

Synthesis and Structure of *N,N'*-Butylene-*N,N'*-hexylenebis(2-oxy-1-naphthaldimine)

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The title compounds were synthesized in the reaction of 2-hydroxy-1-naphthaldehyde and corresponding aliphatic diamines. Standard TGA and DSC procedures were applied to characterize both compounds **1** and **2**. The stereochemistry of the compounds in solid state was determined by the single crystal X-ray diffraction. Compound **1**: C₂₆H₂₄N₂O₂, orthorhombic, space group *Pbca*, *a* = 8.142(2) Å, *b* = 9.097(3) Å, *c* = 28.812(8) Å, *R* = 0.032. Compound **2**: C₂₈H₂₈N₂O₂, orthorhombic, space group *Pbcn*, *a* = 23.738(2) Å, *b* = 8.9933(8) Å, *c* = 10.4206(8) Å, *R* = 0.038. The molecules of both compounds **1** and **2** have an imposed centre of inversion in the middle of the aliphatic chain bridging two naphthaldimine moieties. The well known crystallographic *D*_{2h} bond length pattern as well as quinoid effects were observed in the fused ring system in both cases. In both Schiff bases, the N–H···O type of intramolecular hydrogen bond was established. An additional intermolecular three-centre hydrogen bond links the molecules in **1**.

INTRODUCTION

The structures of the first transition series metal complexes with symmetrically bridged *bis*-bidentate aldimines are known,^{1–5} but less work has been done on the molecular structures of the parent Schiff bases. These Schiff bases are built up of two aldimine moieties separated by a different aliphatic or aromatic chain. The influence of a purely aliphatic bridging

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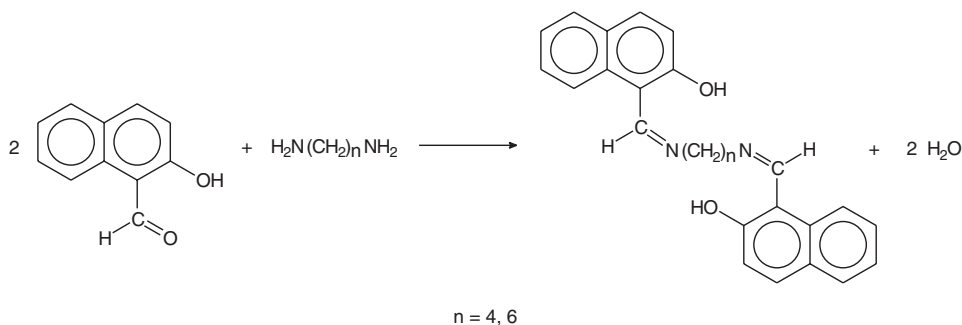
chain on the stereochemistry of this kind of aldimines could be of interest and worth examining. Furthermore, investigation of aryl aldimines having a substituted hydroxy group in position 2 enables examination of the type of intramolecular hydrogen bond. The structures of symmetrically bridged salicylaldimines are known.⁶⁻⁸ The bridging chain in these compounds is not necessarily aliphatic but generally substituted. Among them, there are two cases with unsubstituted aliphatic chains.^{9,10} The title compounds were synthesized and studied in order to investigate the influence of the bridging aliphatic chain length on the stereochemistry of 2-hydroxy-1-naphthaldimines. Both compounds contain a bridging chain consisting of four [*N,N'*-butylenebis(2-oxy-1-naphthaldimine), compound **1**] and six [*N,N'*-hexylenebis(2-oxy-1-naphthaldimine), compound **2**] methylene carbon atoms.

EXPERIMENTAL

Both compounds were synthesized in the same way (Scheme I) by refluxing the methanol solution mixtures of 2-hydroxy-1-naphthaldehyde and either 1,4-diaminobutane or 1,6-diaminohexane (molar ratio 2:1). The Schiff bases were obtained as yellow (compound **1**, yield 90%) and brownish powder precipitates (compound **2**, yield 80%). The compounds were examined by thermogravimetric analysis and differential scanning calorimetry. The DSC measurements were performed by means of a Mettler DSC 30 calorimeter with inert gas-flow (200 cm³/min, Ar) over the samples using aluminium crucibles. The heating rate was 10 °C/min. The TGA measurements were performed on a Mettler TG 50 thermobalance under the conditions similar to the DSC measurements, only at a different heating rate (20 °C/min).

