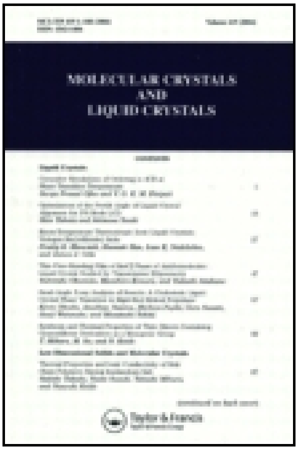

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# The Formation of Isomorphous Packing Structures in Complexes of Dodecyltrimethylammonium Halides Complexes with Rac-1,1′-Bi-2-naphthol

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*The crystal and molecular structures of 1:1 complexes of dodecyltrimethylammonium bromide and chloride adducts of rac-1,1′-Bi-2-naphthol (hereafter, 12TAB/BNP and 12TAC/BNP) have been determined by X-ray diffraction techniques. The crystal data and crystal structures revealed that the complexes crystallize in monoclinic crystal system with space group ($P2_1/a$) and Z-value (4), and also exhibit isomorphous packing structure. The crystal complexes are stabilized by electrostatic interactions, hydrogen bonds, and $C–H \cdots \pi$ interactions. The different lattice parameters as well as the different sizes of the halide ions ($Br^-$ and $Cl^-$) do not prevent both complexes (12TAB/BNP and 12TAC/BNP) from crystallizing with similar packing as well as common hydrogen bonding network and non-bonded interactions. Thus, the entering or leaving groups ($Br^-/Cl^-$) do not alter the existing hydrogen bonding and any other non-bonded interactions and thereby effects isomorphous packing structure.*

**Keywords:** dodecyltrimethylammonium bromide; dodecyltrimethylammonium chloride; H-bonds; Isomorphism; rac-1,1′-Bi-2-naphthol

## 1. INTRODUCTION

The understanding of crystal packing and intermolecular interactions in host-guest chemistry and in the utilization of such understanding in

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the design of new solids with desired physical and chemical properties has increasingly become an area of great interest to many supramolecular chemists [1]. Manipulation of molecular features in host-guest complexes so as to make use of specific interactions (electrostatic, H-bonding, van der Waals, etc.) and molecular packing (similar or different) is essential for future applications in designing supramolecular complexes (solid state complexes). The control of complexes at the molecular level is a field of major interest in molecular design and engineering that has become subject of increasing activity [2].

Crystal structure studies on crystal packing fashions in organic crystal complexes, and in respect to knowing the similarity or differences in packing structures as a result of change in the entering or leaving groups (atoms) will enhance our knowledge in understanding the approach to designing isomorphous organic complexes (similar packing structures), since isomorphism in organic crystal complexes is usually uncommon. In contrast, in inorganic crystal complexes, anions and cations varying in shape from spheres to different polyhedra (trigonal pyramids, tetrahedral, octahedral, etc.) frequently develop similar packing arrays which can be regarded as chemically isotypes.

Isomorphous packing structures between molecules can only be expected if they exhibit simultaneously functional isomorphism; *i.e.*, the entering or leaving groups (atoms) do not alter the existing hydrogen bonding [3] and any other non-bonded interactions. Thus, we were challenged to investigating how the change of halide size in a host molecule as well as shape of guest molecule affect/or not affect the packing structure and intermolecular interactions in organic host-guest complexes.

Based on these objectives we chose to investigate the crystal structures of dodecyltrimethylammonium bromide/chloride (hereafter, 12TAB/12TAC (Fig. 1)) complexes with rac-1,1′-bi-2-naphthol (hereafter, BNP (Fig. 1b) by X-ray diffraction techniques, and compare the packing structures between 12TAB/BNP and 12TAC/BNP complexes in order to know the effects of change of halide size and non-planar shape of the BNP molecule on the packing structures of both complexes, as well as know the existence of isomorphous packing structures between 12TAB/BNP and 12TAC/BNP. The 12TAB and 12TAC molecules were chosen as suitable candidates for complexation with BNP since it has well been known by our group of researchers that monoalkyltrimethylammonium halides form complexes with planar and non-planar aromatic molecules [4–8]. Thus, we found it suitable to study these complexes as per our interest in this research. In this manuscript, we present the isomorphous packing structures of

