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The synthesis and spectroscopic characterization of alkoxy substituted 2,2′-azoquinoxaline and its palladium complex

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Abstract

The synthesis and spectroscopic properties of 5,6-didodecyloxyquinoxaline-2-(1*H*)-one oxime, 2,2′-azobis[5,6-didodecyloxyquinoxaline] and the cyclopalladated complex di-μ-acetato-2,2′-azobis[5,6-didodecyloxyquinoxaline] dipalladium(II) are reported. The novel compounds were characterized by elemental analysis, FT-IR spectroscopy, ^{1}H NMR spectroscopy, UV–vis spectroscopy and mass spectrometry. 5,6-Didodecyloxyquinoxaline-2-(1*H*)-one oxime exhibited liquid crystalline properties, as determined using differential scanning calorimetry, optical polarizing microscopy and X-ray investigation. The compound displayed a discotic mesophase over a large temperature interval that included room temperature. X-ray diffraction revealed that this compound forms a discotic hexagonal ordered columnar mesophase (Col_{ho}) and thus, has potential use as a colored liquid crystal.

Keywords: Oxime; Quinoxaline; Azoquinoxaline; Cyclometallation; Palladium; Liquid crystal

1. Introduction

Oximes and their derivatives are important intermediates in organic synthesis. Besides, they are also of interest as biologically active compounds [1]. Many drugs in the market today have an oxime group in their structure, which is then frequently used for molecular modifications to develop new drugs and for synthesis of many functional groups as a starting point [2]. Oximes have both N and O donor atoms, therefore; they link metals forming chelate complexes. On the other hand, some of the oximes and their metal complexes exhibit liquid crystalline properties [3,4].

The chemistry of cyclometallated complexes is one of the most advanced area of modern organometallic chemistry [5–7]. These compounds are used in organic synthesis [8], catalysis [9,10], and asymmetric synthesis [11]. The cyclometallation reaction with Pd(II) or Pt(II) chlorides was already described more than 30 years ago [12], demonstrating that azobenzene reacts with platinum(II) or palladium(II) chlorides to give complexes with carbon-to-metal σ-bonds. This fact has prompted us to search for similar reactions with quinoxalinone oxime and 2,2′-azoquinoxaline compounds [13–15]. Symmetrically substituted 2,2′-azoquinoxaline synthesized from substituted quinoxaline-2-(1*H*)-one oxime by the template effect of cobalt(II) chloride [16] leads to the formation of dinuclear cyclopalladated complexes with e.g. *p*-substituted azobenzene ligands [17]. Recently, new cyclopalladated complexes have been described and they exhibit some interesting properties such as liquid crystalline [18–21], photochemical and electrochemical properties [22] or show promising cytotoxic activity by interaction with DNA or acting as intercalation agents [23,24].

Interestingly, there is a continuous effort to extend the chemistry of cyclopalladated complexes. Nonetheless, azoquinoxaline cyclopalladated chemistry appears not to have been fully explored especially when compared to the development of azobenzene cyclopalladated complexes.

In this work a general route is described for the synthesis of new 5,6-didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**),

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2,2′-azobis[5,6-didodecyloxyquinoxaline] (**5**) and its di-μ-acetato-2,2′-azobis[5,6-didodecyloxyquinoxaline] dipalladium(II) complex (**6**). 5,6-Didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**) was previously obtained by cyclization of 1,2-diamino-4,5-didodecyloxybenzene with *anti*-chloroglyoxime. The reaction was carried out under argon atmosphere in order to avoid oxidation of 1,2-diamino-4,5-didodecyloxybenzene. 5,6-Didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**) was converted into 2,2′-azobis[5,6-didodecyloxyquinoxaline] (**5**) by the template effect of cobalt(II) chloride and di-μ-acetato-2,2′-azobis[5,6-didodecyloxyquinoxaline] dipalladium(II) was synthesized by the reaction of **5** with $(CH_3COO)_2Pd$. Structures of the new compounds were characterized by elemental analysis, FT-IR spectroscopy, ^{1}H NMR spectroscopy, UV–vis spectroscopy and mass spectrometry. The liquid crystalline properties of compound **4** were investigated with a polarizing microscope, differential scanning calorimeter (DSC) and X-ray diffractometer.

5,6-Didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**) exhibits thermotropic liquid crystalline behaviour forming a discotic hexagonal ordered columnar (Col_{ho}) mesophase in a large temperature interval including room temperature. Compounds **5** and **6** did not exhibit the liquid crystal phase because they produced just one peak in the DSC spectra.

