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The monoalkyltrimethylammonium halides $CH_3(CH_2)_{n-1}N^+(CH_3)_3X^-$ ($X = Br$ or Cl, when $n = 12$, and $X = Br$, when $n = 16$) and rac-1,1′-bi-2-naphthol ($C_{20}H_{14}O_2$) have been found to form 1:1 crystalline complexes. In all the complexes, the host molecules are sandwiched between the BNP molecules in a similar manner. The bent tail region of the alkyl chains in all the host molecules are arranged in a uniquely interdigitated fashion. The bent conformation of the alkyl chains could be attributed to the effect of placing a naphthol plane almost normal to the long alkyl chain axis. The change of the counter anion in the host molecule does not show any significant variation in the packing arrangement observed in the complexes of 12TAB/BNP and 12TAC/BNP or 16TAB/BNP and 16TAC/BNP. The nonplanar shape of the BNP molecule affects the molecular packing of the alkyl chains. The BNP molecule adopts a cisoid conformation in all the complexes. Hydrogen bonds and C-H…π interactions stabilize these crystal structures.

Keywords: monoalkylammonium halides; crystal structure; molecular interactions; molecular recognition; crystal engineering; surfactants

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INTRODUCTION

It has been known that crystalline complexes are formed between monoalkylammonium halides and aromatic molecules through molecular aggregation [1]. Understanding the interactions between two different types of molecules in the complex crystal becomes important in the study of molecular recognition and crystal engineering. Particularly, the molecular recognition in the complex crystal is a subject of considerable interest [2] because of its applications to separation purposes. A few years ago, it was reported [3] that an interaction between a chiral onium salt (cinchonidinium chloride) and a racemic compound (Bis-β-naphthol) accomplishes a successful enantiomeric resolution, but many researchers are mainly interested in exploiting the most economical or cheapest means of resolving racemic compounds using a nonchiral host molecule. However, this constitutes an important and difficult task yet to be achieved. As an initial approach in our investigations, the inclusion crystallization of nonchiral host compound with racemic guest compound was chosen to investigate the molecular recognition between nonchiral host compound (monoalkyltrimethylammonium halides) and racemic guest compound (rac-1,1′-bi-2-naphthol) from the viewpoint of the crystal structure solution and analysis for the purpose of knowing the feasible interactions, molecular packing structures, and space groups of such complexes and comparing with those of monoalkyltrimethylammonium halides and chiral guest molecules (e.g, (R)-(+)-1,1′-bi-2-naphthol). Such a study will be useful for probable future applications (e.g., separation purposes).

Our recent studies [4–6] on molecular aggregation between monoalkyltrimethylammonium halides with either (R)-(+)-1,1′-bi-2-naphthol (hereafter, RBNP) or rac-1,1′-bi-2-naphthol (hereafter, BNP), have shown that a nonchiral amphiphile can form a complex crystal with either a chiral or racemic nonplanar aromatic molecule. In each of these reports on the 1:1 complex crystals of a monoalkyltrimethylammonium halide with BNP, hydrogen bonding, and C-H$\ldots\pi$ interactions were predominant in contributing to the stability of the crystal structures. The packing arrangements found in this study were quite different from those observed in crystal structures of complexes of monoalkyltrimethylammonium halides with planar aromatic molecules [1].

In order to investigate the effect of halide anion (Br^- or Cl^-) on the packing structure, and the effect of the nonplanar shape of the BNP molecule on the alkyl chain packing of host molecules, we prepared four complex crystals viz., dodecyltrimethylammonium bromide with BNP (hereafter, 12TAB/BNP), dodecyltrimethylammonium chloride with BNP (hereafter, 12TAC/BNP), hexadecyltrimethylammonium bromide with

