

EVALUATION OF THE BULKINESS OF PERI-SUBSTITUENTS DISTORTING 1,8-DIMETHYLNAPHTHALENE FRAMEWORK

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ABSTRACT: A systematic investigation on the distortion of a naphthalene ring was conducted using 1,8-dimethylnaphthalene framework, where bulkiness was deliberately increased one-bond-length distanced from the *peri*-position. By introducing bromo groups into the methyl group at the 1- and 8-position, the steric repulsion increased, leading to a distortion of the naphthalene ring, both horizontally and vertically. X-ray crystallography revealed that 1,8-bis(bromomethyl)naphthalene had a vertical distortion with 11.0° of dihedral angle (α) between *peri*-substituents, which disturbed the coplanarity of the naphthalene ring. On the other hand, the dihedral angle of 1,8-bis(dibromomethyl)naphthalene was smaller ($\alpha = 8.3^\circ$) despite the bulkier substituents. In this case, the horizontal distortion of the naphthalene ring increased. These distortions should non-electronically activate the naphthalene framework. Nitration and hydrogenation reactions were conducted to evaluate the reactivity; however, the bulkier 1,8-bis(dibromomethyl)naphthalene was intact under the employed conditions. A DFT calculation suggested that the inertness of the 1,8-bis(dibromomethyl)naphthalene is presumably due to the negative hyperconjugation of the (dibromo)methyl group. These findings open up new avenues for future research in this field, particularly in developing novel activation protocols for aromatic compounds.

KEYWORDS: dis-planarity, *peri*-positions, steric repulsion, naphthalene

1. INTRODUCTION

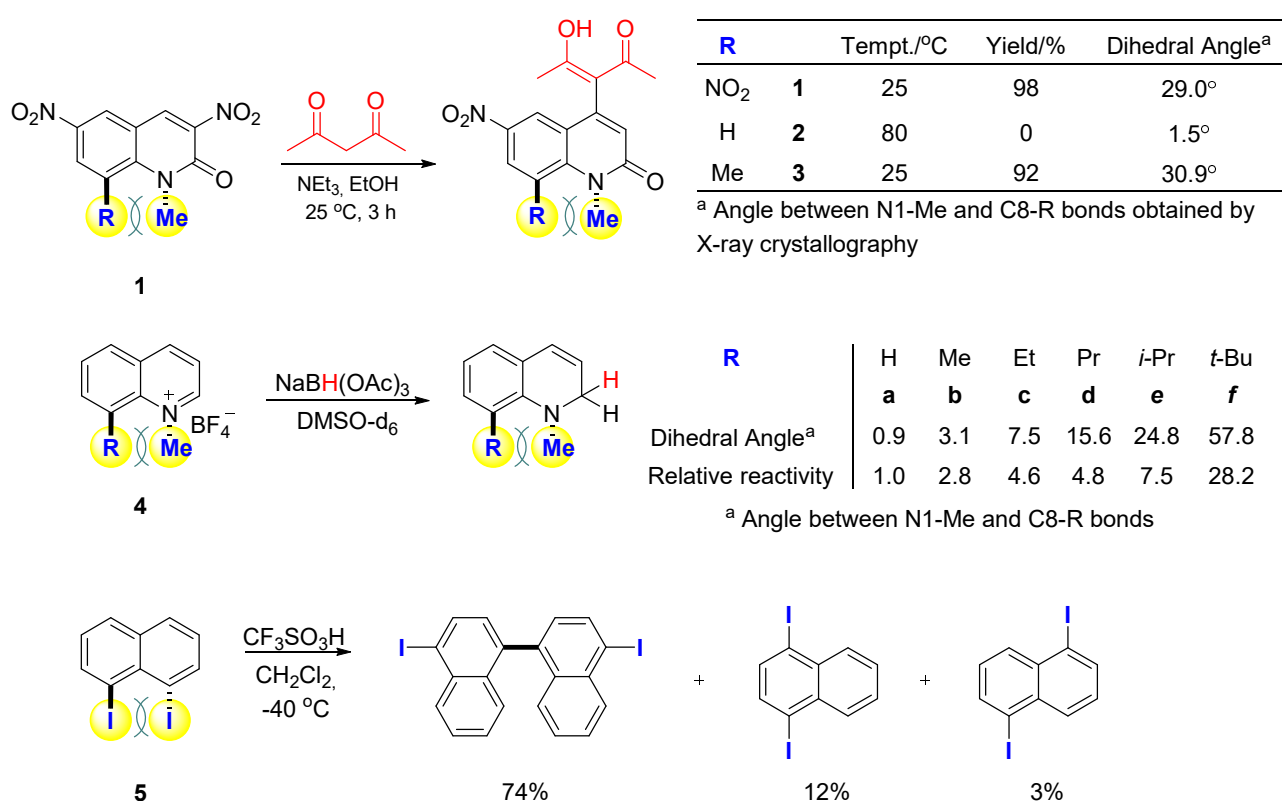
Aromatic compounds have long been at the forefront of material and pharmaceutical sciences. As a result, a multitude of methods for modifying aromatic rings have been devised. The most commonly used reactions include cross-coupling reactions, reactions using benzyne, and electrophilic substitution. In the former two cases, the presence of a reactive carbon-halogen or carbon-sulfonate bond, facilitated by a good leaving group such as halide or sulfonate, is crucial for undergoing the reactions. In the latter case, the substituent effect is a key to promoting the reaction. While introducing an electron-donating or -withdrawing group is the most common activation method for the aromatic ring, the reaction site is often limited due to its directing

