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Steric effects on forming different by-products in the formylation reaction of 2,4-dialkylphenol (dialkyl = *t*-Bu/*t*-Bu, *t*-Bu/Me and Me/Me) proved by their structural and spectral characterizations

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Abstract

In the formylation reaction of 2,4-dialkylphenol (2,4-di-*tert*-butylphenol, 2-*tert*-butyl-4-methylphenol and 2,4-dimethylphenol) in the presence of hexamethylenetetramine, steric effects of alkyl groups play important roles in forming different types of by-products, namely 2,4-di-*tert*-butyl-6-[(6,8-di-*tert*-butyl-2*H*-1,3-benzoxazin-3(4*H*)-yl)methyl]phenol (**1**), 2-*tert*-butyl-4-methyl-6-[(6-*tert*-butyl-8-methyl-2*H*-1,3-benzoxazin-3(4*H*)-yl)methyl]phenol (**2**) and tris(2-hydroxy-3,5-dimethylbenzyl)amine hydrochlorate (**3**). These three compounds are fully characterized and single-crystal structures of **1** and **3** are further elucidated.

Keywords: Formylation reaction; Steric effects; Crystal structures; Hydrogen bonding interactions

1. Introduction

Nitrogen-containing benzoheterocycles and their derivatives are important intermediates for organic synthesis and useful candidates for the studies on biochemistry and medicinal chemistry [1–3]. Tris(2-hydroxy-3,5-dialkyl)amines [4] are typical tripodal ligands and there have been many related reports in the literature. For instance, Holmes et al. reported the structures and dynamic NMR behavior of new classes of silatranes and phosphatranes derived from the tripodal ligand tris(2-hydroxy-3,5-dialkyl)amine [5]. Gallium(III) and Indium(III) complexes of tris(2-mercaptobenzyl)amine and tris(2-hydroxybenzyl)amine [6] and dimethoxy-(tris(2-oxy-3,5-dimethylbenzyl)amine) tantalum(IV) salts [7] have also been documented. With regard to tris(2-hydroxy-3,5-dimethylbenzyl)amine [8], its phenylphosphinate [9] and 2,2′-thiobis(4-methyl-6-*tert*-butylphenyl)phosphonate [10] as well as its Fe(III) complex have been structurally reported previously [11].

In addition to the conventional preparation methods, our experimental results show that 2,4-di-*tert*-butyl-6-[(6,8-di-*tert*-butyl-2*H*-1,3-benzoxazin-3(4*H*)-yl)methyl]phenol (**1**), 2-*tert*-butyl-4-methyl-6-[(6-*tert*-butyl-8-methyl-2*H*-1,3-benzoxazin-3(4*H*)-yl)methyl]phenol (**2**) and tris(2-hydroxy-3,5-dimethylbenzyl)amine hydrochlorate (**3**) can also be yielded as the main by-products in the process of Duff reaction [12] of 2,4-dialkylphenol and hexamethylenetetramine (HMT). However, different by-products can be obtained in different yields due to the use of different dialkyl ring substituents (dialkyl = *t*-Bu/*t*-Bu, *t*-Bu/Me and Me/Me), as can be seen in Scheme 1. In addition, we present herein the single-crystal structures of **1** and **3**.

2. Experimental

2.1. Materials and measurements

Melting points were measured without correction in this work. 2,4-Di-*tert*-butylphenol, 2-*tert*-butyl-4-methylphenol

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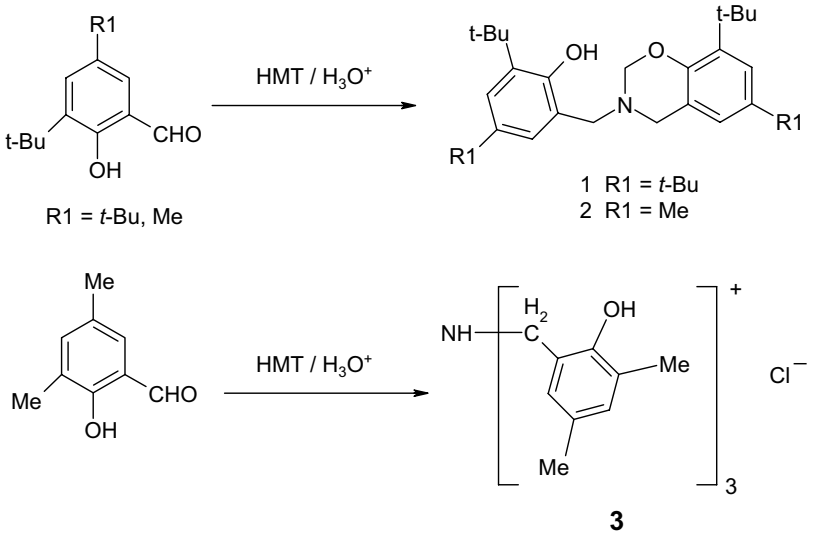


