



MOLECULAR STRUCTURE OF BRIDGED *peri*-AROYL-NAPHTHALENE COMPOUND HAVING CYCLOHEXANE-*cis*-1,2-DIOXY-HINGE MOIETY IN SOLUTION

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The bridged *peri*-aroylnaphthalene compound having cyclohexane-*cis*-1,2-dioxy-hinge moiety connecting two benzoyl groups was successfully synthesized through the nucleophilic aromatic substitution of 1,8-bis(4-fluorobenzoyl)-2,7-dimethoxynaphthalene and cyclohexane-*cis*-1,2-diol. The compound consists of two ingredients in solution. The ingredients were successfully separated by repeated preparative thin layer chromatographical treatments to give stable solids. By the aid of ¹H NMR spectroscopic time course tracing, the ingredients have proved to undergo mutual transformations between them to yield an equilibrium mixture of constant fraction values by standing the individual solution within several weeks, revealing that the ingredients are conformational isomers to each other. Spatial organizations of the isomers of the bridged compound in solution have been elucidated by difference NOE measurement showing rough geometry of the isomers as two “extended” forms.

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Introduction

Non-coplanarly accumulated aromatic ring compounds have received much attention from organic chemists and material scientists. Some of these compounds show characteristic physical and chemical properties including semiconductivity, photochromic nature, and molecular recognition ability probably originated from their unique spatial structures.¹⁻⁵ Therefore, accurate structural understanding of the fine spatial organization and the conformational alteration behaviour of this type of molecules in solution are of great important to design the novel functional molecules of this category as well as crystal structure analysis.⁶⁻⁹

The authors have studied on structure and reaction behaviours of naphthalene derivatives having two aroyl substituents at 1- and 8-positions (*peri*-positions)¹⁰⁻¹³ especially focusing on X-ray crystal structure analysis.¹⁴⁻²² In the single crystal molecular structures of *peri*-aroylnaphthalene compounds aromatic rings accumulate non-coplanarly. Two aroyl groups are situated almost perpendicularly against the naphthalene ring and generally oriented in an opposite direction. Only a few of the *peri*-aroylnaphthalene compounds show *syn*-oriented conformation where the two aroyl groups situate in a same direction.^{19,20,21,22} The X-ray crystal structure of the molecular packing of homologous and analogous compounds as well as the single molecular spatial organization has been determined and analysed comparatively to elucidate hitherto unknown or slighted stabilizing interactions functioning in the crystal of such

non-coplanarly accumulating aromatic ring compounds. In addition, the structures of these compounds in solution have been investigated with the aid of ¹H NMR spectroscopy to reveal the rather dynamic feature of the molecular structure accompanying with the motion capability of the parts in molecule, flexibility, and interconversion behaviour giving the structural parameters such as the rotational barrier of linkages of ketonic bondings.^{12, 13}

Recently, the authors have designed and synthesized intramolecularly bridged homologous molecules, which share terminal benzene ring connected to the benzoyl groups at 1- and 8-positions of the naphthalene ring.²³ And crystal structure of one of the bridged homologues has been clarified [Figure 1, compound **1b**].

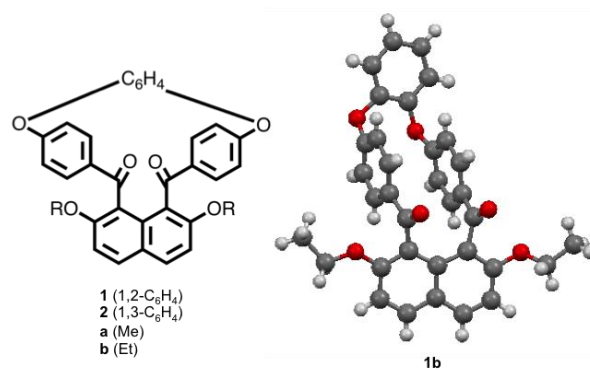


Figure 1. Bridged *peri*-aroylnaphthalene compounds having benzenedioxy-hinge moiety **1** and **2** with the example of crystal structure of compound **1b**

The molecular structure of these molecules in solution has been studied with the aid of ¹H NMR spectroscopy, disclosing the unsymmetrical circumstance of the phenylene ring of the benzoyl groups of the bridged compounds (**1b** and **2b**) against the apparently symmetric shape in chemical formulae (Figure 2).

