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Liquid Crystals

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Properties of liquid-crystalline copper(II) complexes and their binary mixtures

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Properties of liquid-crystalline copper(II) complexes and their binary mixtures†

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Eight new copper compounds with mesogenic properties from the series of the bis{1-[4-*trans*-(4-alkylcyclohexyl)phenyl]-alkylpropane-1,3-dionato} copper(II) complexes have been prepared. The thermal behaviour of these compounds was investigated by means of polarizing microscopy and differential scanning calorimetry (D.S.C.) measurements. The mesophase, which could be found in most of them, is of the monotropic nematic type. This has been confirmed by miscibility experiments. The physical data of a binary mixture with an enantiotropic mesophase are also presented. In addition the crystal structures of two of the compounds are reported.

1. *Introduction*

The practical applications of liquid crystals have led to the demand for materials with special optical and magnetic properties. A new approach in this direction could be the combination of mesomorphic properties with those of transition metal complexes. The first copper-containing liquid crystals were prepared by Giroud-Godquin and Billard [1, 2]. Recent research on such liquid crystals has concentrated mainly on copper(II) phthalocyanines [3–5] which can lead to a new class of one dimensional conductors, Schiff's base complexes [6–9] where the first polymeric systems have been described recently [10], copper(II) alkanoates [11] or copper(II) acetylacetonates [12–15] which show a broad liquid-crystalline range and the appearance of rod- and disc-like mesophases. Of special interest is the preparation of copper(II) complexes with both kinds of mesophases in order to investigate a transition between discotic and rod-like mesophases, as mentioned by Chandrasekhar *et al.* [13].

2. Experimental

1[4-*trans*-(4-pentylcyclohexyl)phenyl]-undecandion-1·3 has been prepared, the other ligands used to prepare the copper(II) complexes were a gift from E. Merck, Darmstadt, F.R. Germany. The preparation of the complexes followed the scheme described in [13, 16, 17]. The elemental analysis data are given in table 1. The types of phases and the transition temperatures were determined using a polarizing microscope (Leitz Orthoplan-Pol) equipped with a hot stage and a controller (Mettler FP82 and FP80). The transition enthalpies were determined with D.S.C. equipment (DuPont Thermal Analyser 990, calibrated with pure indium as standard; see table 2).

Single crystals were obtained by evaporation from a solution of toluene. For X-ray measurements of single crystals of Cu31 and Cu38 an automatic Stoe-Stadi-4 diffractometer with Mo-K_α radiation and also the program package SHELX-76 was used for structure calculations [18]. The crystal structures were solved starting from

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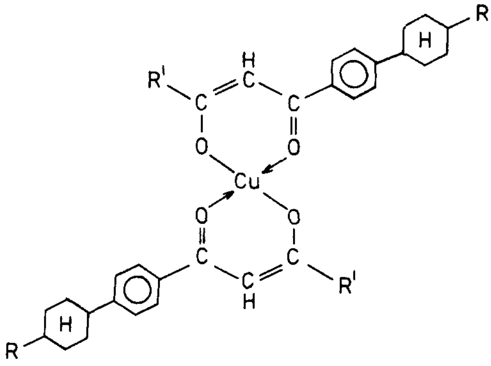
† Part of Dr-Ing. thesis of Bettina Mühlberger, D17, Technische Hochschule Darmstadt.

Table 1. Elemental data for the copper(II) complexes.



Compound	R	R'	Calculated/ per cent C	Calculated/ per cent H	Found/per cent C	Found/per cent H
Cu72	C_7H_{15}	C_2H_5	74·43	9·11	74·09	8·96
Cu51	C_5H_{11}	CH_3	73·03	8·47	73·14	8·57
Cu52	C_5H_{11}	C_2H_5	73·55	8·70	73·66	8·76
Cu58	C_5H_{11}	C_8H_{17}	75·84	9·77	76·13	10·05
Cu31	C_3H_7	CH_3	71·95	7·94	73·02	8·07
Cu33	C_3H_7	C_3H_7	73·06	8·47	73·33	8·43
Cu35	C_3H_7	C_5H_{11}	74·00	8·91	74·16	8·99
Cu36	C_3H_7	C_6H_{13}	74·43	9·11	74·92	9·30
Cu37	C_3H_7	C_7H_{15}	74·82	9·29	75·00	9·24
Cu38	C_3H_7	C_8H_{17}	75·18	9·46	75·02	9·68

Table 2. Thermodynamic data of the compounds investigated; estimated standard deviations for the temperature measurements are ±1·0 K. For the enthalpy measurements we calculated a maximal error of 6 per cent. T_{CI} is the melting and T_{NI} the clearing temperature; ΔH_m is the melting enthalpy.

