# New tridentate ligands based on 2-*tert*-butyl-4-methylphenol: synthesis and structure

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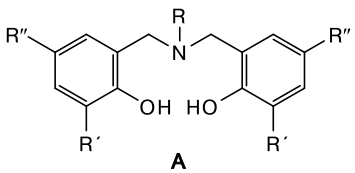
New sterically hindered bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)amine was synthesized based on 2-*tert*-butyl-4-methylphenol. The reaction of the latter with *N*,*N*-dimethylformamide affords *N*,*N*-bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)formamide. According to the X-ray diffraction data, this reaction product occurs in the crystal as a dimer stabilized by a bifurcated hydrogen bond. The intermolecular component of the C=O···H—O hydrogen bond ($l$ = 1.990 Å) between the carbonyl oxygen atom of one molecule and the phenolic hydroxy group of another molecule ensures the formation of dimers. Another component is the intramolecular C=O···H—O hydrogen bond ($l$ = 1.754 Å) between the carbonyl oxygen atom and the phenolic hydroxy group of the same molecule. In the crystal, the dimers are linked *via* C—H...π interactions to form long chains (2.741 Å).

**Key words:** bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)amine, *N*,*N*-bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)formamide, X-ray diffraction.

2-Aminophenol and its derivatives are widely used as ligands.[1—5] Particular attention is given to polydentate ligands. Their stereoelectronic characteristics can be controlled and, consequently, their physicochemical properties and reactivity can be varied by changing the nature of substituents both at the nitrogen atom and at the aromatic ring. Tetradentate ligands based on 2-hydroxybenzylamine $N(CH_2C_6H_nR_{4-n}OH)_3$ are being extensively investigated and are commonly employed in the synthesis of transition metal complexes (see the study[6] and references cited therein) and hypervalent compounds of main group elements (Si, Al, P).[7—15]

Tridentate ligands based on 2-hydroxybenzylamine (**A**), which form complexes with P, Ti, Cu, Fe, Zn, Mo, W, Zr compounds, have received much less attention.




**A**

R = H, Me, $PhCH_2$, $PyCH_2$, $CH_2C(O)OH$
R′, R″ = Me, $Bu^t$

These unique complexes are not only of interest for research in theoretical and structural chemistry but are also of great practical importance as efficient catalysts for organic synthesis and polymer chemistry.[16—25] The synthesis of bis(3,5-di-*tert*-butyl-2-hydroxybenzyl)amine (**A**, R = H, R′ = R″ = $Bu^t$) has recently been reported.[23,26] The free NH group in tridentate ligands not only increases their synthetic potential but also influences their conformational behavior due to the formation of intra- and/or intermolecular hydrogen bonds. Therefore, the development of methods for the synthesis of bis(2-hydroxybenzyl)amine derivatives and investigation of their structures are of interest. The goals of this work are to develop a method for the synthesis of bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)amine (**1**) and to study its structure and properties.

## Results and Discussion

The reaction of 2,4-di-*tert*-butylphenol with hexamethylenetetramine in 85% formic acid affords bis(3,5-di-*tert*-butyl-2-hydroxybenzyl)amine.[23,26] This reaction proceeds through a benzoxazine derivative as an intermediate, which was also isolated and characterized.[23] We applied this procedure[26] to prepare bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)amine (**1**). 2-*tert*-Butyl-4-methylphenol reacts with hexamethylenetetramine to form compound **1** in 37% yield (Scheme 1, method *A*).