Crystallization and X-ray Experiment

The solubility of the compounds is quite different. While compound **1** was found to be a very insoluble material, its homologue, compound **2**, was mainly soluble in the most common organic solvents. Because of its pronounced insolubility, single



Scheme 1

crystals of compound **1** for X-ray diffraction could not be prepared in a simple manner. Compound **1** single crystals of suitable size and diffraction quality were obtained in a reaction procedure employing the liquid-diffusion technique in a U-tube. The methanol solutions of diamine and aldehyde were allowed to diffuse to each other through a chloroform layer. During a period of about a week yellow-brown needle-like crystals with appropriate cross section dimensions appeared in the chloroform layer. Numerous diffusion experiments were made using various concentrations of the reactants. Best results were achieved with a concentration of aldehyde and amine of 0.3 and 1 mol dm⁻³, respectively. Dark red compound **2** single crystals were grown from a slightly oversaturated S,S-dimethylsulphoxyde solution by standing at room temperature for a period of two weeks.

Table I lists general and crystallographic data and the details of data collection and refinement procedure. The X-ray diffraction was performed by means of a Philips PW1100 diffractometer (upgraded by STOE, Germany) using graphite monochro-

TABLE I

General and crystal data and details of the diffraction procedure and structure determination for compounds **1** and **2**

	Compound 1	Compound 2
Empirical formula	C ₂₆ H ₂₄ N ₂ O ₂	C ₂₈ H ₂₈ N ₂ O ₂
Molecular weight, M_r	396.48	424.54
Crystal system and space group	Orthorhombic, <i>Pbca</i> (No. 61)	Orthorhombic, <i>Pbcn</i> (No. 60)
Unit cell parameters:		
	$a = 8.142(2) \text{ \AA}$	$a = 23.738(2) \text{ \AA}$
	$b = 9.097(3) \text{ \AA}$	$b = 8.9933(8) \text{ \AA}$
	$c = 28.812(8) \text{ \AA}$	$c = 10.4206(8) \text{ \AA}$
	$V = 2134.0(11) \text{ \AA}^3$	$V = 2224.6(3) \text{ \AA}^3$
Unit cell contents, Z	4	4
Crystal size/mm ³	0.72 × 0.67 × 0.23	0.75 × 0.64 × 0.60
$D_{\text{calc}}/\text{g cm}^{-3}$	1.234	1.268
μ/mm^{-1}	0.07	0.07
$\lambda(\text{MoK}\alpha)/\text{\AA}$	0.71073	0.71073
2θ range/°	4–54	4–54
Number of measured reflections	2345	2431
Number of observed reflections, $I_{\text{net}} \geq 1.0 \sigma I_{\text{net}}$	843	1268
$F(000)$	840	904
$(\Delta/\sigma)_{\text{max}}$	0.005	0.004
No. of least-squares parameters	136	145
R	0.032	0.038
wR	0.049	0.064
S	1.09	1.14
$\Delta\rho_{\text{min, max}} (\text{e}/\text{\AA}^3)$	-0.08; 0.11	-0.11; 0.20