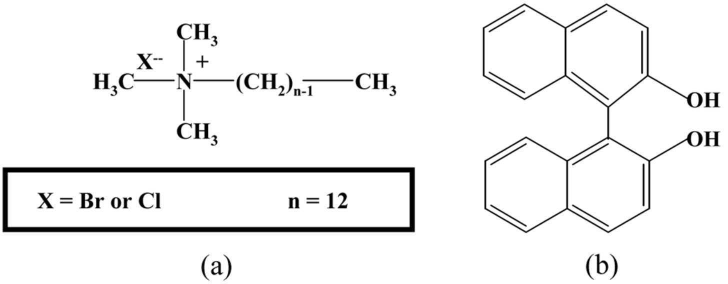


**FIGURE 1** (a) The chemical structure of a monoalkyltrimethylammonium halide (12TAB and 12TAC, where n = 12 and X = Br and Cl, respectively); (b) The chemical structure of Rac-1,1′-Bi-2-naphthol (BNP).

12TAB/BNP and 12TAC/BNP and how they address the aims of our research studies as mentioned. We have chosen the 12TAB or 12TAC molecule as *host* molecule and BNP as *guest* molecule in this manuscript.

## 2. EXPERIMENTAL

### 2.1. Preparation of Dodecyltrimethylammonium Bromide and Chloride Adducts of Rac-1,1′-Bi-2-naphthol

Samples of the two host compounds (12TAB and 12TAC) were purchased from Tokyo Chemical Industry Company Ltd., whilst BNP was purchased from Wako Chemical Industry Ltd., Tokyo, Japan. Single crystals of each of the complexes (12TAB/BNP, 12TAC/BNP) were prepared by treating a host compound with BNP in the molar ratio 1:1 in 8 ml acetone solution at room temperature. The mixture was simultaneously stirred and warmed at 30–40°C for 15 minutes in a glass bottle. It was then covered with a plastic wrap and kept in an incubator at 20°C for a week. Colorless needle-like single crystals were obtained in each case.

### 2.2. X-ray Intensity Data Collection

A four-circle diffractometer (Rigaku AFC5R for 12TAB/BNP and Rigaku AFC7R for 12TAC/BNP) fitted with graphite monochromatized CuK$\alpha$ radiation ($\lambda = 1.5418$ Å) was used for data collection. To obtain the cell constants and the orientation matrix for data collection the setting angles of 25 reflections in the range of $73.66 < 2\theta < 79.98^\circ$ for 12TAB/BNP, and $71.57 < 2\theta < 79.84^\circ$ for 12TAC/BNP were used.

The intensity data were collected at 298 K in the $\omega - 2\theta$ scan mode with scanning widths of $\Delta w = (1.26 + 0.30\tan\theta)^{\circ}$ for 12TAB/BNP and $\Delta w = (1.68 + 0.30\tan\theta)^{\circ}$ for 12TAC/BNP. Three reference reflections were measured after every 100 reflections for 12TAB/BNP and 150 reflections for 12TAC/BNP. Small intensity decreases were observed (0.1% in 12TAB/BNP and 0.34% in 12TAC/BNP), and decay correction was applied in both cases. An empirical absorption correction based on azimuthal scans of several reflections was applied together with Lorentz and polarization corrections in both cases.

