CRYSTAL STRUCTURE OF 2,7-DIBENZYLOXY-1,8-BIS(4-FLUOROBENZOYL)NAPHTHALENE AND THE COMPARISON WITH THE HOMOLOGUES: CONTRIBUTION OF (Ar)C–H…O=C AND (sp3)C–H…π INTERACTIONS TO THE MOLECULAR PACKING

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As a part of our structural studies of 1,8-dibenzoylnaphthalene compounds, one of the highly congested aromatic-rings-accumulated molecules, 2,7-dibenzyloxy-1,8-bis(4-fluorobenzoyl)naphthalene is designed and determined the crystal structure by X-ray crystallography. Through comparison of the single molecular structure and the molecular packing structure with the homologues bearing alkoxy/aryloxy groups (methoxy, ethoxy, butoxy, and phenoxy groups) at the 2,7-positions, the contribution of the non-bonding molecular interactions to stabilization of the molecular packing is estimated. As a common feature of single molecular structure in the series of the compounds, two aroyl groups are attached to the naphthalene ring in a nearly perpendicular fashion. In the crystal packing of these compounds, aromatic hydrogen interacts with carbonyl oxygen to stabilize the spatial organization of non-coplanarly accumulated aromatic rings replacing π…π stacking interaction of benzene rings. Especially, intermolecular (Ar)C–H…O=C interactions between the benzene rings of the aroyl groups and the carbonyl groups of the neighbouring molecules are influential. The pile superposing the naphthalene ring planes constructed mainly by this intermolecular interaction probably plays the most important role for stabilizing the crystal structure of these compounds. In addition to the (Ar)C–H…O=C interactions, butoxy-, phenoxy- and benzyloxy-bearing compounds have intermolecular (sp3)C–H…π interactions between the methylene moieties (benzene rings for phenoxy-bearing homologue) of the 2,7-substituents and the aromatic rings of the neighbouring molecules (aromatic ring: the naphthalene ring for butoxy-bearing homologue; the benzene rings for phenoxy-bearing homologue; the benzene ring of the benzyloxy group for benzyloxy-bearing homologue). Furthermore, the effective contribution of (Ar)C–H…O=C interaction depends on the kind of the 2,7-substituents. The existence of intermolecular (sp3)C–H…π interactions by 2,7-substituents is plausible to weaken the contribution of intermolecular (Ar)C–H…O=C interactions to the molecular packing. The stability of the molecular accumulation in the crystal is rationally interpreted from the viewpoint of the complementary combination of (Ar)C–H…O=C and (sp3)C–H…π interactions. The order of the total stability of the crystals of these compounds estimated on the basis of melting point and density is in good agreement of the number of these superior interactions, (Ar)C–H…O=C and (sp3)C–H…π interactions.

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Introduction

Molecules of congested non-coplanarly-accumulated aromatic rings structure such as binaphthyl[1-4] and biphenyl[5-8] have attracted the interests of the chemists in organic chemistry and material chemistry fields as unique building blocks affording characteristic optic and electronic properties in addition to construction of chiral molecular scaffold[9-13] for organic synthesis.

To integrate information about stabilization factors of crystalline accumulation state of organic molecules is significantly important as fundamental study for development of such functional organic materials.
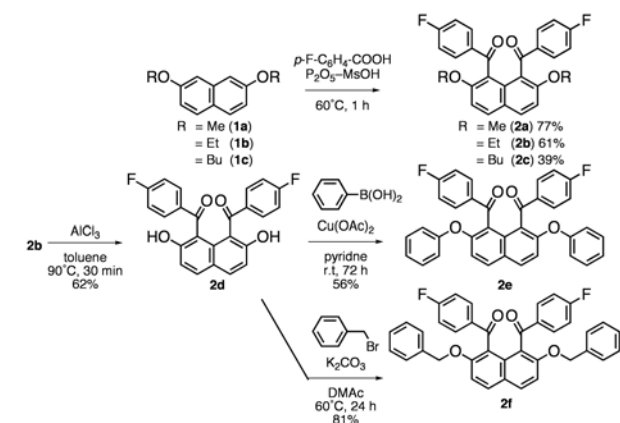
Recently, the authors have reported *peri*(1,8)-selective diaroylation of naphthalene derivatives.[14,15] According to the X-ray crystal structure analyses of the resulting 1,8-diaroylnaphthalene compounds, they have been proved to have characteristic structure of non-coplanarly aromatic-rings-accumulated structures.[16-18] As single molecular structure, two aroyl groups are attached to the naphthalene ring in an almost perpendicular fashion and oriented in an opposite direction. The highly congested circumstance leading non-coplanar spatial organization of aromatic rings accumulation of these molecules means the reduced development of conjugation inevitably resulting in relatively lower contribution of interaction originated from conjugated planes. Instead, it affords the opportunity to reveal the normally hidden interactions.[19,20]