property. Therefore, despite the vast number of reported reactions, the development of a novel activation protocol remains a relatively unexplored field.

In our previous work, we studied the functionalization of the 1-methyl-2-quinolone framework where we found that 1-methyl-3,6,8-trinitro-2-quinolone (**1**) exhibits unusually high reactivities [1–3]. For instance, when **1** was allowed to react with 2,4-pentanedione, *cine*-substitution efficiently proceeded on the pyridone moiety at room temperature. Furthermore, 3,6-dinitroquinolone **3** exhibited similar reactivity to **1** even though the 8-substituent was replaced by an electron-donating methyl group. In contrast, no reaction occurred for 8-unsubstituted 3,6-dinitroquinolone **2** even under

heating conditions. Since the 8-position is distant from the reaction site and this reaction was not influenced by the electronic property of the 8-substituent, the steric effect of the substituent was speculated to be the determining factor. Indeed, X-ray crystallography of **1** showed that the dihedral angle between N1–Me and C8–NO₂ bonds was 25°. According to these outcomes, we concluded that steric repulsion between the 1-methyl group and 8-substituent distorts the quinolone framework, decreases the aromaticity, turning it

non-electronically activated, by which the pyridine moiety serves as an activated nitroalkene (**Scheme 1a**) [4]. Following these results, a systematic study on the correlation between steric repulsion and reactivity was performed using 1-methyl-8-alkyl-quinolinium salts **4a–f** (**Scheme 1b**) [5]. As the 8-substituent becomes bulkier, the dihedral angles between N1–Me and C8–R bonds become longer, inducing distortion of the quinoline ring. Accordingly, highly distorted quinolinium salts **4e** and **4f** underwent reactions with NaBH(OAc)₃ efficiently (**Scheme 1b**).



Scheme 1. (a) Non-electronic activation of 8-substituted MeQone derivatives; (b) Comparing reactivities of 8-alkylated 1-methylquinolinium salts; (c) Homocoupling and halogen dance reaction of 1,8-diiodonaphthalene.

More recently, we have also reported the transformation of naphthalene using steric repulsion of iodo groups with large atomic radius. While 1,5-diiodonaphthalene remained unreactive, a modest structure of 1,8-diiodonaphthalene (**5**) was reactive under the same conditions to undergo the rearrangement of the iodo group (halogen dance

reaction) and homocoupling leading to binaphthyl (**Scheme 1c**) [6]. Ultimately, to the best of our knowledge, a systematic study between the bulkiness of *peri*-substituents and distortion of the naphthalene ring has not been systematically studied. Therefore, this report discusses the topic using 1,8-dimethylnaphthalene **7** as a model compound, in

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which the bulkiness of the *peri*-substituents was increased by sequential bromination of the methyl groups. Next, we investigated the reactivity of the distorted naphthalene ring using the classic nitration and hydrogenation reactions.