We expected that an alternative method (*B*) based on the reaction of 3-*tert*-butyl-2-hydroxy-5-methylbenzyl chloride (**2**) with 3-*tert*-butyl-2-hydroxy-5-methylbenzyl-

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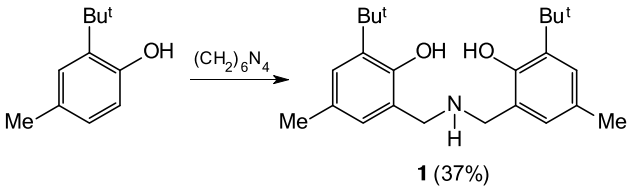
**Scheme 1**




**1** (37%)

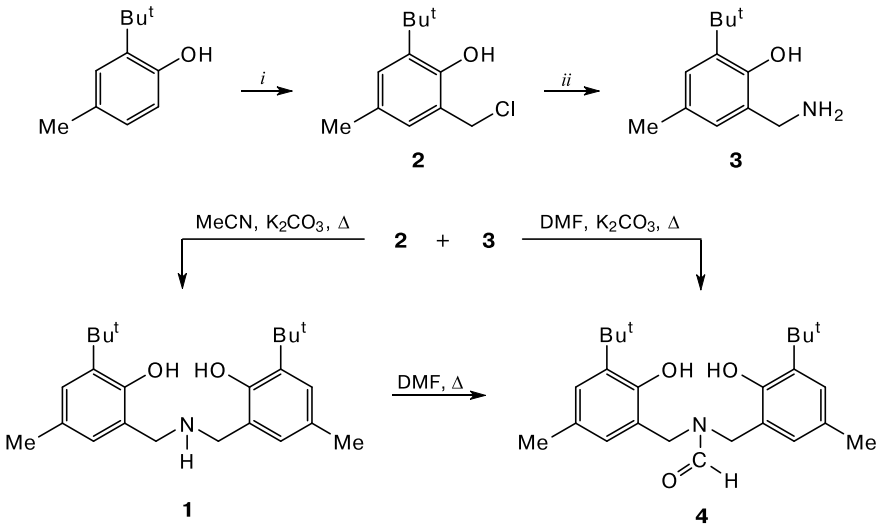
**Reagents and conditions:** 1) 85% HCOOH; 2) HCl—$HOCH_2$-$CH_2OH$; 3) KOH.

amine (**3**) would allow the synthesis of amine **1** in higher yield (Scheme 2). Compound **2** was prepared by the chloromethylation of 2-*tert*-butyl-4-methylphenol in toluene. Unfortunately, the amination of compound **2** with aqueous ammonia was not selective and gave a mixture of compound **3** and bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)amine (**1**). Hence, we synthesized 3-*tert*-butyl-2-hydroxy-5-methylbenzylamine **3** through a multistep process (see Scheme 2). The reaction of benzylamine **3** with benzyl chloride **2** in acetonitrile in the presence of potassium carbonate as a base produced compound **1** in 72% yield.

However, an attempt to improve the yield of compound **1** using another solvent led to an unexpected result. The heating of equimolar amounts of compounds **2** and **3** in DMF affords *N*,*N*-bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)formamide (**4**) (68% yield) as the formylation product of compound **1** (see Scheme 2). Actually, the heating of compound **1** with DMF in a sealed tube for 10 h at 150 °C resulted in the formylation of compound **1**, and formamide **4** was isolated in a similar yield (62%). The structures of all the synthesized compounds were confirmed by NMR spectroscopy; their composition, by elemental analysis.

We obtained crystals of compound **4** suitable for X-ray diffraction. The interatomic distances of formamide **4** are given in Fig. 1. In the crystal, formamide **4** occurs as a dimer formed through intermolecular C=O···H—O hydrogen bonds ($l$ = 1.990 Å) between the carbonyl oxygen atom of one molecule and the phenolic hydroxy group of another molecule (Fig. 2). This bond is the intermolecular component of the bifurcated hydrogen bond. Another component of this bond is the intramolecular C=O···H—O hydrogen bond ($l$ = 1.754 Å) between the carbonyl oxygen atom and the phenolic hydroxy group of the same molecule. The oxygen atoms that link two molecules of the dimer, O(1) and O(3), lie almost in one plane; the distance between these atoms is 2.705(3) Å. The intramolecular distance between the oxygen atoms, O(1)...O(2), is 2.643(4) Å, and the intramolecular O(1)...H—O(2) angle is 172.3°. Both the intra- and intermolecular contacts are smaller than the sum of the van der Waals radii of the corresponding atoms. The formamide molecule contains a trivalent nitrogen atom, which lies nearly within the plane formed by the carbon atoms and deviates from it only slightly (by 0.013 Å). The deviation of the nitrogen atom from the plane passing through three carbon atoms of *N*-(3,5-di-*tert*-butyl-2-hydroxyphenyl)-*N*-methylformamide[27] is even smaller, and it differs from the corresponding distance in the *N*,*N*-diformylformamide (triformamide) molecule $N[C(O)H]_3$, in which the nitrogen atom is within the CHO plane.[28] The interatomic N—C distances between the nitrogen atom and the carbonyl groups of the dimer are similar within the experimental error