The authors have intended to disclose the stabilization factors of non-coplanarly aromatic-rings-accumulated compounds through investigation of the spatial organization of single molecule, the molecular accumulation structure, and the non-bonding molecular interactions in 1,8-diaroylnaphthalene homologues. In the course of these

studies,[21-23] the authors have designed and synthesized 1,8-bis(4-fluorobenzoyl)-2,7-dialkoxynaphthalene compounds followed by determination of the crystal structures, e.g., 1,8-bis(4-fluorobenzoyl)-2,7-di*methoxy*naphthalene,[24] 2,7-di*ethoxy*-1,8-bis(4-fluorobenzoyl)naphthalene,[25] 2,7-di*butoxy*-1,8-bis(4-fluorobenzoyl)naphthalene,[26] and 1,8-bis(4-fluorobenzoyl)-2,7-di*phenoxy*naphthalene.[27] Herein, the authors will report crystal structure of 2,7-di*benzyloxy*-1,8-bis(4-fluorobenzoyl)naphthalene and discuss the contribution of the non-bonding molecular interactions to stabilization of the molecular packing through comparison of the crystal structures of the methoxy-, ethoxy-, butoxy-, and phenoxy-bearing homologues in detail.

Figure 1. Molecular structure of 2,7-dibenzyloxy-1,8-bis(4-fluorobenzoyl)naphthalene, showing 50% probability displacement ellipsoids.
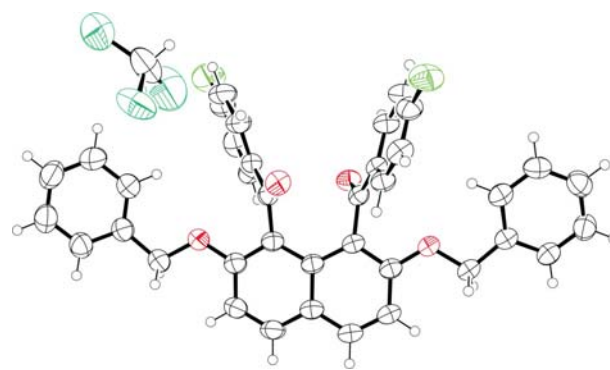
Results and Discussion

The synthetic routes of the benzyloxy-bearing compound (**2f**) and the homologues **2a**(OMe), **2b**(OEt), **2c**(OBu), and **2e**(OPh) are summarized in the Scheme. The benzyloxy-bearing compound (**2f**) was synthesized via nucleophilic substitution reaction of 1,8-bis(4-fluorobenzoyl)-2,7-dihydroxynaphthalene (**2d**) with bromomethylbenzene. The homologous 1,8-bis(4-fluorobenzoyl)naphthalene compounds bearing methoxy (**2a**), ethoxy (**2b**), butoxy (**2c**), and phenoxy-bearing (**2e**) groups at the 2,7-positions, were synthesized on the basis of literatures.[24-27]


Scheme 1. Synthesis of 2,7-dibenzyloxy-1,8-bis(4-fluorobenzoyl)naphthalene and the homologous compounds.