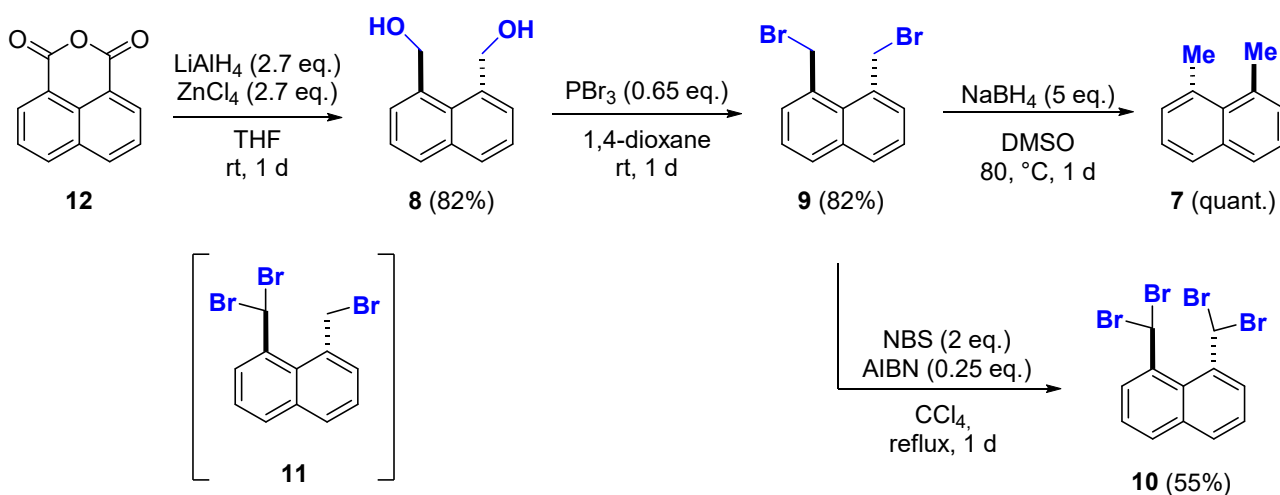
2. METHODOLOGY

2.1. Synthesis of 1,8-disubstituted naphthalene

According to the works of literature [7,8], 1,8-disubstituted naphthalenes **7–10** were prepared from 1,8-naphthalic anhydride **12** (Scheme 2). Reduction of **12** with LiAlH₄ in the presence of ZnCl₄ afforded bis(hydroxymethyl) derivative **8**. The

hydroxy groups were efficiently converted to bromomethyl groups upon heating with PBr₃, which led to bis(bromomethyl) derivative **9**.

Next, the subsequent reduction of **9** with NaBH₄ yielded 1,8-dimethyl derivative **7** quantitatively [9]. Bromination of the benzyl positions in **9** using *N*-bromosuccinimide (NBS) proceeded to afford bis(dibromomethyl) derivative **10**; however, an intermediately-expected product as tribrominated **11** was not detected even when an equimolar amount of NBS was used. Although direct synthesis of **11** from **7** or **8** was also attempted using NBS, tribrominated product **11** remains undetectable [10].



Scheme 2. Preparation of 1,8-disubstituted naphthalenes **7–10**.

2.2. Crystal Structure Study

Since four kinds of 1,8-disubstituted naphthalenes **7–10** were in hand, their single crystals were subjected to X-ray crystallography. Data interpretation was carried out using Gauss view 9.0. The numbering of atoms (Figure 1) and ORTEP views from top and side directions were shown in Figure 2, respectively. Selected crystal parameters for naphthalenes are also presented in Table 1.

2.3. Ring Distortions Evaluation

The distortion of the naphthalene ring was evaluated from three parameters. Namely, (a) vertical

distortion, (b) horizontal distortion, and (c) bond length and interatom distance [11]. In this section, these parameters are compared and discussed for naphthalenes **7–10**.

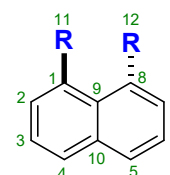
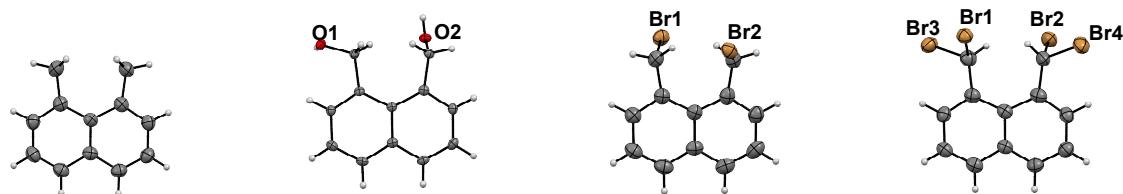
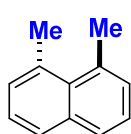
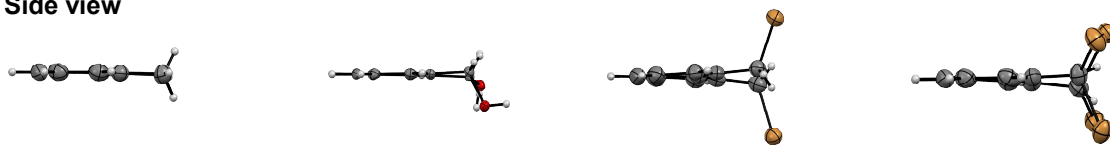


Figure 1. The order of atoms in the crystal structures of naphthalene derivatives.

