

Crystal Structure and Solution Structural Dynamic Feature of 1,8-Dibenzoyl-2,7-Dimethoxynaphthalene

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ABSTRACT

The crystal structure and the dynamic feature of molecular structure in solution for 1,8-dibenzoyl-2,7-dimethoxynaphthalene are revealed by X-ray crystallographic analysis and VT-NMR measurements. In crystal, the molecule of the title compound is located on a twofold rotation axis. The two benzoyl groups are situated in an opposite direction. The dihedral angle between the mean planes of the phenyl ring and the naphthalene ring system is $80.25(6)^\circ$. The benzene ring and carbonyl moiety in each benzoyl group are almost coplanar. The molecular packing is stabilized by weak C–H...O hydrogen bonds and a π - π stacking interaction between the benzene rings [centroid-centroid and interplanar distances of 3.6383(10) and 3.294 Å, respectively]. In solution, the temperature-dependent rotation behavior of the C–C bond between the benzene ring and the ketonic carbonyl group has been observed by ^1H VT-NMR measurements. Furthermore, comparison of the C–C bond rotation behavior between the benzene ring and the carbonyl group with 1-benzoyl-2,7-dimethoxynaphthalene has clarified that the C–C bond between the ketonic carbonyl group and the naphthalene ring rotates slower than the 1,8-dibenzoylated homologue.

Keywords: Non-Coplanarly Accumulated Aromatic Rings Molecules; Crystal Structural Features; Bond Rotation Behavior in Solution; X-Ray Crystallography; VT-NMR Spectroscopy

1. Introduction

Non-coplanarly accumulated aromatic rings molecules, such as binaphthyl and biphenyl compounds, have attracted significant attention because of their characteristic properties, unique shapes, and various applications [1-7]. *Peri*-substituted naphthalenes have also received much attention as unique structured aromatic core compounds for variety of the functional materials [8-12]. Therefore, the structural analyses have been actively performed [13-15]. Recently, the authors' group has revealed that diaroxylation at 1,8-positions of 2,7-dimethoxynaphthalene smoothly proceeds [16,17]. According to X-ray crystal structural study, the obtained 1,8-diaroynaphthalene has unique non-coplanar alignment of aromatic rings [18]. The curious reversible aroylation behavior of the naphthalene derivative [16] and chemospecific and regioselective ethereal alkyl-oxygen bond cleavage reaction of aroylated naphthalenes [19] can be explained on the basis of the structural features of the aroylated naphthalenes. Under these circumstances, the authors have undertaken the structural studies of the aroylnaphthalene compounds [20-26] with investigation of the formation and the related reaction behaviors.

In this article, the crystallographical structural charac-

teristics and the dynamic feature in solution structure of a 1,8-diaroxygenated naphthalene derivative having two methoxy groups at the 2,7-positions are described as the most simple homologue of monoaroxygenated derivative.

2. Experimental

All reagents were of commercial quality and were used as received. Solvents were dried and purified using standard techniques. Pentoxide-methanesulfonic acid (P_2O_5 -MsOH) was prepared according to literature [27].

2.1. Measurements

^1H 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_4Si (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. High-resolution FAB mass spectra were recorded on a JEOL MStation (MS700) ion trap mass spectrometer in positive ion mode.

2.2. Synthetic Procedure of the Title Compound

The title compound was synthesized via direct condensation mediated by P_2O_5 -MsOH of 2,7-dimethoxynaphthalene with benzoic acid (**Scheme 1**). To a mixture of 2,7-dimethoxynaphthalene (0.200 mmol, 37.6 mg) and benzoic acid (0.440 mmol, 174 mg), P_2O_5 -MsOH (0.88 mL) was added by portions at rt. After the reaction mixture was stirred at 60 °C for 3 h, it was poured into iced water (20 mL) and the mixture was extracted with $CHCl_3$ (15 mL \times 3). The combined extracts were washed with 2 M NaOH aq and followed by sat NaCl aq. The organic layers thus obtained were dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure to give powdery product. Isolation of the title compound was carried out by column chromatography [hexane: AcOEt = 2:1] (1,8-diaroylnaphthalene 63%; 3-monoaroylnaphthalene 19%; 1-monoaroylnaphthalene 3%). Colorless single crystals suitable for X-ray diffraction were obtained by recrystallization from ethanol.