Figure 1 gives ORTEP representation of the molecular structure of the benzyloxy-bearing compound (**2f**), as determined by the structured X-ray analysis.[28] In the benzyloxy-bearing compound (**2f**), two benzoyl groups at the 1,8-positions of the naphthalene ring system are aligned almost antiparallel, the naphthalene ring system makes dihedral angles of 71.25(9)° and 70.09(8)° with the benzene rings of the aroyl groups. The benzene rings of the benzyloxy groups are oriented in a perpendicular fashion to the benzene ring of the neighbouring aroyl groups [dihedral angles = 88.46(13)° and 87.06(12)°]. The benzene rings of the benzyloxy groups are connected with the benzene rings of the neighbouring aroyl groups by *intra*molecular C–H…π interactions [2.89 Å and 2.97 Å]. Consequently, the dihedral angle between the benzene ring of the benzyloxy group and the benzene ring of the aroyl group is closer to 90° than that between the benzene ring of the aroyl group and the naphthalene ring.
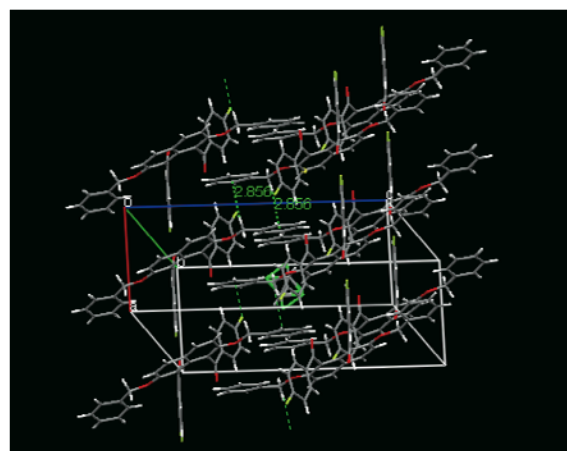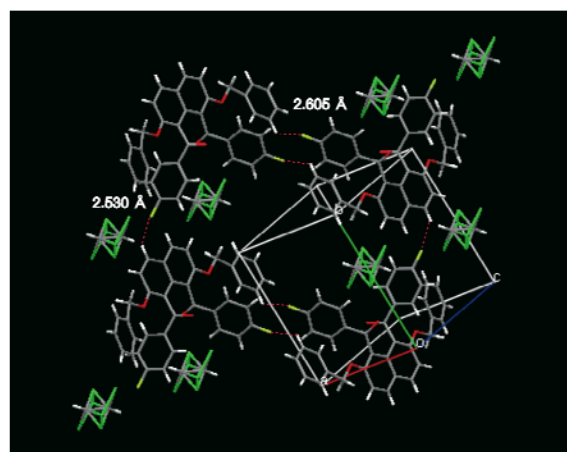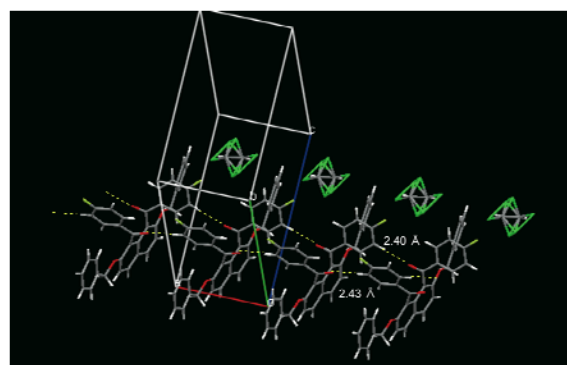
Figure 2. Observed molecular interactions of 2,7-dibenzyloxy-1,8-bis(4-fluorobenzoyl)naphthalene: C–H…O=C interactions between aromatic C–Hs and carbonyl groups (top), two types of C–H…F interactions (middle), and C–H…π interactions between methylene C–Hs and benzene rings of benzyloxy groups (bottom).
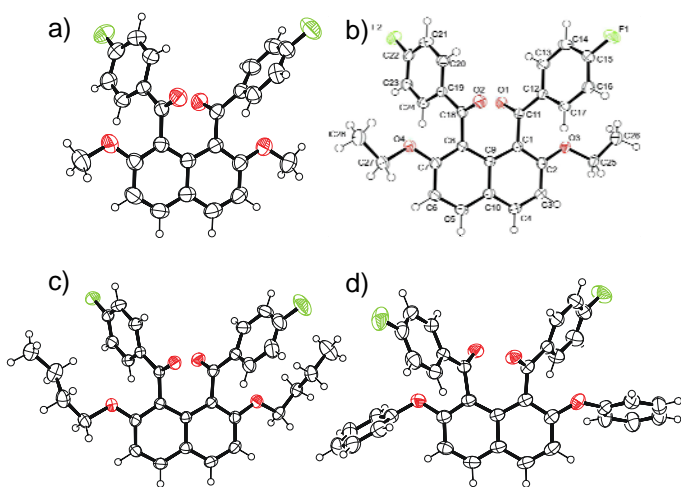
The chloroform molecule is disordered over two positions with site occupancies of 0.50 and 0.50.

In the crystal, three kinds of intermolecular interactions are observed, i.e., C–H…O=C interaction, C–H…F interaction, and C–H…π interaction. The duplicate C–H…O=C interactions between the benzene rings of the aroyl groups and the carbonyl groups arrange the molecules along *a*-axis (**Figure 2**, top).