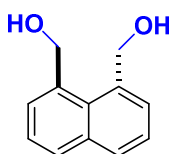
Front view



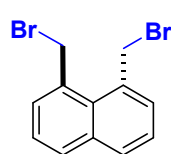
Side view



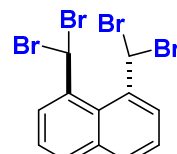
7



8



9



10

Figure 2. ORTEP drawing of naphthalene derivatives **7–10** with displacement ellipsoids at the 50% probability level.

Table 1. Selected crystallographic information for crystal structures **7–10**.

	7 (R = CH ₃)	8 (R = CH ₂ OH)	9 (R = CH ₂ Br)	10 (R = CHBr ₂)
Empirical formula	C ₁₂ H ₁₂	C ₁₂ H ₁₂ O ₂	C ₁₂ H ₁₀ Br ₂	C ₁₂ H ₈ Br ₄
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>n</i>	<i>P</i> 2 ₁ / <i>n</i>	<i>C</i> 2/ <i>c</i>	<i>P</i> 2 ₁ / <i>n</i>
<i>a</i> (Å)	9.5783(6)	8.4875(4)	23.3250(11)	10.1755(4)
<i>b</i> (Å)	6.8687(4)	4.8113(2)	7.5802(3)	6.7237(2)
<i>c</i> (Å)	13.2814(10)	22.4730(9)	12.3748(6)	19.3037(8)
β (°)	92.066(7)	94.379(4)	95.479(4)	100.207(4)
<i>Z</i>	4	4	8	4
Goodness of fit	1.051	1.098	1.097	1.031
<i>R</i> ₁ [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.054,	<i>R</i> ₁ = 0.041,	<i>R</i> ₁ = 0.036,	<i>R</i> ₁ = 0.048,
<i>wR</i> ₂ (all data)	<i>wR</i> ₂ = 0.160	<i>wR</i> ₂ = 0.110	<i>wR</i> ₂ = 0.107	<i>wR</i> ₂ = 0.132
CCDC number	2266182	2266183	2266184	2266185

3. RESULTS AND DISCUSSIONS

3.1 Vertical distortion

The vertical distortion of the naphthalene framework was estimated by comparison of three dihedral angles α – γ (Table 2). Referring to our previous work [4,5], we initially assessed the vertical distortion using a dihedral angle α between C1–C11 and C8–C12 bonds. In the case of dimethyl

naphthalene **7**, the dihedral angle α is 0.7°, indicating that the coplanarity remains. Introducing a hydroxy group to the methyl groups distorted the naphthalene ring to some extent. More importantly, the larger bromo groups distorted the naphthalene ring to a greater degree as can be seen with the dihedral angle α reaching 11.0°. Interestingly, bis(dibromomethyl)naphthalene **10** is less distorted

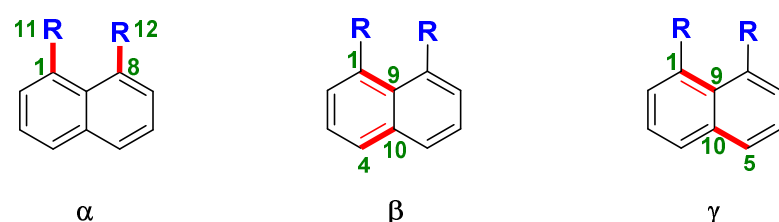
than **9**, even though two bromine atoms are introduced to the methyl groups.

Another parameter to evaluate the vertical distortion called dihedral angles β (between C1–C9 and C4–C10 bonds) and γ (between C1–C9 and C4–C10 bonds) were compared [12]. While the dihedral angle β and γ of dimethylnaphthalene **7** are undoubtedly small ($\beta = 0.5^\circ$ and $\gamma = 0.1^\circ$), those

values of other naphthalenes **8–10** increase depending on the bulkiness of substituents.

Meanwhile, the dihedral angles **10** were not larger than **9**, which showed a similar tendency with α . Since substituting the bulkier dibromomethyl groups should induce more prominent ring distortion, an alternative parameter to evaluate the distortion was required.

Table 2. Comparison of dihedral angles α (between C1–C11 and C8–C12 bonds), β (between C1–C9 and C4–C10 bonds), and γ (between C1–C9 and C5–C10 bonds) in naphthalene **7–10**.



