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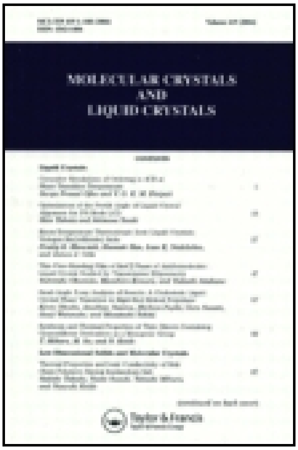

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Discotic Liquid Crystals of Transition Metal Complexes 7[1] : Dimer Discotic Rectangular Ordered Mesophase in Bis(p-n- alkoxybiphenylbutane-1,3-dionato)copper (II) Complexes

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Discotic Liquid Crystals of Transition Metal Complexes 7[1]: Dimer Discotic Rectangular Ordered Mesophase in Bis(*p*-*n*-alkoxybiphenylbutane-1,3-dionato)copper (II) Complexes

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A novel series of bis(*p*-*n*-alkoxybiphenylbutane-1,3-dionato)copper(II) complexes (*n*-alkoxy = $C_nH_{2n+1}O$, $n = 8 \sim 12$, 16, 18) have been synthesized and characterized. The C_nO-Cu complexes for $n = 9 \sim 12$, 16, and 18 have a unique dimer discotic rectangular ordered columnar (D_{ro}) mesophase. The X-ray diffraction powder pattern of the D_{ro} mesophase of the $C_{16}O$-Cu complex at 185°C gave the intradimer distance 5.12 Å, the interdimer distance in the column 9.40 Å, and the two-dimensional rectangular lattice constants $a = 46.8$ Å, $b = 16.7$ Å. The type of the present D_{ro} mesophase could be assigned as D_{ro} (C2/m).

Keywords: discotic liquid crystal, metal complex, dimer, β-diketonato copper(II)

I. INTRODUCTION

The first rectangular columnar (D_r) mesogen of an organic transition metal complex was recently reported by Ohta *et al.*—the octadodecyltetrapyrazinoporphyrazine-copper(II) complex has a rectangular disordered columnar (D_{rd}) mesophase.[2] So far as we know, this D_{rd} mesogen is the only example in the organic transition metal complexes. Destrade *et al.* classified discotic rectangular *disordered* columnar (D_{rd}) mesophases into different three types (Figure 1).[3] However, in this classification, a rectangular *ordered* columnar (D_{ro}) mesophase was not included.

In the present work, a new series of bis(*p*-*n*-alkoxybiphenylbutane-1,3-dionato)-copper(II) complexes (*n*-alkoxy = $C_nH_{2n+1}O$, $n = 8 \sim 12$, 16, 18; abbreviated as C_nO-Cu) has been synthesized. Among them, the C_nO-Cu ($n = 9 \sim 12$, 16, 18) complexes have D_{ro} mesophases. The C_nO-Cu complexes form dimers; the dimers pile up to form a column with the periodicity $h = 9.40$ Å, in the mesophase. The first D_{ro} mesophase was obtained in the penta-*O*-*n*-alkanoylglucopylanose deriv-

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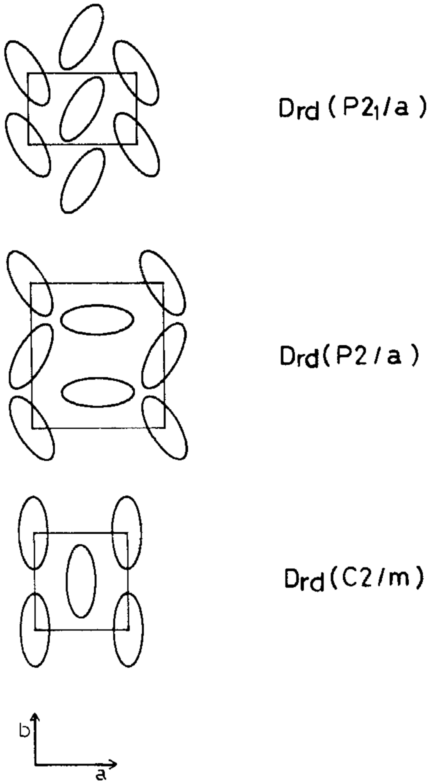

FIGURE 1 Three D_{rd} columnar mesophases classified by Destrade *et al.*[3]

atives.[4] The present C_nO-Cu complexes give another example of the D_{ro} mesophase. In this paper, we wish to report on this unique dimer discotic rectangular ordered columnar (D_{ro}) mesophase of the C_nO-Cu complexes.

II. EXPERIMENTAL

II-1. Synthesis

The coordination scheme of the present bis(*p*-*n*-alkoxybiphenyl butane-1,3-dionato)copper(II) complexes (C_nO-Cu: *n*-alkoxy = $C_nH_{2n+1}O$, $n = 8 \sim 12, 16, 18$) is shown in Scheme 1. The C_nO-Cu complexes were obtained from *p*-*n*-alkoxybiphenylbutane-1,3-dione (abbreviated as C_nO-Lig) as a starting material described in the previous paper.[5] In Table I are listed elemental analysis data, the yields, and recrystallization solvents of C_nO-Cu. The detailed procedure is described for $C_{16}O$-Cu as follows.