## 2.3. Determination and Refinement of the Crystal Structures of 12TAB/BNP and 12TAC/BNP Complexes

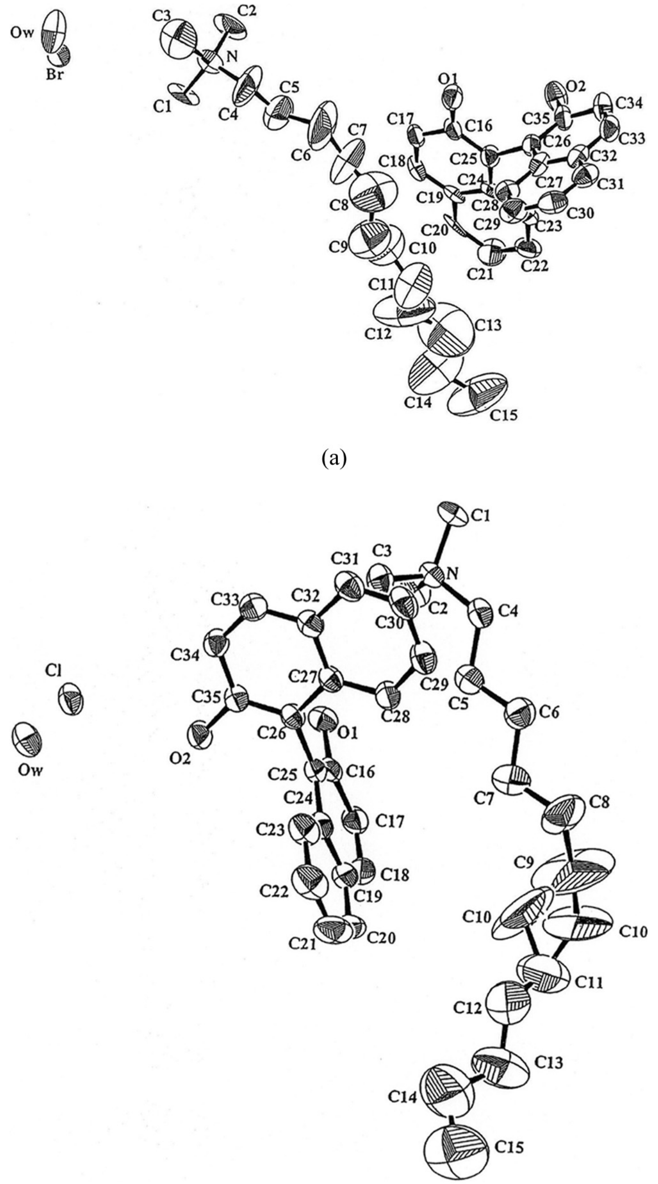
Both crystal structures (12TAB/BNP and 12TAC/BNP) were solved by direct methods (SIR92) [9] and expanded using Fourier techniques (DIRDIF94) [10]. The non-hydrogen atoms were refined anisotropically. The hydrogen atoms attached to O1, O2 of BNP, and Ow of water in 12TAB/BNP and 12TAC/BNP complexes could not be located in difference Fourier maps, but were included at calculated positions, with O–H = 0.86 Å. The orientation of hydrogen atoms of hydroxyl oxygen atoms were determined according to the best hydrogen bond to the oxygen atom. All other H atoms were placed in idealized positions with C–H distances of 0.99 Å for the secondary ($CH_2$) group and 1.00Å for the tertiary (CH) group. The H atoms were treated using a riding model. With the exception of C–C′ joining the two naphthol moieties, all the C–C bonds in the host and guest molecules were restrained to 1.520 and 1.390 Å, respectively, with weight 0.0005 to obtain appropriate bond lengths in the host and guest molecules. The final cycles of full-matrix least-squares refinement were based on 2089 observed reflections, 361 variable parameters for 12TAB/BNP BNP complex. In the case of 12TAC/BNP, the complex showed a statistical structure (Fig. 3b) since an additional peak close to C10 was observed in the difference Fourier map. This peak was assigned as C10′ with occupancy of 0.5 while the occupancy of C10 was changed to 0.5. Refinement of this structure including occupancies of these atoms improved the $R$-factor. The final cycles of full-matrix least-squares refinement were based on 2942 observed reflections, 370 variable parameters. The function minimized in both cases was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma^2(F_o)$. The final $R$ and $Rw$ factors were 0.096 and 0.127 for 12TAB/BNP, and 0.098 and 0.144 for 12TAC/BNP, respectively. The maximum and minimum peaks on the final difference map were 0.38 and $-0.43e^-/Å^3$ for 12TAB/BNP and 0.49 and $-0.49e^-/Å^3$ for 12TAC/BNP. Atomic scattering factors were taken from Cromer and Waber [11]. All calculations on data collection, structure determination, and refinement were performed using the TeXsan crystallographic software package of the Molecular Structure Corporation [12]. The experimental parameters, atomic coordinates, bond lengths, bond angles, torsional angles, and thermal parameters for both complexes can be obtained upon request from the *Director, Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, CB2 1EZ, UK*. The deposit numbers are *160236* and *160237* for 12TAB/BNP and 12TAC/BNP complexes, respectively.