Scheme 1. Synthetic routes for compounds **1**–**3**.

and 2,4-dimethylphenol were purchased as commercial chemicals from Aldrich. All other solvents and reagents were of analytical grade and used without further purification. Analyses for carbon, hydrogen and nitrogen were performed on a Perkin-Elmer 1400C analyzer. Infrared spectra (4000–400 cm^{-1}) were recorded with a Nicolet FT-IR 170X spectrophotometer on KBr disks. ^1H NMR spectra were obtained in a Bruker 500 MHz NMR spectrometer. Electrospray ionization mass spectra (ESI-MS) were recorded on a Finnigan MAT SSQ 710 mass spectrometer in a scan range of 100–1200 amu. The samples were determined by injecting the diluted methanol solutions using 1:1 methanol/water solvent as the mobile phase. The spray voltage and capillary temperature were set at 4.5 kV and 200 °C, respectively.

2.2. Synthesis of 2,4-di-tert-butyl-6-[(6,8-di-tert-butyl-2H-1,3-benzoxazin-3(4H)-yl)methyl]phenol (*1*)

The preparation method of **1** is the same as that of 3,5-di-*tert*-2-hydroxybenzaldehyde reported by Larrow et al. [13]. The crude product (mainly a mixture of 3,5-di-*tert*-2-hydroxybenzaldehyde and **1**) was suspended in a warm (55 °C) methanol solution and a white powder was separated by vacuum filtration which afforded compound **1** in a yield of ~35%. 3,5-Di-*tert*-butyl-2-hydroxybenzaldehyde could be separated from the filtrate by cooling to 0 °C and subsequent sucking in a yield of ~45%. Mp 167–168 °C. Anal. Calcd for $C_{31}H_{47}NO_2$: C, 79.95; H, 10.17; N, 3.01%. Found: C, 79.79; H, 9.96; N, 2.82%. IR (KBr disk, cm^{-1}): 3300 (OH), 2960, 2904, 2869 and 1390 (*t*-Bu), 1231 (C—O). ^1H NMR (500 MHz, $CDCl_3$, 298 K, TMS): δ 1.28 (s, 9H, *t*-Bu), 1.32 (s, 9H, *t*-Bu), 1.36 (s, 9H, *t*-Bu), 1.57 (s, 9H, *t*-Bu), 4.10 (s, 4H, N—CH_2—Ar), 4.87 (s, 2H, Ar—O—CH_2—N), 6.84 (s, 1H, Ar—OH), 7.23–7.32 (m, 4H, Ar—H). ESI-MS: *m*/*z* 466.3 $[C_{31}H_{47}NO_2+H]^+$ (100%). Single crystals suitable for X-ray analysis were grown from ethanol by slow evaporation at room temperature in air.

2.3. Synthesis of 2-tert-butyl-4-methyl-6-[(6-tert-butyl-8-methyl-2H-1,3-benzoxazin-3(4H)-yl)methyl]phenol (*2*)

The synthesis of 3-*tert*-butyl-5-methyl-2-hydroxybenzaldehyde is the same as 3,5-di-*tert*-2-hydroxybenzaldehyde in a yield of ~35% except that CH_3COOH was used to replace CF_3COOH. Compound **2** is not isolated because of the synthetic difficulties (<10% yield) but an ESI-MS peak at 382.1 corresponding to the cation of $[C_{25}H_{35}NO_2+H]^+$ (100%) verifies the presence of **2** in the residue.