1,8-benzoyl-2,7-dimethoxynaphthalene: Colorless needle (EtOH); m.p. = 530 K; IR (KBr): 1665, 1626 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): 3.68 (6H, s), 7.21 (2H, d, J = 9.2 Hz), 7.34 (4H, dd, J = 7.6, 7.6 Hz), 7.49 (2H, t, J = 7.4 Hz), 7.70 (4H, d, J = 7.4 Hz), 7.95 (2H, d, J = 9.2 Hz) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): 56.40, 111.24, 121.47, 125.55, 127.95, 129.09, 129.84, 132.03, 132.64, 138.61, 156.28, 196.875 ppm. The above melting point and spectral data are compatible with the literature [28].

1-benzoyl-2,7-dimethoxynaphthalene: Colorless plate (Hexane + methylenechloride); m.p. = 358.5 - 362 K; IR (KBr): 1663, 1627 cm^{-1} ; 1H NMR δ (400 MHz, $CDCl_3$): 3.71 (3H, s), 3.79 (3H, s), 6.80 (1H, d, J = 2.8 Hz), 7.01 (1H, dd, J = 9.2, 2.8 Hz), 7.17 (1H, d, J = 9.2 Hz), 7.43 (2H, t, J = 8.0 Hz), 7.57 (1H, t, J = 8.2 Hz), 7.72 (1H, d, J = 8.0 Hz), 7.84 - 7.89 (3H, m) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): 55.09, 56.24, 102.02, 110.17, 117.01, 121.67, 124.29, 128.48, 129.44, 129.62, 130.96, 132.98, 133.31, 137.98, 154.93, 158.77, 198.07 ppm; HRMS (FAB; *m*-nitrobenzyl alcohol [*m*-NBA]) m/z : $[M + H]^+$; Calcd for $C_{19}H_{17}O_3$, 293.3365; found, 293.1185.

2.3. X-Ray Crystallography

For the crystal structure determination, the single-crystal



Scheme 1. Synthetic reaction of the title compound: P_2O_5 -MsOH mediated direct condensation of 2,7-dimethoxynaphthalene with benzoic acid.

of the compound $C_{26}H_{20}O_4$ was used for data collection on a four-circle Rigaku R-Axis RAPID diffractometer (equipped with a two-dimensional area IP detector). The graphite-mono-chromated Cu $K\alpha$ radiation ($\lambda = 1.54187$ 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* [29] software. The data reduction was performed using *CrystalStructure* [30]. The structures were solved by direct methods using *SIR2004* [31] and refined by a full-matrix least-squares procedure using the program *SHELXL97* [32]. 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 $U_{iso}(H) = 1.2U_{eq}(C)$. Molecular structure of the compound showing the atomic numbering scheme is shown in **Figure 1**. The crystallography details for the structures determination of the compound are displayed presented in **Table 1**.

2.4. Variable Temperature NMR

Variable temperature 1H NMR spectra were recorded on an FT-NMR operating at 400 MHz. Chemical shift values were reported in parts per million (ppm) relative to $(CH_3)_4Si$ (TMS). The solvent used in all cases was $CDCl_3$ and CS_2 (1:4 v/v) solution. Low temperature spectra were obtained with the use of a JEOL cooling system.