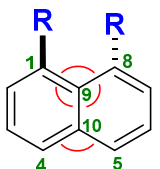
R		$\alpha/^\circ$	$\beta/^\circ$	$\gamma/^\circ$
CH ₃	7	0.7	0.5	0.1
CH ₂ OH	8	4.6	2.1	1.5
CH ₂ Br	9	11.0	4.1	4.9
CHBr ₂	10	8.3	3.5	3.1

3.2. Horizontal distortion

Bond angles around the C9 position shown in **Table 3** can also be reviewed to compare the degree of distortion as the sum of three angles equals 360° . Three atoms connected to C9 carbon are located in the same plane, albeit the angles were not equally divided. All compounds have wider angles δ (outside of the naphthalene ring) than the standard sp^2 bond angle (120°), while the other two angles ϵ and η (inside of the naphthalene ring) are narrower. This

fact indicates that the angle was pushed C1 and C8 apart by steric repulsion between the *peri*-substituents. Accordingly, the outer angle θ at the opposite side (C4–C10–C5 angle) became smaller. Notably, each compound exhibited similar bond angle tendencies regardless of the bulkiness of the substituents. This result suggests that the first distortion occurs horizontally by absorbing repulsive energy when the *peri*-substituents become bulky.

Table 3. Bond angles around the C9 and C10 carbons in **7–10**.



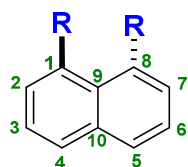
R		$\delta/^\circ$	$\epsilon/^\circ$	$\eta/^\circ$	$\theta/^\circ$
CH ₃	7	126.3(1)	116.6(9)	117.1(9)	118.2(1)
CH ₂ OH	8	126.3(2)	117.1(2)	116.7(2)	118.5(3)
CH ₂ Br	9	128.4(6)	115.1(5)	116.5(5)	117.6(2)
CHBr ₂	10	125.5(1)	117.2(1)	117.3(1)	118.8(2)

3.3. Bond length and interatomic distances

Steric repulsion between *peri*-substituents should affect not only bond angles but also bond distances and interatomic distances. Some selected bond lengths and interatomic distances are shown in **Tables 4** and **5**, respectively. While bond lengths of **7–9** resemble each other, a partial stretch of the inner bond length (C1–C9) of bis(dibromomethyl) derivative **10** was observed, which is about 0.035 Å longer than any other naphthalenes **7–9**. On the other hand, the outer bond lengths (C1–C2, C2–C3, C4–C10) of **10** are shorter than those of other derivatives. Probably, due to these longer bond

lengths, vertical dihedral angles α – γ in **10** are smaller than those of **9**. Interatomic distances also indicate high ring distortion of naphthalene **10**, that is, C1---C8 and C2---C9 were longer than others. Additionally, the distance between C4 and C5 on the opposite side is shorter than those of other substrates, which is a result of a compression of the naphthalene ring. Since C11---C12 distances are longer in naphthalenes **8–10** than **7**, the *peri*-substituents repel each other to absorb repulsive energy, which causes horizontal and vertical distortions. When the relaxation reaches its limits, the naphthalene scaffold is distorted.

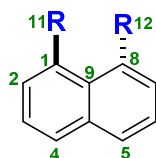
Table 4. Bond lengths (Å) in the naphthalene ring in **7–10**.



	7 (R = CH ₃)	8 (R = CH ₂ OH)	9 (R = CH ₂ Br)	10 (R = CHBr ₂)
C1–C2	1.375(2)	1.380(2)	1.380(4)	1.370(1)
C2–C3	1.407(2)	1.407(2)	1.404(4)	1.380(1)
C3–C4	1.358(2)	1.364(2)	1.358(4)	1.370(1)
C4–C10	1.419(2)	1.421(2)	1.423(4)	1.407(9)
C10–C9	1.442(2)	1.435(1)	1.430(4)	1.434(9)
C9–C1	1.445(2)	1.447(2)	1.446(4)	1.481(9)
C10–C5	1.418(2)	1.422(2)	1.422(4)	1.414(9)
C5–C6	1.358(2)	1.361(2)	1.360(5)	1.340(1)
C6–C7	1.409(2)	1.407(2)	1.400(5)	1.410(1)
C7–C8	1.377(2)	1.380(2)	1.382(4)	1.370(1)
C8–C9	1.444(2)	1.446(2)	1.446(4)	1.434(9)

Values smaller than others are colored in blue, and values larger than the others are colored in red.

Table 5. Selected interatom distances (Å) in **7–10**.



	7 (R = CH ₃)	8 (R = CH ₂ OH)	9 (R = CH ₂ Br)	10 (R = CHBr ₂)
C1–C8	2.569(2)	2.580(2)	2.580(4)	2.625(9)
C2–C9	2.429(2)	2.444(2)	2.440(4)	2.459(9)
C4–C5	2.442(2)	2.440(2)	2.445(5)	2.413(9)
C11–C12	2.961(2)	2.984(2)	3.058(4)	2.987(9)

Values smaller than others are colored in blue, and values larger than the others are colored in red.