## 3. RESULTS AND DISCUSSION

### 3.1. Molecular Geometry of 12TAB/BNP and 12TAC/BNP Complexes

The molecular structures of 12TAB/BNP and 12TAC/BNP complexes with atomic numbering are shown in Figs. 2a and b. In both complexes, the host-guest ratio exists as 1:1. The asymmetric unit of the respective complexes consists of one host, BNP, and a water molecule. The hydrophilic head group of the host molecules is rigid, whilst the hydrophobic tail part is not. The naphthol groups in the BNP molecule have a rigid aromatic ring conformation, and the BNP molecule is nonplanar due to the effect of steric influences of the adjacent neighboring oxygen atoms on each of the naphthol moieties. The bond lengths, bond angles, and torsion angles in both host molecules (12TAB and 12TAC) are within the ranges reported on both compounds [13–17]. In all the complexes, the sum of the bond angles in the benzene rings of the naphthol groups are generally between 719.9 and 720°, and those of the angles about C atoms are also between 359.9 and 360°. This suggests that the benzene rings do not deviate from the regular hexagon shape. In both complexes, the deviations of the hydroxyl oxygen atoms from each plane of the naphthalene rings (1) and (2) are generally between 0.02 and $-0.01$Å, respectively. The bond lengths and angles in the naphthalene rings show typical conjugated double bond character in both complexes.

In the 12TAB molecule, the average C$-$C bond length, and C$-$C$-$C bond angle are 1.520(5) Å and 112.0(5)°, respectively. The average N$-$C bond length and C$-$N$-$C bond angle are 1.532(4) Å and 109.5(3), respectively. The C$-$C$-$C$-$C torsion angles are in the range of $180 \pm 43°$, with the exceptions of C6$-$C7$-$C8$-$C9 ($-68(2)°$) and C7$-$C8$-$C9$-$C10 ($-67(2)°$), which suggests a *gauche* conformation about C7$-$C8 and C8$-$C9. The mean deviation from plane in naphthol plane (1) and naphthol plane (2) are 0.015 and 0.016 Å, respectively



(a)

(b)

**FIGURE 2** (a) The molecular structure and atomic numbering of 1:1 complex of 12TAB/BNP. The 40% probability ellipsoids are shown. Hydrogen atoms omitted for clarity; (b) The molecular structure and atomic numbering of 1:1 complex of 12TAC/BNP. The 40% probability ellipsoids are shown. Hydrogen atoms omitted for clarity.

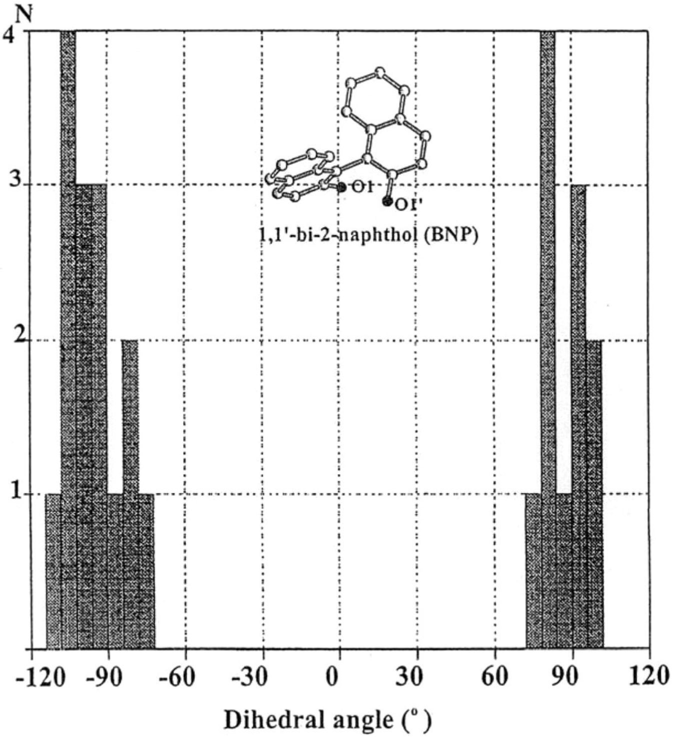


**FIGURE 3** Number of observations (N) plotted against dihedral angles in BNP for 26 crystal structures obtained from Cambridge Structure Database (CSD).

which suggests that the aromatic rings have good planarity. The dihedral angle between the two naphthol planes defined by O1, C16,...C25 and O2, C35,...C26 in BNP is 86.2(8)°. The OH groups of the BNP molecule adopt the *cis* mode, thus the BNP molecule adopts the *cisoid* [18] conformation. The C–C bond connecting two naphthol rings is in a length of 1.518(8) Å and longer than the one observed in the corresponding value for *cisoid* racemate conformation (1.475 Å) [19]. The average value of C–C bond length and C–C–C bond angles in naphthalene ring (1) are 1.391(4) Å and 120.2(6)°, whilst in naphthalene (2), the values are 1.392(4) Å and 120.5(5)°. These values in the two naphthalene rings do not differ significantly from each other, and none of them is deformed. The C–O bond lengths are 1.38(1) Å and 1.346(7) Å for O1–C16 and O2–C35, respectively, and the average value (1.363(4) Å) is similar to the usual value for C–O bond length (1.36(2) Å) [20] in phenols.