Compound	$T_{CI}/°C$	$\Delta H_m/kJ\,mol^{-1}$	$T_{NI}/°C$
Cu72	194	(*a*)	187
	196	(*a*)	
Cu51	224	54	
Cu52	196	45	191
Cu58	127	(*b*)	129
	135	(*b*)	
Cu31	246	50	
Cu33	193	45	169
Cu35	169	46	148
Cu36	157	47	141
Cu37	143	45	128
Cu38	134	61	131
	148	45	

(*a*) We have not been able to separate the D.S.C. peaks.

(*b*) We have found different enthalpies with different heating rates because of the double melting behaviour of this compound.

Table 3. Experimental conditions for the structure determination and crystal structure data of Cu38 and Cu31. Estimated standard deviations are given in parantheses.

	Cu38	Cu31
Crystal habit	Prism	Prism
Crystal size/mm^3	0·18 × 0·3 × 0·8	0·22 × 0·28 × 0·4
Wavelength (Mo-K_α)/Å	0·710 69	0·710 69
Monochromator	Graphite	Graphite
Temperature/K	299	298
Absorption coefficient/cm^{-1}	4.6	5·99
$(\sin\theta/\lambda)_{max}$/Å^{-1}	0·538	0·538
Number of reflections measured	12 857	9432
Symmetry independent reflections	6391	4859
Reflections considered	5812	4122
Number of free parameters	488	376
$F(000)$	902	678
$R(F)$	0·0685	0·1658
$R_w(F)$	0·0670	0·1564
Molecular formula	$C_{52}H_{78}O_4Cu$	$C_{38}H_{50}O_4Cu$
Space group	$P\bar{1}$	$P\bar{1}$
Z	2	2
a/Å	23·340(4)	13·916(6)
b/Å	10·722(2)	11·992(5)
c/Å	10·079(2)	11·712(5)
α/degrees	83·47(1)	74·80(2)
β/degrees	88·21(1)	85·85(2)
γ/degrees	77·10(1)	78·30(2)
Volume of the unit cell/Å^3	2442·7	1846·6
ϱ/g cm^{-3}	1·129	1·140

the copper positions obtained from a Patterson synthesis. The positions of all other atoms, except the hydrogen, were obtained by successive Fourier methods and least squares refinements. The hydrogen atoms were given their idealized positions with fixed isotropic thermal parameters. Isotropic temperature factors have been refined for the terminal carbon atoms, anisotropic ones for the other non-hydrogen atoms. Table 3 shows the crystallographic data for Cu38 and Cu31. The atomic positional parameters and the equivalent temperature factors are summarized in tables 4 and 5, respectively. The Fo/Fc lists have been deposited with the British Library, Document Supply Centre; copies of these tables, comprising 26 pages, can be obtained from the British Library by using the procedure described at the end of this issue and by quoting the number SUP 16510. For the X-ray investigations on powder samples of Cu58 a Stoe horizontal focussing diffractometer in an extended version was used [19]. For heating a specially designed oven was mounted on the goniometer head.

3. Results and discussion

3.1. *Thermodynamic data*

With the exception of Cu51 and Cu31 all of the compounds show monotropic nematic phases (see table 2) analogous to the bis[1-(*p*-*n*-decyl-biphenyl)3-(*p*-substituted phenyl)propane-1,3-dionato]-copper(II) complexes, described by Chandrasekhar *et al.* [13]. In contrast with these compounds, the copper β-diketonates which are substituted by two alkylated phenyl rings have discotic mesophases with very low melting points [20, 21]. For Cu51 and Cu31 spontaneous decomposition at

Table 4. Atomic positional parameters and equivalent temperature parameters for Cu38 with standard deviations in parentheses.