2.4. Synthesis of tris(2-hydroxy-3,5-dimethylbenzyl)amine hydrochlorate (*3*)

The synthesis of 3,5-dimethyl-2-hydroxybenzaldehyde is the same as above-mentioned two aldehydes. Hydrochlorate **3** was obtained as precipitate from the water/ether mixture. The precipitate was collected and recrystallized by ethanol/water in a yield of ~40%. Mp 210–211 °C. Anal. Calcd for $C_{27}H_{34}ClNO_3$: C, 71.11; H, 7.51; N, 3.07%. Found: C, 71.03; H, 7.38; N, 2.97%. IR (KBr disk, cm^{-1}): 3241 (N—H), 3111, 3011, 2970, 2916, 2862, 1492, 1443, 1422, 1387 (CH_3), 1249, 1195 (C—O). ^1H NMR (500 MHz, $CDCl_3$, 298 K, TMS): δ 2.24 (s, 18H, CH_3), 4.08 (s, 6H, CH_2), 6.79 (s, 3H, Ar—OH), 7.03 (s, 3H, Ar—H), 7.29 (s, 3H, Ar—H), 9.28 (s, 1H, N—H). ESI-MS: *m*/*z* 420.2 $[C_{27}H_{34}ClNO_3-Cl]^+$ (100%). Single crystals suitable for X-ray analysis were grown from a mixture of ethanol and water in a 2:1 (v/v) ratio by slow evaporation at room temperature in air.

2.5. X-ray data collection and solution

Single-crystal samples of **1** and **3** were glue-covered and mounted on glass fibers and then used for data collection using graphite mono-chromated Mo Kα radiation ($\lambda = 0.71073$ Å). Crystallographic data of **1** were collected at 291(2) K on a Bruker SMART 1 K CCD diffractometer, while those of **3** were collected at 100(2) K on a Rigaku Mercury CCD area-detector, using graphite mono-chromated Mo Kα radiation ($\lambda = 0.71073$ Å). In the case of Rigaku system, the original data files generated by CRYSTALCLEAR [14] were transformed to SHELXTL97 format by TEXSAN program [15].

The crystal systems of **1** and **3** were determined by laue symmetry and the space groups were assigned on the basis of systematic absences using XPREP. Absorption corrections were performed to all data and the structures were solved by direct methods and refined by full-matrix least-squares method on F^2_{obs} by using the SHELXTL-PC software package [16]. All non-H atoms were anisotropically refined and all hydrogen atoms were inserted in the calculated positions assigned fixed isotropic thermal parameters and allowed to ride on their respective parent atoms. As for the crystal data of **3**, it was difficult to grow good single crystals especially for smaller ones (samples in large size and high R_{int} values were found) and the room temperature data were not good enough although we have tried many times. That is why we collected the low temperature data for **3** at 100 K. Only several "C alerts" were reported at this time when checked by Platon, which indicated that the structure mode of **3** was reliable. The summary of the crystal data, experimental details and refinement results for **1** and **3** is listed in Table 1.

Table 1
Summary of crystal data, experimental details and refinement results for **1** and **3**