2. Experimental

2.1. Materials

Ethanol, $CHCl_3$, methanol, DMF, dichloromethane and acetone were dried as described by Perrin and Armarego [25]. $CoCl_2\cdot 6H_2O$ and $(CH_3COO)_2Pd$ were purchased from Fluka. Catechol, 1-bromododecane, CH_3COOH 100%, HNO_3 65%, HNO_3 100%, Pd/C 10%, hydrazine hydrate, anhydrous Na_2SO_4 and K_2CO_3 were purchased from Merck. *anti*-Chloroglyoxime [26,27], 1,2-didodecyloxybenzene (**1**) [28], 1,2-dinitro-4,5-didodecyloxybenzene (**2**) [28], 1,2-diamino-4,5-didodecyloxybenzene (**3**) [28] were synthesized and purified as described in the literature.

2.2. Equipment

Elemental analyses were obtained using a Carlo Erba 1106 instrument. Infrared spectra in KBr pellets were recorded on a Bio-Rad FTS 175C FT-IR spectrophotometer. Optical spectra in the UV–vis region were recorded with a Schimadzu 2001 UV–vis spectrophotometer using 1 cm path length cuvettes at room temperature. The mass spectra were acquired on a LCQ-ion trap (Thermo Finnigan), equipped with an electrospray source. Electrospray full scan spectra, in the range of *m/z* 50–2000 amu or *m/z* 2000–3000 amu, were obtained by infusion through fused silica tubing at a rate of 2–10 μL min^{-1}. The solutions were analysed in positive mode. The LCQ calibration (*m/z* 50–2000) was achieved according to the standard calibration procedure from the manufacturer (mixture of caffeine, MRFA and Ultramark 1621). An ES-Tuning Mix solution (Agilent) was used to calibrate the spectrometer between 2000 and 3000 amu. The temperature of the heated capillary of the LCQ was set to the range of 180–200 °C, the ion spray voltage was in the range of 1–7 kV with an injection time of 5–200 ms. ^{1}H NMR spectra were recorded in $CDCl_3$ and DMSO-d_6 solutions on a Bruker 500 MHz spectrometer using TMS as an internal reference. Transition temperatures were determined with scan rates of 10 °C min^{-1} using a Mettler Toledo Star^e Thermal Analysis System/DSC 822^e System differential scanning calorimeter calibrated with indium from 3 to 4 mg samples under a nitrogen atmosphere. Optical textures were observed with a polarizing microscope Leitz Wetzler Orthoplan-pol equipped with a hot stage Linkam TMS 93 and temperature-controller Linkam LNP. X-ray measurements were performed with Cu Kα radiation using a Rigaku Kristalloflex diffractometer (D_{max} 2200) at room temperature.

2.3. Synthesis

2.3.1. 5,6-Didodecyloxyquinoxaline-2-(1H)-one oxime (4)

To a suspension of compound **3** (2.60 g, 5.46 mmol) in dry EtOH (70 ml) a solution of mono *anti*-chloroglyoxime (0.66 g, 5.46 mmol) in dry EtOH (50 ml) was added under stirring in an argon atmosphere. Stirring was continued overnight at room temperature. The colour of the solution turned from light yellow to dark yellow. The solid was filtered off and washed with cold EtOH to give a dark yellow solid. Purity of the product **4** was controlled by thin layer chromatography (silicagel; 25:1; CH_2Cl_2:MeOH). The obtained product is soluble in CH_2Cl_2 and $CHCl_3$ at room temperature and in hot benzene, DMSO, DMF, 1,4-dioxane, acetone, hexane, EtOH, MeOH and CCl_4. Yield 1.9 g, (64%), m.p. 99 °C. FT-IR (KBr tablet, ν_{max}/cm^{-1}) 3419 (NH), 3330 (OH), 3040 (ArCH), 2956–2852 (CH), 1622 (C=N), 1514 (C–C aromatic), 1281–1238 (Ar–O–C), 952 (N–O). ^{1}H NMR δ (ppm) (DMSO-d_6) 9.98 (s, 1H, OH), 9.70 (s, 1H, NH), 7.45 (s, 1H, N=CH), 6.7 (s, 2H, Ar-H), 4.01–3.98 (t, 4H, Ar-OCH_2), 1.85–1.81 (m, 4H, β-CH_2), 1.46–1.26 (b, 36H, CH_2), 0.88 (t, 6H, CH_3). Calcd. for $C_{32}H_{55}N_3O_3$: C 72.55, H 10.46, N 7.93; found: C 72.07, H 10.92, N 7.38. UV–vis (chloroform): λ_{max}, nm (log ε) 367 (4.05), 249 (4.44). ESI (*m/z*) 530.5 $[M+H]^+$.