In the 12TAC molecule, the average bond lengths and angles are 1.516(5) Å for C–C, 112.0(4)° for C–C–C, 1.500(6) Å for N–C; and 109.5(4)° for C–N–C, respectively. These values are not significantly different from those in the 12TAB complex and others reported [17]. The C–C–C–C torsion angles in the alkyl chain are in the range of 180 ± 42°, with the exceptions of C7–C8–C9–C10 (−56.3(8)°) and C7–C8–C9–C10′ (−58.4(9)°) which suggests a *gauche* conformation about C8–C9. The mean deviation from plane in naphthol plane (1) and (2) are 0.058 Å and 0.043 Å, respectively, which also suggests that the aromatic rings are planar. The dihedral angle between the two naphthol planes defined by one oxygen atom and ten atoms in BNP is 80.7(6)° and the BNP molecule has *cisoid* [18] conformation with OH groups adopting the *cis* mode. The C–C bond connecting two naphthol rings is in a length of 1.489(6) Å and is longer than the corresponding *cisoid* racemate conformation (1.475) Å [19]. The average value of C–C bond length and C–C–C bond angles in naphthalene ring (1) are 1.400(7) Å and 120.3(5)°, whilst in naphthalene (2), the values are 1.389(4) Å and 120.3(4)°. The values in the two naphthalene rings do not differ significantly from each other and suggest that both naphthalene rings are normal. The C–O bond lengths are 1.367(6) Å and 1.389(5) Å for O1–C16 and O2–C35, respectively, and the average value 1.378(5) Å is slightly longer the usual value for C–O bond length (1.36(2) Å) [20] in phenols.

As can be observed in the histogram shown in Fig. 3, the number of observations plotted against dihedral angles in BNP, and in its organic complexes with $R$-factor $<0.10$ revealed that the dihedral angles obtained in these complexes agreed well with the corresponding values (90 ± 24°) obtained by statistical analysis for 26 crystal structures from Cambridge Structure Database (CSD, Version 5.12, April 1997 release). An inspection of the C–C, C–O bond lengths and C–C–C, O–C–C bond angles of the BNP molecule in both complexes compared with those values in the 26 crystal structures retrieved from the CSD [13–19] showed that the bond lengths and angles are within the range of accepted values and are in agreement with those retrieved from the CSD [13–19]. The atoms in the host molecules show significant thermal motion in the atoms from the bent region to the terminal part of the alkyl chain. The reason for the large temperature factors could be attributed to the fact that BNP molecules are rigid and creates a cavity in which the flexible alkyl chains serve as space fillers and thereby increases the thermal motion of the atoms affected.

## 3.2. Host-Guest Interactions and Isomorphous Packing Structures in 12TAB/BNP and 12TAC/BNP Complexes

In both complexes (12TAB/BNP and 12TAC/BNP) the formula unit per unit cell is 4. Thus, the total number of host and guest molecules in the corresponding cells is 8 (Figs. 4a and b). The complexes crystallize in the same crystal system (*monoclinic*) with space group as $P2_1/a$ (Table 1). The crystal structures for both complexes viewed along the *b*-axis are shown in Figs. 4a and b. The crystal complexes are stabilized by electrostatic interactions, strong and weak hydrogen bonds. The coulombic interactions exists between the bromide anion and charged nitrogen; $Br \cdots N^+$ (4.74 Å), with the $Br \cdots N^+$ distance being higher than that reported on the crystal structure of 12TAB (3.3 Å) [13]. In 12TAB/BNP (Fig. 5a), the distance between the bromide anion and oxygen atom of BNP ($O1 \cdots Br$) is 3.158(8) Å), meaning the formation of hydrogen bond, although the hydrogen atom attached to the O1 atom of BNP could not be located in the difference Fourier map. The bromide anion also forms hydrogen bond with the oxygen atom of water ($Ow \cdots Br$, 3.247(7) Å). A strong hydrogen bond formation was observed in the interaction between O2 of BNP and oxygen of the water molecule ($O2 \cdots Ow$, 2.660(7) Å). A short $C\text{-}H \cdots C$ distance (2.96 Å) in Fig. 5a was observed between a hydrogen atom (H19) of a methylene group of the alkyl chain and a carbon atom (C23) of one of the naphthol moieties, which is shorter than the van der Waals