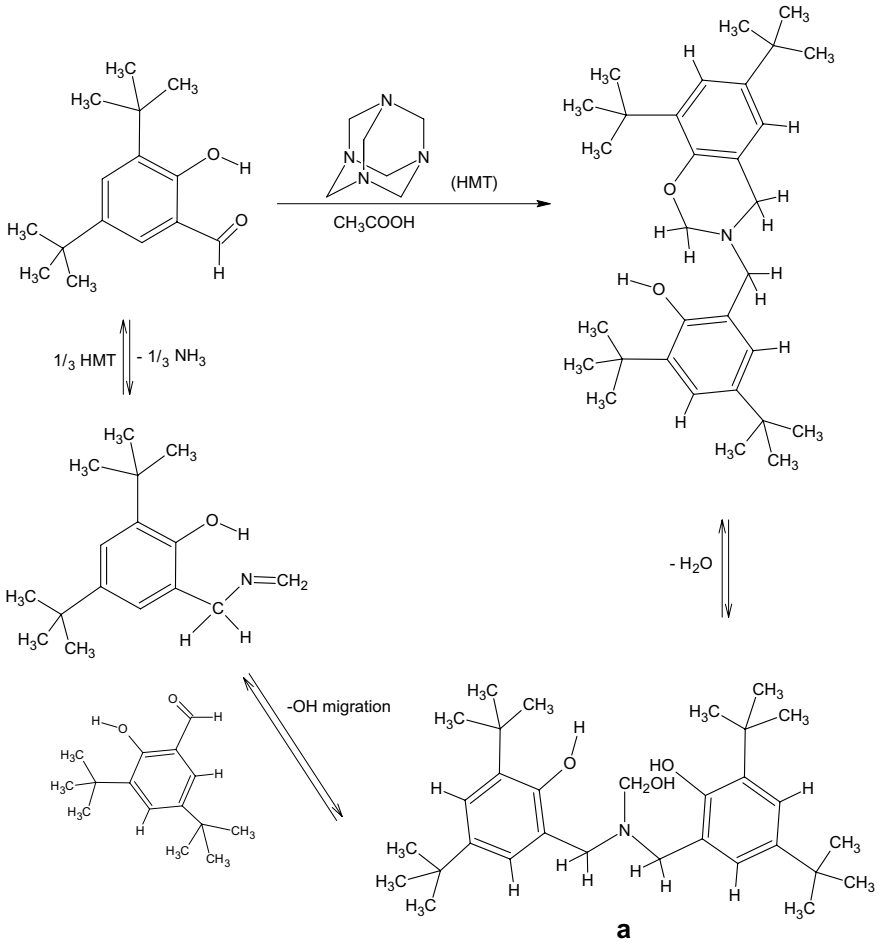
Compound	**1**	**3**
Empirical formula	$C_{31}H_{47}NO_2$	$C_{27}H_{34}NClO_3$
Formula weight	465.70	456.00
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/c$	$Fdd2$
Unit cell dimensions	11.0245(13) Å	25.878(5) Å
	18.770(2) Å	46.488(9) Å
	28.840(4) Å	8.485(2) Å
	$\beta = 97.247(3)°$	$\beta = 90°$
V/Å^3	5920.3(12)	10208(4)
Z	8	16
D/g cm^{-3}	1.045	1.187
Absorption coefficient/mm^{-1}	0.064	0.177
Crystal size [mm]	0.20 × 0.30 × 0.40	0.20 × 0.30 × 0.40
Temperature [K]	291(2)	100(2)
θ Range for data collection	1.79–26.00	3.06–25.00
Index ranges	$-13 \leqslant h \leqslant 8$	$-30 \leqslant h \leqslant 30$
	$-22 \leqslant k \leqslant 23$	$-49 \leqslant k \leqslant 54$
	$-35 \leqslant l \leqslant 34$	$-10 \leqslant l \leqslant 8$
No. of reflections collected	31,324	21,282
No. of independent reflections/ parameters	11,639/637	4334/298
Absorption correction	Multi-scan	Multi-scan
Observed data [$I > 2\sigma(I)$]	8041	4076
Goodness-of-fit on F^2	1.187	1.160
F (000)	2048	3904
Flack parameter	–	0.01(11)
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0553$	$R_1 = 0.0748$
	$wR_2 = 0.1452$	$wR_2 = 0.1774$
R indices (all data)	$R_1 = 0.0812$	$R_1 = 0.0793$
	$wR_1 = 0.1503$	$wR_1 = 0.1799$
Largest diff. peak and hole (e Å^{-3})	0.152/−0.149	0.383/−0.300

$R_1 = \Sigma||Fo| - |Fc||/\Sigma|Fo|$, $wR_2 = [\Sigma[w(Fo^2 - Fc^2)^2]/\Sigma w(Fo^2)^2]^{1/2}$.