Bis(p-n-hexadecyloxybiphenylbutane-1,3-dionato)copper(II) complex (*$C_{16}O$-Cu*). To an ethanolic solution of 0.20 g (0.42 mmol) of $C_{16}O$-Lig at 50°C, ca. 1 ml of

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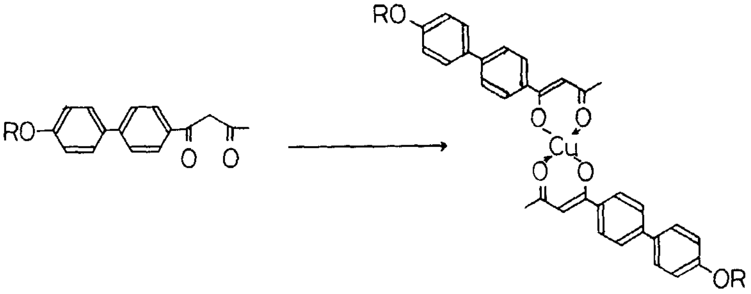

CnO - Cu

R = CnH2n+1 ; n = 8~12,16,18

SCHEME 1 Coordination scheme of bis(*p*-*n*-alkoxybiphenylbutane-1,3-dionato)copper(II) complexes, C_nO-Cu.

an aqueous solution (28%) of ammonium hydroxide and then an aqueous solution of 0.040 g (0.21 mmol) of cupric chloride dihydrate were added. The resulting green-gray precipitate was collected, washed with water and a small portion of ethanol several times and air dried. Recrystallization from benzene gave green powder of the complex (0.11 g, 52%).

For these C_nO-Cu complexes, C and H elemental analyses were satisfactory (Table I).

II-2. Measurements

The phase transition behaviors of these compounds synthesized here were observed with a polarizing microscope equipped with a heating plate controlled by a ther-

TABLE I

Elemental analysis data, yields, and recrystallization solvents for CnO-Cu.

n	Elemental analysis Found(calcd.)% C	H	Yield (%)	Recrystallization solvent
8	72.56(72.45)	7.36(7.38)	47	acetone:benzene = 2:3 (v/v)
9	73.01(73.09)	7.50(7.68)	49	acetone:benzene = 2:3
10	73.42(73.57)	7.82(7.86)	46	acetone:benzene = 2:3
11	73.81(73.90)	8.03(8.08)	49	acetone:benzene = 2:3
12	74.18(74.24)	8.23(8.32)	50	acetone:benzene = 2:3
16	75.44(75.36)	8.90(8.94)	52	benzene
18	75.97(76.17)	9.19(9.32)	51	chloroform

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moregulator, FP80 and FP82, and measured with a differential scanning calorimeter, Rigaku Thermoflex TG-DSC.

The X-ray diffraction powder patterns were employed to characterize the mesophases in the present compounds. The patterns were measured with Cu-Kα radiation, using a Rigaku Geigerflex equipped with a hand-made heating plate controlled by a thermoregulator.[6]

TABLE II

Phase transition temperatures (T) and enthalpy changes (ΔH) of C_nO-Cu complexes.

n	Phase[a] $\xrightarrow{T^{o}C[\Delta H(kcal/mol)]}$ Phase
8	$K \xrightarrow{224.0[4.2]}$ I.L.(decomp)
9	$K^{b} \xrightarrow{193.0[2.0]} D_{ro} \xrightarrow{213.2[4.0]}$ I.L.(decomp)
10	$K^{b} \xrightarrow{161.4[1.3]} D_{ro} \xrightarrow{211.8[8.1]}$ I.L.(decomp)
11	$K^{b} \xrightarrow{151.0[1.5]} D_{ro} \xrightarrow{210.5[8.5]}$ I.L.(decomp)
12	$K^{b} \xrightarrow{135.1[1.2]} D_{ro} \xrightarrow{208.8[9.9]}$ I.L.(decomp)
16	$K \xrightarrow{127.0[7.4]} D_{ro} \xrightarrow{199.9[10.1]}$ I.L.[c]
18	$K \xrightarrow{130.9[8.7]} D_{ro} \xrightarrow{196.5[11.1]}$ I.L.[c]

[a] Phase nomenclature: K = crystal, D_{ro} = discotic rectangular ordered columnar mesophase, and I.L. = isotropic liquid.

[b] For *n* = 9–12, several unidentified phase transitions were observed before melting to the mesophase only by DSC.

[c] These isotropic liquids are more stable than the others, and don't decompose during several heating and cooling cycles.