mated MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). Cell dimensions were determined using fitting angles for 21 (compound **1**) and 35 (compound **2**) general strong reflections in the range $18^\circ < 2\theta < 25^\circ$ and $20^\circ < 2\theta < 35^\circ$, respectively. The reflections were collected by the ω -scan technique in the range $4^\circ \leq 2\theta \leq 54^\circ$. Single crystals used in diffraction experiment had the dimensions $0.72 \times 0.67 \times 0.23 \text{ mm}^3$ (compound **1**) and $0.75 \times 0.64 \times 0.60 \text{ mm}^3$ (compound **2**). Due to the low intensities of the collected data, the criterion for observed and in the refinement used reflections was $I_{\text{net}} \geq 1\sigma I_{\text{net}}$. The structures were solved by direct methods and refined anisotropically for all non-hydrogen atoms using the full-matrix least-squares refinement. Stereochemical rules for Csp³ and Csp² were used in calculation of the H-atom positions. They were placed 0.95 Å apart from the attached carbon atoms. Hydrogen atoms involved in the intramolecular hydrogen bonding were located according to the difference Fourier maps calculated at R factor values below 0.05. All hydrogen atoms were included in structure factor calculations with isotropic thermal parameter $1.2 \times U_{\text{eq}}$ of the corresponding carbon atom. The refinements converged at $R = 0.032$ (compound **1**) and $R = 0.038$ (compound **2**). Atomic scattering factors for neutral atomic species were taken from the *International Tables for X-ray Crystallography*.¹¹ The final difference Fourier maps were featureless. Computer programs used: DIF4,¹² REDU4¹³ (data collection, cell refinement and Lp-corrections); NRCVAX94¹⁴ (solving, refinement and preparation of tables for publication); ORTEPIII¹⁵ (molecular graphics).

RESULTS AND DISCUSSION

Discussion of the Structures

ORTEPIII drawings of compound **1** and **2** discrete molecules in general orientation are depicted in Figure 1. Table II lists the fractional coordinates along with equivalent isotropic thermal displacement parameters for non-H atoms. Valence bond lengths and bond angles are summarized in Table III.

Both compounds are built up of discrete molecules lying in the 4 (a) special position of space group *Pbca* (compound **1**) and space group *Pbcn* (compound **2**), respectively, with the imposed inversion centre placed in the middle of the aliphatic chain. The existence of internal molecular symmetry in both compounds is manifested in a specific stereochemical way with two parallel (but not co-planar) naphthaldimine moieties being bridged with either 4- or 6-membered aliphatic chain. This could be named a »stepped« conformation (Figure 2). All aliphatic carbon atoms in compound **1** are coplanar due to the inversion centre symmetry operation. The dihedral angle between the plane through these atoms and the plane defined by the naphthalene ring moiety is $93.4(1)^\circ$. A similar conformation is observed in compound **2** with the corresponding dihedral angle amounting to $96.6(1)^\circ$, but, as it might be expected, the longer aliphatic chain decreases the symmetry of the central part of the molecule. Namely, all six methylene carbon atoms are not situated in the mean least-squares plane with min. and max. discrepancies being $0.033(3)$ and $0.064(3)$ Å, respectively.

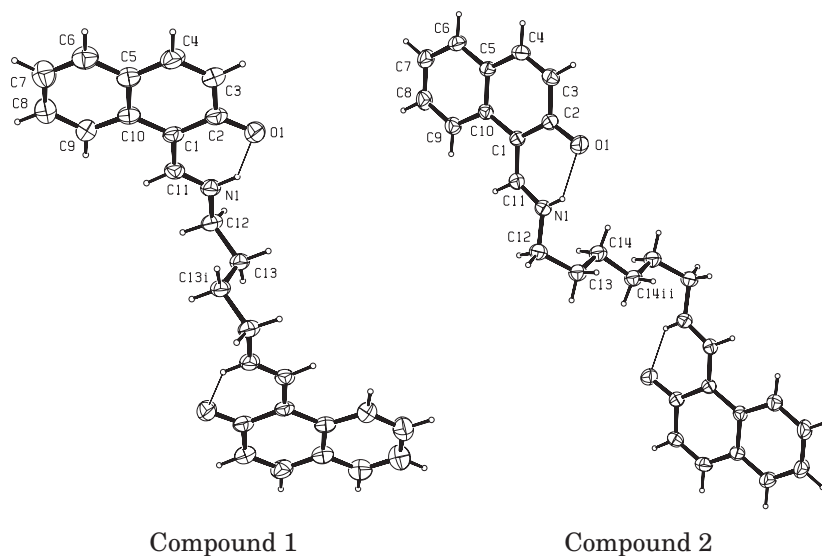


Figure 1. An ORTEPIII presentation of compound **1** and compound **2** molecules in general orientation with our crystallographic atom numbering scheme. The non-hydrogen atom thermal ellipsoids are drawn at the 30% probability level. Small open circles of the arbitrary radius represent the hydrogen atoms. Symmetry codes: (i) $2 - x, -y, -z$; (ii) $1 - x, -y, -z$.