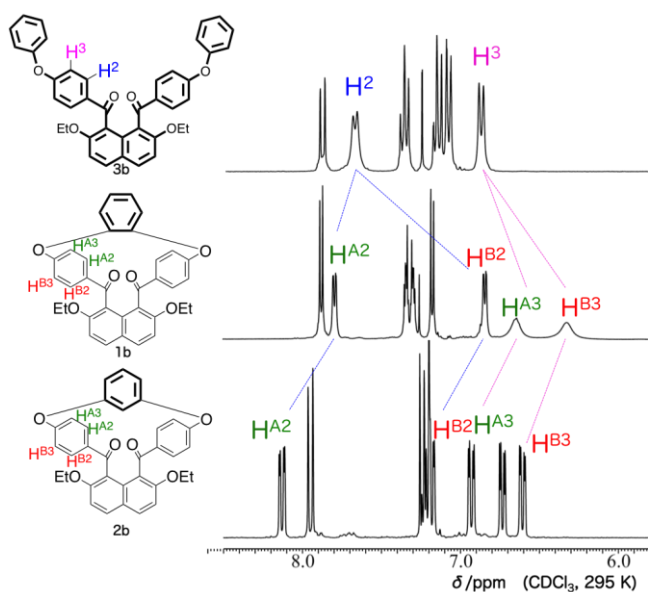


Figure 2. ^1H NMR spectra of compounds **3b**, **1b**, and **2b**.

These results indicate that the spatial mobility of the aromatic rings in the benzoyl groups is strongly restricted. In a natural consequence of design and synthesis of bridged *peri*-aroylnaphthalene compounds, those having cyclohexanedioxy-hinge moiety in place of benzenedioxy unit (**4**) as the objected homologous bridged molecules [Figure 3] were triggered. In this paper, the authors report the synthesis of novel-bridged *peri*-aroylnaphthalene compound having cyclohexane-*cis*-1,2-dioxy-hinge unit (compound **4**) and discuss the structural characteristic especially focusing on the existence of two stable conformational isomers, isolation of the ingredients, the spatial organization of them, and interconversion behaviours with the aid of ^1H NMR spectroscopy.

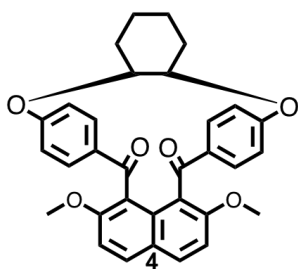


Figure 3. Bridged *peri*-aroylnaphthalene compound **4**.

Experimental

All reagents were of commercial quality and were used as received. Solvents were dried and purified using standard techniques.²⁴ 2,7-Dimethoxynaphthalene, the bridged *peri*-aroylnaphthalene compounds connected with 1,2-benzenedioxy moiety (**1**) and 1,3-benzenedioxy one (**2**), and 2,7-diethoxy-1,8-bis(4-phenoxybenzoyl)naphthalene (**3**) were prepared according to literatures.^{22,23,25,26}

Measurement

^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 (100 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.