3.4. Mechanism of Distortions

To summarize our discussions above, the ring distortion occurs in the following order: 1) horizontal distortion, 2) vertical distortion, and 3) elongation of the bond and interatomic distances as their bulkiness increases (**Figure 3**). In detail, when two substituents exist at the *peri*-positions, the outside-ring angles around C9 δ become larger than the sp^2 standard angle (120°), while the inside-ring angles ϵ and η become smaller (horizontal distortion). Consequently, the planarity of the plane is considered to still intact for all compounds.

However, larger steric repulsion disturbs the coplanarity of the naphthalene ring (dihedral angle α) upon introducing hydroxy or bromo groups into the methyl groups (vertical distortion). When the substituent becomes bulkier, interatomic distances **X** and **Y** become longer, and bond length **C** also elongates due to the steric repulsion between *peri*-substituents (elongation of bond and interatomic distances). Thus, the bond lengths represented by **A** and **B**, along with the interatomic distance denoted by **Z** on the opposite side experience a reduction in length due to compression.

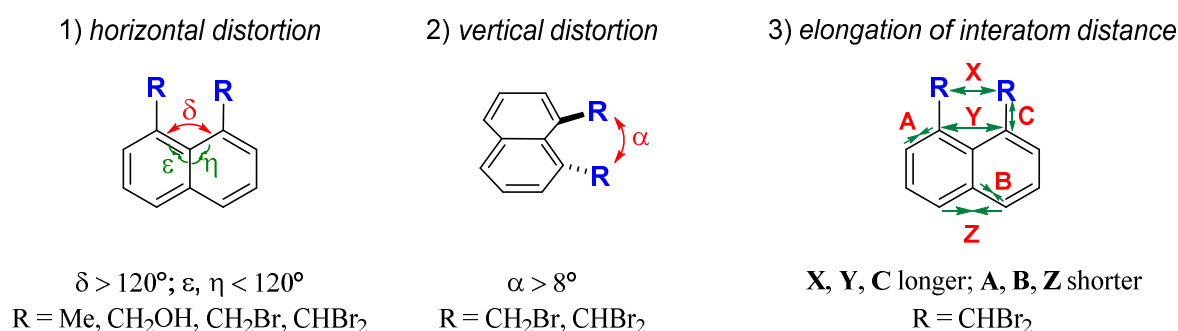


Figure 3. Three kinds of distortion of the naphthalene ring.

Table 6. Nitration of 1,8-disubstituted naphthalenes **7**, **9** and **10**.

Entry	Naphthalene		Yield/%		Recovery/%
	R		2-nitro	4-nitro	
1	CH ₃	7	44	56	0
2	CH ₂ Br	9	0	12	62
3	CHBr ₂	10	0	0	61

3.5. Nitration Reaction

When 1,8-dimethylnaphthalene **7** was reacted with HNO₃ in acetic anhydride, the nitration proceeded at room temperature to afford 2- and

4-nitrated products in 44% and 56% yields, respectively (**Table 6**, Entry 1) [13]. However, brominated naphthalene **9** was less reactive, which led to a 4-nitrated product with only a 12% yield

The 8th International Symposium on Frontier Technology (ISFT)

(Entry 2). Moreover, tetrabrominated naphthalene **10** was recovered intact under the conditions employed (Entry 3). These results contradicted our expectation, presumably due to the electronic effect of bromomethyl groups, in which the electron-withdrawing inductive effect diminishes the electron density of the naphthalene ring.

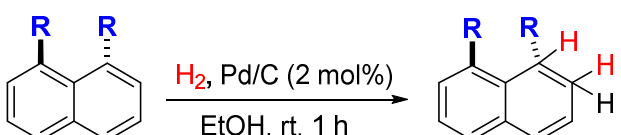
3.6. Hydrogenation Reaction

Catalytic hydrogenation was chosen for another reactivity test, presuming it should be less influenced

by the electronic effect of the substituents. Naphthalenes were stirred under H₂ with atmospheric pressure in the presence of a Pd/C catalyst in ethanol at room temperature for 1 h (**Table 8**).

While naphthalenes **7**, **8** and **10** were intact under the conditions, bromomethyl substituted naphthalene **9** underwent debromination to afford dimethylnaphthalene **7**. Although the reaction proceeded at substituents, the high reactivity of **9** is interesting to compare with the inertness of **10**.

Table 8. Hydrogenation on 1,8-disubstituted naphthalenes **7–10**.



Entry	R		Yield/%	Recovery/%
1	CH ₃	7	0	99
2	CH ₂ OH	8	0	98
3 ^a	CH ₂ Br	9	0	21
4 ^b	CHBr ₂	10	0	99

^a Dimethylnaphthalene **7** was obtained in 68% yield.