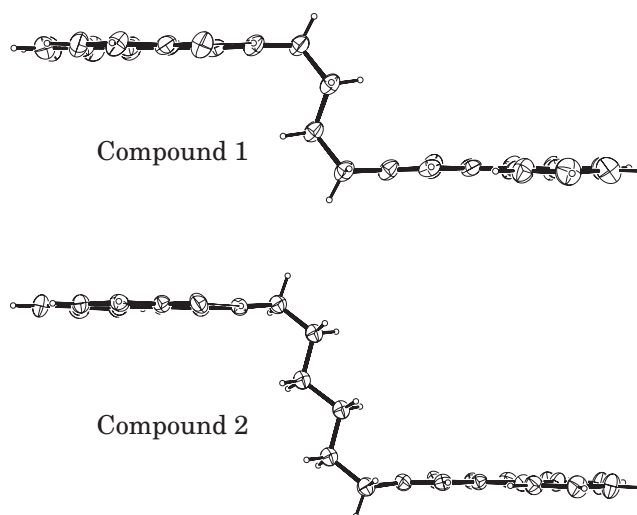


Figure 2. The spatial relationship between terminal naphthaldimine moieties and the central bridging aliphatic chain in compounds **1** and **2**.

TABLE II
 Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
 for compounds **1** and **2**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*
Compound 1				
O1	0.4779(2)	-0.0292(2)	0.0536(1)	0.0713(7)
N1	0.7234(2)	0.1309(2)	0.0267(1)	0.0570(8)
C1	0.5830(3)	0.1677(3)	0.0988(1)	0.0518(8)
C2	0.4703(3)	0.0507(3)	0.0897(1)	0.0590(9)
C3	0.3452(4)	0.0245(3)	0.1244(1)	0.0756(11)
C4	0.3328(4)	0.1048(4)	0.1632(1)	0.0760(11)
C5	0.4439(3)	0.2216(3)	0.1733(1)	0.0647(10)
C6	0.4300(4)	0.3034(4)	0.2145(1)	0.0771(12)
C7	0.5317(4)	0.4164(4)	0.2239(1)	0.0856(14)
C8	0.6529(4)	0.4518(4)	0.1922(1)	0.0861(14)
C9	0.6721(4)	0.3734(3)	0.1522(1)	0.0729(11)
C10	0.5696(3)	0.2558(3)	0.1412(1)	0.0564(9)
C11	0.7064(3)	0.1971(3)	0.0662(1)	0.0542(9)
C12	0.8475(3)	0.1622(3)	-0.0079(1)	0.0602(9)
C13	0.9469(3)	0.0265(3)	-0.0194(1)	0.0563(8)
H1N1	0.6525	0.0596	0.0197	
Compound 2				
O1	0.4574(1)	0.3917(2)	0.1411(1)	0.0613(6)
N1	0.4031(1)	0.2455(2)	0.3160(2)	0.0489(6)
C1	0.3750(1)	0.2531(2)	0.0945(2)	0.0416(5)
C2	0.4206(1)	0.3471(2)	0.0594(2)	0.0459(6)
C3	0.4258(1)	0.3912(2)	-0.0730(2)	0.0510(7)
C4	0.3883(1)	0.3457(3)	-0.1617(2)	0.0532(7)
C5	0.3414(1)	0.2526(2)	-0.1303(2)	0.0464(6)
C6	0.3020(1)	0.2089(3)	-0.2239(2)	0.0593(8)
C7	0.2564(1)	0.1233(3)	-0.1931(2)	0.0657(10)
C8	0.2494(1)	0.0764(3)	-0.0680(2)	0.0637(9)
C9	0.2873(1)	0.1163(2)	0.0256(2)	0.0536(7)
C10	0.3343(1)	0.2056(2)	-0.0018(2)	0.0423(6)
C11	0.3698(1)	0.2073(2)	0.2238(2)	0.0459(7)
C12	0.3924(1)	0.2058(3)	0.4493(2)	0.0572(8)
C13	0.4446(1)	0.1561(3)	0.5209(2)	0.0570(8)
C14	0.4761(1)	0.0310(3)	0.4580(2)	0.0573(8)
H1N1	0.4298	0.2981	0.2964	