Synthesis of bridged *peri*-aroylnaphthalene **4**

To a solution of 1,8-bis(4-fluorobenzoyl)-2,7-dimethoxynaphthalene (0.3 mmol, 130.7 mg) in *N,N*-dimethylacetamide (7.5 mL), NaH (1.5 mmol, 60 mg) and *cis*-1,2-cyclohexanediol (0.3 mmol, 35 mg) were added and the resulting solution was stirred at 423 K for 24 h. The reaction mixture was poured into aqueous 2 M HCl (75 mL) at rt resulting in formation of pale yellow precipitates. The precipitates were collected by filtration and dried in vacuo, giving crude product (156 mg; conv. 32%). The crude material was purified by column chromatography repeatedly [silica gel, toluene : AcOEt = 3 : 1 (two times) and CHCl_3 : AcOEt = 20 : 1 (ten times)] to give two ingredients of conformers of the target compound (**4**) (isolated yields **I**: 10%; **II**: 18%). Isolated conformer **I**: ^1H NMR δ (300 MHz, CDCl_3): 7.93 (2H, d, J = 9.0 Hz), 7.83 (2H, dd, J = 8.7 Hz and 2.7 Hz), 7.23 (2H, d, J = 9.0 Hz), 6.81 (2H, dd, J = 8.7 Hz and 2.7 Hz), 6.62 (2H, dd, J = 8.7 Hz and 2.7 Hz), 6.27 (2H, dd, J = 8.7 Hz and 2.7 Hz), 4.64–4.73 (2H, m), 3.78 (6H, s), 1.42–2.19 (8H, m) ppm; ^{13}C NMR δ (100 MHz, CDCl_3): 22.15, 29.78, 56.89, 111.30, 113.95, 122.09, 122.17, 125.09, 125.59, 129.14, 130.80, 131.47, 131.99, 134.00, 156.23, 162.14, 193.67 ppm; IR ν (KBr): 2937, 2859 (C–H, cyclohexane), 1670 (C=O), 1598, 1511, 1460 (Ar, naphthalene), 1260, 1240 (C–O–C) cm^{-1} ; HRMS (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{32}\text{H}_{29}\text{O}_6$, 509.2000 found 509.1964. Isolated conformer **II**: ^1H NMR δ (300 MHz, CDCl_3): 7.95 (2H, d, J = 9.0 Hz), 7.81 (2H, dd, J = 8.4 Hz and 2.4 Hz), 7.23 (2H, d, J = 9.0 Hz), 6.87 (2H, dd, J = 8.4 Hz and 2.4 Hz), 6.60 (2H, dd, J = 8.4 Hz and 2.4 Hz), 6.37 (2H, dd, J = 8.4 Hz and 2.4 Hz), 4.70–4.78 (2H, m), 3.78 (6H, s), 1.46–2.22 (8H, m) ppm; ^{13}C NMR δ (100 MHz, CDCl_3): 22.14, 29.86, 56.87, 111.30, 115.48, 120.65, 122.00, 125.49, 130.10, 131.66, 132.06, 134.41, 156.35, 162.38, 193.88 ppm; IR ν (KBr): 2935, 2859 (C–H, cyclohexane), 1671 (C=O), 1596, 1510, 1460 (Ar, naphthalene), 1261, 1238 (C–O–C) cm^{-1} ; HRMS (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{32}\text{H}_{29}\text{O}_6$, 509.2000 found 509.1964.

Results and Discussion

The authors attempted the synthesis of the newly designed bridged *peri*-aroylnaphthalene compound having a cyclohexane-*cis*-1,2-dioxy-hinge connecting at the edge of the aroyl groups by two synthetic protocols: one is the dual $\text{S}_{\text{N}}2$ reaction of 1,8-bis(4-hydroxybenzoyl)-2,7-dimethoxynaphthalene compound with dibromocyclohexane and the other is dual $\text{S}_{\text{N}}\text{Ar}$ reaction of 1,8-bis(4-fluorobenzoyl)naphthalene analogue with dihydroxy-

cyclohexane in the presence of base. Though the former approach gave a complex mixture comprised of significant amount of undesirable products of cyclohexenyl ether compounds, the latter choice of the starting materials constructed the targeted bridged *peri*-arylnaphthalene structure in a rather good conversion. Under the optimized conditions that are almost same with the synthetic reaction conditions for catechol- or resorcinol-hinge *peri*-arylnaphthalene analogues briefly reported in the preceding paper, the reaction of 1,8-bis(4-fluorobenzoyl)naphthalene and *cis*-1,2-cyclohexanediol was allowed to proceed in a satisfied conversion, still accompanying with several types of by-products such as 2 : 1 adduct of *peri*-arylnaphthalene substrate and *cis*-1,2-cyclohexanediol molecule, and 2,7-dimethoxynaphthalene, and so on. Furthermore, the ^1H NMR spectrum of the crude product shows the existence of two major ingredients of the structures possibly fit as cyclohexane-*cis*-1,2-dioxy-hinge bridged *peri*-arylnaphthalene (compound **4**) molecules. In solution, two isolated ingredients were observed apparently as stable species at rt by ^1H NMR spectrometrically.