The C–H…F interactions make molecular alignments along *bc*-diagonal (**Figure 2**, middle). The C–H…F interactions between the naphthalene rings and fluoro groups (2.530 Å) are formed between same enantiomeric isomers, whereas the duplicate C–H…F interactions between the benzene rings and the fluoro groups (2.605 Å) are connected with the counterpart isomers.
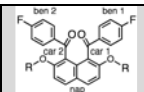
Furthermore, the C–H…π interactions between the methylene moieties and the benzene rings of the benzyloxy groups stack the benzyloxy groups into columnar structure along *a*-axis [C–H…*Cg* distance = 2.856 Å] (**Figure 2**, bottom).

These structural features of the benzyloxy-bearing compound (**2f**) are compared with the homologues. **Figure 3** displays ORTEP representation structures of homologues **2a**(OMe), **2b**(OEt), **2c**(OBu), and **2e**(OPh). **Table 1** shows the selected dihedral angles in the benzyloxy-bearing molecule and the homologues. The spatial structures of all compounds are apparently similar. The benzene rings of the aroyl groups are non-coplanarly situated to the naphthalene rings. However, these molecules are different each other from the viewpoint of the spatial symmetry. The difference in the dihedral angles between the benzene rings of the aroyl groups and the naphthalene ring in the molecule is smaller in the order of molecules **2a**(OMe), **2b**(OEt), **2f**(OBn), **2e**(OPh), and **2c**(OBu). On the other hand, the dihedral angles between the benzene rings are larger with decreasing of the difference, i.e., **2b**(OEt) < **2a**(OMe) < **2f**(OBn) < **2c**(OBu) < **2e**(OPh).

Table 1. Selected dihedral angles of molecules **2a**, **2b**, **2c**, **2e**, and **2f**

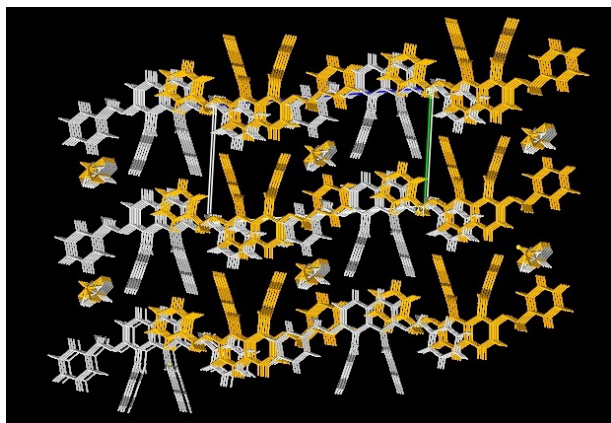|	2a (OMe)	2b (OEt)	2f (OBn)	2e (OPh)	2c (OBu)
nap–ben1	66.87	70.24	71.23	72.07	74.54
nap–ben2	88.09	67.28	70.10	73.24	74.40
nap–ben3	-	-	20.33	62.49	-
nap–ben4	-	-	13.90	77.96	-
ben1–ben2	32.34	14.12	36.66	49.01	44.60
ben1–ben3	-	-	88.46	54.78	-
ben1–ben4	-	-	75.16	48.25	-
ben2–ben3	-	-	87.06	80.54	-
ben2–ben4	-	-	81.38	86.72	-
ben3–ben4	-	-	20.54	44.35	-
car1-nap	72.02	69.33	60.67	62.87	65.70
car2-nap	64.90	67.16	62.56	65.79	63.20
ben1-car1	14.55	2.89	14.96	17.58	12.42
ben2-car2	16.39	2.441	9.35	16.70	19.30

The abbreviations displayed in the left column are designated as aromatic ring moieties in molecules **2a**, **2b**, **2c**, **2e**, and **2f**, which are indicated on the molecular figure at the upper left of this Table.

Benzyloxy-bearing molecule **2f** and the homologue **2e**(OPh) have benzene rings in the 2,7-substituents. In the case of the benzyloxy-bearing molecule (**2f**), the benzene rings of the benzyloxy group are symmetrically situated against the benzene rings of the aroyl groups. Homologues **2a**(OMe) and **2e**(OPh) have apparently distinguishable structural feature from the others in view of the symmetry of the spatial property. Single molecular structure of compounds **2c**(OBu) and **2f**(OBn) can be regarded as a rather simple extension for homologue **2b**(OEt). Moreover, compound **2f**(OBn) seems to be more similar to compound **2c**(OBu) than compound **2b**(OEt) from the viewpoint of the spatial organization of single molecule.