$$*U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} \mathbf{a}_i^* \mathbf{a}_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

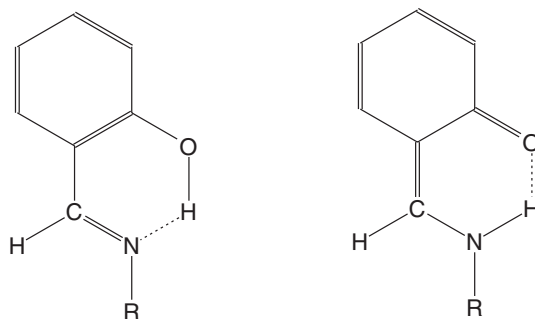
TABLE III
 Interatomic bond distances (Å) and valence bond angles (°)
 for compounds **1** and **2**

	Compound 1	Compound 2
	Bond distances (Å)	
O1–C2	1.270(3)	1.284(3)
N1–C11	1.296(3)	1.291(3)
N1–C12	1.447(3)	1.457(3)
C1–C2	1.429(4)	1.421(3)
C1–C10	1.465(4)	1.456(3)
C1–C11	1.401(3)	1.415(3)
C2–C3	1.447(4)	1.441(3)
C3–C4	1.338(5)	1.346(3)
C4–C5	1.426(4)	1.432(3)
C5–C6	1.407(4)	1.407(3)
C5–C10	1.414(4)	1.414(3)
C6–C7	1.348(5)	1.367(4)
C7–C8	1.383(5)	1.380(4)
C8–C9	1.362(5)	1.374(3)
C9–C10	1.394(4)	1.405(3)
C12–C13	1.513(3)	1.514(3)
C13–C13 ⁱ	1.493(5)	
C13–C14		1.501(3)
C14–C14 ⁱⁱ		1.539(4)
	Bond angles (°)	
O1–C2–C1	123.0(2)	122.3(2)
O1–C2–C3	120.4(2)	119.3(2)
C1–C2–C3	116.7(3)	118.4(2)
C2–C3–C4	122.7(3)	121.1(2)
C3–C4–C5	122.0(3)	122.4(2)
C4–C5–C6	121.1(3)	121.5(2)
C4–C5–C10	119.3(3)	118.9(2)
C6–C5–C10	119.7(3)	119.6(2)
C5–C6–C7	121.5(3)	121.4(2)
C6–C7–C8	118.9(3)	119.3(2)
C7–C8–C9	121.2(3)	120.8(2)
C8–C9–C10	121.7(3)	121.7(2)
C5–C10–C9	116.9(3)	117.2(2)
C1–C10–C5	118.6(2)	119.1(2)
C1–C10–C9	124.5(2)	123.7(2)
C2–C1–C10	120.8(2)	120.1(2)

TABLE III. Continued

	Compound 1	Compound 2
C10–C1–C11	120.5(2)	120.9(2)
C2–C1–C11	118.7(2)	118.9(2)
N1–C11–C1	125.3(2)	125.3(2)
C11–N1–C12	126.0(2)	122.5(2)
N1–C12–C13	111.3(2)	113.5(2)
C12–C13–C13	114.2(2)	
C12–C13–C14		114.4(2)
C13–C14–C14 ⁱⁱ		113.0(2)