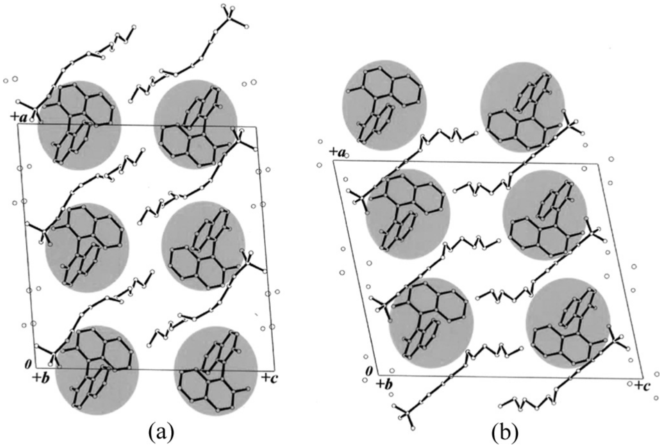
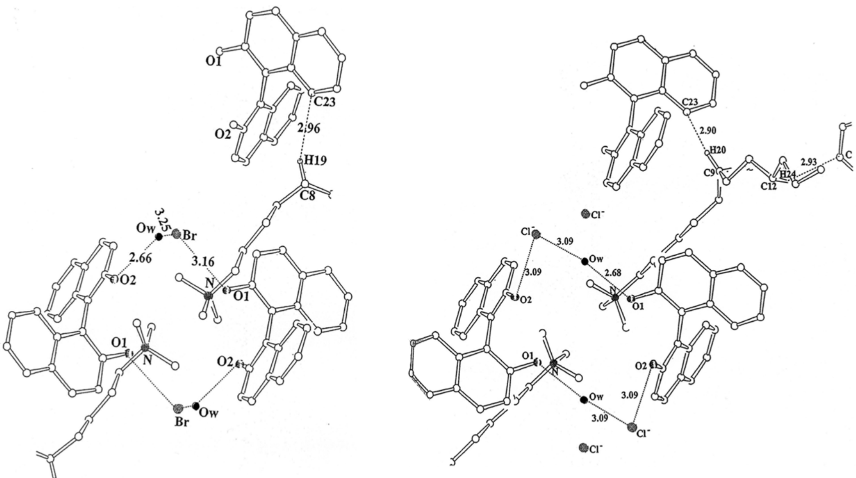


**FIGURE 4** The crystal structure of (a) 1:1 complex of 12TAB/BNP and (b) 1:1 complex of 12TAC/BNP. The isomorphous packing structure between 12TAB/BNP and 12TAC/BNP.

**TABLE 1** Crystal Data of 12TAB/BNP and 12TAC/BNP

| Complex | 12TAB/BNP | 12TAC/BNP |
|---|---|---|
| Molecular formula | $C_{35}H_{46}O_3N^+Br^-$ | $C_{35}H_{46}O_3N^+C1^-$ |
| Crystal system | Monoclinic | Monoclinic |
| Formula | 608.66 | 564.21 |
| Space group | $P2_1/a$ | $P2_1/a$ |
| $a$/Å | 19.299(2) | 18.237(4) |
| $b$/Å | 9.699(8) | 8.782(1) |
| $c$/Å | 18.359(2) | 21.458(4) |
| $\beta$/° | 95.140(8) | 102.52(1) |
| Volume/Å$^3$ | 3422.8(5) | 3354(1) |
| Z | 4 | 4 |
| D*calc*/gcm$^{-3}$ | 1.18 | 1.12 |
| F(000) | 1288 | 1216 |
| *m.p*/°C | 152.3 | 119.1 |

distance (2.97 Å) [21]. Thus C–H$\cdots\pi$ interactions can be inferred to also stabilize the crystal structure. The C$\cdots$C intermolecular contacts observed between the methyl groups and the aromatic rings, and shorter than van der Waals distances (3.54 Å) [21] are C2$\cdots$C32; 3.53(1), C3$\cdots$C35; 3.37(2), and C3$\cdots$C34; 3.14(1) Å.