The two major ingredients of the structures were successfully isolated by repeated preparative thin layer chromatography (PTLC) on silica-gel as almost pure forms (Figure 4, top; isolated ingredient **I**, Figure 5, top; isolated ingredient **II**). For each of the ingredients, four kinds of protons for the benzene ring of the benzoyl moiety are distinguished as respective chemical shifts (see Figure 2). The authors have recently reported ^1H NMR spectroscopic structural analysis of phenylene-hinge-bridged *peri*-arylnaphthalene compounds **1** and **2**. The spectra of phenylene-hinge-bridged *peri*-aryloxybenzoylnaphthalene compounds show unique non-equivalency in chemical shift that the protons of the phenylene rings in the oxybenzoyl groups at 1- and 8-positions appear at δ 7.80, 6.84, 6.54–6.74 (broad), 6.20–6.40 (broad) ppm for compound **1b** (catechol-hinge derivative) and δ 8.14, 7.18, 6.94, and 6.62 ppm for compound **2b** (resorcinol-hinge derivative) [Figure 2], whereas for non-bridged compound **3b** a couple of equivalent signals at 7.66 ppm and 6.86 ppm are observed. The ^1H NMR spectra of the isolated ingredients show non-equivalency in chemical shift that the protons of the phenylene-hinge appear at δ 7.83, 6.81, 6.62, and 6.27 ppm for isolated ingredient **I** and at δ 7.81, 6.87, 6.60, and 6.37 ppm for isolated ingredient **II**. The resemble signals feature manifests that the two ingredients have essentially the same situation of the phenylene ring of oxybenzoyl groups to each other and also with the phenylene bridged *peri*-aryloxybenzoylnaphthalene compounds. Consequently, both of the ingredients have quite large similarity in structure, having more intimate relationship than structural isomers. Accordingly, bridged *peri*-arylnaphthalene compound **4** is proved composed of two independent structurally isomeric molecules.

The findings prompted the authors to follow the structure transformation behaviour of the two independent molecules in solution. First, the authors attempted to observe rather long ranged time-course of the product distribution by ^1H NMR spectroscopy (Figures 4 and 5). After standing of CDCl_3 solutions containing each of the ingredients at rt for 2 weeks, the counter ingredient formed. Then, the ratio of the ingredients in solution varied gradually. Standing for additional 6 weeks, the molecular ratio became equally constant value (ingredient **I** : ingredient **II** = 67 : 33) in both

solutions initially contain one of almost pure ingredient solely. The time-course of the interconversion behaviour also showed that the structural transformation of one ingredient (ingredient **II**) to the other one (**I**) proceeds much slowly. Based on the facts described above, the ingredients are considered as conformational isomers having high energy barrier against interconversion.

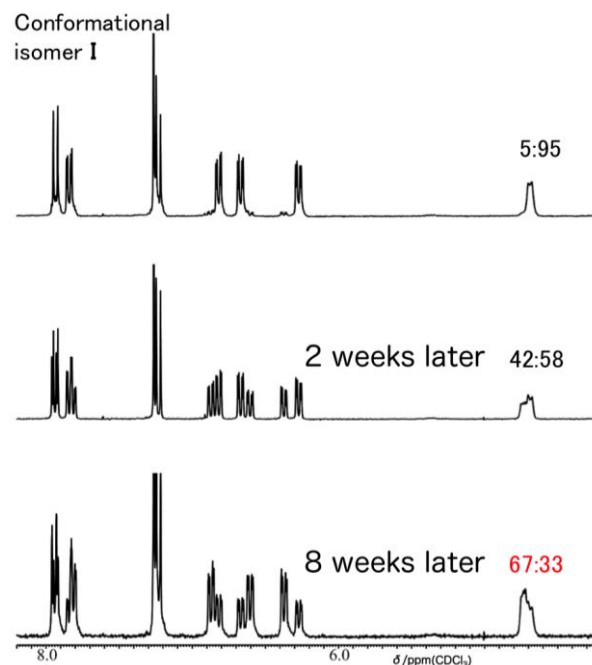


Figure 4. Time-course of ^1H NMR spectra of compound **4** (conformational isomer **I**).