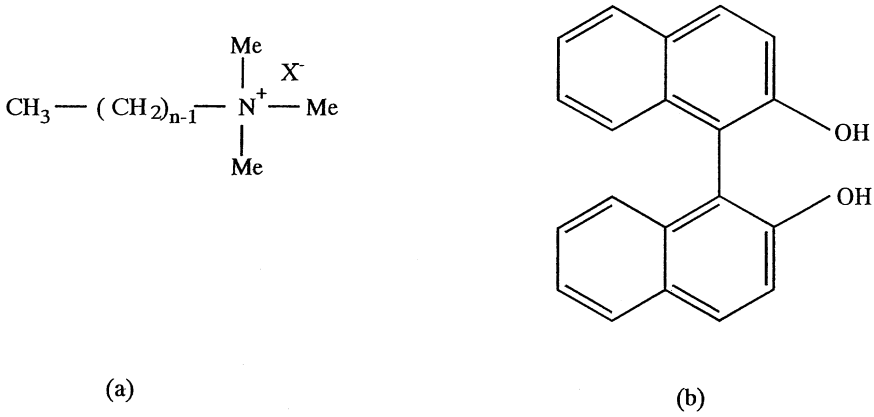


FIGURE 1 Chemical structures of host amphiphile and guest aromatic molecule. (a) Host amphiphiles: n = 12, X = Br, Dodecyltrimethylammonium bromide (12TAB); n = 12, X = Cl, Dodecyltrimethylammonium chloride (12TAC); n = 16, X = Br, Hexadecyltrimethylammonium bromide (16TAB); n = 16, X = Cl, Hexadecyltrimethylammonium chloride (16TAC). (b) Guest aromatic molecule: Rac-1,1′-bi-2-naphthol (BNP).

BNP (hereafter, 16TAB/BNP), and hexadecyltrimethylammonium chloride with BNP (hereafter, 16TAC/BNP) and determined their crystal structures. The crystal structure of 16TAC/BNP has already been reported [6]. In this study, we discuss the crystal structures of 12TAB/BNP, 12TAC/BNP, and 16TAB/BNP. The 12TAB, 12TAC, and 16TAB in each complex is termed the *host* molecule, while the BNP is termed the *guest* molecule (Figs. 1a and 1b, respectively).

EXPERIMENTAL

Preparation of 12TAB/BNP, 12TAC/BNP, and 16TAB/BNP

Samples of the three host compounds 12TAB, 12TAC, and 16TAB were purchased from Tokyo Chemical Industry Company, Ltd. (Tokyo, Japan) and BNP was obtained from Wako Chemical Industry Ltd. (Tokyo, Japan). Single crystals of each of the complexes were prepared by treating a host compound with BNP in the molar ratio of 1:1 in acetone solution at room temperature. The mixture was simultaneously stirred and warmed at 303–313 K for 15 min in a glass bottle. It was then covered with a plastic wrap and kept in an incubator at 293 K for a week. Colorless needle-like single crystals were obtained in each case.

X-ray Data Collection

A four-circle diffractometer (Rigaku AFC5R for 12TAB/BNP and 16TAB/BNP, and AFC7R for 12TAC/BNP) fitted with graphite monochromatized CuKα 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 $71 < 2\theta < 80^\circ$ were used. The intensity data were collected at 298 K in the $\omega - 2\theta$ scan mode. Three reference reflections were measured after every 100 or 150 reflections. Small intensity decreases of less than 1.5% were observed and decay correction was applied. An empirical absorption correction based on azimuthal scans of several reflections was applied in every case. The parameters for data collection and crystal data are summarized in Table 1.

Determination and Refinement of the Crystal Structure

The crystal structures were solved by direct methods (SIR92) [8] and expanded using Fourier techniques [9]. The nonhydrogen atoms were refined anisotropically. The hydrogen atoms attached to O1, O2 of BNP, and Ow of water in all the complexes could not be located in difference Fourier maps, thus they were not included in the calculation. The remaining hydrogen atoms were introduced by geometrical calculations but not refined. The 12TAC/BNP complex showed a positional disorder of C10 (Fig. 2b). The site occupation factors of C10 and C10′ are 0.5 and 0.5, respectively. 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 the observed reflections (Table 1), 361, 370, and 397 variable parameters, and 54, 20, and 40 geometrical restraints for 12TAB/BNP, 12TAC/BNP, and 16TAB/BNP, respectively. The function minimized in all cases was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma^2(F_o)$. The final R and R_w factors were 0.109 and 0.127 for 12TAB/BNP, 0.098 and 0.148 for 12TAC/BNP, and 0.086 and 0.089 for 16TAB/BNP.

Atomic scattering factors were taken from Cromer and Waber [10]. All calculations on data collection, structure determination, and refinement were performed using the teXsan crystallographic software package of the Molecular Structure Corporation [7]. The final atomic coordinates, anisotropic thermal parameters, bond lengths, and angles and tables of observed and calculated structure factors for the three complexes have been deposited at the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge, CBZ 1EZ, UK. The deposition reference numbers of CCDC are 160236 (12TAB/BNP), 160237 (12TAC/BNP), and 160238 (16TAB/BNP).