3. Results and Discussion

The title compound was synthesized via direct condensation

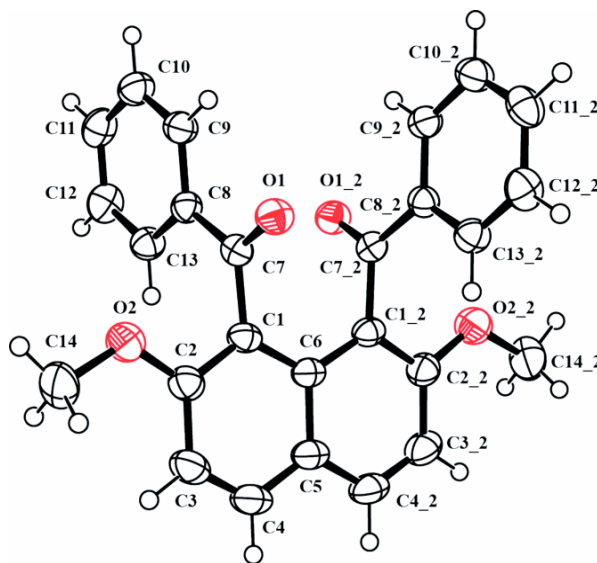


Figure 1. Molecular structure of the title compound, with the atom-labeling scheme and displacement ellipsoids drawn at the 50% probability level. The symbol “_2” refers to symmetry code: $-x, y, -z + 1/2$.

mediated by P₂O₅—MsOH of 2,7-dimethoxynaphthalene with benzoic acid (see Experimental section). **Table 1** shows the crystallographic data of the title compound. **Table 2** shows selected bond lengths and angles. **Table 3** gives selected torsion angles. **Figure 1** gives ORTEP representation of the molecular structure of the title compound, as determined by the structured X-ray analysis [33]. The molecule of the title compound lies across a crystallographic 2-fold axis so that the asymmetric unit contains one half of the molecules. Thus, the two benzoyl groups are situated in an opposite direction (anti-orientation). The benzoyl groups are twisted away from the naphthalene moiety, and the dihedral angle is 80.25(6)°. The torsion angle between the carbonyl group and the naphthalene ring is -76.73(16)° [C6—C1—C7—O1] and that between the carbonyl group and the phenyl group is 179.76(13)° [O1—C7—C8—C13].

Table 1. Crystallographic data and structure refinement parameters.

Empirical formula	C ₂₆ H ₂₀ O ₄
Formula weight	396.43 g·mol ⁻¹
Crystal shape, colour	Needle, colorless
Temperature	93(2) K
Wavelength	1.54187 Å
Crystal system	Monoclinic
Space group	C 2/c
Unit cell dimensions	a = 13.9677 (4) Å b = 10.2145 (3) Å c = 14.6966 (4) Å β = 109.711(2)°
Volume	1973.95 (10) Å ³
Z, calculated density	4, 1.334 Mg·m ⁻³
Absorption coefficient	0.72 mm ⁻¹
F(000)	832
Crystal size	0.50 × 0.10 × 0.10 mm
Theta range for data collection	3.2° to 68.1°
Limiting indices	-16 ≤ h ≤ 16 -12 ≤ k ≤ 12 -17 ≤ l ≤ 17
Reflections collected/unique	17362/1807 [Rint = 0.027]
Completeness to theta = 68.21°	99.6%
Max. and min transmission	0.930 and 0.838
Refinement method	Full-matrix least-squares on F ²
Data/restraints/parameters	1807/0/139
Goodness-of-fit on F ²	1.08
Final R indices [I > 2 sigma (I)]	R1 = 0.039, wR2 = 0.107
R indices (all data)	R1 = 0.047, wR2 = 0.115
Largest diff. peak and hole	0.19 e·Å ⁻³ and -0.21 e·Å ⁻³

Table 2. Selected bond lengths (Å) and angles (°).

O1—C7	1.2197 (16)	C2—C1—C7	115.70 (13)
C1—C2	1.382 (2)	C6—C1—C7	123.36 (13)
C1—C6	1.4264 (17)	O1—C7—C8	121.63 (13)
C1—C7	1.5158 (19)	O1—C7—C1	118.49 (12)
C7—C8	1.4814 (19)	C8—C7—C1	119.88 (12)
C8—C13	1.3908 (19)	C13—C8—C7	122.02 (13)
C8—C9	1.396 (2)	C9—C8—C7	118.88 (13)

Table 3. Selected torsion angles (°).