Figure 4 and **Figure 5** show the crystal packing structures of the benzyloxy-bearing compound (**2f**) and the homologues when the naphthalene rings are placed in parallel fashion against the paper. Molecules are color-coded according to symmetry operation. There are two different types of atrope isomeric situations of the aroyl groups against the naphthalene ring, i.e., *R,R*-isomer and *S,S*-one. Pink-colored molecules and yellow-colored ones express *R,R*-isomers, whereas white-colored molecules and green-colored ones display *S,S*-isomers. The overlapping of *R,R*-isomers and *S,S*-ones seems to become smaller in the order of compounds **2a**(OMe), **2e**(OPh), **2b**(OEt), **2f**(OBn), and **2c** (OBu). In the cases of compounds **2b**(OEt), **2c**(OBu), and **2f**(OBn), column structure composed of same enantiomeric isomers seems to interpenetrate with the neighbouring columns composed of the counterpart isomers. These results are supposed the authors that molecules **2a**(OMe) and **2e**(OPh) form intermolecular interactions between *R,R*-isomer and *S,S*-isomer, whereas molecules **2b**(OEt), **2c**(OBu), and **2f**(OBn) have interactions between same enantiomeric isomers and those between the counterpart isomers in the crystals.

Figure 3. Molecular structures of 1,8-bis(4-fluorobenzoyl)naphthalene homologues bearing alkoxy/aryloxy groups at the 2,7-positions, showing 50% probability displacement ellipsoids: a) molecule **2a** (OMe), b) molecule **2b** (OEt), c) molecule **2c** (OBu), d) molecule **2e** (OPh).

The observed non-bonding interactions in the molecular packing of the compounds are summarized in **Table 2**. The underlined C–H…X designates the highlighted interactions elucidated on the basis of shorter distance of two atoms less than the sum of the van der Waals radii. In the case of C–H…π interactions, the distances less than 3.0 Å are regarded as effective interactions. As the common features in these molecular packings, two kinds of non-bonding molecular interactions are observed, i.e., intermolecular $_{(Ar)}$C–H…O=C interactions between the carbonyl groups and the benzene rings of the aroyl groups (or the naphthalene ring) and intermolecular $_{(sp^3)}$C–H…π interactions between the methylene moieties of the 2,7-substituents and the naphthalene rings (or $_{(Ar)}$C–H…π interactions between the benzene rings of phenoxy groups). The former interactions are observed in all compounds. The latter interactions are observed in compounds **2c**(OBu), **2e**(OPh), and **2f**(OBn). The intermolecular $_{(Ar)}$C–H…O=C interactions are observed between *R,R*-isomers and *S,S*-isomers in compounds **2a** (OMe) and **2e**(OPh), whereas the interactions are formed between same enantiomeric isomers giving columnar structures for compounds **2b**(OEt), **2c**(OBu), and **2f**(OBn). The intermolecular C–H…π interactions are observed as the interpenetration of columns in compounds **2c**(OBu), **2e**(OPh) and **2f**(OBn). The intermolecular $_{(Ar)}$C–H…O=C interactions are weakened in the order of compounds **2b** (OEt) > **2e**(OPh), **2f**(OBn) > **2a**(OMe) >> **2c**(OBu) on the basis of distance between H and O atoms. On the other hand, the intermolecular $_{(sp3)}$C–H…π distances in compound are strengthened in the order of compounds **2c**(OBu) > **2f** (OBn) > **2e**(OPh). Therefore, the $_{(Ar)}$C–H…O=C interactions and $_{(sp3)}$C–H…π ones are regarded in complementary relationship for compounds **2c** (OBu), **2e**(OPh), and **2f**(OBn). Moreover, *intra*molecular C–H…π interactions between the benzene rings of the aroyl groups and the 2,7-substituents are formed in molecules **2c**(OBu) and **2f**(OBn).

F**igure 4.** Molecular packing of 2,7-dibenzyloxy-1,8-bis(4-fluorobenzoyl) naphthalene compounds, viewed down *a*-axis.

