograph Shimadzu GC-9A equipped with a flame-ionization detector, steel column 2000 × 3 mm, stationary phase SE-30 (5%) on carrier Chromoton N-AW-HMDS, ramp 50–270°C, heating rate 8 deg/min, carrier gas helium. GC-MS analyses were performed on a chromatograph Shimadzu GC 2010 with a detector GCMS-QP2010 Ultra, capillary column Supelco 5 ms (60 m × 0.25 mm × 0.25 μm), carrier gas helium. Mass spectrum of compound **7** was recorded on an instrument Bruker MALDI TOF Autoflex III. IR spectra were taken on a spectrophotometer Bruker Vertex-70V from mulls in mineral oil. ^{1}H and ^{13}C NMR spectra were registered on a spectrometer Bruker Avance III HD 500 (500, 125 MHz) in $CDCl_3$, internal reference TMS, homo- and heteronuclear 2D experiments were carried out using standard pulse sequences of Bruker. Melting points were measured on an apparatus PHMK 80/2617 (Kofler hot stage). The reaction monitoring was done by TLC on Sorbfil plates, eluent cyclohexane–$CHCl_3$–EtOAc, 1 : 2 : 10, development in iodine vapor.

XRD analysis of the single crystal of compound **8** was performed on an automatic diffractometer XCalibur Eos, equipped with a CCD-detector and a source of MoK_α-radiation (graphite monochromator, λ 0.71073 Å, ω-scanning, $2\theta_{max}$°). Collection of data and processing of results was performed using the program Crys-Alis^{Pro}Oxford Diffraction Ltd. [16]. The structure was solved by the direct method and refined using SHELX program [17] in OLEX2 software [18] in full-matrix least-squares method in anisotropic approximation for nonhydrogen atoms. Hydrogen atoms were localized from difference Fourier synthesis and were included in the refinement with fixed positions and thermal parameters.

Microscopic fungi used as test cultures were taken from the collection of Ufa Biological Institute of the Russian Academy of Sciences.

Aminomethylation of pyrrole (indole, carbazole) with reagent 1. General procedure. In a glass reactor was charged 0.07 mL (1 mmol) of pyrrole (indole, carbazole) and 0.66 mL (5 mmol) of bisamine **1**. The mixture was stirred at heating for 6 h in the presence of 5 mol % of the desired catalyst (Tables 1–3). On cooling the mixture was passed through a bed of SiO_2, then evaporated on a rotary evaporator.

***N*-(Dimethylaminomethyl)pyrrole (2).** Yield 43%, resinous substance. IR spectrum, ν, cm^{-1}: 3202, 3129, 3100, 2974, 2443, 1669, 1252, 1174, 1143, 1096, 1014, 843, 718, 611. ^{1}H NMR spectrum, δ, ppm: 2.23 s (6H, CH_3), 3.49 s (2H, CH_2), 5.28 m (2H$_{arom}$), 6.82 m (2H$_{arom}$). ^{13}C NMR spectrum, δ, ppm: 44.36 (CH_3), 71.10 (CH_2), 110.97 (C$_{arom}$), 122.29 (C$_{arom}$).

Mass spectrum, *m/z* (I_{rel}, %): 124 (10) $[M]^+$, 80 (20), 58 (10), 44 (100). *M* 124.10 [19].

***N*,*N*-Dimethyl-1-(1*H*-pyrrol-2-yl)methanamine (3).** Yield 96%, resinous substance. IR spectrum, ν, cm^{-1}: 3377, 3100, 1669, 1252, 1174, 1143, 1096, 1014, 843, 718. ^1H NMR spectrum, δ, ppm: 2.26 s (6H, CH_3), 3.46 s (2H, CH_2), 6.11 s (1H$_{arom}$), 6.13 s (1H$_{arom}$), 6.73 s (1H$_{arom}$), 9.27 s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 44.96 (CH_3), 56.63 (CH_2), 107.12 (C_{arom}), 107.53 (C_{arom}), 117.85 ($C_{arom}N$), 129.02 ($C_{arom}N$). Mass spectrum, *m/z* (I_{rel}, %): 124 (30) $[M]^+$, 80 (100), 53 (15), 44 (25). *M* 124.10 [6].

***N*,*N*-Dimethyl-1-(1*H*-pyrrol-3-yl)methanamine (4).** Yield 38%, resinous substance. IR spectrum, ν, cm^{-1}: 3376, 3205, 3128, 3115, 2970, 2443, 1662, 1252, 1174, 1143, 1096, 1014, 845, 731, 614. ^1H NMR spectrum, δ, ppm: 2.22 s (6H, CH_3), 3.39 s (2H, CH_2), 5.28 s (1H$_{arom}$), 5.93 m (1H$_{arom}$), 6.82 m (1H$_{arom}$), 8.94 s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 44.35 (CH_3), 55.63 (CH_2), 106.37 (C_{arom}), 107.12 (C_{arom}), 117.85 ($C_{arom}N$), 128.95 ($C_{arom}N$). Mass spectrum, *m/z* (I_{rel}, %): 124 (15) $[M]^+$, 80 (10), 58 (100), 42 (15). Found, %: C 67.86; H 9.64; N 22.48. $C_7H_{12}N_2$. Calculated, %: C 67.70; H 9.74; N 22.56. *M* 124.10.