**Scheme 2**




**Reagents and conditions:** *i*. 1) HCl—$PhCH_3$, 2) $(CH_2O)_n$; *ii*. 1) $Me_3SiCl$, $Et_3N$, $PhCH_3$; 2) $(Me_3Si)_2NLi$; 3) MeOH, Δ, 10 h.

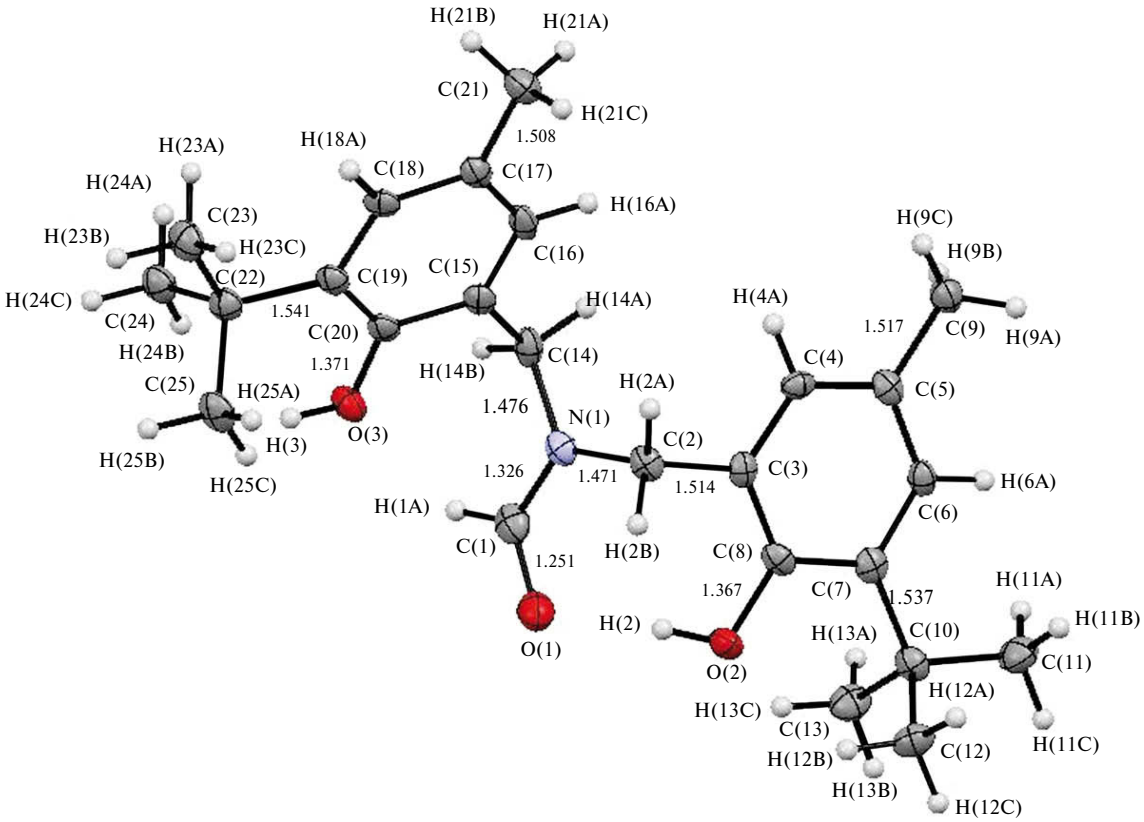



**Fig. 1.** Molecular structure of formamide **4**.

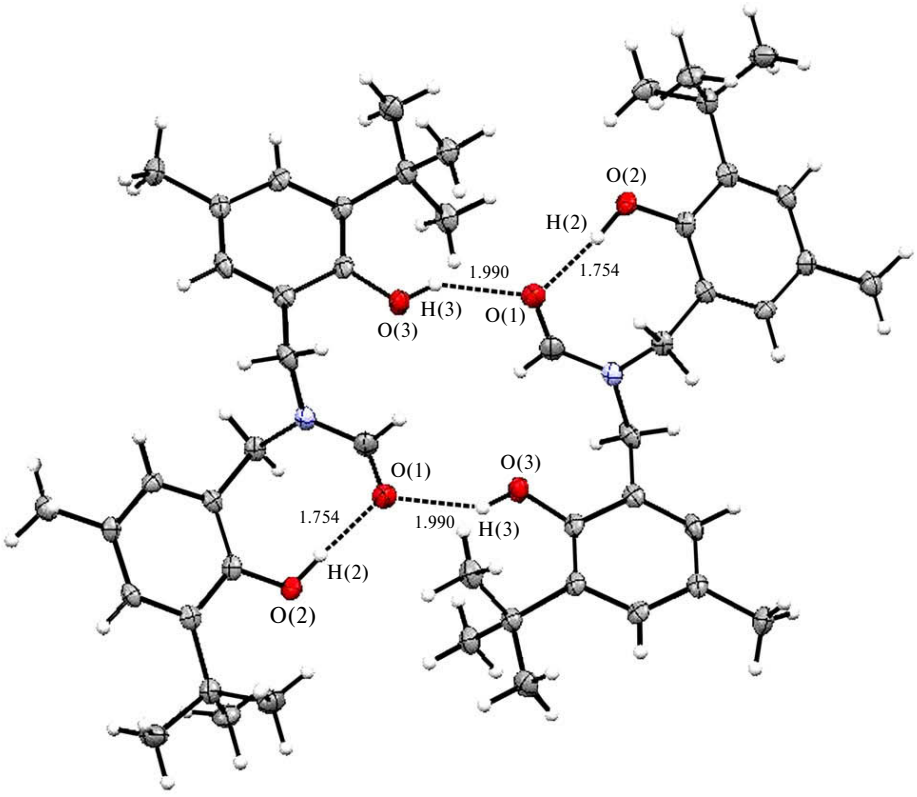



**Fig. 2.** Dimer of formamide **4**.

(1.471 and 1.476 Å; 2σ = 0.006—0.008 Å). The N—C=O and C=O bond lengths are 1.326 and 1.251 Å, respectively, and are in the range typical of the amide moiety of carboxamides. Thus, the N—C=O and C=O bond lengths of 3-phenylpropionic acid *N*,*N*-dibenzylamide are 1.350 and 1.244 Å, respectively.[29] The hydrogen bond in compound **4** leads to a decrease in the N—C=O bond length and an increase in the C=O bond length compared to the corresponding values in the compound $PhCH_2CH_2C(O)N(CH_2Ph)_2$.

The nitrogen atom in compound **4** is bound to two almost equivalent benzyl moieties and the formyl group HC=O. The benzene carbon atoms lie in one plane, the deviations of the carbon atoms from the plane being within 0.001—0.007 Å. The angle between the planes is 31.83°.

The dimers of the formamide are linked to form infinite polymer chains (Fig. 3) through interactions between the methylene groups of the $C(O)N—CH_2$ moiety and the π system of the aromatic ring of the adjacent molecule. The CH···π distance between the H atom and the centroid of the aromatic ring is 2.741 Å (Fig. 4). Such interactions are described in the literature (see, *e.g.*, the study[30]).