Atom	x/a	y/b	z/c	$U_{eq}/\text{Å}^2 \times 10^3$
Cu1	0·5000	0·5000	0·0000	56
C1	0·8186(4)	−0·4033(8)	0·4714(9)	170(*a*)
C2	0·7540(3)	−0·3623(6)	0·4628(8)	118
C3	0·7314(2)	−0·2258(5)	0·4240(6)	97
C4	0·6642(2)	−0·1865(4)	0·4180(6)	84
C5	0·6423(2)	−0·0461(5)	0·3657(5)	77
C6	0·5759(2)	0·0001(4)	0·3636(5)	70
C7	0·5587(2)	0·1409(4)	0·3026(5)	67
C8	0·4945(2)	0·1962(4)	0·2996(5)	71
C9	0·4771(2)	0·3275(4)	0·2221(4)	59
O1	0·5088(1)	0·3456(3)	0·1190(3)	65
C10	0·4294(2)	0·4161(4)	0·2618(4)	58
C11	0·4040(2)	0·5314(4)	0·1858(4)	54
O2	0·4254(1)	0·5716(3)	0·0741(3)	59
C12	0·3479(2)	0·6153(4)	0·2270(4)	54
C13	0·3172(2)	0·7099(4)	0·1354(5)	64
C14	0·2643(2)	0·7881(4)	0·1675(5)	72
C15	0·2404(2)	0·7743(5)	0·2942(5)	69
C16	0·2717(2)	0·6817(5)	0·3863(5)	81
C17	0·3243(2)	0·6036(5)	0·3547(5)	74
C18	0·1820(2)	0·8599(5)	0·3330(6)	89
C19	0·1905(2)	0·9827(6)	0·3744(8)	127
C20	0·1322(3)	1·0672(8)	0·4182(9)	154
C21	0·0868(3)	1·0900(7)	0·3223(8)	130
C22	0·0779(3)	0·9707(9)	0·2805(11)	181
C23	0·1358(3)	0·8849(8)	0·2351(10)	184
C24	0·0256(4)	1·1713(9)	0·3707(11)	179
C25	0·0205(8)	1·2851(16)	0·3760(21)	373(*a*)
C26	−0·0448(7)	1·3602(13)	0·3956(16)	331(*a*)
Cu2	0·5000	0·5000	0·5000	58
C27	0·0650(5)	1·1599(9)	0·8508(11)	217(*a*)
C28	0·0897(3)	1·0243(7)	0·8380(9)	147
C29	0·1553(2)	0·9911(6)	0·8157(7)	108
C30	0·1802(2)	0·8500(5)	0·8122(6)	92
C31	0·2449(2)	0·8143(5)	0·7821(5)	77
C32	0·2713(2)	0·6719(4)	0·7960(5)	68
C33	0·3361(2)	0·6365(4)	0·7565(5)	65
C34	0·3634(2)	0·4954(4)	0·7802(5)	64
C35	0·4220(2)	0·4540(4)	0·7151(4)	56
O3	0·4303(1)	0·5233(3)	0·6071(3)	66
C36	0·4619(2)	0·3451(4)	0·7682(4)	57
C37	0·5140(2)	0·2897(4)	0·7077(4)	52
O4	0·5317(1)	0·3367(3)	0·5954(3)	62
C38	0·5533(2)	0·1688(4)	0·7671(4)	52
C39	0·6005(2)	0·1101(4)	0·6915(5)	62
C40	0·6391(2)	−0·0002(4)	0·7423(5)	69
C41	0·6331(2)	−0·0591(4)	0·8695(5)	61
C42	0·5863(2)	0·0001(4)	0·9451(5)	71
C43	0·5473(2)	0·1111(4)	0·8969(5)	70
C44	0·6763(2)	−0·1789(4)	0·9266(5)	71
C45	0·6978(2)	−0·2738(4)	0·8276(6)	92
C46	0·7433(3)	−0·3913(5)	0·8855(6)	93
C47	0·7947(2)	−0·3534(5)	0·9402(6)	89
C48	0·7735(3)	−0·2638(6)	1·0442(7)	120
C49	0·7280(3)	−0·1446(5)	0·9897(6)	101
C50	0·8422(3)	−0·4691(5)	0·9984(8)	130
C51	0·8784(6)	−0·5347(13)	0·8980(15)	275(*a*)
C52	0·9310(8)	−0·6016(17)	0·9582(19)	406(*a*)

(*a*) U_{iso}.

Table 5. Atomic positional parameters and equivalent temperature parameters for Cu31 with standard deviations in parentheses.

Atom	x/a	y/b	z/c	$U_{\mathrm{eq}}/\text{Å}^2 \times 10^3$
Cu	0·4583(2)	0·3303(2)	0·4604(2)	67
C1	0·475(2)	0·405(2)	0·786(2)	99
C2	0·439(1)	0·422(2)	0·660(2)	72
O1	0·4742(9)	0·3344(10)	0·6183(10)	78
C3	0·372(1)	0·524(2)	0·603(2)	75
C4	0·337(1)	0·543(2)	0·494(2)	62
O2	0·3640(9)	0·4705(11)	0·4288(10)	76
C5	0·262(1)	0·649(2)	0·445(2)	65
C6	0·233(2)	0·670(2)	0·332(2)	105
C7	0·162(2)	0·765(2)	0·281(2)	129
C8	0·121(2)	0·845(2)	0·340(2)	89
C9	0·153(2)	0·830(2)	0·446(2)	113
C10	0·221(2)	0·729(2)	0·501(2)	91
C11	0·049(2)	0·957(2)	0·285(2)	148
C12	0·069(2)	1·029(2)	0·177(2)	140
C13	−0·007(2)	1·125(2)	0·122(2)	164
C14	−0·113(3)	1·113(3)	0·154(3)	170
C15	−0·131(2)	1·053(3)	0·258(3)	217
C16	−0·054(2)	0·956(2)	0·317(3)	170
C17	−0·113(4)	1·230(5)	0·079(7)	591(*a*)
C18	−0·220(3)	1·260(3)	0·081(4)	227(*a*)
C19	−0·302(2)	1·385(2)	0·011(3)	132(*a*)
C20	0·447(2)	0·258(2)	0·127(2)	96
C21	0·482(1)	0·244(2)	0·250(2)	74
O3	0·4438(9)	0·3295(10)	0·2999(10)	72
C22	0·542(1)	0·141(2)	0·306(1)	67
C23	0·574(1)	0·115(2)	0·424(2)	64
O4	0·5492(8)	0·1797(10)	0·4935(11)	73
C24	0·639(1)	0·005(1)	0·475(2)	59
C25	0·680(1)	−0·013(2)	0·584(2)	80
C26	0·739(2)	−0·117(2)	0·636(2)	86
C27	0·763(1)	−0·211(2)	0·581(2)	77
C28	0·721(1)	−0·196(2)	0·467(2)	82
C29	0·661(1)	−0·086(2)	0·417(2)	78
C30	0·833(2)	−0·329(2)	0·632(2)	102
C31	0·935(2)	−0·322(3)	0·582(3)	124
C32	1·007(2)	−0·451(2)	0·625(3)	214
C33	1·007(2)	−0·484(2)	0·761(3)	188
C34	0·907(2)	−0·488(3)	0·812(3)	164
C35	0·838(2)	−0·353(2)	0·773(2)	165
C36	1·068(2)	−0·599(2)	0·824(3)	182
C37	1·158(3)	−0·585(4)	0·806(4)	319(*a*)
C38	1·204(6)	−0·724(5)	0·880(6)	592(*a*)