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III. RESULTS AND DISCUSSION

The C_nO-Cu complexes have a mesophase for $n = 9 \sim 12, 16, 18$. In Table II are summarized the phase transition temperatures and phase transition enthalpy changes for the C_nO-Cu complexes. All transition temperatures of the complexes are plotted against the number of carbon atoms in the alkoxy chains (n) in Figure 2. For $n = 9 \sim 12$, several unidentified phase transitions denoted with X marks in Figure 2 were observed before melting from the crystalline phase to the mesophase only by DSC measurements. These complexes gradually decompose at ca. 200°C. Thereby,

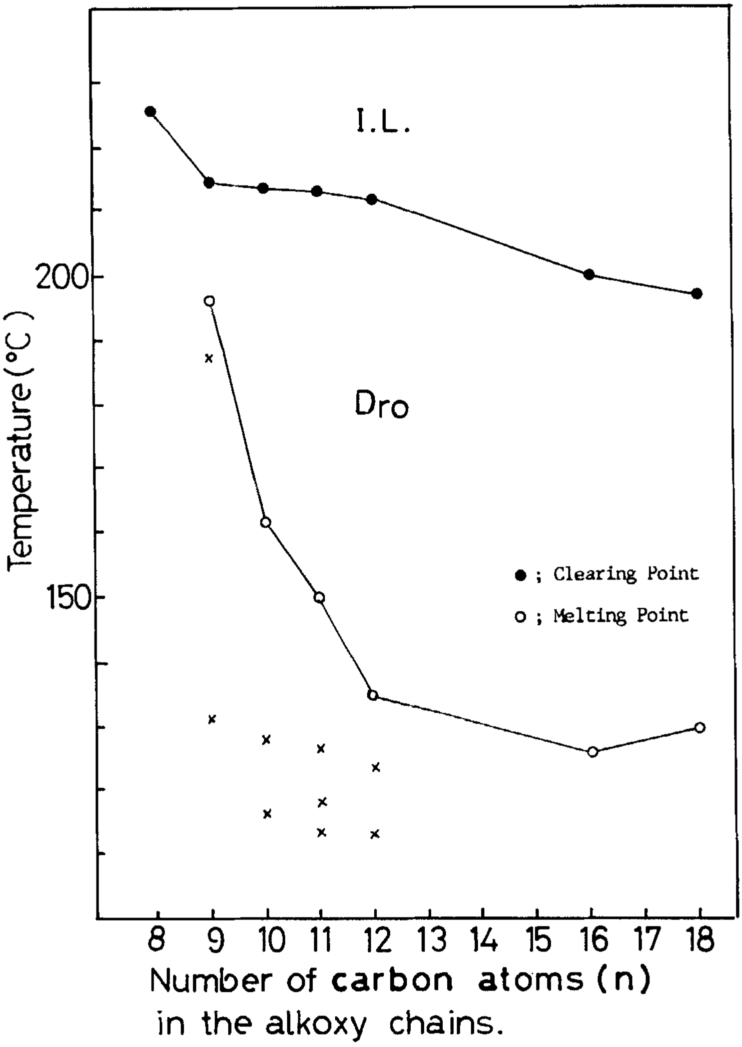

FIGURE 2 Phase transition temperatures vs. number of carbon atoms in the alkoxy chain for C_nO-Cu.

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the X-marked peaks could not be identified by microscopic observations in the cooling stages. The temperature range of the mesophase become wider with the increase of the carbon number (n). It is the widest for $n = 12$ ($\Delta T = 73.7$°C). The present mesophase is characterized for $C_{16}O$-Cu in detail as follows.

Dimer discotic rectangular ordered columnar (D_{ro}) *mesophase in* $C_{16}O$*-Cu.* When the $C_{16}O$-Cu complex is heated from room temperature, it shows a crystal-mesophase transition at 127.0°C (m.p.), and then a mesophase-isotropic liquid at 199.9°C (c.p.). When the isotropic liquid was cooled to 199.0°C, the mesophase gave a mosaic texture as shown in Figure 3. The X-ray diffraction powder pattern of the mesophase at 185°C is shown in Figure 4. All of the spacings observed in this pattern are listed in Table IIIa. Surprisingly, these spacings completely correspond to the reflections from a two-dimensional rectangular lattice. Figure 5 shows the reciprocal lattice of the corresponding two-dimensional rectangular system which was described assuming that two narrow peaks (1 and 2) in Figure 4 are (200) and (110) reflections from the lattice, respectively; the reciprocal lattice constants $a^* = 1/46.8$ Å, $b^* = 1/16.7$ Å. All of the spacings for the mesophase in $C_{18}O$-Cu at 180°C can also be indexed to the reflections from a two-dimensional rectangular lattice (Table IIIb). Thus, these X-ray diffraction patterns are compatible with those of discotic rectangular columnar (D_r) mesophases.

However, two questions arise: Q1, the C_nO-Cu complexes can not be expected to be discotic because of the nearly rod-like molecular shapes; Q2, the spacing of 9.40 Å for $C_{16}O$-Cu was assigned to a (500) reflection, but the assignment is contrary