3. Results and discussion

3.1. Synthesis and spectral characterizations

The structures of **1** and **3** are confirmed by the combination of the results of EA (ratio of C, H, N), FT-IR, ^{1}H NMR and ESI-MS (M+1 peak). In our experiments, we applied Larrow's method [13] to synthesize 3,5-dialkyl-2-hydroxybenzaldehydes (dialkyl = *t*-Bu/*t*-Bu, *t*-Bu/Me and Me/Me), and it is found that the resulting 3,5-dialkyl-2-hydroxybenzaldehydes can further react with HMT to different extent. In the case of starting material 2,4-di-*tert*-butylphenol, 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde is the main product whose structure has been reported previously in our group [17]. However, some of produced 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde molecules can further react with HMT under our experimental condition. The proposed mechanism for the formation of compound **1** is shown in Scheme 2. HMT molecule can be decomposed into CH_3—NH_2 first by losing NH_3 molecule under the acidic condition, and then CH_3—NH_2 is added to the C=O group of 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde. Finally, one water molecule is removed by β-elimination and the Ph—CH_2—N=CH_2 intermediate (isomerising to Ph—CH=N—CH_3) is formed. At the next reversible step, one aldehyde group from another 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde molecule is added to the imine nitrogen atom forming intermediate **a**, and finally the six-membered ring contraction occurs and *N*,*O*-benzoheterocycle **1** is formed. Because of the strong spatial crowding effects of two bulky *tert*-butyl groups, the reaction stops here and **1** is the main by-product in a yield of ~35%. By contrast, for the starting materials of 2-*tert*-butyl-4-methylphenol, the main by-product is also the six-membered *N*,*O*-benzoheterocycle **2**. But the yield is very low (<15%) and it is hard to isolate from the mixture. As for 2,4-dimethylphenol, the yielded 3,5-dimethyl-2-hydroxybenzaldehyde can form intermediate **a** first, nevertheless it can further react with another 2,4-dimethylphenol molecule to form **3** in a yield of ~40%, overwhelming the formation of *N*,*O*-benzoheterocycle. On considering the ESI-MS peak corresponding to 1,3-benzoxazine motif is not observed in the case of compound **3**, which is due to the low steric hindrance of two phenyl-substituted methyl groups. One can conclude that the steric effects are vital in the formylation reaction of 2,4-dialkylphenol in forming different by-products.



Scheme 2. Possible mechanism for the formation of compound **1**.

In our experiments, it is no doubt that the nitrogen percentage in EA results comes from the HMT molecule. More importantly, it is deduced that the most intense peaks in the ES-MS spectra of **1**, **2** and **3** demonstrate different by-products. The isotopic distributions of **1** and **2** are calculated by using the numbers and natural isotopic abundance of atoms in $[C_{31}H_{47}NO_2+H]^+$ for **1** and $[C_{25}H_{35}NO_2+H]^+$ for **2** [18]. The fitting results are analogous to the experimental ones, further confirming the existence of the expected species (Fig. 1). Moreover, single-crystal structures of **1** and **3** are performed by using X-ray diffraction method, which will be discussed below.

3.2. Crystal structure of (*1*)

The atom-numbering scheme of compound **1** is shown in Fig. 2 where two crystallographically independent molecules are found per asymmetric unit. All the phenolic rings are essentially coplanar and the dihedral angles of two phenol rings in each molecule are a little bit different, which are 63.2(2)° (between ring C1 to C6 and ring C18 to C23 with the mean deviation from plane as 0.0021 and 0.0019 Å) and 55.1(2)° (between ring C1A to C6A and ring C18A to C23A with the mean deviation from plane as 0.0013 and 0.0011 Å), respectively. In addition, two phenol rings in the same molecule are nearly parallel to their respective counterparts with the dihedral angles of 19.3(2)° (between ring C1 to C6 and ring C1A to C6A) and 9.2(2)° (between ring C18 to C23 and ring C18A to C23A). In each of the independent molecule, the six-membered *N*,*O*-benzoheterocycle is not planar, and the related torsion angles are slightly different from each other. They are N1—C16—O1—C1, 48.3(2)°; C16—O1—C1—C2, 164.8(3)°; O1—C1—C2—C15, 176.5(3)°; C1—C2—C15—N1, 165.7(3); C2—C15—N1—C16, 46.2(3)°; C15—N1—C16—O1, 114.0(3)°; and N1A—C16A—O1A—C1A, 121.1(2)°; C16A—O1A—C1A—C2A, 23.2(3)°; O1A—C1A—C2A—C15A, 175.0(3)°; C1A—C2A—C15A—N1A, 17.3(3)°; C2A—C15A—N1A—C16A, 135.5(3)°; C15A—N1A—C16A—O1A, 72.1(3)°, respectively.