^b Reaction conditions: 40 °C, cat. 20 mol%, 1 h

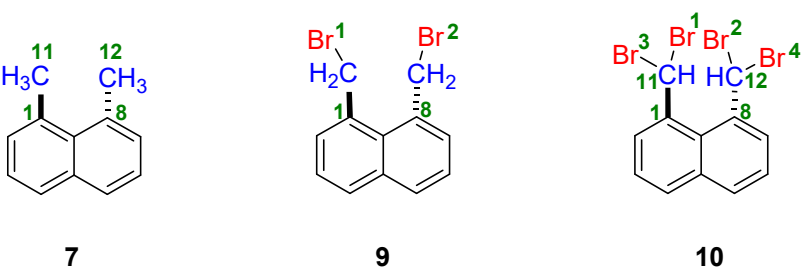
3.7. Another effect of bromo groups

Among *peri*-substituted naphthalenes **7–10**, only **9** exhibited high reactivity for hydrogenation. From a viewpoint of distortion, tetrabrominated naphthalene **10** is prominently considered more reactive. However, **10**, in fact, is less reactive than **9**. One possible reason why the catalytic surface is not approachable during the hydrogenation reaction could be due to the steric hindrance caused by the dibromomethyl group. Nevertheless, another reason should be considered to explain the unusual reactivity of **9**. To obtain insight, density functional theory (DFT) calculations for **7**, **9**, and **10** were performed. The calculated structural parameters were quite similar to the actual parameters observed in crystallography (**Table 9**).

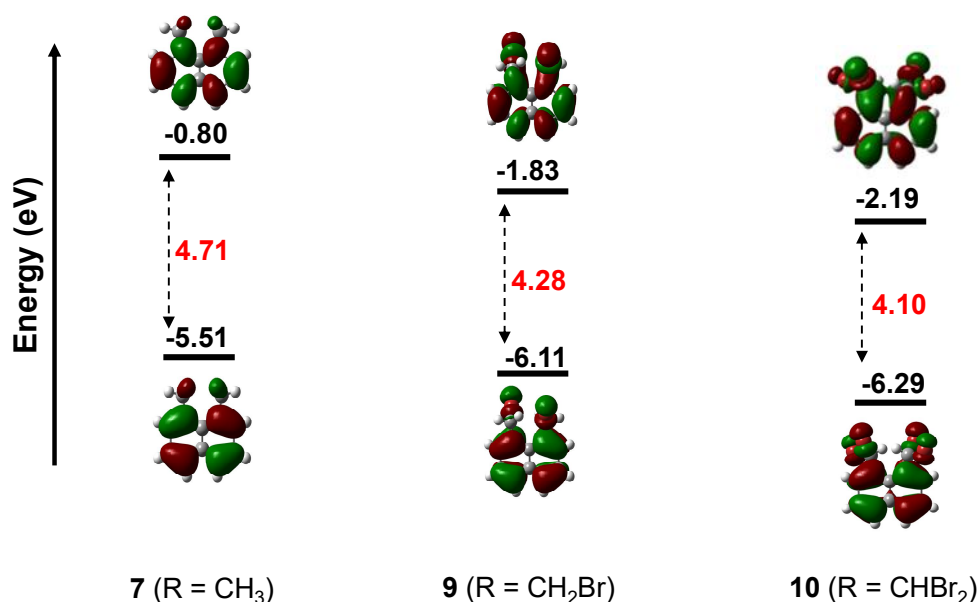
Characteristically, the bond lengths between the substituent and the ring carbon (C1–C11 and

C8–C12) are shorter in the case of **9**, indicating these bonds possess double bond properties. The carbon–bromine bonds (C11–Br1 and C12–Br2) of brominated naphthalenes **9** and **10** were orthogonal to the naphthalene ring and elongated. On the other hand, the second carbon–bromine bonds (C11–Br3 and C12–Br4) in **10** are the shortest values.

The HOMO levels were lowered as bromo groups were introduced (**Figure 4**). These results indicate that the carbon–bromine bonds interact with π -system of the naphthalene ring, and π electrons were delocalized to the anti-bonding orbital of the carbon–bromine bond (σ^*), which facilitates the bond fission to show a high reactivity of **9**. However, the introduction of the second bromine atom increases the steric hindrance, which results in the inertness of **10**.

Table 9. Comparison of actual and calculated C–C and C–Br bond lengths (Å) in **7**, **9** and **10**.