2.3.2. 2,2′-Azobis[5,6-didodecyloxyquinoxaline] (5)

To a suspension of compound **4** (1.91 g, 3.60 mmol) in dry EtOH (100 ml) a solution of $CoCl_2\cdot 6H_2O$ (0.42 g, 1.80 mmol) in dry EtOH (20 ml) was added dropwise in an argon atmosphere. The colour of the reaction mixture turned from dark yellow to brown. After addition, the mixture was refluxed overnight and filtered while still hot. The obtained solid was dissolved in CH_2Cl_2 for purification and re-precipitation occurred when the solution was poured into acetone. The orange-yellow product was washed with acetone, dried, and its purity was controlled by thin layer chromatography (silicagel; 50:1; CH_2Cl_2:MeOH). The product is soluble in CH_2Cl_2 and $CHCl_3$ at room temperature and in hot DMSO, DMF, CCl_4, benzene, 1,4-dioxane, hexane, and pyridine. Yield 1.37 g (74%); m.p. 146 °C. FT-IR (KBr tablet, ν_{max}/cm^{-1}) 3042 (ArCH), 2955–2852 (CH), 1614 (C=N),1538 (C–C aromatic), 1503

(N=N), 1242–1210 (Ar–O–C). ^{1}H NMR δ (ppm) ($CDCl_3$) 9.42 (s, 2H, N=CH), 7.53 (s, 2H, Ar-H), 7.44 (s, 2H, Ar-H), 4.25–4.21 (t, 8H, Ar-OCH_2), 1.97–1.94 (m, 8H, β-CH_2), 1.61–1.27 (br, 72H, CH_2), 0.88 (t, 12H, CH_3). Calcd. for $C_{64}H_{106}N_6O_4$: C 75.10, H 10.44, N 8.21; found: C 74.92, H 10.86, N 8.03. UV–vis (chloroform): λ_{max}, nm (log ε) 430 (4.60), 288 (4.48), 238 (4.39). ESI (*m*/*z*) 1023.8 $[M+H]^+$.

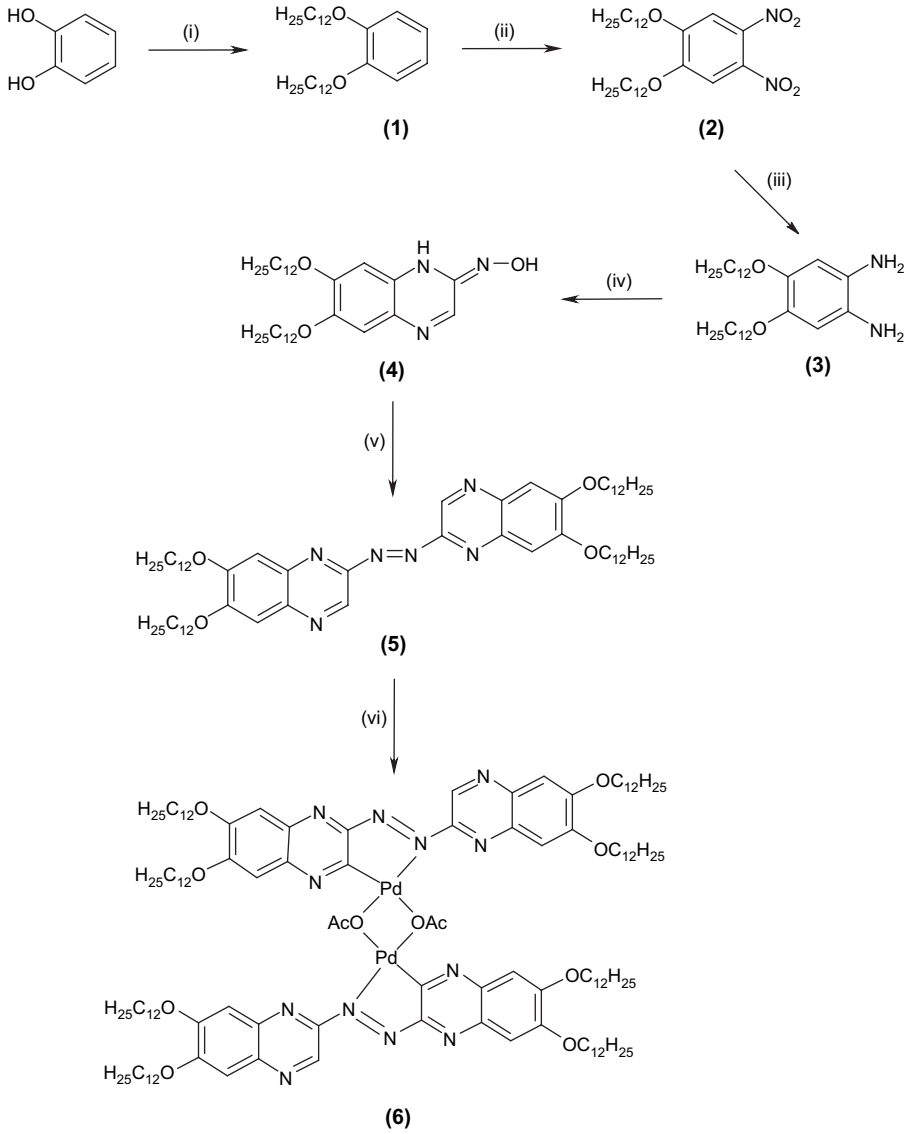
*2.3.3. Di-μ-acetato-2,2′-azobis[5,6-didodecyloxyquinoxaline]dipalladium(II) (**6**)*