In **1** and **2**, the *quinoid* phenomenon (Scheme II) was established.¹⁶ This effect was observed in many aromatic compounds containing a hydroxy group in *ortho* position to electron withdrawing substituents (e.g., –CHO, –NO₂).^{17,18} The *quinoid* effect in naphthalene fragment is accompanied by a rather peculiar crystallographic D_{2h} bond length pattern.¹⁹ Namely, the molecules of both compounds contain a significantly short C3–C4 bond length [1.338(5) Å (**1**); 1.346(3) Å (**2**)]. At the same time, C1–C2, C1–C10



Scheme 2

and C2–C3 bond distances were considerably lengthened to 1.429(4), 1.465(4), 1.447(4) Å (**1**), and 1.421(3), 1.456(3), 1.441(3) Å (**2**), respectively. The chelate ring bond length motif indicates the significant delocalization of π electrons. The C2–O1 and N1–C11 bond distances [1.270(3) Å, 1.296(3) Å (**1**); 1.284(3) Å, 1.291(3) Å (**2**)] are intermediar between single and double carbon to oxygen (1.362 Å, 1.222 Å) and carbon to nitrogen bond lengths (1.339 Å, 1.279 Å), respectively.¹⁹

The Schiff base derivatives having substituted an $-OH$ in position 2 to the aldimine group exist either as keto or enol tautomers. Depending on the tautomers present two extreme types of intramolecular hydrogen bond are possible: $O \cdots H-N$ in the case of keto and $O-H \cdots N$ in the case of enol tautomer. Sometimes, both keto and enol tautomers coexist, but not necessarily in the same amounts. Our crystallographic studies have shown that in different N-substituted naphthaldimines the keto form dominates with the $N-H \cdots O$ type of hydrogen bond.²⁰⁻²³ Neither **1** nor **2** are exceptions to this rule. The position of the H atom was in all cases unambiguously determined by the X-ray experiment. In **1**, the $N-H$ and $O \cdots H$ distances are 0.891 Å and 1.906 Å, while the corresponding distances in **2** are 0.816 Å and 1.939 Å, respectively. Another feature of the chelate ring formed by the intramolecular hydrogen linkage is the very short stereochemically induced $O \cdots N$ 'bite' distance amounting to 2.592(3) Å (compound **1**) and 2.591(2) Å (compound **2**), respectively. Although the keto tautomer prevails in naphthaldimines, there are no known salicylaldimines having the same tautomeric form in the solid state.

The C1, C2, C11, N1 and O1 chelate ring atoms in compound **1** as well as in compound **2**, lie essentially all in the same plane. The deviation of the compound **2** H-atom from the chelate plane is negligible (0.008 Å), while in compound **1** the H-atom is placed 0.055 Å away from the best plane calculated through the non-H chelate ring atoms. This large discrepancy is caused by the existence of a three-centre intermolecular hydrogen bond in compound **1**.²⁴ These bonds connect the neighboring molecules in the crystal *via* the N-imino hydrogen atom (Figure 3). The N1 nitrogen atom of a **1** molecule is thus hydrogen bonded to two O1 oxygen atoms, one from the same, and the other (symmetry operator: $-x + 1, -y, -z$) from the neighboring molecule at a distance 2.982 Å. In this way, the compound **1** molecules are associated into infinite chains spreading along the crystallographic $\{100\}$ direction.

It can be finally concluded that, in spite of the differences in molecular structures and crystal packing, both compounds **1** and **2** show a large similarity. The molecules possess an unusually high degree of symmetry in the solid state despite the presence of a flexible aliphatic chain. However, the lengthening of the bridging chain from four to six carbon atoms prevents the molecules from intermolecular hydrogen bonding. The difference in the intermolecular bonding (hydrogen bonding *vs.* van der Waals attractions) results in most of the macroscopic differences (melting point, solubility etc.) between compounds. It should be noted that the self-assembling of the molecules by the intermolecular hydrogen bonding is effected by their *bis*-bidentate configuration, combined with an appropriate aliphatic chain length.