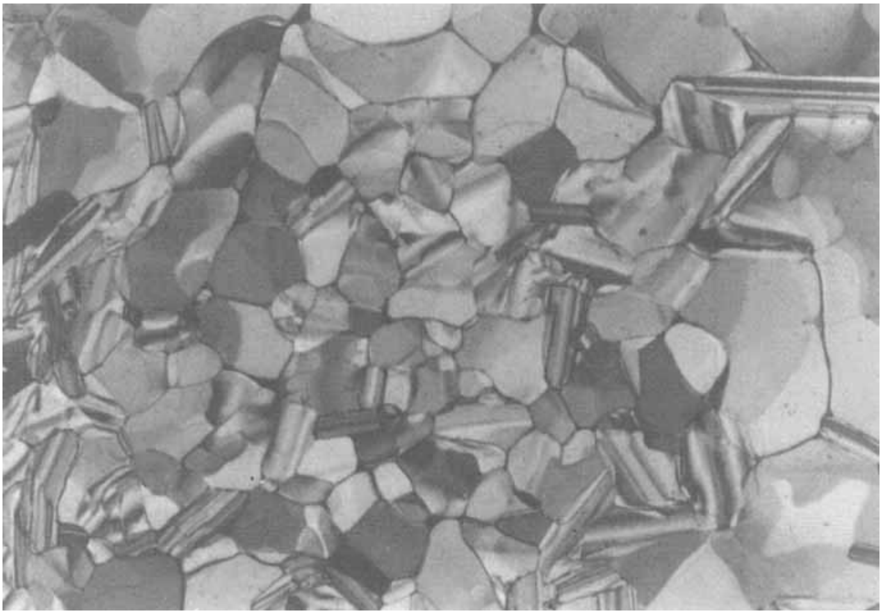

FIGURE 3 The mosaic texture of bis(p-n-hexadecyloxybiphenylbutane-1,3-dionato)copper(II) complexe, $C_{16}O$-Cu, obtained from the isotropic liquid on cooling to 199.0°C. See Color Plate V.

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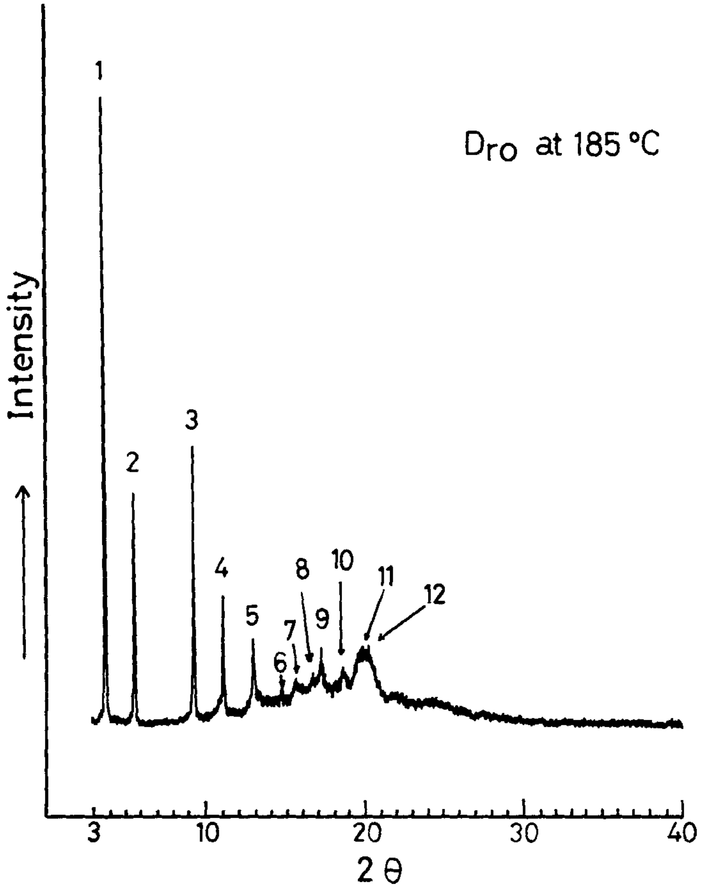

FIGURE 4 X-Ray diffraction powder pattern of the $C_{16}O$-Cu complex at 185°C.

to the extinction rule of two-dimensional rectangular lattices, h0: $h = 2n + 1$, 0k: $k = 2n + 1$ for P21/a[7]; $h + k = 2n + 1$ for C2/m.

For Q1: As shown in Figure 6, the Corey-Pauling-Koltum (CPK) models of this $C_{16}O$-Cu complex are not discotic but nearly rod-like. Nevertheless, assuming that the complexes form dimers as illustrated in Figure 8-1, the shapes of the dimers can be regarded to be discotic. Usha *et al.* reported a homologous bis[1,3-di(*p*-*n*-octyloxyphenyl)propane-1,3-dionato]copper(II) complex which forms dimers in the crystalline state.[8] Therefore, it can be assumed that the present C_nO-Cu complexes also form dimers. Moreover, Mühlberger *et al.* reported another homologous bis{[4-trans-(4-alkylcyclo-hexyl)phenyl]-alkylpropane-1,3-dionato}copper(II) complex, the single-crystal X-ray diffraction data of which gave the intermolecular distance 5.04 Å.[9] The narrow peaks at 5.12 and 5.11 Å were observed for $C_{16}O$-Cu and $C_{18}O$-Cu, respectively (Figure 4 and Table III). Probably, these peaks correspond to the intradimer distance.