(*a*) U_{iso}.

temperatures above 200°C is responsible for the absence of a mesophase. Microscopic observations show that all liquid-crystalline phases exhibit the typical nematic Schlieren texture. The melting and clearing points of the Cu3n series show the typical odd–even effect.

Cu72 has two melting points. This means that this complex crystallizes into two solid state modifications. Virgin crystals of Cu38 melted at 134°C (c1 phase). After recrystallization by cooling a melting point of 148°C (c2 phase) was found. This means that Cu38 has two polymorphs, the thermodynamically stable c2 modification and the metastable c1 form, which can also be studied by comparing the melting enthalpies (see table 2).

Three endothermic D.S.C. peaks can be found by heating virgin crystals of Cu58; one at 120°C, the second at 127°C and the last at 135°C. After resolidification we found only the second and third peaks, which was confirmed by X-ray experiments. Two different crystalline phases at room temperature were found, the c1 phase for the virgin crystals and the c2 phase after resolidification of the melt, in addition to the high temperature c3 phase. Further to the solid polymorphism this complex exhibits the so called unusual double melting behaviour [12]. On heating above 127°C the crystalline c2 phase melts into a nematic mesophase but the solid c3 phase recrystallizes immediately from this mesophase, which is isotropic at 129°C. The melting point of the c3 phase is at 135°C. Figure 1 shows the typical D.S.C. thermograms of this double melting behaviour. The ratio of peak IV (due to the melting point of the c3 phase) to peak I (due to the melting point of the c2 phase) increases with decreasing heating rates, which is characteristic of double melting behaviour. The clearing of the mesophase is responsible for peak II and the recrystallization

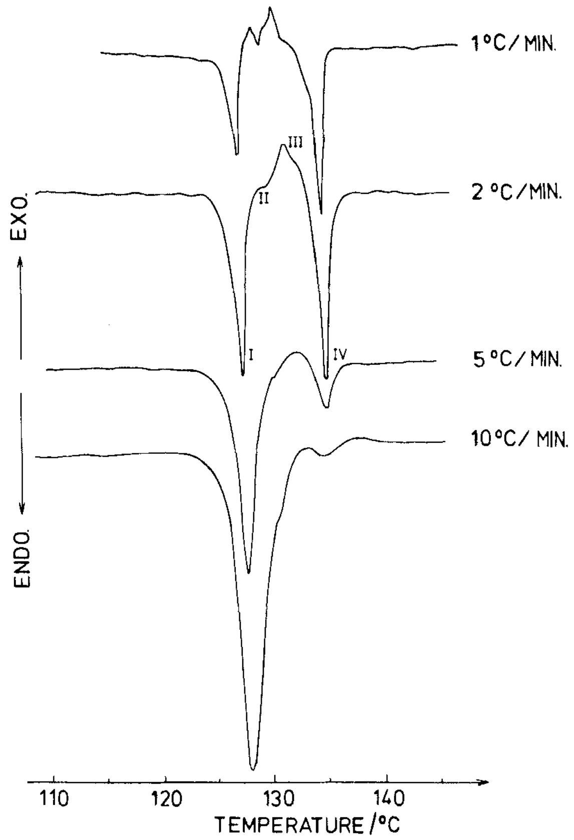


Figure 1. Differential scanning calorimetry thermograms of previously melted Cu58 crystals for different heating rates. Peaks denoted with I, II, III and IV in this figure are explained in the text.