The melting points lowers in the order of compounds **2b** (OEt) > **2a**(OMe)>> **2e**(OPh) >> **2f**(OBn) >> **2c**(OBu). The melting points of compounds **2b**(OEt) and **2a**(OMe) are nearly equal, however, there are three drastic droppings in the melting points between compounds **2a**(OMe) and **2e** (OPh), between compounds **2e**(OPh) and **2f**(OBn), and between compounds **2f**(OBn) and **2c**(OBu). The first dropping between compounds **2a**(OMe) and **2e**(OPh) indicates that existence of C–H…π interaction weakens the contribution of intermolecular C–H…O=C interaction to stabilize the molecular packing.

a)

b)

c)

d)
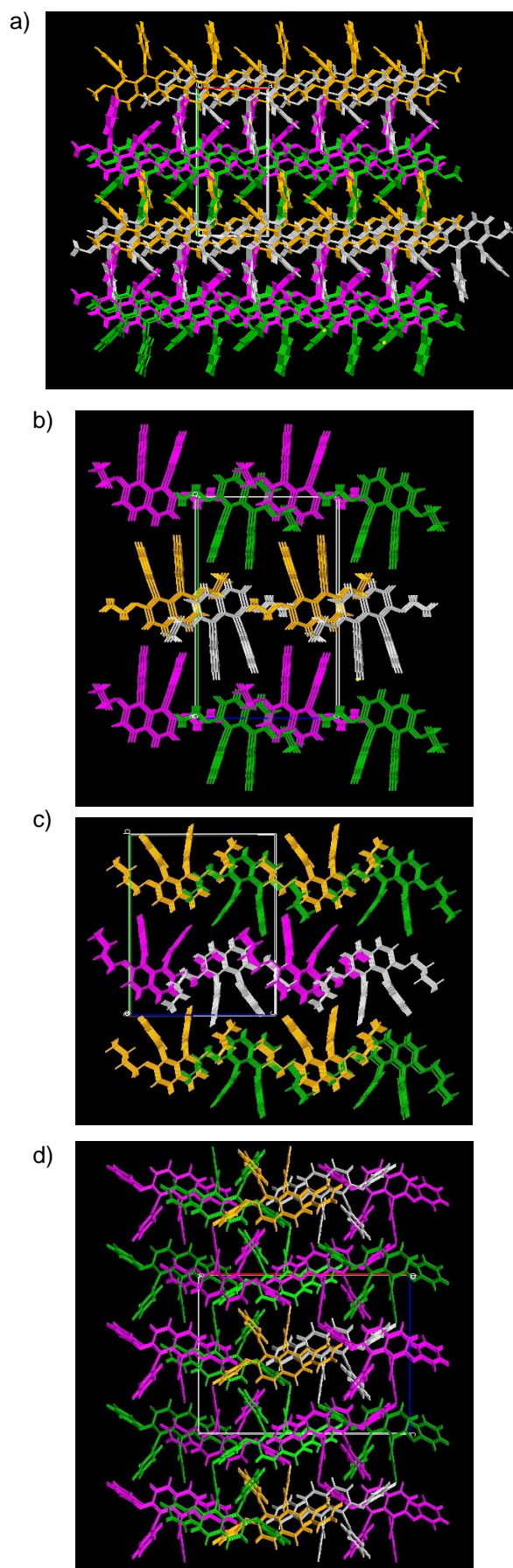
Figure 5. Molecular packing of 1,8-bis(4-fluorobenzoyl)naphthalene compounds bearing alkoxy groups at 2,7-positions: a) compound **2a** (OMe), b) compound **2b** (OEt), c) compound **2c** (OBu), d) compound **2e** (OPh).

Table 2. Non-bonding distances in molecules **2a**, **2b**, **2c**, **2e**, and **2f** with the melting point and the density

C–H...X	2b (OEt)	2a (OMe)	2e (OPh)	2f (OBn)	2c (OBu)
between same isomers					
C–H$_{Ar}$…O=C$_{car1}$	2.30	-	-	2.40	-
C–H$_{Ar}$…O=C$_{car2}$	2.37	-	-	2.43	2.70
C–H$_{nap}$…F$_1$	2.44	-	-	2.53	2.66
C–H$_{nap}$…F$_2$	-	-	-	-	2.50
between the counterpart isomers					
C–H$_{Ar}$…O=C$_{car1}$	-	2.54, 2.69	-	-	-
C–H$_{nao}$…O=C$_{car1}$	-	-	2.40	-	-
C–H$_{sp3}$…O=C$_{car1}$	-	-	-	-	2.64
C–H$_{Ar}$…π$_{2,7-Ar}$	none	none	2.80	-	none
C–H$_{sp3}$… π$_{2,7-Ar}$	none	none	-	2.86	none
C–H$_{sp3}$…π$_{nap}$	-	-	-	-	2.54
C–H$_{Ar}$…F$_2$	-	-	-	2.60	-
intramolecular					
C–H$_{sp3}$…π$_{1,8-Ar}$	-	-	-	-	2.79
C–H$_{2,7-Ar}$…π$_{1,8-Ar}$	none	none	-	2.89	none
C–H$_{2,7-Ar}$…π$_{1,8-Ar}$	none	none	-	2.97	none
M.p (˚C)	210	195	168	127	92
Density (g cm³)	1.361	1.427	1.348	1.36	1.261