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TABLE III

X-ray diffraction data: a) the mesophase of $C_{16}O$-Cu at 185°C, b) the mesophase of $C_{18}O$-Cu at 180°C.

a) $C_{16}O$-Cu: T = 185°C *a* = 46.8 Å, *b* = 16.7 Å

Peak No.	Measured lattice spacing(Å)	Calculated lattice spacing(Å)	(h k l)	
1	23.38	23.4	(2 0 0)	
2	15.71	15.7	(1 1 0)	
3	9.40	9.35	(5 0 0)	a,a$^+$,b
4	7.86	7.86	(2 2 0)	
5	6.75	6.79	(4 2 0)	
6	5.94	5.85	(8 0 0)	
7	5.67	5.69	(6 2 0)	
8	5.25	5.24	(3 3 0)	
9	5.12	5.21	(7 2 0)	a,c
10	4.74	4.70	(5 3 0)	
11	4.53	4.53	(6 3 0)	a
12	4.40	4.41	(9 2 0)	a

b) $C_{18}O$-Cu: T = 180°C a = 50.4 Å, b = 18.0 Å

Peak No.	Measured lattice spacing(Å)	Calculated lattice spacing(Å)	(h k l)	
1	25.19	25.2	(2 0 0)	
2	16.93	16.9	(1 1 0)	
3	10.10	10.1	(5 0 0)	a,a$^+$,b
4	8.43	8.47	(2 2 0)	
5	6.75	6.69	(7 1 0)	
6	5.11	5.15	(5 3 0)	c
7	4.49	4.49	(0 4 0)	

a These indexations are not consistent with the extinction rule of a two-dimensional rectangular lattice of C2/m : h+k=2n+1.

a^+ This indexation is not consistent also with the extinction rule of a two-dimensional rectangular lattice of $P2_1/a$: h0: h=2n+1, k0: k=2n+1.[7]

b This reflection corresponds to an interdimer distance.

c This reflection corresponds to an intradimer distance.

For Q2: The spacing at 9.40 Å assigned to the (500) reflection is contrary to the extinction rule of two-dimensional rectangular lattices.[7] Therefore, it can be assumed that the spacing at 9.40 Å is an inter-disc (dimer) distance (*h*) in a column—the present mesophase can be assumed to be a discotic rectangular ordered columnar (D_{ro}) mesophase.

The two assumptions mentioned above are estimated by a calculation of the density of the mesophase in the $C_{16}O$-Cu complex. The number (*Z*) of the molecules

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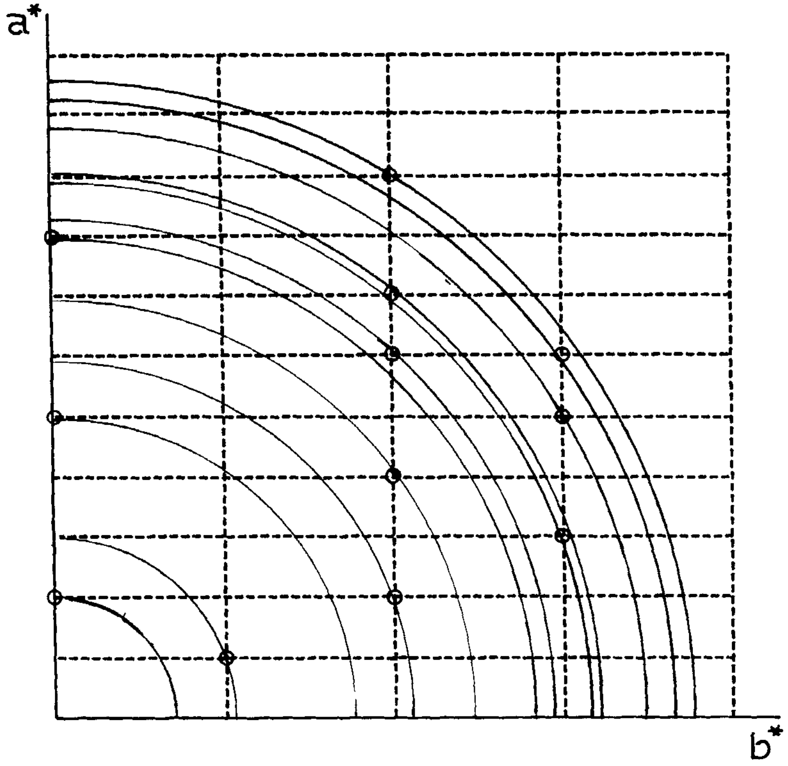

FIGURE 5 The reciprocal lattice of the rectangular system for $C_{16}O$-Cu at 185°C, $a^* = 1/46.8$ Å, $b^* = 1/16.7$ Å.

in the two-dimensional rectangular *ab* lattice with the inter-disc distance *h* (a = 46.8 Å, b = 16.7 Å, and h = 9.40 Å) can be calculated by the following equations.

$$\rho = (M/L)/(V/Z)$$

$$Z = \rho VL/M$$

where M = the molecular weight, L = avogadoro constant, and V = the molecular volume.

$$\begin{aligned} V &= a \times b \times h \\ &= 46.8 \times 16.7 \times 9.40 \\ &= 7330 \text{ Å}^3 \\ &= 7330 \times 10^{-24} \text{ cm}^3 \end{aligned}$$

Usha *et al.* reported that the density (ρ) of the homologous bis[1,3-di(*p*-*n*-octylphenyl)propane-1,3-dionato]copper(II) complex is 1.16 g/cm^3 in the crystalline state.[8]