O1—C7—C8—C13	179.76 (13)
O1—C7—C8—C9	0.4 (2)
C2—C1—C7—O1	97.99 (16)
C6—C1—C7—O1	-76.73 (16)

In the crystal structure, the molecular packing of the title compound is mainly stabilized by van der Waals interaction. In addition, the packing of the molecule is stabilized by relatively weak C—H...O hydrogen bonding, namely, C12—H12...O1ⁱ [symmetry code: i) x, -y + 1, z + 1/2], C14—H14B...O1ⁱⁱ [symmetry code: ii) -x + 1/2, y - 1/2, -z + 1/2], and a π-π stacking interaction [centroid-centroid and interplanar distances of 3.6383 (10) and 3.294 Å, respectively]. In the packing, the molecules are arranged by C—H...O hydrogen bonding along the c axis of the unit cell, and by a π-π stacking interaction perpendicular the bc plane of the unit cell (**Figures 2** and **3**). The nonbonding distances are listed in **Table 4**.

The dynamic behavior of the title compound in solution was estimated by variable temperature NMR (VT-NMR) measurement performed in chloroform-*d* and CS₂ (1:4, v/v) solution from 293 to 173 K. **Figure 4(a)** shows the ¹H VT-NMR spectra of the title compound in the aromatic region. At 293 K, two signals of δ 7.21 and 7.95 ppm are assigned the protons at 3(6)- and 4(5)-positions of the naphthalene ring, respectively. The signals of δ 7.34, 7.49, and 7.70 ppm are assigned to the protons at *m*-positions, *p*-position, and *o*-positions of benzoyl groups. The signals of δ 7.21 and 7.95 ppm (at 293 K) are scarcely changed from 293 to 173 K. On the other hand, the figures of the three signals of δ 7.34, 7.49, and 7.70 ppm (at 293 K) are drastically changed. The signal of δ 7.49 ppm (at 293 K) is broadened from 293 to 233 K then sharpened from 213 to 173 K. The signals of δ 7.34 and 7.70 ppm (at 293 K) are broadened from 293 to 213 K and each of them splits into two broad signals at 193 K. The both of the pairs of broad signals are sharpened again at 173 K.

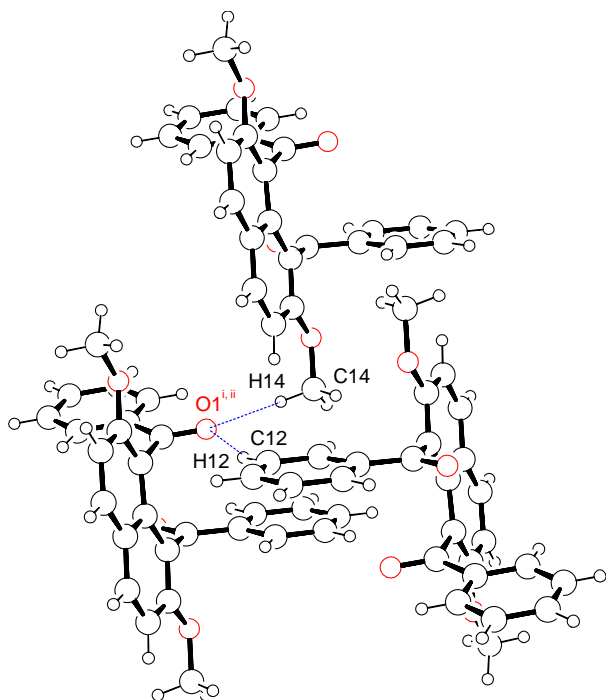


Figure 2. C—H...O interactions of methoxy group and benzene ring with carbonyl group [Symmetry codes: (i) $x, -y + 1, z + 1/2$; (ii) $-x + 1/2, y - 1/2, -z + 1/2$].

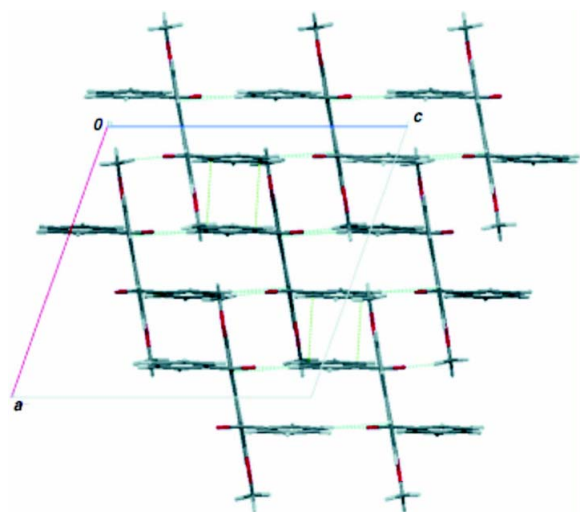


Figure 3. A partial packing diagram of the title compound, viewed down the b axis. The dashed lines indicate hydrogen bonds (blue dashed lines) and π - π stacking interactions (green lines).