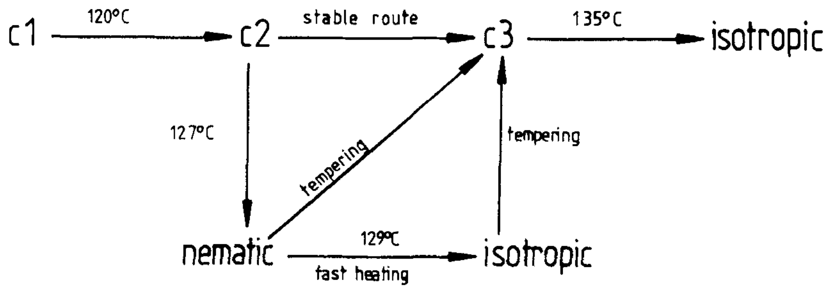


Figure 2. Phase transitions for Cu58 on heating.

of the c3 phase for the exothermic peak III. The polymorphism of Cu58 is shown in figure 2.

3.2. *Single crystal data*

The copper atoms for Cu38 are positioned on the inversion centre of symmetry at 0·5, 0·5, 0 for molecule A (Cu1) and 0·5, 0·5, 0·5 for molecule B (Cu2), which agrees with P$\bar{1}$ symmetry. Therefore, we have found two crystallographically different molecules for Cu38 (molecule A and molecule B), whereas for Cu31 the copper atoms also occupy general positions (see tables 3–5). Some relevant bond distances and angles are listed in table 6. The molecular structures of the crystallographically

Table 6. Relevant bond distances and bond angles for Cu38 and Cu31; standard deviations are given in parantheses.

	Bond distances/Å		Bond angles/degrees	
Cu38				
Molecule A	Cu1–O1	1·906(3)	O1–Cu1–O2	92·4(1)
	Cu1–O2	1·905(3)	Cu1–O1–C9	126·1(3)
	O1–C9	1·279(5)	O1–C9–C10	124·8(4)
	O2–C11	1·286(5)	C9–C10–C11	124·5(4)
	C9–C10	1·374(6)	O2–C11–C10	123·3(3)
	C10–C11	1·397(6)	Cu1–O2–C11	126·3(3)
Molecule B	Cu2–O3	1·909(3)	O3–Cu3–O4	92·1(1)
	Cu2–O4	1·903(3)	Cu2–O3–C35	126·1(3)
	O3–C35	1·281(5)	O3–C35–C36	124·5(4)
	O4–C37	1·280(5)	C35–C36–C37	125·2(4)
	C35–C36	1·385(6)	O4–C37–C36	123·0(4)
	C36–C37	1·378(6)	Cu2–O4–C37	127·3(3)
Cu31	Cu–O1	1·89(1)	O1–Cu–O2	93·3(5)
	Cu–O2	1·88(1)	Cu–O1–C2	124(1)
	O1–C2	1·27(2)	O1–C2–C3	126(2)
	O2–C4	1·29(2)	C2–C3–C4	125(2)
	C2–C3	1·42(2)	O2–C4–C3	122(2)
	C3–C4	1·34(2)	Cu–O2–C4	128(1)
	Cu–O3	1·91(1)	O3–Cu–O4	92·8(5)
	Cu–O4	1·92(1)	Cu–O3–C21	126(1)
	O3–C21	1·31(2)	O3–C21–C22	125(1)
	O4–C23	1·25(2)	C21–C22–C23	124(2)
	C21–C22	1·38(2)	O4–C22–C23	126(2)
	C22–C23	1·42(2)	Cu–O4–C23	126(1)