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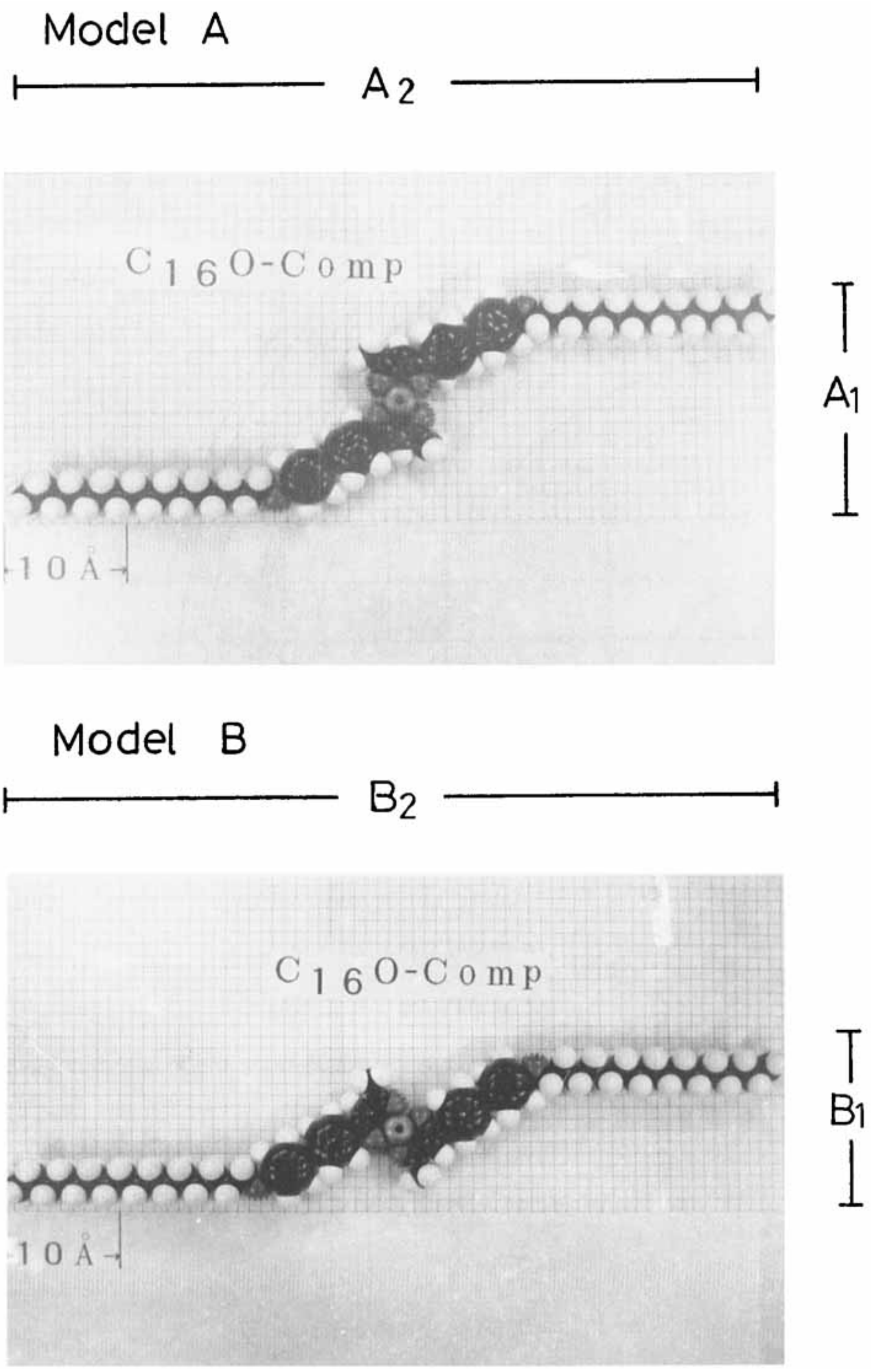

FIGURE 6 Corey-Pualing-Koltun (CPK) molecular models of $C_{16}O$-Cu.

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The density (ρ) in the mesophase of $C_{16}O$-Cu at 185°C is assumed as 0.95 g/cm^3 smaller than 1.16 g/cm^3, because the mesophase at 185°C swells from the crystalline phase at r.t.

$$Z = \frac{0.95 \times 7330 \times 10^{-24} \times 6.02205 \times 10^{23}}{1018.97}$$

$$= 4.1$$

Therefore, about four molecules exist in the *abh* lattice of the $C_{16}O$-Cu complex.