TABLE 1 Crystal Data and Data Collection Details for 12TAB/BNP, 12TAC/BNP, 16TAB/BNP, and 16TAC/BNP Complexes

Complex	12TAB/BNP	12TAC/BNP	16TAB/BNP	16TAC/BNP[6]
Chemical formula	$C_{15}H_{34}N^+Br^-$. $C_{20}H_{14}O_2.H_2O$	$C_{15}H_{34}N^+Cl^-$. $C_{20}H_{14}O_2.H_2O$	$C_{19}H_{42}N^+Br^-$. $C_{20}H_{14}O_2.H_2O$	$C_{19}H_{42}N^+Cl^-$. $C_{20}H_{14}O_2.H_2O$
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Formula weight	612.69	568.24	668.80	624.35
Space group	$P2_{1/a}$	$P2_{1/a}$	12/a	12/a
a/Å	19.299(2)	18.237(4)	19.300(5)	18.249(2)
b/Å	9.6996(8)	8.782(1)	9.619(2)	8.757(2)
c/Å	18.359(2)	21.458(4)	45.103(6)	48.029(3)
β/°	95.140(8)	102.52(1)	111.488(7)	101.145(6)
Volume/$\dot{A}^3$	3422.8(5)	3354(1)	7790(2)	7530(1)
Z	4	4	8	8
Dcalc/g cm^{-3}	1.190	1.120	1.140	1.100
F(000)	1288	1216	2832	2720
Crystal dimensions/mm	$0.20 \times 0.20 \times 0.10$	$0.20 \times 0.20 \times 0.25$	$0.20 \times 0.20 \times 0.20$	$0.20 \times 0.20 \times 0.20$
μ(CuKα)/cm^{-1}	18.79	12.5	16.96	11.55
Maximum 2θ/°	120	120	120	130
No. of reflections measured	Total: 2552	Total: 5542	Total: 6267	Total: 6993
	Unique: 2552	Unique: 5356	Unique: 6166	Unique: 6898
No. of reflections used in calculation	2089 [$I > \sigma(I)$]	2942 [$I > \sigma(I)$]	3335 [$I > \sigma(I)$]	2578 [$I > 2.5\sigma(I)$]
R-factor	0.109	0.098	0.086	0.075
Rw	0.127	0.148	0.089	0.066
Goodness-of-fit	2.11	1.65	2.00	1.84
Δ/eÅ^{-3} (min/max)	−0.43/0.38	−0.49/0.49	−0.29/0.40	−0.15/0.15
Absorption correction	Psi-scan	Psi-scan	Psi-scan	Psi-scan
Absorption range	0.990–1.00	0.963–1.00	0.820–1.00	0.867–1.00

RESULTS AND DISCUSSION

Molecular Structures

The molecular structures with atomic numbering are shown in Figures 2a–2c. The asymmetric unit of the respective crystal consists of one host molecule, BNP, and water. The ammonium cation and halide anion (Br^- or Cl^-) constitute the hydrophilic region, and the alkyl chain constitutes the hydrophobic region of the host molecule. The hydrophilic head group of the host molecules is rigid, while the hydrophobic tail part is not. The naphthol groups in the BNP molecule have a rigid aromatic ring conformation, and the shape of the BNP molecule is nonplanar due to the steric effect of the two adjacent neighboring oxygen atoms on each of the naphthol moieties.