Table 4. Nonbonding distances and related geometrical parameters in 1,8-dibenzoyl-2,7-dimethoxynaphthalene ($^\circ$).

D—H...A	D—H (Å)	H...A (Å)	D...A (Å)	D—H...A (deg.)
C12—H12...O1 ⁽ⁱ⁾	0.95	2.60	3.4987 (19)	159
C14—H14B...O1 ⁽ⁱⁱ⁾	0.98	2.39	3.344 (2)	164

Symmetry codes: (i) $x, -y + 1, z + 1/2$; (ii) $-x + 1/2, y - 1/2, -z + 1/2$.

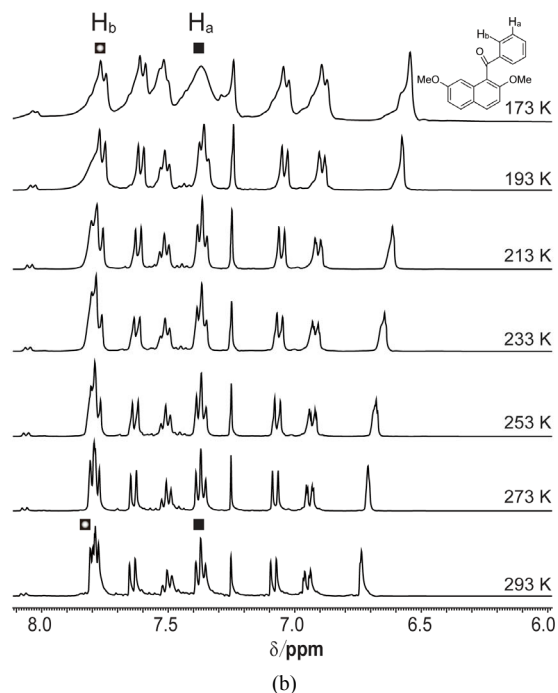
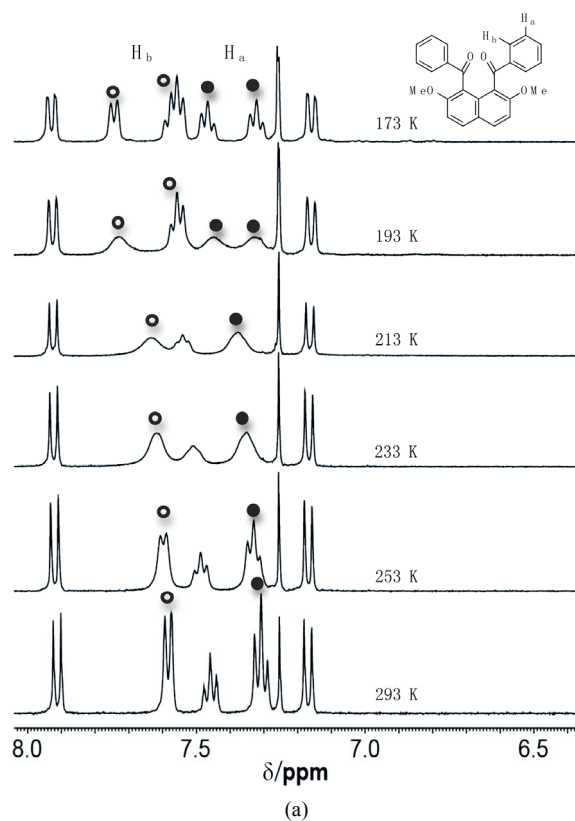


Figure 4. Variable-temperature ^1H NMR study in $\text{CDCl}_3/\text{CS}_2$ (1:4 v/v): (a) the title compound (6.5 - 8.0 ppm); (b) 1-benzoyl-2,7-dimethoxynaphthalene (6.0 - 8.0 ppm).