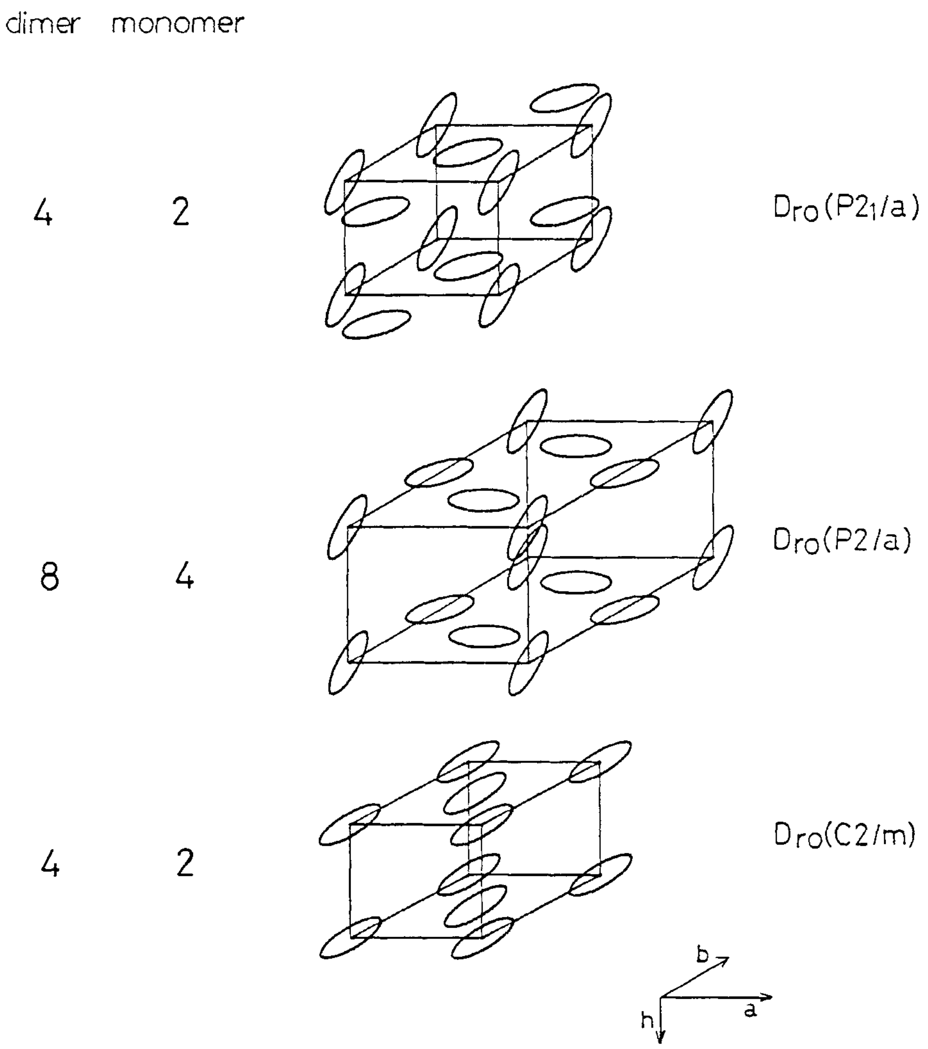


FIGURE 7 The three types of D_{ro} mesophases. The number of complexes in the *abh* lattice.

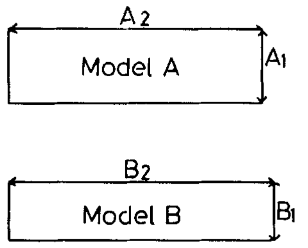
Hereupon, it still remains a question as to which type of rectangular lattice (D_{ro}(C2/m), D_{ro}(P2/a), or D_{ro}($P2_1$/a)) this rectangular-ordered (D_{ro}) mesophase belongs to, as illustrated in Figure 7. The number of the molecules in an *abh* lattice of D_{ro}($P2_1$/a) and/or D_{ro}(C2/m) is two when the complexes exist as monomers, and four for the dimers. The number of the molecules in an *abh* lattice of D_{ro}(P2/a) is four when the complexes exist as monomers, and eight for the dimers. Since the number (Z) of the molecules in a rectangular-ordered lattice is four as calculated above, the mesophase type is D_{ro}(P2/a) when the complexes exist as monomers, and D_{ro}(C2/m) or D_{ro}($P2_1$/a) when they exist as dimers.

The type of the D_{ro} mesophase is estimated from the molecular sizes of the present complexes. The molecular sizes measured with the CPK models in Figure 6 are listed in Table IV: for Model A, A_1 = 18.5 Å, A_2 = 63.0 Å; for Model B, B_1 = 14.0 Å, B_2 = 66.0 Å in C_{16}O-Cu. When the mesophase type is D_{ro}(P2/a), a two-molecular length is necessary in the *b*-axis direction and a one-molecular length in the *a*-axis direction. Taking into account the observed values, *a* = 46.8 Å, *b* = 16.7 Å, the molecules could not be compacted in the lattice even if the molecules might interdigitate to a great extent. Hence, the possibility of the D_{ro}(P2/a) mesophase type can be excluded; that is, it has been proven that the complexes exist as the dimers. Therefore, the mesophase type is either D_{ro}($P2_1$/a) or D_{ro}(C2/m)

TABLE IV

The molecular sizes measured from the C.P.K. molecular models of the C_{16}O-Cu and C_{18}O-Cu complexes.