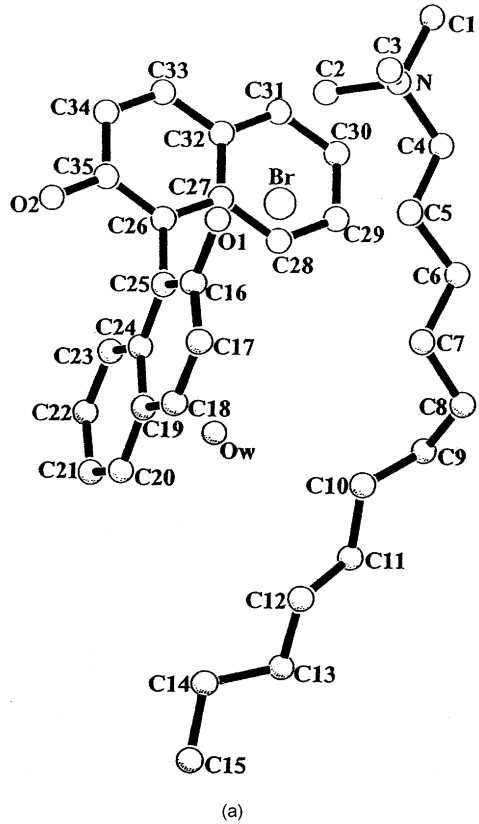


FIGURE 2 Molecular structure and atomic numbering of (a) 12TAB/BNP, (b) 12TAC/BNP, and (c) 16TAB/BNP. Hydrogen atoms are omitted for clarity of the structure.

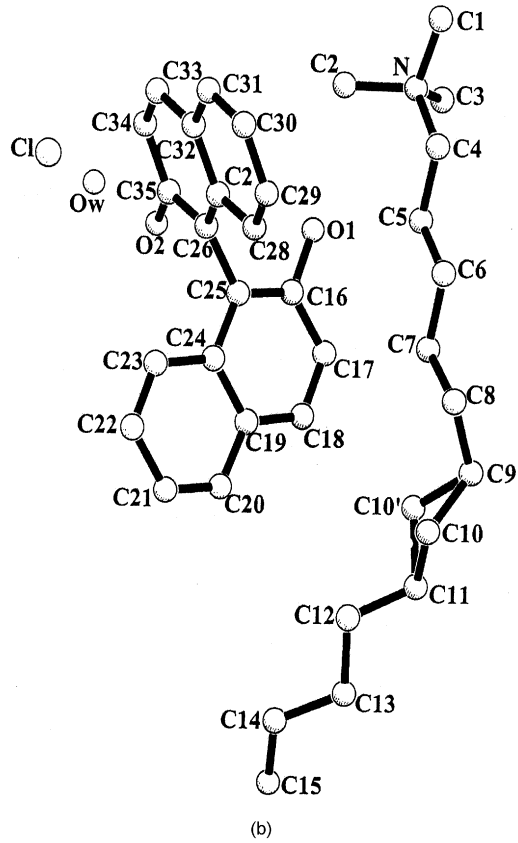


FIGURE 2 Continued.

The bond lengths and bond angles are within the ranges reported on similar compounds [11,12]. The torsion angles, which deviated more than 30° from 180°, are: 61(2)° for C7-C8, −67(1)° for C8-C9, −143(2)° for C9-C10, and −142(4)° for C12-C13, in 12TAB. In 12TAC, they are −148.5(6)° for C7-C8, 54.6(8)° for C8-C9, 139.9(7)° for C9-C10, −140.1(7)° for C9-C10′, −138.2(7)° for C10-C11, 139.9(7)° for C10′-C11, and 144(1)° for C11-C12. Similarly, in 16TAB, those are 61(2)° for C6-C7, −146(1)° for C9-C10, and −92(1)° for C10-C11. These values suggest that the alkyl chains of these host molecules do not have fully extended *trans* conformation but have at least one *gauche* conformation. The dihedral angle between the two naphthol planes defined by one oxygen atom and ten carbon atoms in BNP for 12TAB/BNP, 12TAC/BNP, and 16TAB/BNP complexes are 86.2(8)°, 80.7(6)°, and 89.4(8)°, respectively. These values agreed well with the