In summary, we synthesized new sterically hindered bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)amine (**1**) based on 2-*tert*-butyl-4-methylphenol. Compound **1** reacts with *N*,*N*-dimethylformamide on heating to form *N*,*N*-bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)formamide (**4**). According to the X-ray diffraction data, compound **4** occurs in the crystal as a dimer formed through an intermolecular component of the bifurcated C=O···H—O hydro-

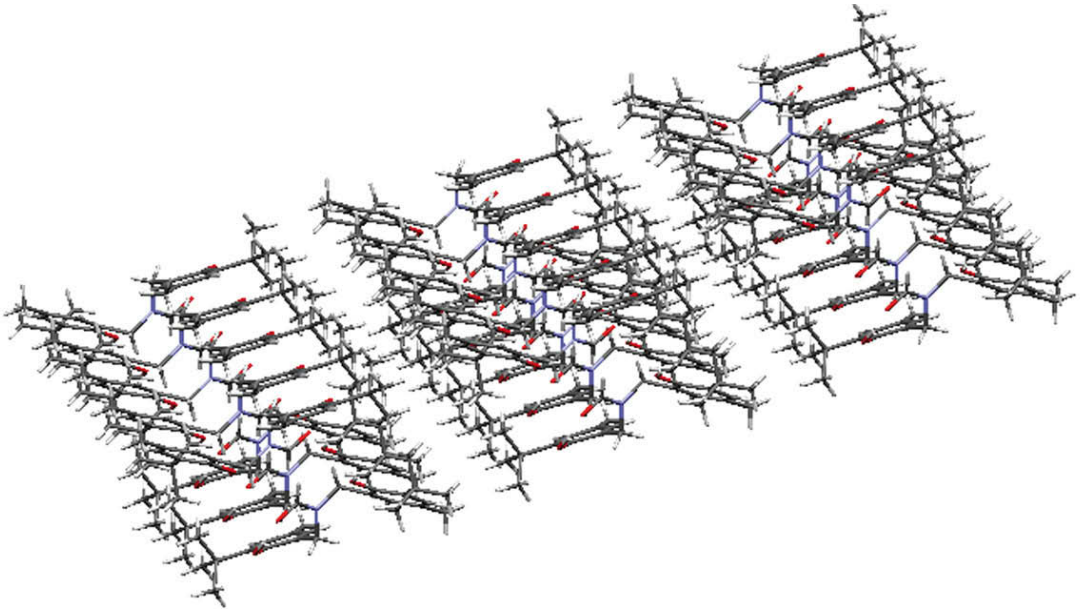


**Fig. 3**. Chains of dimers of compound **4**.

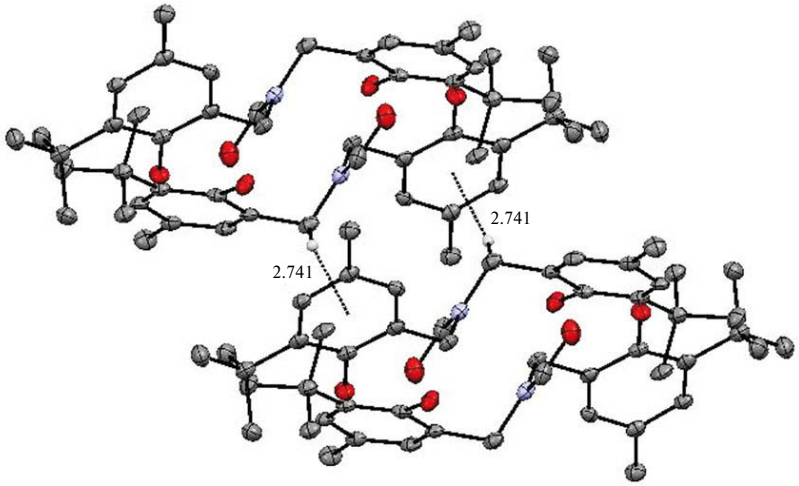



**Fig. 4.** The CH···π contacts in compound **4**.

gen bond between the carbonyl oxygen atom of one molecule and the phenolic hydroxy group of another molecule. The second component of the bifurcated hydrogen bond is the intramolecular C=O···H—O hydrogen bond between the carbonyl oxygen atom and the phenolic hydroxyl group of the same molecule. The dimers are linked by CH···π interactions (2.741 Å) to form long chains. Based on the literature data on the structures and properties of related compounds, it can be suggested that the new compounds are potential tridentate ligands.