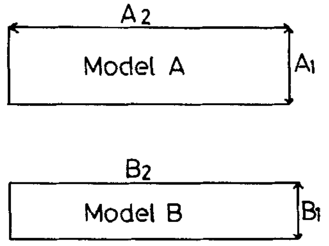
C_{16}O-Cu



A_1 = 18.5 Å
A_2 = 63.0 Å

B_1 = 14.0 Å
B_2 = 66.0 Å

C_{18}O-Cu



A_1 = 18.5 Å
A_2 = 69.0 Å

B_1 = 14.0 Å
B_2 = 72.0A

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and the absence of a peak corresponding to the (210) plane (Table III) suggests that the mesophase type is D_{ro}(C2/m).[10]

The structure of the D_{ro}(C2/m) mesophase mentioned above is illustrated in Figure 8. The complexes form dimers (Figure 8-1). The intradimer distance and the interdimer distance in the column are 5.12 and 9.40 Å, respectively. The cores of the dimers are inclined to the column axis (Figure 8-2). A schematic representation of the structure in this D_{ro}(C2/m) mesophase is shown in Figure 8-3.

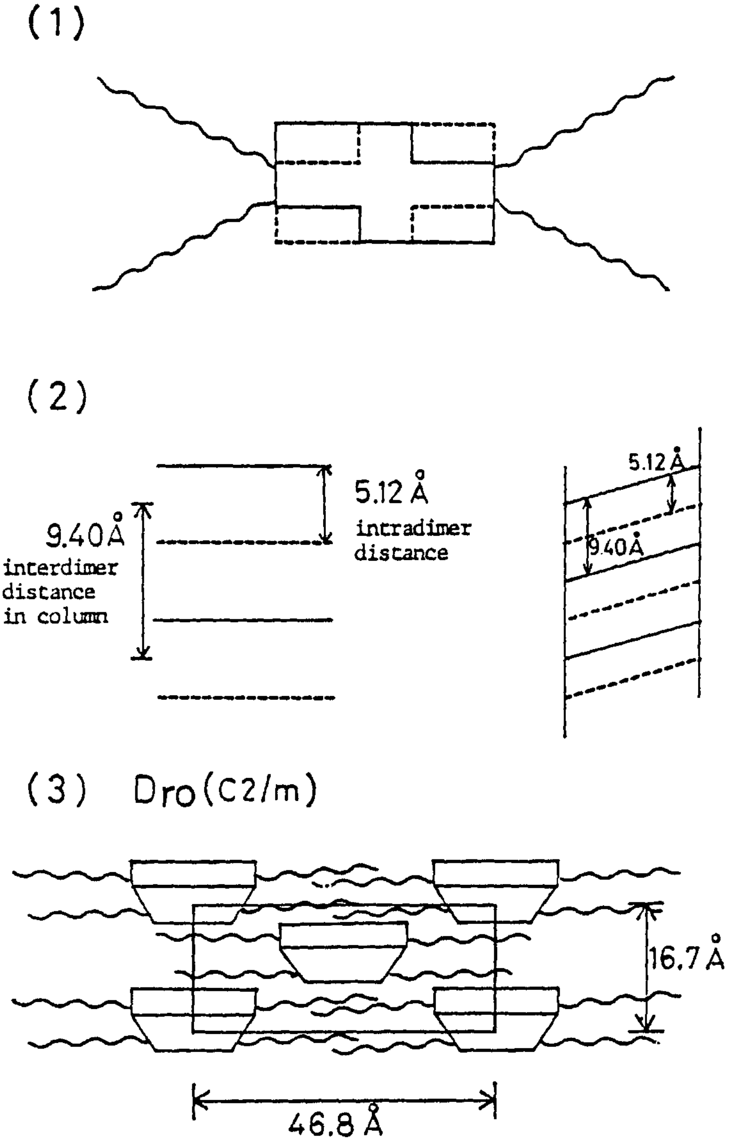

FIGURE 8 Schematic representation of the structure of the D_{ro}(C2/m) mesophase in the C_{16}O-Cu complexes.

Levelut reported that a discotic mesogen needs more than six peripheral chains in a disc-like molecule.[11] However, Kawada *et al.* reported the first example in which only a two-sided chain-substituted compound shows discotic columnar mesomorphism.[12] Furthermore, Praefcke *et al.* reported that two long alkyl-chain-substituted S,S-dialkyl dithioacetals of aldoses form the multimers to exhibit hexagonal columnar mesomorphism.[13] The present two long chain-substituted C_nO-Cu complexes form dimers with four chains to exhibit columnar mesomorphism, as mentioned above. From these facts, we think that the appearance of the discotic mesophase depends not on the number of the side chains in a disc-like molecule but on the number of the peripheral chains in a form of the aggregated molecules.

IV. CONCLUSION

A novel series of bis(*p*-*n*-alkoxybiphenylbutane-1,3-dionato)copper(II) complexes (*n*-alkoxy = $C_nH_{2n+1}O$, $n = 8 \sim 12$, 16, 18) have been synthesized. It was found that the C_nO-Cu complexes for $n = 9 \sim 12$, 16, 18 have a unique dimer discotic rectangular-ordered columnar (D_{ro}) mesophase. The D_{ro} mesophase of the $C_{16}O$-Cu complex at 185°C gave the intradimer distance 5.12 Å, the interdimer distance in the column 9.40 Å, and the two-dimensional rectangular lattice constants a = 46.8 Å, b = 16.7 Å. The type of the present D_{ro} mesophase can be assigned as $D_{ro}(C2/m)$.

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