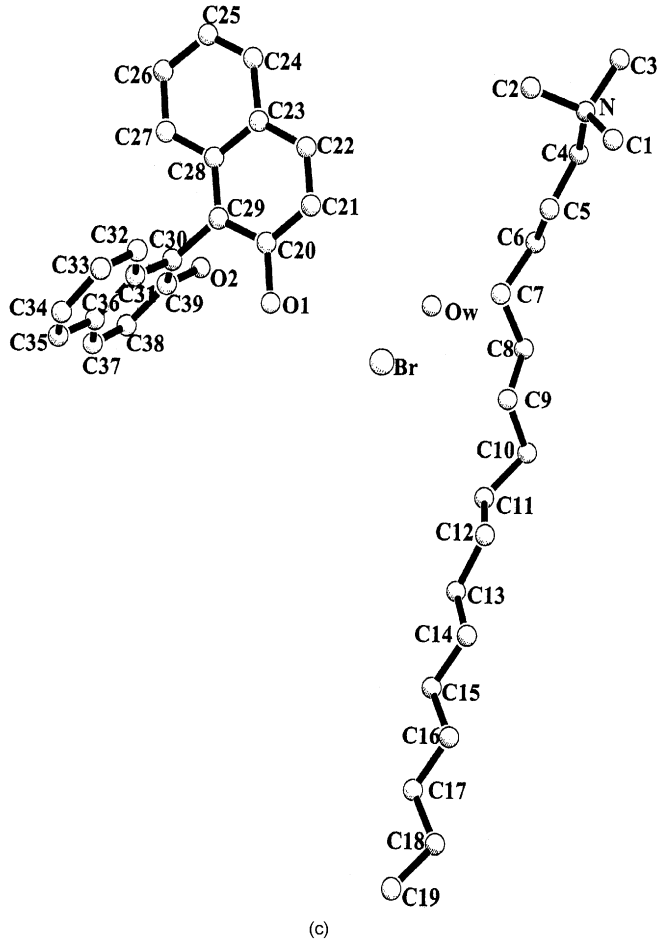


FIGURE 2 Continued.

corresponding values ($90 \pm 24°$) obtained by a statistical analysis for 27 crystal structures of BNP and its complexes retrieved from the Cambridge Structural Database [13]. In all the complexes, the BNP molecule adopts the *cisoid* conformation [14,15].

Intermolecular Interactions and Packing Structure in the Complexes

The crystal structures of 12TAB/BNP, 12TAC/BNP, and 16TAB/BNP, and 16TAC/BNP [6] viewed along the *b*-axis are shown in Figures 3a–3d. The crystal complex is stabilized by hydrogen bonds between the OH of BNP or

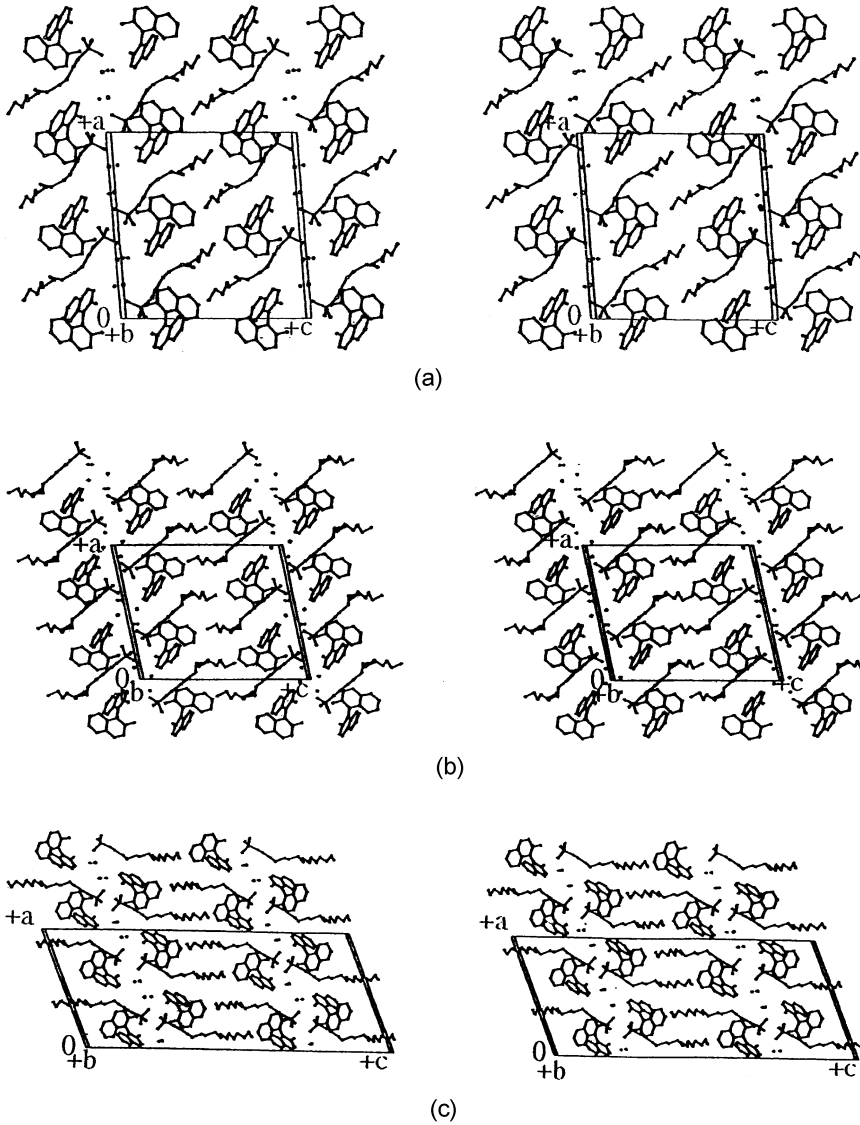
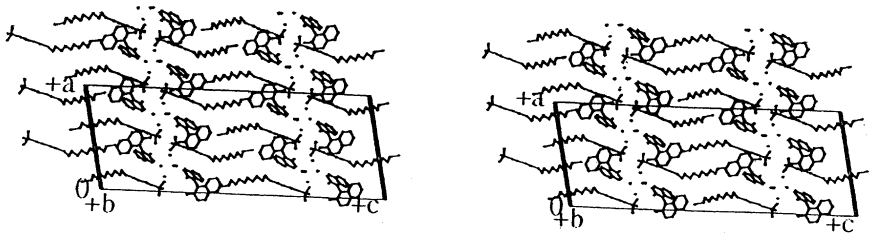