## Experimental

The $^1$H and $^{13}$C NMR spectra were recorded on a Bruker DPX 400 spectrometer (400.13 and 100.61 MHz, respectively) in $CDCl_3$ with $SiMe_4$ as the internal standard. The solvents were prepared by standard procedures[31] and stored over 4 Å molecular sieves, which were activated for 6 h on heating (290±10 °C (1 Torr)).

**X-ray diffraction study** of the crystal and molecular structure of formamide **4** was performed on a Bruker automated diffractometer. The X-ray diffraction data collection statistics are given in Table 1. The refinement of the unit cell parameters and preliminary X-ray diffraction data processing were performed using the SHELX TL, version 5.1, program package. The structure was refined by the full-matrix least-squares method with anisotropic displacement parameters for nonhydrogen atoms. All hydrogen atoms were found in difference Fourier maps and refined using a riding model to $R = 0.063$. The CIF file containing the complete structural data was deposited with the Cambridge Crystallographic Data Centre (CCDC 2032153) and is available at www.ccdc.cam.ac.uk/data_request/cif.

**(3-*tert*-Butyl-2-hydroxy-5-methylbenzyl)amine (1). Method *A*.** A mixture of 2-*tert*-butyl-4-methylphenol (1.64 g, 10 mmol) and hexamethylenetetramine (2.8 g, 20 mmol) in 85% formic acid (100 mL) was refluxed for 2 h, resulting in the formation of two layers. The lower layer (yellowish oil) was separated by decantation and evacuated (~3 Torr) at room temperature for 3 h. The residue was dissolved in ethylene glycol (100 mL), and concentrated hydrochloric acid (40 mL) was added. The mixture was warmed in an oil bath (130±5 °C) for 12 h and then cooled. The precipitate was filtered off and washed with distilled water (2×10 mL). The resulting hydrochloride of compound **1** was added portionwise with vigorous stirring to a mixture of a KOH solution (3.5 g in 30 mL of water) and diethyl ether (50 mL). After 30 min, the ethereal layer was separated and dried with $MgSO_4$. The solvent was removed using a rotary evaporator, and the residue was evacuated (~2 Torr) at room temperature for 4 h. Yellowish viscous oil was obtained in a yield of 1.37 g (37%); attempts to crystallize this compound failed.

**Method *B*.** Equimolar amounts of 3-*tert*-butyl-2-hydroxy-5-methylbenzyl chloride (**2**) (0.43 g, 2 mmol) and 3-*tert*-butyl-2-hydroxy-5-methylbenzylamine (**3**) (0.39 g, 2 mmol) were dissolved in MeCN, and $K_2CO_3$ (1 g, 7.2 mol, excess) was added. The reaction mixture was refluxed with vigorous stirring for 30 h and then poured to cold water (10 mL). Compound **1** was extracted with diethyl ether (3×5 mL). The solvent was removed using a rotary evaporator, and the residue was evacuated (~1 Torr) at room temperature for 6 h. Yellowish viscous oil was obtained in a yield of 0.53 g (72%).