(a) (b)

**FIGURE 5** (a) Hydrogen bond interactions in the 12TAB/BNP (viewed along *b*-axis). The broken lines represent C–H$\cdots\pi$ interactions, whilst the dotted lines represent hydrogen bonds; (b) Hydrogen bond interactions in the 12TAC/BNP (viewed along *b*-axis). The broken lines represent C–H$\cdots\pi$ interactions, whilst the dotted lines represent hydrogen bonds.

The coulombic interactions that exist between chloride anion and charged nitrogen have the shortest distance of $Cl \cdots N^+$ being 4.12 Å, and is similar to the one observed in 12TAC [15]. In the 12TAC/BNP BNP complex (Fig. 5b), the hydrogen bonds formation exist between, $O2 \cdots Cl$, 3.089(3) Å, $Ow \cdots Cl$, 3.088(4) Å and $O1 \cdots Ow$, 2.680(5) Å. The crystal structure is also stabilized by $C\text{-}H \cdots \pi$ interactions and attributed to the following short distances, *viz*; $C21 \cdots H24$; 2.93 Å, $C23 \cdots H20$; 2.90 Å (Fig. 5b). The $C \cdots C$ intermolecular distances shorter than the van der Wals distance (3.54 Å) [21] are $C2 \cdots C33$; 3.3373(7), $C16 \cdots C22$; 3.54(1) Å. This shows the interactions between the carbon atoms in both the alkyl chain and the methyl groups and those of the carbon atoms in the aromatic rings.

The shortest guest-guest intermolecular distance in the neighboring guest molecules of each of the complexes are 3.79 and 3.94 Å for 12TAB/BNP and 12TAC/BNP, respectively, whilst those of host–host intermolecular distances are 4.01 and 4.25 for 12TAB/BNP and 12TAC/BNP, respectively. It can be inferred from these $C \cdots C$ intermolecular distances of the guest-guest and host-host compounds that the host molecules are rather more closely trapped between the rigid BNP molecules, thus the packing of the BNP molecules is the driving force behind the packing of the alkyl chains. In both complexes, the host molecules are arranged in a unique interdigitated fashion (Figs. 4a and b) in the bent tail region of the alkyl chain viewed in the *ac*-plane, but the type of arrangement is quite different from those observed in complexes of monoalkylammonium halides with planar aromatic molecules [13–17].

It can be deduced from the crystal data (Table 1) and crystal structures (Figs. 4a and b) that the complexes have common space group ($P2_1/a$) and Z-value (4), and also exhibit isomorphous packing structure. The different lattice parameters as well as the different sizes of the halides do not prevent them from crystallizing with similar packing as well as similar existing hydrogen bonding and non-bonded interactions. This suggests that the difference in the size and volume of the halogen atoms as well as difference in lattice parameters cannot alter the common packing and intermolecular interactions in both complexes. However, in complexes involving monoalkyltrimethylammonium halides with planar aromatic molecules, *e.g., para*-Phenylphenol (*p*-PP), the substitution of the counter anion changes the packing arrangement, intermolecular interactions, and also the host-guest ratio. Such examples are seen in 12TAB/*p*-PP, 12TAC/*p*-PP, 14TAB/*p*-PP, and 14TAC/*p*-PP complexes where the host/guest ratios are 2:1,1:1, 2:1, and 1:1, respectively [15,17]. The comparisons suggest that the difference in size or volume of the halide ions do not have any effect on the isomorphous packing structures in 12TAB/BNP and 12TAC/BNP, and the formation of the isomorphous packing structures between both complexes could be attributed to the effect of hydrogen bonding network and non-bonded interactions involved in the complexes of 12TAB/BNP and 12TAC/BNP.

## CONCLUSION

The different sizes of the halide ions ($Br^-$ and $Cl^-$) do not prevent complexes of 12TAB/BNP and 12TAC/BNP from crystallizing with similar packing as well as common hydrogen bonding network and non-bonded interactions. The entering or leaving groups ($Br^-/Cl^-$) do not alter the existing hydrogen bonding and any other non-bonded interactions, and thereby effects isomorphous packing structure.

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