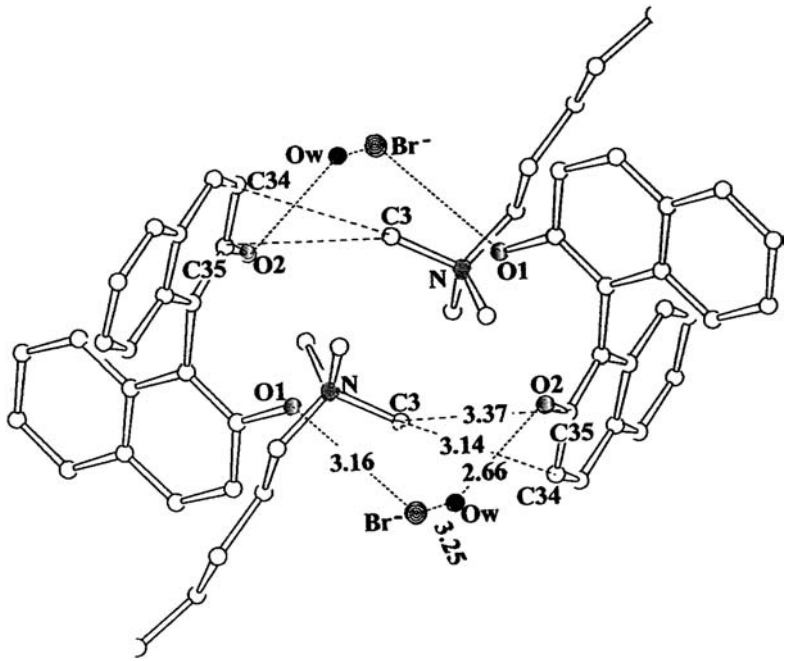
FIGURE 3 Stereoscopic view of (a) 12TAB/BNP, (b) 12TAC/BNP, (c) 16TAB/BNP, and (d) 16TAC/BNP [6] viewed along the *b*-axis.



(d)

FIGURE 3 Continued.

the water molecule and oxygen atom and the halide anion, and weak hydrogen bonds between C-H of the host molecule and π-system of BNP. All the crystal structures of the four crystal complexes have a similar hydrogen bond network. In 12TAB/BNP (Fig. 4a), the Coulombic interactions exist



(a)

FIGURE 4 Hydrogen bonds and C···C nonbonded interactions observed in (a) 12TAB/BNP, (b) 12TAC/BNP, and (c) 16TAB/BNP (part of Figures 3a, 3b, and 3c) complexes. The dotted lines represent hydrogen bonds, broken lines represent C···C nonbonded contacts. The Cl^-, Br^-, N^+, Ow, O1, O2 are explicitly colored for clarity.