**Table 1.** X-ray diffraction data collection and structure refinement statistics for compound **4**

| Parameter | Value |
|---|---|
| Molecular formula | $C_{25}H_{35}NO_3$ |
| $M$ | 397.54 |
| $T$/K | 100 |
| Crystal system | Triclinic |
| Space group | $P\bar{1}$ |
| Unit cell parameters | |
| $a$/Å | 9.030(2) |
| $b$/Å | 10.181(3) |
| $c$/Å | 14.160(4) |
| α/deg | 92.284(6) |
| β/deg | 103.511(5) |
| γ/deg | 15.707(5) |
| $V$/Å$^3$ | 1125.5 (1) |
| $Z$ | 2 |
| $d_{calc}$/g cm$^{-3}$ | 1.173 |
| μ/mm$^{-1}$ | 0.076 |
| Crystal size/mm | 0.10×0.08×0.03 |
| $\theta_{min}$—$\theta_{max}$/deg | 2.25—26.02 |
| $T_{min}/T_{max}$ | 0.9925/0.9977 |
| $F(000)$ | 432 |
| λ/Å | 0.71073 |
| $2\theta_{max}$/deg | 52 |
| Ranges of indices | $-11 \leqslant h \leqslant 11$, $-12 \leqslant k \leqslant 10$, $-17 \leqslant l \leqslant 17$ |
| Number of reflections | |
| measured | 8374 |
| unique | 4353 |
| with $I > 2\sigma(I)$ | 2365 |
| ($R_{int}$) | (0.0617) |
| Number of refined parameters | 272 |
| GOOF | 0.948 |
| $R$ factors based on $F^2 > 2\sigma(F^2)$ | |
| $R_1$ | 0.0633 |
| $wR_2$ | 0.1278 |
| $R$ factors based on all reflections | |
| $R_1$ | 0.1318 |
| $wR_2$ | 0.1533 |
| Residual electron density ($\Delta\rho_{min}/\Delta\rho_{max}$)/e Å$^{-3}$ | −0.236/0.300 |

$^1$H NMR ($CDCl_3$), δ: 1.36 (s, 18 H, 2 Bu$^t$); 2.22 (s, 6 H, 2 Me); 3.56 (s, 4 H, 2 $CH_2$); 5.64 (br.s, 1 H, NH); 6.72 (s, 2 H, 2 $CH_{Ar}$); 6.96 (s, 2 H, 2 $CH_{Ar}$); 8.54 (br.s, 2 H, 2 OH). $^{13}$C NMR ($CDCl_3$), δ: 24.8 (Me); 30.7 ($\underline{Me}_3C$); 36.2 ($\underline{C}Me_3$); 52.4 ($CH_2$); 122.6, 123.8, 124.8, 137.4, 142.3, 151.8 (Ar). Found (%): C, 77.82; H, 9.39; N, 3.86. $C_{24}H_{35}NO_2$. Calculated (%): C, 78.00; H, 9.55; N, 3.79.

**3-*tert*-Butyl-2-hydroxy-5-methylbenzyl chloride (2).** Hydrogen chloride was bubbled through a solution of 2-*tert*-butyl-4-methylphenol (3.28 g, 20 mmol) in toluene (25 mL), cooled to −5±2 °C, for 15 min. Then paraformaldehyde (0.75 g, 25 mmol, excess) was added with stirring. The reaction mixture was gradually warmed to room temperature, and hydrogen chloride

was bubbled for two hours. Water (10 mL) was added to the reaction mixture. The organic layer was separated, washed with water (3×5 mL), and dried with $K_2CO_3$. The solvent was removed using a rotary evaporator, and the residue was evacuated (~3 Torr) at room temperature for 6 h. Yellowish oil was obtained in a yield of 3.78 g (89%) and was used without additional purification. $^1$H NMR ($CDCl_3$), δ: 1.37 (s, 9 H, $Bu^t$); 2.26 (s, 3 H, Me); 4.62 (s, 2 H, $CH_2$); 5.43 (br.s, 1 H, OH); 6.92 (s, 1 H, $CH_{Ar}$); 7.14 (s, 1 H, $CH_{Ar}$). $^{13}$C NMR ($CDCl_3$), δ: 20.6 (Me); 31.4 ($\underline{Me}_3C$); 35.8 ($\underline{C}Me_3$); 42.7 ($CH_2$); 123.9, 124.6, 128.3, 138.7, 143.5, 152.4 (Ar). Found (%): C, 67.93; H, 8.25. $C_{12}H_{17}ClO$. Calculated (%): C, 67.76; H, 8.06.