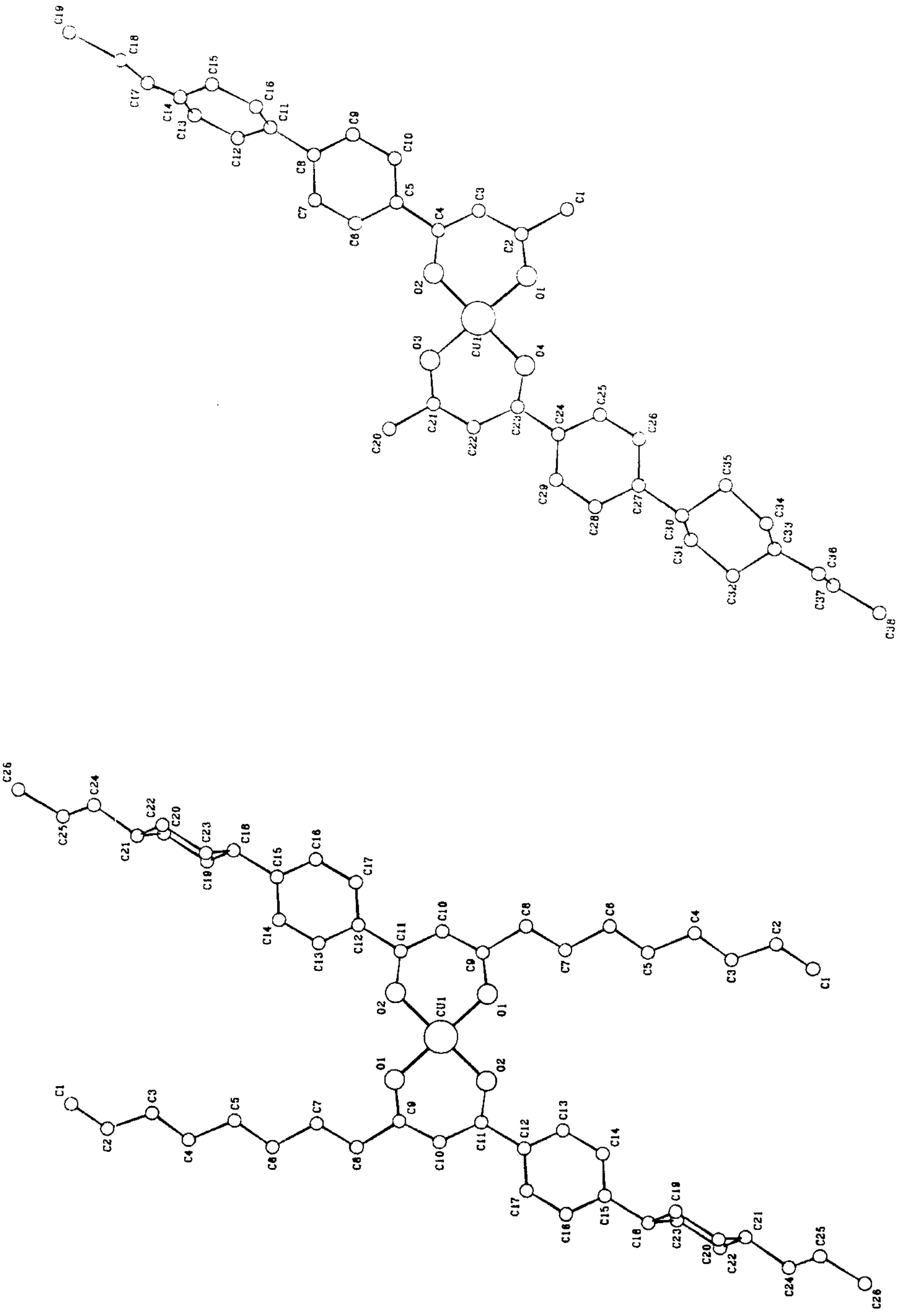


Figure 3. Molecular structures: (*left*) Cu38, molecule A; (*right*) Cu31.