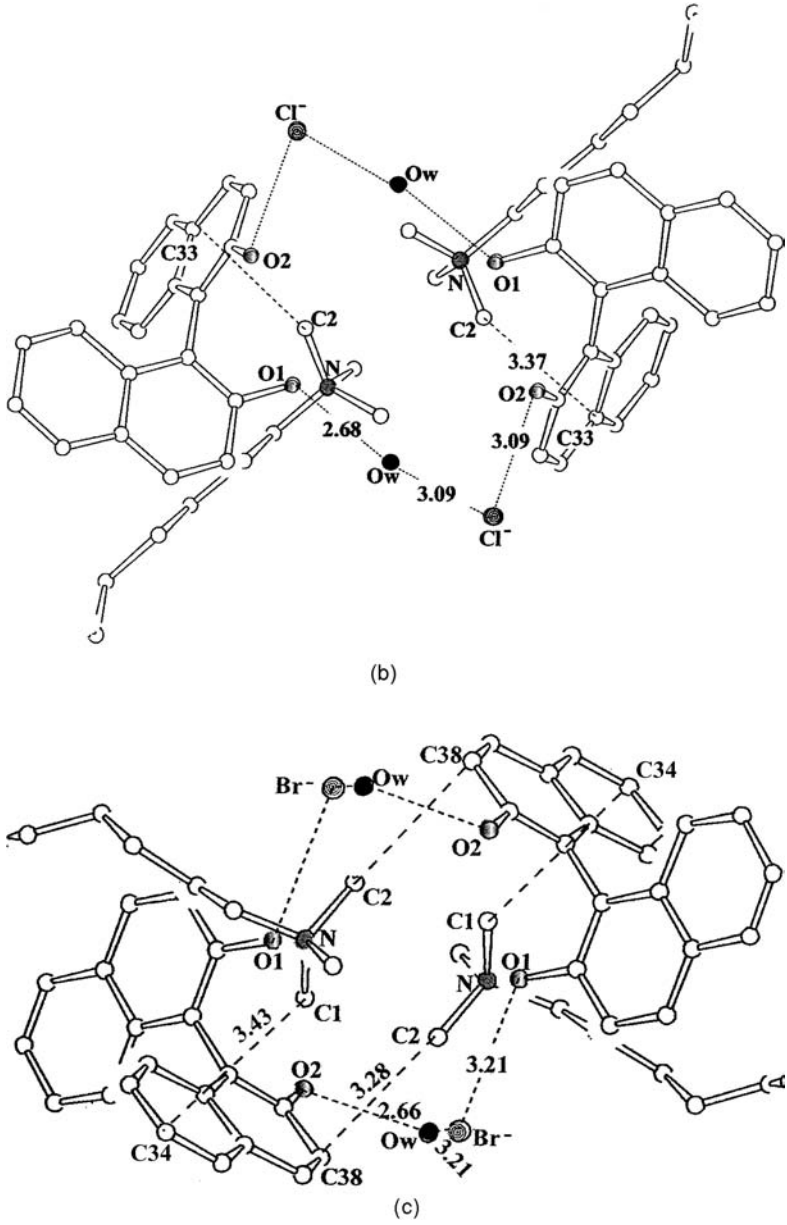


FIGURE 4 Continued.

between the bromide anion and charged nitrogen and have the shortest distance of $Br^- \ldots N^+$, being 4.74(2) Å, which is longer than that reported on the crystal structure of 12TAB (3.3 Å) [12]. In all of these 1:1 complexes, the guest molecule utilizes its OH groups to form a hydrogen bond with either halide anions or water. Since the hydroxyl H-atoms of BNP and those of water molecules were not located in the difference map, the criterion for hydrogen bonding was based on the short contact distances between non-H atoms with values less than the sum of their van der Waals radii. In 12TAB/BNP (Fig. 4a), the distance between the bromide anion and oxygen atom of BNP (O1 ... Br) is 3.158(8) Å, suggesting a hydrogen bond. The bromide anion also forms a hydrogen bond with water (Ow ... Br, 3.247(7) Å). A strong hydrogen bond is found in the interaction between the other OH groups of BNP and the oxygen of the water molecule (O2 ... Ow, 2.660(7) Å). The short C-H ... C distances observed between some hydrogen atoms of the naphthol moieties were found to be shorter than the sum of the van der Waals distance of 2.90 Å [16]. These are; C35 ... H7, 2.855 Å and C34 ... H9, 2.713 Å. These C ... H values between the methyl groups and π-system of the naphthol groups are within the range of values (2.62–2.90 Å) reported by Umezawa et al. [17] on statistical analysis studies (search on Cambridge Structure Database results for 19921 entries with YCH_3/Ar intermolecular contacts less than 2.90 Å, where Y = any non-hydrogen atom) on the H/C interatomic distance for intermolecular YCH_3/Ar interaction. The corresponding C ... C nonbonded contact distances with values less than the sum of the van der Waals distance (3.40 Å) [16] are C3 ... C35, 3.37(2) Å and C3 ... C34, 3.14(1) Å.