The second dropping between compounds **2e**(OPh) and **2f**(OBn) suggests that the contribution of the $_{(sp3)}$C–H…π interactions to the molecular packing competes with that of the $_{(Ar)}$C–H…O=C interactions. The third dropping between compounds **2f**(OBn) and **2c**(OBu) is essentially regarded as a continuation of the second dropping between compounds **2e**(OPh) and **2f**(OBn). In consequence of this, the *intra*molecular interaction character shown as *intra*molecular $_{(sp3)}$C–H…π interaction or $_{(2,7-Ar)}$C–H…π interaction seems to be more strengthened relatively to the intermolecular interaction character in the molecular packing of compounds **2f**(OBn) and **2c**(OBu). The number of C–H…F interactions seems to increase with emphasizing of the *intra*molecular interaction character. The relationship between melting point and molecular interactions is also in agreement with the density.

In 1,8-bis(4-fluorobenzoyl)naphthalene homologues, two aroyl groups are situated essentially perpendicularly against the naphthalene ring. The non-coplanarly accumulated-aromatic-rings structure indicates that 1,8-bis(4-fluorobenzoyl)naphthalene homologues avoid the internal steric repulsions *intra*molecularly in the first place. Furthermore, intermolecular $_{(Ar)}$C–H…O=C interactions are observed in the crystal packing of all of the compounds. The difference in the dihedral angles of two aroyl groups with the naphthalene ring is larger in the order of homologues **2a**(OMe) < **2b**(OEt) < **2f**(OBn) < **2e**(OPh) < **2c** (OBu). The order apparently correlates with the $_{(Ar)}$C–H…O=C distances. The relationship between the dihedral angle of the benzene rings and the $_{(Ar)}$C–H…O=C distance makes the authors naturally envision that formation of intermolecular $_{(Ar)}$C–H…O=C interactions is disturbed with increasing the *intra*molecular repulsion by replacing the 2,7-substituents. When the 2,7-substituents form effective interactions such as intermolecular $_{(sp3)}$C–H…π interactions, their contribution to the molecular packing precedes that of intermolecular $_{(Ar)}$C–H…O=C interactions. In the case of methoxy-bearing compound (**2a**), the melting point is high as same as ethoxy-bearing compound (**2b**), whereas the $_{(Ar)}$C–H…O=C interactions is for weaker than ethoxy-bearing compound (**2b**). Small size of the methoxy groups at the 2,7-positions presumably allows the molecule to be in close proximity to the adjacent molecules. Under these circumstances, weak van der Waals interactions cooperatively contribute to stabilize the molecular packing. In consequence of this, alternative arrangement of *R,R*-isomer and *S,S*-one in compound **2a**(OMe) thus obtained might be sticky packed as suggested in density in Table 2.

Conclusively, the crystal structure of 2,7-dibenzyloxy-1,8-bis(4-fluorobenzoyl) naphthalene is determined. Through the comparison of the methoxy-, ethoxy-, butoxy-, and phenoxy group bearing homologues, 1,8-bis(4-fluorobenzoyl)naphthalene compounds bearing 2,7-dialkoxy/diaryloxy groups are analyzed to elucidate the governing factors that determine the structural feature in crystal. For these non-coplanarly aromatic-rings-accumulated compounds, the single molecular structure and the molecular packing feature are well interpreted that the whole stability is governed by intermolecular $_{(Ar)}$C–H…O=C and $_{(sp3)}$C–H…π interactions instead of π…π stacking interaction. The *intra*molecular avoidance of the steric hindrance at the first place presumably determines the single molecular structure. The *intra*molecular $_{(sp3)}$C–H…π interaction or $_{(2,7-Ar)}$C–H…π interaction contributes single molecular structure more stable. In other words, absence of *intra*molecular $_{(sp3)}$C–H…π interaction or $_{(2,7-Ar)}$C–H…π interaction in single molecular structure directly means larger contribution or emphasis of the intermolecular interaction character in the molecular accumulation structure. The mode of overlapping feature to give columnar structure is also determined at the same manner. Under the allowance of above requisites, the $_{(Ar)}$C–H…O=C interactions are placed to the maximum number in the crystalline molecular accumulation. This explanation is in good agreement with the melting point and the density of these crystals.