1,1'-(1*H*-Pyrrole-2,5-diyl)bis(*N*,*N*-dimethylmethanamine) (5). Yield 92%, resinous substance. IR spectrum, ν, cm^{-1}: 3459, 3140, 1661, 1252, 1174, 1142, 1097, 1041, 845, 767. ^1H NMR spectrum, δ, ppm: 2.20 s (12H, CH_3), 3.37 s (4H, CH_2), 5.92 s (2H$_{arom}$), 9.04 s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 44.99 (CH_3), 56.67 (CH_2), 107.08 (C_{arom}), 128.97 ($C_{arom}N$). Mass spectrum, *m/z* (I_{rel}, %): 181 (15) $[M]^+$, 137 (55) 93 (100) 80 (10), 58 (85), 42 (20). *M* 181.16 [6].

***N*-(1*H*-Indol-3-ylmethyl)-*N*,*N*-dimethylamine (6).** Yield 24%, resinous substance. IR spectrum, ν, cm^{-1}: 3408, 3049, 1676, 1614, 1557, 1513, 1237, 1180, 1154, 1098, 1016, 849, 806, 723, 626, 570, 426. ^1H NMR spectrum, δ, ppm: 2.26 s (6H, CH_3), 3.56 s (2H, CH_2), 6.91 s (1H$_{arom}$), 7.08, 7.12, 7.53, 7.70 m (4H, C_{arom}). ^{13}C NMR spectrum, δ, ppm: 45.16 (CH_3), 53.93 (CH_2), 111.12 (C_{arom}), 112.27 (C_{arom}), 119.5 ($C_{arom}N$), 120.2 (C_{arom}), 123.90, 127.80, 136.30. Mass spectrum, *m/z* (I_{rel}, %): 174 (5) $[M]^+$, 130 (5), 58 (100), 42 (5). *M* 174.12 [4].

1,1'-(1*H*-Indole-1,3-diyl)bis(*N*,*N*-dimethylmethanamine) (7). Yield 79%, resinous substance. IR spectrum, ν, cm^{-1}: 3467, 3051, 2973, 2941, 2244, 2198, 1670, 1614, 1324, 1265, 1239, 1155, 1097, 1042, 909, 849, 805, 733, 643. ^1H NMR spectrum, δ, ppm: 2.33 s (12H, CH_3), 3.68 s and 4.74 s (4H, CH_2), 7.14–7.75 m (5H, C_{arom}). ^{13}C NMR spectrum, δ, ppm: 42.73 and 45.32 (CH_3), 54.49 and 68.65 (CH_2), 110.00 (C_{arom}), 112.13 (C_{arom}), 119.37 (C_{arom}), 119.45, 121.83, 128.11, 128.54, 137.28 (CN_{arom}). Mass spectrum, *m/z* (I_{rel}, %): 230 [*M* – H]. *M* 231.17 [20].

1-(9*H*-Carbazol-9-yl)-*N*,*N*-dimethylmethanamine (8). Yield 54%, colorless needle crystals. R_f 0.77, mp 67–68°C. Mass spectrum, *m/z* (I_{rel}, %): 224 (10) $[M]^+$, 58 (100), 42 (5) [12].

9-[(Dimethylamino)methyl]-9*H*-carbazole-3-carbaldehyde (9). Yield 32%, yellow oily substance. R_f 0.15, eluent cyclohexane–$CHCl_3$–EtOAc, 1 : 2 : 10. IR spectrum, ν, cm^{-1}: 1768, 1599, 1239, 1037, 928, 858, 756, 574. ^1H NMR spectrum, δ, ppm: 2.50 s (6H, CH_3), 3.93 s (2H, CH_2), 7.21–8.09 m (7H, CH_{arom}), 9.35 s (1H, CH). ^{13}C NMR spectrum, δ, ppm: 43.07 (CH_3), 65.74 (CH_2), 109.48 (C_{arom}), 110.78 (C_{arom}), 119.34 (C_{arom}), 120.08 (C_{arom}), 125.55 (C_{arom}), 125.76 (C_{arom}), 139.83 (CN_{arom}), 162.74 (C_{arom}), 195.04 (CO). Found, %: C 76.04; H 6.56; N 11.19. $C_{16}H_{16}N_2O$. Calculated, %: C 76.16; H 6.39; N 11.10.

Crystallographic parameters and details of refinement of the crystal structure of amine 8. $C_{15}H_{16}N_2$. *M* 224.30. Crystal lattice orthorhombic, space group 19: $P2_12_12_1$. Unit cell parameters: *a* 5.2971(10), *b* 13.249(2), *c* 17.886(3) Å, α = β = γ = 90°, *V* 1255.3(3) $Å^3$, *Z* 5; *T* 293(2) K, μ(Mo$K_α$) 0.088 mm^{-1}, d_{calc} 1.484 g/cm^3. 2380 independent reflections were observed (R_{int} 0286) in the range of indices $-3 \le h \le 7$, $-18 \le k \le 16$, $-24 \le l \le 22$. The final values of convergence factors are as follows: R_1 0.0483 for 2380 independent reflections with $I > 2\sigma(I)$, wR_2 0.0905 for all independent reflections. Complete XRD data are deposited in Cambridge Crystallographic Data Centre (CCDC 1551084).

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