In 12TAC/BNP (Figure 4b), the Coulombic interactions that exist between the chloride anion and charged nitrogen have the shortest distance of $Cl^- \ldots N^+$, being 4.12 Å (2). In this complex, the hydrogen bonds formation exists between O2 ... Cl, 3.089(3) Å, Ow ... Cl, 3.088(4) Å, and O1 ... Ow, 2.680(5) Å. The crystal structure is also stabilized by C-H ... π interactions, and the C ... H interaction observed was between C33 ... H4 with a distance of 2.911 Å and the corresponding C2 ... C33 distance is 3.372(7) Å.

In 16TAB/BNP (Fig. 4c), Coulombic interactions also occur between the bromide anion and charged nitrogen with the shortest distance of $N^+ \ldots Br^-$ being 4.84 Å(1). The hydrogen bonds are O1 ... Br, 3.205(4) Å, Ow ... Br, 3.213(6) Å, O2 ... Ow, 2.690(7) Å. The H ... C interatomic distance for the NCH_3/Ar interactions observed were C36 ... H2, 2.880 Å, and C38 ... H4, 2.773 Å. Their corresponding C ... C nonbonded contact distances are C1 ... C34 3.429(8) Å and C2 ... C38 3.28(9) Å, respectively. Similar kinds of interactions between the host and guest molecules were also observed in 16TAC/BNP [6]. In all the complexes, the C-H ... π interactions are enhanced when the C-H group of the alkyl chain lies above or

below a naphthol moiety or when the C-H group points towards a naphthol group. The shortest distance between guest-guest molecules in 12TAB/BNP, 12TAC/BNP, and 16TAB/BNP complexes are 3.79, 3.94, and 3.77 Å, respectively, while those of host-host molecules are 4.01, 4.25, and 4.60 Å. This suggests that the BNP molecules are closer to each other than those of the host molecules.

In all the complexes, the host molecules are arranged in a unique interdigitated fashion (Figs. 3a–3d) in the bent tail region of the alkyl chain when viewed in the *ac*-plane. This type of arrangement is quite different from those observed in complexes of monoalkylammonium halides with planar aromatic molecules [1,16,18–20]. The host molecules are trapped between the BNP molecules in the *ac*-plane in a similar packing fashion in all the complexes (Figs. 3a–3d). The packing mode of the BNP molecules contributes to the driving force behind the packing of the host molecules. It can be deduced from the crystal structures (Figs. 3a–3d) that the packing structure in complexes with $n = 12$ are similar just as well as those with $n = 16$. The similarities in the crystal structures can be attributed to the manner in which the BNP molecule is similarly arranged along the *a* and *c*-axes (Figs. 3a–3d) in all the complexes. The change of the counter anion in the host molecule does not change the packing structure observed in 12TAB/BNP and 12TAC/BNP or 16TAB/BNP and 16TAC/BNP complexes. On the other hand, in complexes involving amphiphilic molecules with a planar aromatic molecule, e.g., *p*-Phenylphenol (*p*-PP), the substitution of the counter anion changes the packing structure 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 ratio is 2:1, 1:1, 2:1, and 1:1, respectively [12,18].

The bent conformation in the alkyl chains could be attributed to the effect of placing a naphthol plane almost normal to the long alkyl chain axis. Thus, the nonplanar shape of the BNP molecule seems to affect the molecular packing of the alkyl chains.

CONCLUSIONS

The host molecules are sandwiched between the BNP molecules in a similar manner in all the complexes. The replacement of Br by Cl or vice-versa does not alter the packing arrangement in the nTAB/BNP and nTAC/BNP complexes ($n = 12$ or 16). The host-guest ratio (1:1) of the complexes is also consistent and does not vary even when the counter anion is changed. The nonplanar shape of the BNP molecule contributes to the bent structure of the alkyl chain. The BNP molecule adopts the

cisoid conformation in all the complexes. Hydrogen bonds and C-H $\ldots \pi$ interactions stabilize these crystal structures.

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