Experimentals

All reagents were of commercial quality and were used as received. Solvents were dried and purified using standard techniques.[29] 2,7-dimethoxynaphthalene (**1a**) and 2,7-diethoxynaphthalene (**1b**) were prepared according to literatures.[30, 31]

Measurements

^{1}H NMR spectra were recorded on a JEOL JNM-AL300 spectrometer (300 MHz) and a JEOL ECX400 spectrometer (400 MHz). Chemical shifts are expressed in ppm relative to internal standard of Me$_4$Si (δ 0.00). ^{13}C NMR spectra were recorded on a JEOL JNM-AL300 spectrometer (75 MHz). Chemical shifts are expressed in ppm relative to internal standard of CDCl$_3$ (δ 77.0). IR spectra were recorded on a JASCO FT/IR-4100 spectrometer. Elemental analyses were performed on a Yanaco CHN CORDER MT-5 analyzer.

High-resolution FAB mass spectra were recorded on a JEOL MStation (MS700) ion trap mass spectrometer in positive ion mode.

X-ray Crystallography

For the crystal structure determination, the single-crystal of the compound **2e** was used for data collection on a four-circle Rigaku RAXIS RAPID diffractometer (equipped with a two-dimensional area IP detector). The graphite-mono-chromated Cu Kα radiation (λ = 1.54187 Å) was used for data collection. The lattice parameters were determined by the least-squares methods on the basis of all reflections with $F^2 > 2\sigma(F^2)$. The data collection and cell refinement were performed using *PROCESS-AUTO* software. The data reduction was performed using *CrystalStructure*. The structures were solved by direct methods using *SIR2004* and refined by a full-matrix least-squares procedure using the program *SHELXL97*. All H atoms were found in a difference map and were subsequently refined as riding atoms, with the aromatic C–H = 0.95 Å and methyl C–H = 0.98 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Synthesis of 2,7-dibenzyloxy-1,8-bis(4-fluorobenzoyl) naphthalene (2f)

To a 10 mL flask, 1,8-bis(4-fluorobenzoyl)-2,7-dihydroxynaphthalene (**2d**, 1.0 mmol, 404 mg), benzylbromide (5.0 mmol, 855 mg), potassium carbonate (5.0 mmol, 691 mg), DMAc (2.0 mL) were placed. The reaction mixture was stirred at 60 °C for 24 h. After the reaction, the mixture was extracted with AcOEt. The combined extracts were washed with brine. The organic layers thus obtained were dried over anhydrous MgSO₄. The solvent was removed under reduced pressure to give cake. The crude product was purified recrystallization from CHCl₃–MeOH (81% isolated yield). Furthermore, the isolated product was crystallized from methanol to give single-crystal.

^{1}H NMR δ (300 MHz, CDCl₃) : 5.00 (4H, s), 6.89-6.92 (4H, m), 7.01 (4H, t, *J*= 8.2 Hz) 7.16-7.20 (6H, m), 7.22 (2H, d, *J*= 8.9 Hz), 7.76 (4H, dd, *J*= 8.2, 5.4 Hz), 7.91 (2H, d, *J*= 8.9 Hz) ppm; ^{13}C NMR δ (75 MHz, CDCl₃) :70.81, 112.32, 115.13 (d, $^2J_{C-F}$= 22.4 Hz), 121.60, 125.70, 126.59, 127.71, 128.26, 130.24, 131.70 (d, $^3J_{C-F}$= 9.3 Hz), 132.25, 135.47 (d, $^4J_{C-F}$= 2.8 Hz), 136.10, 155.45, 165.56 (d, $^1J_{C-F}$= 253.6 Hz), 195.71 ppm; IR (KBr) : 1656 (C=O), 1595, 1505, 1454 (Ar, naphthalene), 1236 (=C−O−C) cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₃₈H₂₇F₂O₄, 585.1877 found, 585.1854. m.p.=127˚C

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