The structure of **1** is further stabilized by intramolecular hydrogen bonds. Intramolecular O—H...N hydrogen bonding interactions are observed between the phenol oxygen atoms (O2 and O2A) and the nitrogen atoms (N1 and N1A) (Table 2), forming fused six-membered hydrogen-bond rings. However, there are no π–π stacking interactions between adjacent molecules in its crystal packing, mainly due to the steric repulsions of the *tert*-butyl groups.

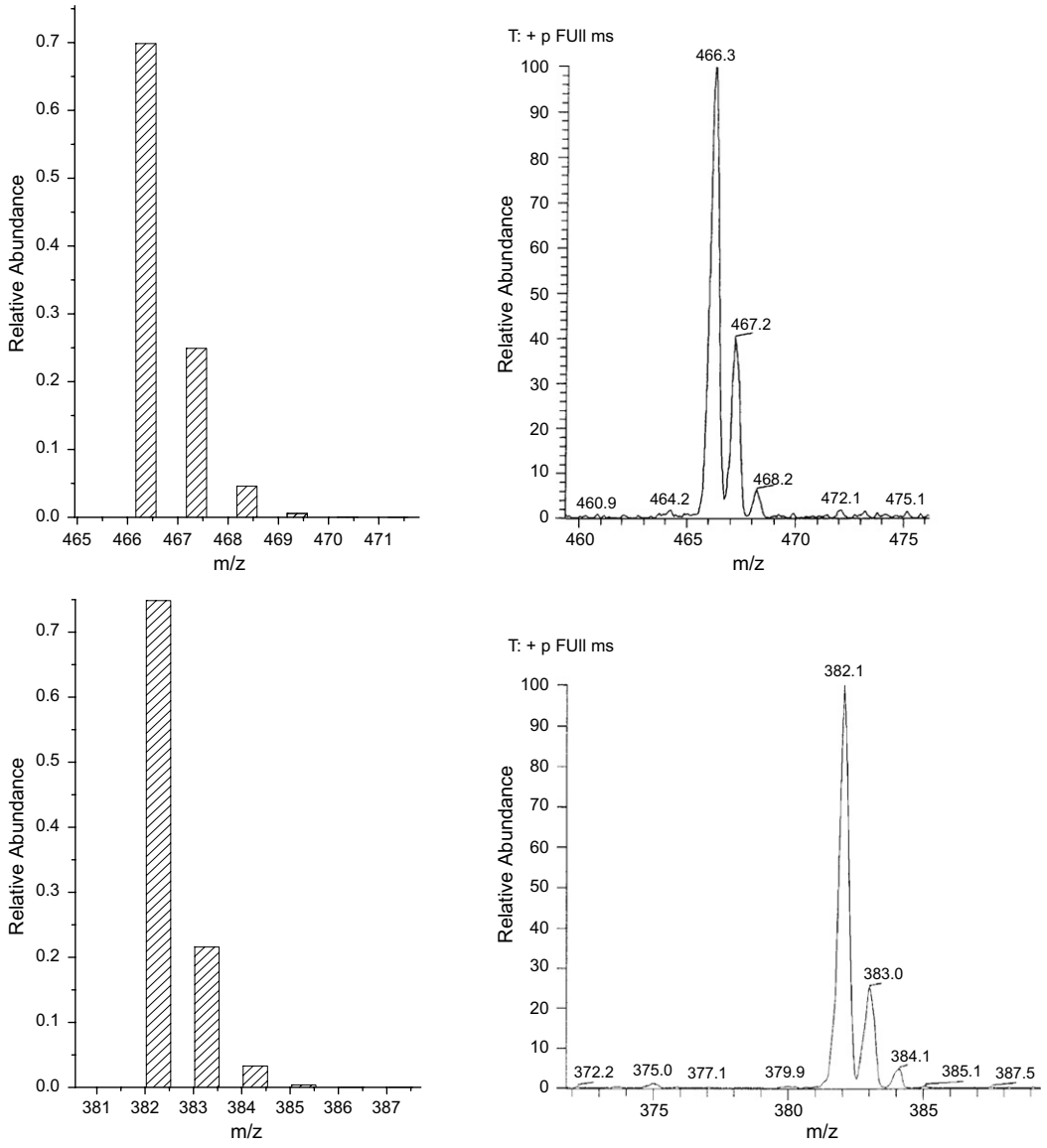


Fig. 1. The ESI-MS spectra of compounds **1** and **2** obtained in positive mode around *m*/*z* 466.3 $[C_{31}H_{47}NO_2+H]^+$ and *m*/*z* 382.1 $[C_{25}H_{35}NO_2+H]^+$, together with the corresponding calculated IDPs for comparison.

*3.3. Crystal structure of (**3**)*

As illustrated in Fig. 3, the X-ray determination results of **3** reveal that tris(2-hydroxy-3,5-dimethylbenzyl)amine is in its protonated form, which is countered by a chloride anion. The chloride anion in **3** is believed to play a key role in packing the orthorhombic non-centrosymmetric space group *Fdd2* which can be verified by a low Flack parameter of 0.01(11), since neutral tris(2-hydroxy-3,5-dimethylbenzyl)amine crystallizes in the monoclinic centrosymmetric space group $P2_1/c$ by contrast. All the phenolic rings in **3** are approximately planar and the bond lengths and angles fall within the normal ranges [9–10]. The dihedral angles between the three phenyl rings are 56.5(3)° (between ring C1 to C6 with the mean deviation from plane of 0.0143 Å and ring C10 to C15 with the mean deviation from plane of 0.0044 Å), 108.9(3)° (between ring C1 to C6 and ring C19 to C24 with the mean deviation from plane of 0.0065 Å) and 55.0(3)° (between ring C10 to C15 and ring C19 to C24), respectively.

Hydrogen bonding interactions are the most important feature in **3**. The chloride anion links three phenolic oxygen atoms of the same molecule by O—H…Cl hydrogen contacts with similar O…Cl separations of 2.35, 2.37 and 2.39 Å and O—H…Cl angles of 139.0, 146.0 and 142.0°, respectively. In addition, intramolecular N—H…O type of hydrogen contacts is found between the protonated tertiary nitrogen atom and three phenolic oxygen atoms with N…O separations of 2.16, 2.24 and 2.46 Å. However, no O—H…O H-bond is observed in the crystal packing of **3**, which is totally different from neutral tris(2-hydroxy-3,5-dimethylbenzyl)amine where one of three OH groups directs outward from the tripodal amino group and links another neighboring oxygen atom by O—H…O hydrogen

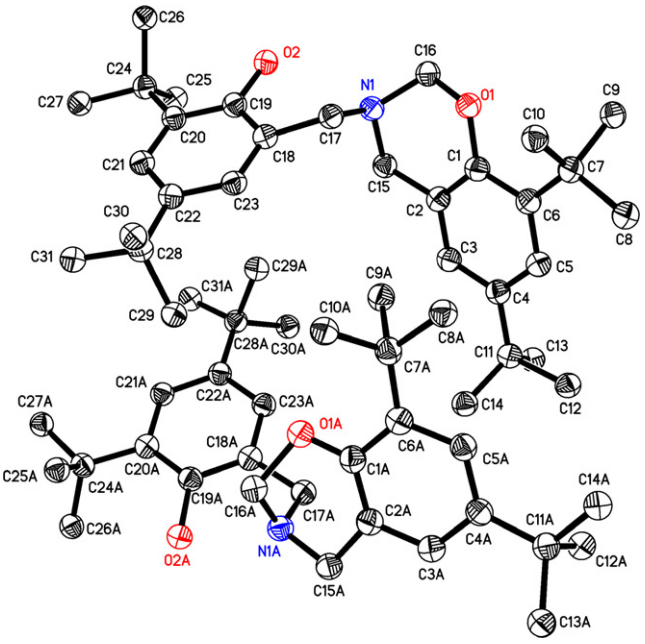


Fig. 2. The molecular structure of the asymmetric unit of **1**, showing 30% thermal probability displacement ellipsoids and the atom-numbering scheme. Hydrogen atoms are omitted for clarity.