The sequent signal changes for the protons at o - and m -positions of the benzoyl group as shown in the VT-NMR spectra of the title compound suggest that the C—C

bond rotation between benzene ring and ketonic carbonyl group is gradually slowed down with decreasing temperature then the magnetically non-equivalent environment is made on the benzene ring. The rotation barrier of the C(benzene)–C(carbonyl) bond was calculated as $\Delta G = 9.63$ kcal/mol on the basis of the detailed VT-NMR measurements (**Figure 5**) [34].

In a similar manner, VT-NMR measurement of the homologous compound of 1-benzoyl-2,7-dimethoxynaphthalene was carried out (**Figure 4(b)**). For spectrum at 293 K, the signals of δ 6.80, 7.01, 7.17, and 7.72 ppm are assigned to the protons at 8-, 6-, 5-, and 3-positions of the naphthalene ring, respectively. The two signals of δ 7.43 and 7.57 ppm are assigned to the protons at *m*-positions and *p*-position of the benzoyl groups. The signals from δ 7.84 to 7.89 ppm are overlapped by the two kinds of signals, the proton at 4-position of the naphthalene ring and the protons at *o*-positions of the benzoyl group. The signals assigned to the protons of the benzoyl group are broadened in the temperature range from 293 to 173 K. However, no split behavior is observed at 173 K.

The title compound and the homologue have two kinds of the C–C bonds allowed to rotate, *i.e.*, C(benzene)–C(carbonyl) and C(naphthalene)–C(carbonyl) bonds. The steric environment around the neighboring carbonyl groups in the title compound is highly congested as shown in **Figure 1**. Naturally, the rotation behavior of C(naphthalene)–C(carbonyl) bond should be slower than 1-benzoylnaphthalene homologue. In the consequence, the temperature-dependent C(benzene)–C(carbonyl) bond rotation behavior might be observed. In other words, C(naphthalene)–C(carbonyl) bond in the title compound rotates slowly in the temperature range that the C(benzene)–C(carbonyl) bond is allowed to rotate freely.

4. Conclusion

Conclusively, the crystal structural shapes and the dy-

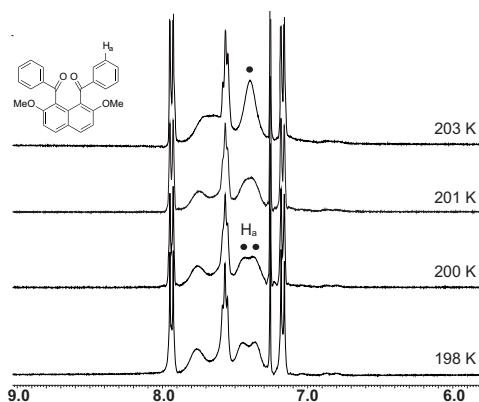


Figure 5. The temperature-dependence of ^1H NMR signals of the title compound in $\text{CDCl}_3/\text{CS}_2$ (1:4 v/v) from 203 to 198 K (6.0 - 9.0 ppm).

namic feature of solution structure of 1,8-dibenzoyl-2,7-dimethoxynaphthalene are clarified. In crystal, the aryl groups of the compound are perpendicularly attached to the naphthalene ring core and situated in an opposite direction. The two types of C–H...O interactions of ketonic carbonyl group with benzene ring and methoxy group and π - π interactions between benzene rings mainly stabilize the molecular packing. According to VT-NMR study, the split signals assigned to the *o*- and *m*-protons of the benzene ring are observed from 193 to 173 K. Comparison of the dynamic behavior with 1-benzoylnaphthalene homologue shows that the C–C bond rotation between ketonic carbonyl group and naphthalene ring in the title compound is enough slow to detect the rotation of the C–C bond rotation between benzene ring and ketonic carbonyl group. The structural information of the title compound in solid state and solution affords some hitherto-unknown aspects in molecular formula and intramolecular rotation properties relationship of these non-coplanarly accumulated aromatic rings molecules.

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