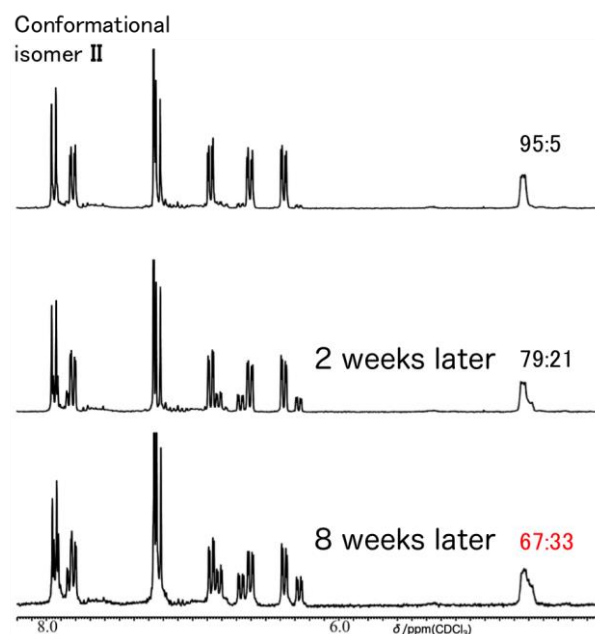


Figure 5. Time-course of ^1H NMR spectra of compound **4** (conformational isomer **II**).

From the standpoint of linkage flexibility of the molecular formulae, this means that there are possible two modes of structural conversion for this equilibrium: one is the inversion of linkages at cyclohexanedioxy-hinge moiety and

the other is reversion of ketonic carbonyl-groups. Accordingly, there are possible four independent isomeric molecules in solution (Figure 6). Two of the extended-type derivatives bear the cyclohexanedioxy-hinge situated outward from the aroyl groups. On the other hand, in other two independent molecules, the cyclohexanedioxy-hinge overspreads the aroyl groups to make the molecular figure as categorized “Bent-type”. The other category of classification of the conformational isomers is *exo/endo*: the former designates that the cyclohexane and naphthalene rings situate on the same side against the plane of *o*-benzoyl and C(=O)-phenylene bonds of the two benzoyl groups.

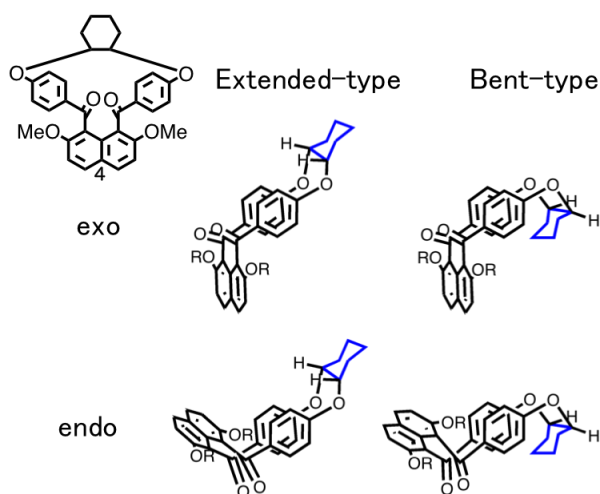


Figure 6. Plausible structure of conformation for bridged compound **4** in solution.

In other words, the interconversion between “Extended-type” structure and “Bent-type” one depends on the mobility of the linkage of cyclohexane ring and ethereal oxygen and the exchange between “*exo*” structure and “*endo*” one on the flexibility of the ketonic carbonyl bonds as displayed in Figure 6. Therefore, specification of two conformers from four candidates targeted required.

Finally, the spatial organization of cyclohexanedioxy-hinge bridged *peri*-aroylnaphthalene compound has been elucidated by the aid of difference Nucleus Overhauser Effect (NOE) measurement (Figures 7 and 8). In all cases, the signals of the protons at the *m*-positions of the benzoyl groups are correlated with the corresponding neighbouring signal of the *o*-positions of the benzoyl groups (Figure 7: δ 6.27 and 6.81 ppm, 6.66 and 7.81 ppm; Figure 8: δ 6.37 and 6.87 ppm, 6.61 and 7.81 ppm). Moreover, the signal of the methine protons of the 1- and 2-positions of the cyclohexanedioxy-hinge correlates with one of the protons at the *m*-position of the benzoyl groups. In the case of isolated conformer **I**, the signals of the *m*-positions of the benzoyl groups at the higher magnetic field correlate with the signal of the protons of the 1- and 2-positions of the cyclohexanedioxy-hinge moiety (Figure 7: δ 6.27 and 4.69 ppm). In the case of isolated conformer **II**, however, the signals of the *m*-position of the benzoyl groups at the low magnetic field correlate with the signal of protons at the 1- and 2-positions of the cyclohexanedioxy-hinge moiety (Figure 8: δ 6.60 and 4.73 ppm). On the other hand, there are no correlated signals of protons of the methylene moiety

of cyclohexanedioxy-hinge moiety with the signals of the *m*-position of the benzoyl groups at higher magnetic field (Figure 8: 6.37 ppm).