Table 2
Hydrogen bonding interactions (Å, °) in compounds **1** and **3**

D—H...A	D—H	H...A	D—A	∠DHA	Symmetry code
1					
O2—H2A...N1	0.96	2.10	2.705(2)	120.0	
O2A—H2B...N1A	0.96	1.81	2.552(2)	132.0	
3					
O1—H1...Cl1	0.82	2.35	3.019(4)	139.0	
O2—H2...Cl1	0.82	2.39	3.071(3)	142.0	
O3—H3...Cl1	0.82	2.37	3.089(4)	146.0	
N1—H1A...O1	0.90	2.16	2.816(4)	129.0	
N1—H1A...O2	0.90	2.24	2.864(4)	127.0	
N1—H1A...O3	0.90	2.46	3.035(4)	123.0	
C16—H16B...Cl1	0.97	2.72	3.586(4)	149.0	x, y, $-1+z$
C25—H25A...Cl1	0.97	2.77	3.548(4)	138.0	x, y, $-1+z$

contact and no C_3 symmetry is observed. So one can conclude that the presence of Cl^- counterion in **3** results in the formation of a packing structure with higher symmetry where every OH group is bonded toward the chloride anion and the hydrogen atom boned to tertiary nitrogen atom at both ends via three O—H...Cl and O—H...O hydrogen bonds. Furthermore, weak C—H...Cl hydrogen bonds can be observed in compound **3** between the two of three methylene groups (C16 and C25) and the chloride anion.

4. Conclusion

In summary, steric effects on forming different by-products in the formylation reaction of 2,4-dialkylphenol (dialkyl = *t*-Bu/*t*-Bu, *t*-Bu/Me and Me/Me) in addition to the expected 3,5-dialkyl-2-hydroxybenzaldehydes are discussed in this paper. In the case of starting materials 2,4-di-*tert*-butylphenol and 2-*tert*-butyl-4-methylphenol, the resulted aldehydes can further react with HMT under our experimental condition and the main by-products **1** and **2** are formed with different yields. In contrast, for the starting material 2,4-dimethylphenol, the yielded intermediate **a** can further react with one 2,4-dimethylphenol molecule to form **3** due to the relatively low steric hindrance of two phenyl-substituted methyl groups. This work supplies a practical method to synthesize 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde and **1** as well as 3,5-dimethyl-2-hydroxybenzaldehyde and **3** in medium yields at the same time, where two new single-crystal structures of **1** and **3** are included.

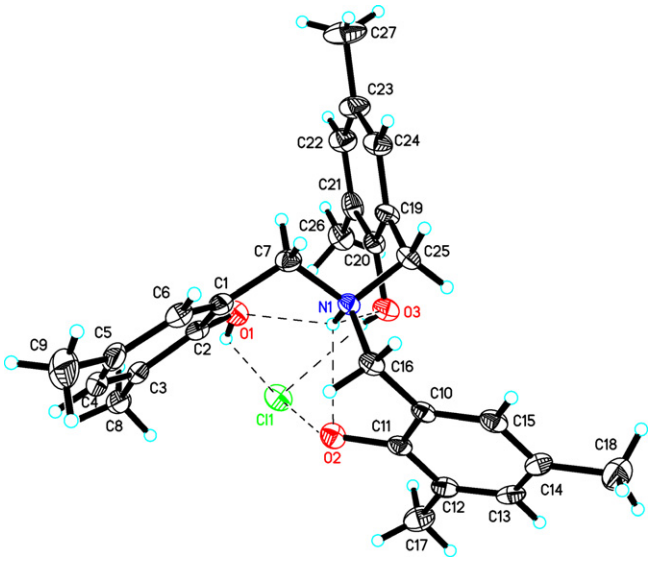


Fig. 3. ORTEP diagram (30% thermal probability) of the molecular structure of **3** with the atom-numbering scheme. H atoms are shown as small spheres of arbitrary radii and intramolecular hydrogen bonds are shown as dashed lines.

5. Supplementary material

CCDC reference numbers 643316 and 643317 for compounds **1** and **3** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; Fax: (International) +44 1223/336 033; E-mail: deposit@ccdc.cam.ac.uk].

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2007.10.035.

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