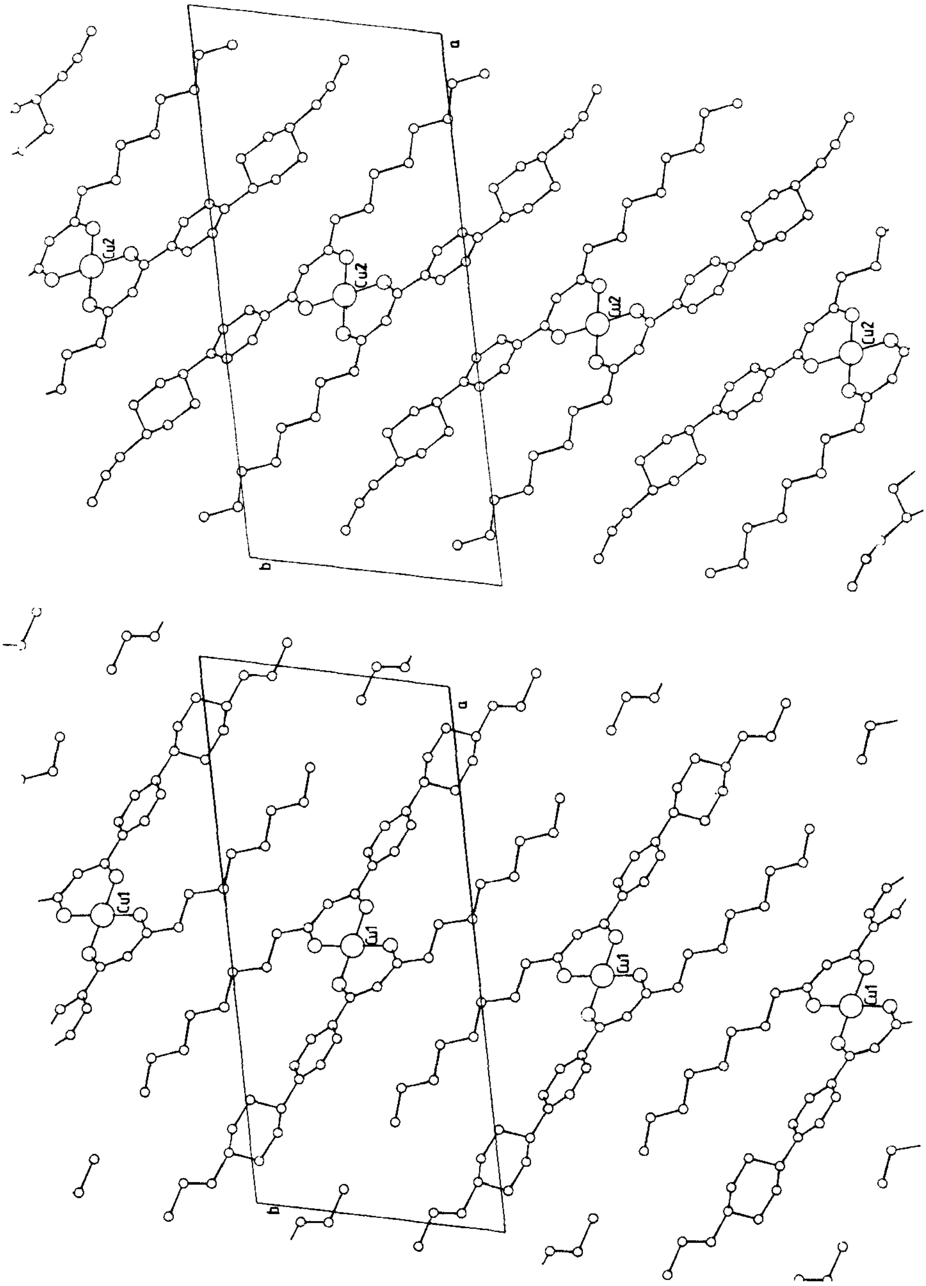


Figure 4. Projection of the crystal structure of Cu38 along [001]: (*left*) molecule A; (*right*) molecule B.

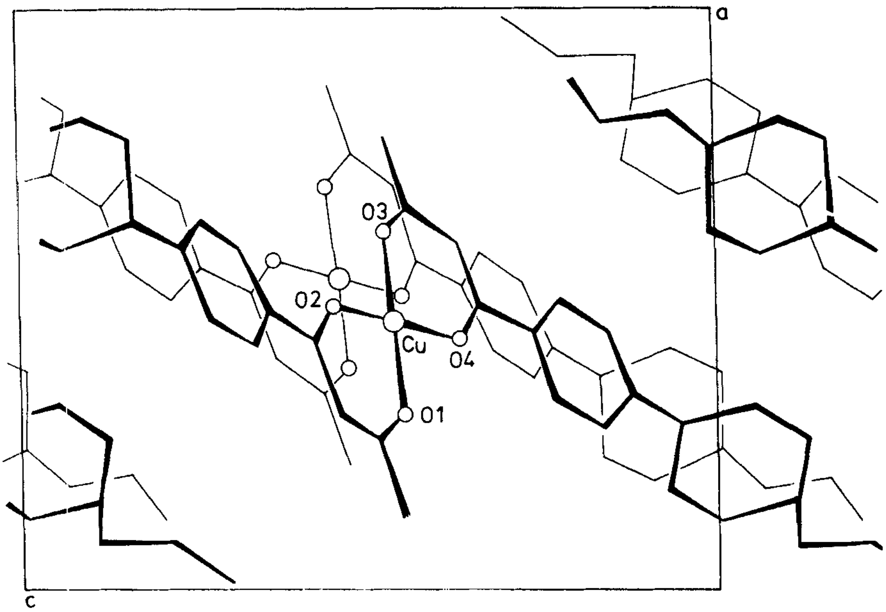


Figure 5. Projection of the crystal structure of Cu31 along [010].

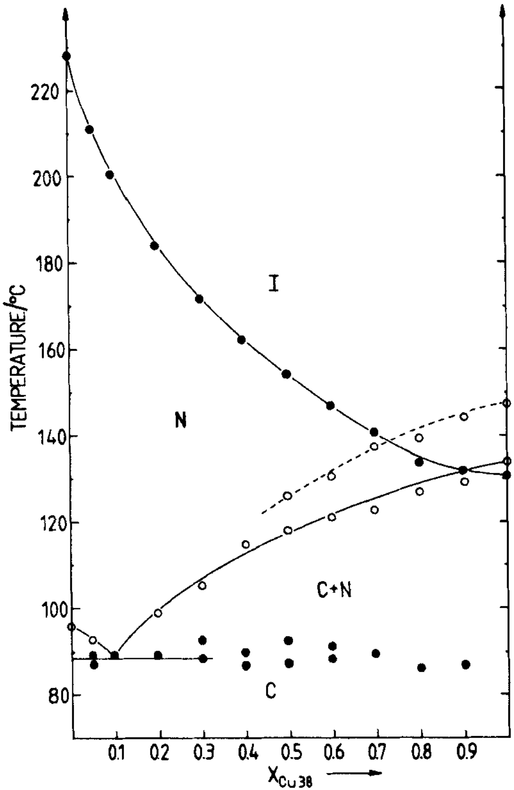


Figure 6. Phase diagram for the binary system Cu38–BCH5CN.

different molecules A and B of Cu38 as well as Cu31 are quite similar (see figure 3). For Cu38 the copper atoms, which are situated on the inversion centre, are surrounded by four oxygen atoms in a strictly square planar coordination. The phenyl rings are slightly tilted against the copper core (21° for molecule A and 15° for molecule B). The fully extended octyl chains are tilted against the copper–oxygen core by 40° for molecule A and 41° for molecule B. Therefore the whole molecule cannot be described as planar. We have also observed very high temperature factors in the region of the terminal propylgroups (see table 4).