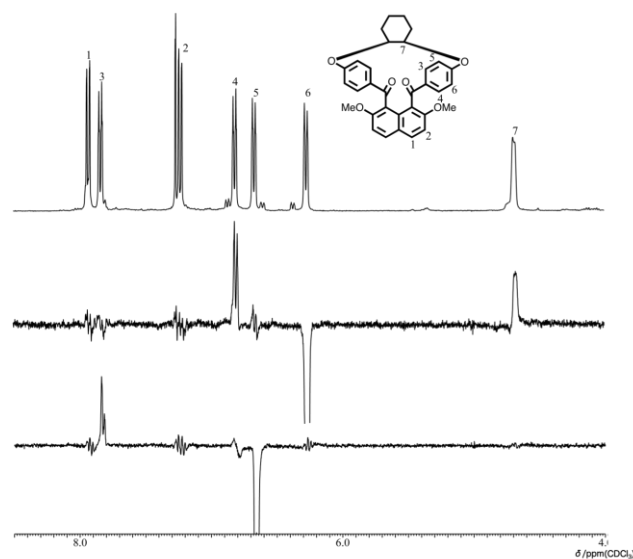


Figure 7. Difference NOE spectra of compound **4** (conformer **I**).

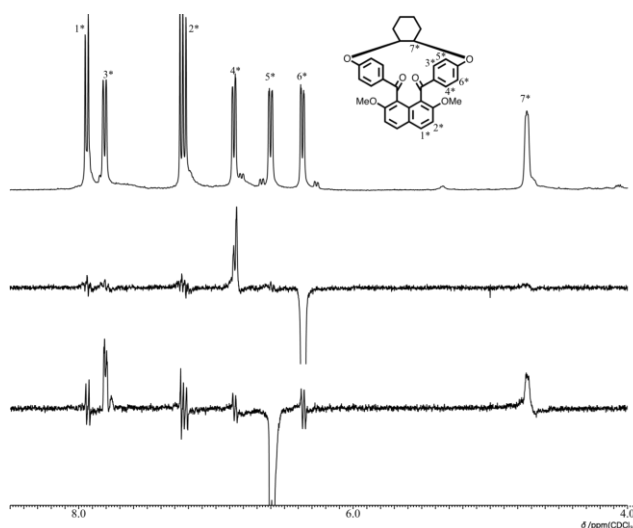


Figure 8. Difference NOE spectra of compound **4** (conformer **II**).

Accordingly, the presence/absence of correlation between the 1- and 2-positioned protons of the cyclohexanedioxy-hinge and *m*-positioned protons of the benzoyl moiety disclosed the conformation of the isomers, “Extended-type” or “Bent-type.” Based on the difference NOE spectra, both of the isolated conformer have correlation between 1- or 2-proton of cyclobutane ring and *m*-proton of benzoyl moiety, the authors conclude that there are no conformers for “Bent-type” in compound **4**. No correlation of the protons at the 1- and 2-position of cyclohexanedioxy-hinge with the signals of the protons of the benzoyl groups should be observed if the shapes of conformer were “Bent-type.”

Here, the authors have referred the spatial organization elucidation of bridged *peri*-aroylnaphthalene compound bearing butylene-hinge by the aid of NOESY measurement (see Note). The results show that one of the *m*-positioned

protons of the benzoyl group appeared at split chemical shifts and the higher field one corresponds to the *m*-positioned proton more closely to the naphthalene ring. According to the above observation, isolated conformer **I** is probably categorized as “*endo*-type” in the extended-classification. In the case of isolated conformer **I**, the signal of protons at the 1- and 2-positions of cyclohexane-hinge has correlation with the higher field signal of a proton at *m*-position of the benzoyl group. So, the protons of cyclohexanedioxy-hinge of isolated conformer **I** have orientation toward the naphthalene ring (Figure 9, *endo*-type). On the other hand, in the case of isolated conformer **II**, the protons at the 1- and 2-positions of cyclohexanedioxy-hinge correlate with the lower field signal of the protons at the *m*-position of the benzoyl groups (Figure 9, *exo*-type).