R			C11–Br1	C11–Br3	C12–Br2	C12–Br4	C1–C11	C8–C12
CH ₃	7	crystal	—	—	—	—	1.507(2)	1.513(2)
		calcd.	—	—	—	—	1.517	1.517
CH ₂ Br	9	crystal	1.981(3)	—	1.996(3)	—	1.499(4)	1.489(4)
		calcd.	2.020	—	2.021	—	1.496	1.496
CHBr ₂	10	crystal	1.986(8)	1.956(7)	1.977(7)	1.947(8)	1.510(1)	1.490(1)
		calcd.	1.996	1.976	1.996	1.976	1.502	1.502

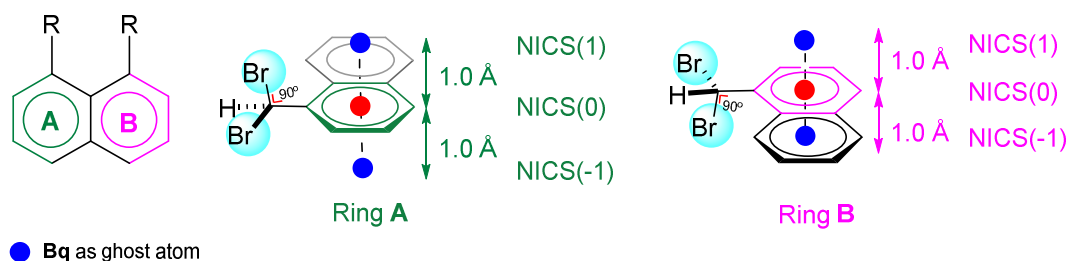
**Figure 4.** Homo and LUMO levels (eV) of **7**, **9** and **10** were calculated at B3LYP/6-31g(d,p).

3.8. NICS evaluation

NICS calculations were performed to estimate the aromaticity degree for naphthalenes **7**, **9**, and **10** (**Table 10**). Due to the unique carbon-bromine bond orientation on the ring plane, two different values, NICS(-1) and NICS(1), were also taken into account. NICS(0) values of bis(bromomethyl)naphthalene **9** and bis(dibromomethyl) naphthalene **10** were estimated to $\delta = 9.9$ ppm and $\delta = 9.3$ ppm, respectively, which are in lower fields than that of

dimethylnaphthalene **7** ($\delta = 9.0$). In the case of NICS(1 or -1), a ghost atom (Bq) on the side with the orthogonal bromo group showed a higher field shift than that on the other side. To avoid the electronic effect of the bromo group, NICS(1 or -1) values without an orthogonal bromo group (NICS(-1) for ring A and NICS(1) for ring B) were compared. As a result, **7** shows the greatest up-field shift among the naphthalenes. Although these results should indicate a higher degree of magnetic

anisotropy of **7** than **9** and **10**, the **10** and **9** show an up-field shift via increasing ring electron-withdrawing effect of the bromo group, as mentioned in the last section, must be taken into consideration. At least the comparison of

Table 10. NICS calculation for **7**, **9** and **10**

R	Ring A			Ring B		
	NICS(-1)	NICS(0)	NICS(1)	NICS(-1)	NICS(0)	NICS(1)
CH ₃	7	-11.1	-9.0	-11.1	-9.0	-11.1
CH ₂ Br	9	-11.6	-9.9	-11.1	-9.9	-11.6
CHBr ₂	10	-11.7	-9.3	-10.7	-9.3	-11.7

4. CONCLUSION

The correlation between the bulkiness of *peri*-substituents and distortion of the naphthalene ring was studied. The X-ray crystallography showed 1,8-bis(bromomethyl)naphthalene **9** possesses a vertical strain with 11.0° dihedral angle between *peri*-substituents, disturbing the coplanarity of the naphthalene ring. On the other hand, the vertical distortion of 1,8-bis(dibromomethyl)naphthalene **10** was smaller (8.3°) even though the substituents became bulkier. In this case, horizontal distortion was also observed besides vertical strain, *that is*, an inner bond distance of C1–C9 is longer, and outer bond distances (C1–C2, C2–C3, C4–C10) are shorter than those of other derivatives. Interatomic distances (C1–C8, C2–C9) of **10** are longer than those of **7–9**, and the distance between C4 and C5 is shorter than those of other substrates.

The distorted naphthalene ring is expected to show higher reactivity because of the decreased aromaticity. To confirm this, we studied two reactions, nitration and hydrogenation using **7–10**. However, a systematic evaluation method for

non-electronic activation was not established because the inductive electron-withdrawing and steric effects were still influenced. Although it is necessary to investigate further in the evaluation of the reactivity, the correlation between the bulkiness of the *peri*-substituents and distortion of the naphthalene ring will be useful information for researchers who study the physical properties of aromatic compounds and their modification.

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