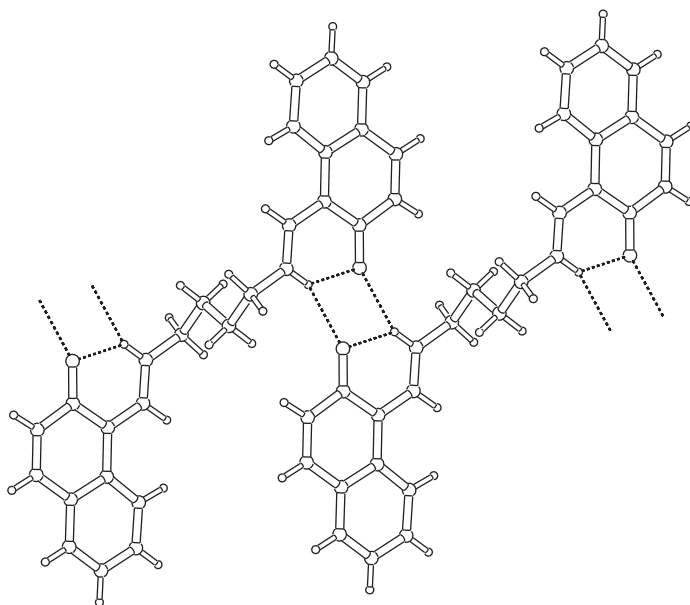


Figure. 3. A PLUTON96 drawing showing self-assembly of compound **1** molecules by three-centre intermolecular hydrogen bonding.

Thermal Analyses

By DSC measurements, the melting points of compounds **1** and **2** were determined at 234.5 and 175.3 °C, respectively. The melting point enthalpies are 49.9(2) kJ/mol (compound **1**) and 48.3(2) kJ/mol (compound **2**). In contrast to the higher melting point temperature and a larger melting point enthalpy of compound **1**, the decomposition temperature of compound **2** is higher (323.7 °C) than that of compound **1** (300.3 °C). An insight into the molecular interactions in the solid state, revealed by X-ray diffraction, supplied in both cases the explanation for the melting point and enthalpy values. Namely, the crystals of compound **1** are built up of molecules inter-linked by hydrogen bonds in the form of infinite ribbons, while the compound **2** crystals are made of discrete molecules held together by normal van der Waals attractions (for details, see crystallographic discussion).

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SAŽETAK

Priprava i struktura *N,N'*-butilen- i *N,N'*-heksilenbis(2-oksi-1-naftaldimina)

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N,N'-Butilenbis(2-oksi-1-naftaldimin) (spoj **1**) i *N,N'*-heksilenbis(2-oksi-1-naftaldimin) (spoj **2**) priređeni su reakcijom 2-hidroksi-1-naftaldehida i odgovarajućih alifatskih diamina. Standardni postupci TGA i DSC primijenjeni su za pobliže opisivanje spoja **1** i spoja **2**. Stereokemija spojeva u krutom stanju određena je raspršenjem rentgenskih zraka na monokristalu. Spoj **1**: C₂₆H₂₄N₂O₂, rompski sustav, prostorna grupa

$Pbca$, $a = 8.142(2)$ Å, $b = 9.097(3)$ Å, $c = 28.812(8)$ Å, $R = 0.032$. Spoj **2**: $C_{28}H_{28}N_2O_2$, rombski sustav, prostorna grupa $Pbcn$, $a = 23.738(2)$ Å, $b = 8.9933(8)$ Å, $c = 10.4206(8)$ Å, $R = 0.038$. Obje molekule posjeduju središte inverzije u sredini alifatskog lanca koji premošćuje terminalne naftaldiminske skupine. Naftalenski prstenovi pokazuju uz kinoidni efekt i dobro poznati raspored duljina veza koji odgovara kristalografskoj simetriji D_{2h} . U obadvije Schiffove baze prisutna je unutarmolekulska vodikova veza $N-H \cdots O$. Molekule spoja **1** dodatno su povezane međumolekulskim tri-centričnim vodikovim vezama.

Crystallographic data for the structures reported in this paper have been deposited at **Cambridge Crystallographic Data Centre**, 12 Union Road, Cambridge CB2 IEZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk) and can be obtained on request, free of charge, by quoting the publication citation and the **deposition number 100397**.