**3-*tert*-Butyl-2-hydroxy-5-methylbenzylamine (3).** A solution of $Me_3SiCl$ (1.08 g, 10 mmol) in toluene (10 mL) was slowly added dropwise with vigorous stirring to a solution of compound **2** (2.13 g, 10 mmol) and triethylamine (1.50 g, 15 mmol, excess) in anhydrous toluene (30 mL) under an argon atmosphere. The reaction mixture was stirred at room temperature for 30 min, refluxed for 1 h, cooled, and filtered off (back filtration). The precipitate was washed with toluene (2×5 mL), and a solution of $(Me_3Si)_2NLi$ (2 g, 12 mmol) in toluene was slowly added dropwise to the combined filtrate (−78 °C). The reaction mixture was stirred at this temperature for 1 h. Then the mixture was slowly warmed to room temperature and kept for 16 h. To remove the LiCl precipitate, the solution was filtered through a ~1-cm thick Celite 521 layer. Methanol (10 mL) was added to the filtrate, and the reaction mixture was refluxed for 8 h. Volatile substances were removed using a rotary evaporator, and the residue was evacuated (~1 Torr) at room temperature for 6 h. Viscous oil was obtained in a yield of 1.58 g (82%) and was used without additional purification. $^1$H NMR ($CDCl_3$), δ: 1.38 (s, 9 H, $Bu^t$); 2.24 (s, 3 H, Me); 4.29 (s, 2 H, $CH_2$); 5.84 (br.s, 2 H, $NH_2$); 6.94 (s, 1 H, $CH_{Ar}$); 7.02 (s, 1 H, $CH_{Ar}$); 8.12 (br.s, 1 H, OH). $^{13}$C NMR ($CDCl_3$), δ: 20.8 (Me); 31.7 ($\underline{Me}_3C$); 35.9 ($\underline{C}Me_3$); 44.3 ($CH_2$); 127.3, 127.9, 129.5, 138.8, 142.2, 153.2 (Ar). Found (%): C, 74.68; H, 10.06; N, 7.19. $C_{12}H_{19}NO$. Calculated (%): C, 74.57; H, 9.91; N, 7.25.

***N*,*N*-Bis(3-*tert*-butyl-2-hydroxy-5-methylbenzyl)formamide (4).** Compound **1** (0.74 g, 2 mmol) and DMF (1 mL) were added with a syringe to a tube pre-filled with argon. The tube was degassed, evacuated, and sealed. The sealed tube was placed in a protective jacket and heated at 150 °C for 10 h. Then the tube was cooled and opened. The content was poured to a mixture of water (5 mL) and *o*-xylene (10 mL). The organic layer was separated and dried over 4 Å molecular sieves. The solvent was removed using a rotary evaporator, and the residue was crystallized from ethanol. Compound **4** was obtained in a yield of 0.49 g (62%). $^1$H NMR ($CDCl_3$), δ: 1.38 (s, 9 H, $Bu^t$); 1.40 (s, 9 H, $Bu^t$); 2.21 (s, 3 H, Me); 2.31 (s, 3 H, Me); 4.26 (s, 2 H, $CH_2$); 4.42 (s, 2 H, $CH_2$); 5.18 (br.s, 1 H, OH); 6.66 (s, 1 H, $CH_{Ar}$); 6.89 (s, 1 H, $CH_{Ar}$); 7.03 (s, 1 H, $CH_{Ar}$); 7.08 (s, 1 H, $CH_{Ar}$); 8.22 (s, 1 H, HC=O); 8.97 (s, 1 H, OH). $^{13}$C NMR ($CDCl_3$), δ: 20.72, 20.87 (Me); 29.62, 30.31 ($\underline{Me}_3C$); 33.80, 34.89 ($\underline{C}Me_3$); 42.94, 47.78 ($CH_2$); 122.14, 122.57, 127.45, 127.93, 128.14, 128.96, 129.65, 129.75, 135.85, 137.99, 151.18, 152.51 (Ar); 165.04 (C=O). Found (%): C, 75.69; H, 8.92; N, 3.44. $C_{25}H_{35}NO_3$. Calculated (%): C, 75.53; H, 8.87; N, 3.52.

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This paper does not contain descriptions of studies on animals or humans.

The authors declare no competing interests.

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