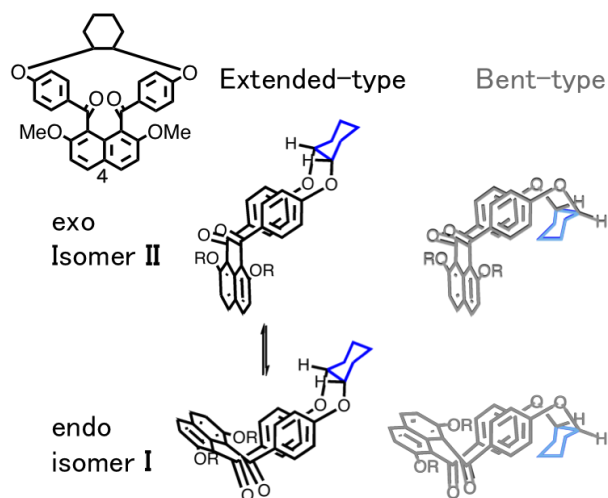


Figure 9. Plausible structure of compound **4** in solution.

The authors consider that the conformers in solution are going back and forth between *exo*-type and *endo*-type in extended-conformation (Figure 9). Based on pretty slow transformation of the cyclohexanedioxy-hinge compound **4**, two of four conformational isomers are isolated and characterized. In the case of bridged *peri*-aroylnaphthalene compound bearing catechol-hinge, although no transformation is observed like bridged compound bearing cyclohexanedioxy-hinge in solution, the shapes of the signals of the protons at the *m*-position of the benzoyl groups are broad. The shapes are interpreted as displaying a vertical motion of the catechol-hinge (Figure 10).

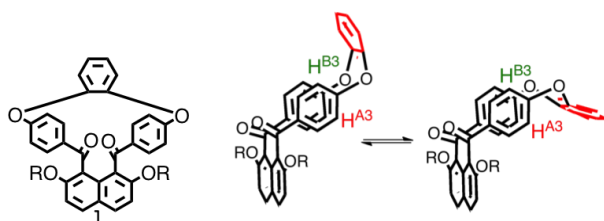


Figure 10. Plausible structure of compound **1** in solution.

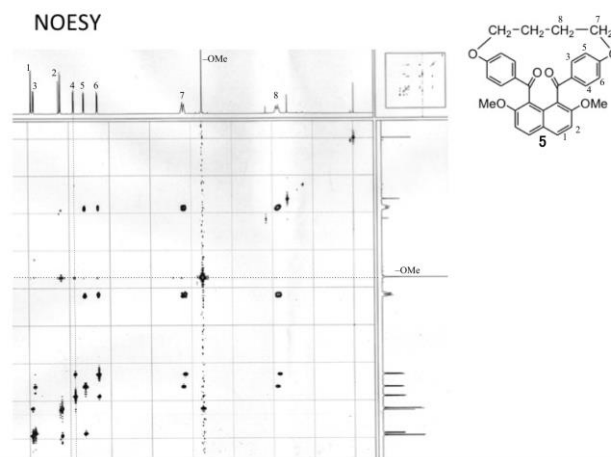


Figure 11. Nucleus overhauser effect correlated spectra of bridged *peri*-aroylnaphthalene compound bearing butylene-hinge moiety (**5**).

Conclusion

The authors synthesized novel bridged *peri*-aroylnaphthalene compound bearing cyclohexane-*cis*-1,2-dioxy-hinge moiety at the terminal carbons of the benzoyl groups. The compound has two stable conformational isomers in solution that can be successfully separated. Though two isomers are sufficiently stable in solid. Furthermore, the structures of the isomers have been revealed as two extended-type conformers. Tracing of the time-course of these conformers by ^1H NMR spectroscopy has revealed interconversion of the isomers to each other proceeds slowly in solution finally giving a mixture of constant ratio.

Note

A signal of methoxy groups at the 2- and 7-positions of naphthalene ring correlate not only with the signal of the protons of the β -position of the naphthalene rings but also with the signal of the protons at the *o*-position of the benzoyl groups, which shows that the protons situate toward the naphthalene ring rather than outward. The latter signal of one of the *o*-position of the benzoyl groups correlates with one of the signals of the protons at the *m*-position of the benzoyl group that is deviated to the higher field in the aromatic region. The observation that one of the signals of the *m*-positioned protons of the benzoyl groups has correlation with the protons of CH_3O -moiety suggests that a proton attributing to the signal at the higher field situates in more close position with the naphthalene ring.

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