Cu31 cannot be described as planar either. The angle between the idealized plane through the copper–oxygen core and the phenyl ring (C5–C10) is 3°; for the second phenyl ring (C24–C29) it is 12°. This time we have numerous problems because of the extraordinarily large temperature factors in the region of the terminal carbon atoms, and so we had to fix some bond distances, which had a negative influence on the *R* value.

Figure 4 represents the structural arrangement of molecule A (Cu38) in the crystalline state seen along the crystallographic *c* axis. Molecules of type B also show a very similar arrangement related to the centre of symmetry. The crystal structure consists of layers stacked along the crystallographic *c* axis with Cu1 at 0·5, 0·5, 0 and Cu2 at 0·5, 0·5, 0·5. The Cu1–Cu2 distance is 5·04 Å. For Cu31 we have found a layer structure again. The layers are stacked perpendicular to the *ab* plane. The molecules

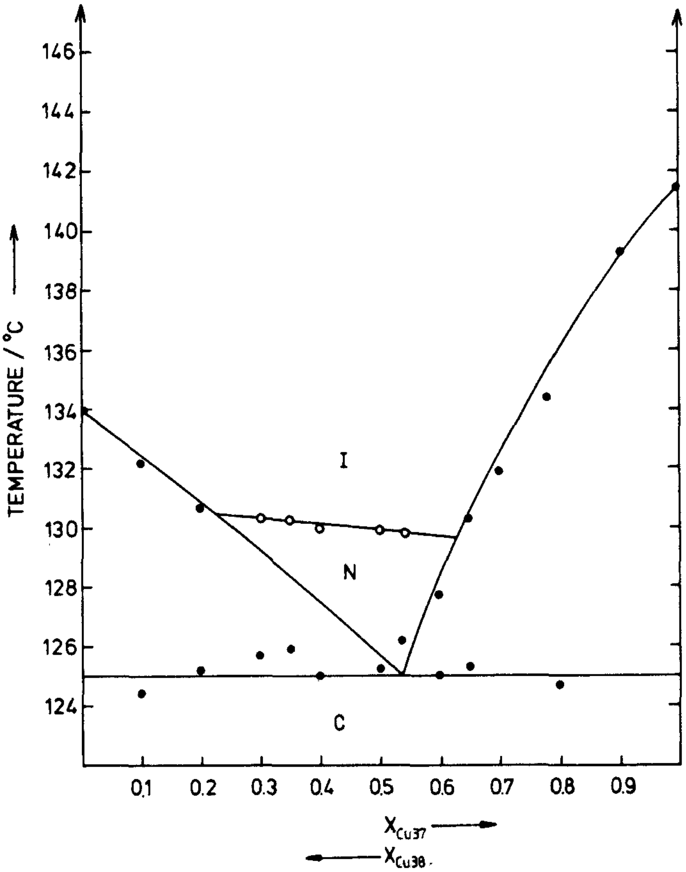


Figure 7. Phase diagram for the binary system Cu38–Cu37.

are slightly shifted against each other within the planes. The shortest copper–copper distance between the two layers is 4·777 Å. The structural arrangement of Cu31 seen along the crystallographic *b* axis is shown in figure 5.

3.3. *The binary mixtures*

3.3.1. *Binary mixture of Cu38–BCH5CN*

The phase diagram of Cu38 and 4(4″-*n*-pentylcyclohexyl)-4′-cyanobiphenyl (BCH5CN) is shown in figure 6. The nematic phases of BCH5CN and CU38 are continuously miscible but the nematic phase range is smaller than expected. Moreover, the two solid phases of Cu38 are present within this binary system. Therefore two eutectic points and two melting curves for Cu38 are observed. However, an exact correlation between the melting curves and the eutectic points is not possible.

3.3.2. *The binary mixtures Cu38–Cu37*

The mesophases are always monotropic nematic. A binary mixture of Cu37 and Cu38 has therefore been investigated to find an enantiotropic liquid-crystalline phase. Figure 7 shows the resultant phase diagram with an eutectic point at 54 mol % Cu37 and the expected enantiotropic nematic phase. It should be mentioned that the thermal stability of these copper(II) complexes above 150°C is small, and so more data cannot be presented.

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