Compound **5** (0.85 g, 0.84 mmol) was dissolved in dry DMF (115 ml) at 100 °C in argon atmosphere and $(CH_3COO)_2Pd$ (0.19 g, 0.84 mmol) was added. The reaction mixture was refluxed overnight at 100 °C. The resulting purple mixture was filtered and the filtrate was cooled down to room temperature. A purple precipitate formed on addition of water (150 ml). The product was filtered off, and washed with water and DMF. The crude product was purified using a preparative silicagel plate (75:1; CH_2Cl_2:MeOH). Yield 0.17 g (18%), m.p. > 300 °C. FT-IR (KBr tablet, ν_{max}/cm^{-1}) 3044 (ArCH), 2952–2854 (CH), 1612 (C=N), 1523 (C–C aromatic), 1504 (N=N), 1247 (Ar–O–C). ^{1}H NMR δ (ppm) ($CDCl_3$) 8.28 (s, 2H, N=CH), 7.45 (s, 4H, Ar-H), 7.35 (s, 4H, Ar-H), 4.17 (t, 16H, Ar-OCH_2), 2.42–0.92 (br, 166H, CH_2 and $COOCH_3$), 0.88 (t, 24H, CH_3). Calcd. for $C_{132}H_{216}N_{12}O_{12}Pd_2$: C 66.72, H 9.16, N 7.07; found: C 66.98, H 9.46, N 7.11. UV–vis (chloroform): λ_{max}, nm (log ε) 549 (4.33), 241 (4.90).

3. Results and discussion

3.1. Synthesis and characterization

In this work, we describe a general route for the synthesis of substituted quinoxaline-2-(1*H*)-one oxime (**4**), 2,2′-azoquinoxaline (**5**), and a symmetrical binuclear cyclopalladated



Scheme 1. Synthesis of azoquinoxaline and its palladium complex. (i) 1-Bromododecan, dry DMF and K_2CO_3; (ii) HNO_3 65%, HNO_3 100%, CH_2Cl_2:CH_3COOH; (iii) hydrazine hydrate, Pd/C 10%, EtOH; (iv) *anti*-chloroglyoxime, EtOH; (v) $CoCl_2 \cdot 6H_2O$, EtOH; (vi) $Pd(CH_3COO)_2$, DMF.

azoquinoxaline complex (**6**) (Scheme 1). As in the case of some other substituted 2,2′-azoquinoxalines, a rational method for the synthesis of this type of compound is to start from 1,2-diaminobenzene derivatives. 1,2-Diamino-didodecyloxybenzene (**3**) was synthesized by reduction of 1,2-dinitro-4,5-didodecyloxybenzene (**2**) which was obtained by the nitration of 1,2-didodecyloxybenzene (**1**) according to the literature [28]. 5,6-Didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**) was obtained by the cyclization reaction of 1,2-diamino-4,5-didodecyloxybenzene (**3**) with s-*trans*-chloroethanedial dioxime without addition of any base. The synthesis of 5,6-didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**) was carried out under argon atmosphere in order to avoid oxidation of 1,2-diamino-4,5-didodecyloxybenzene (**3**) in air. The yield of this cyclization reaction between the two bifunctional reagents is relatively high (64%). A template reaction of 5,6-didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**) with $CoCl_2 \cdot 6H_2O$ in ethanol gave the azo compound 2,2′-azobis[5,6-didodecyloxyquinoxaline] (**5**). The preparation of the symmetrical binuclear cyclopalladated 2,2′-azobis[5,6-didodecyloxyquinoxaline] complex **6** was accomplished by reaction of 2,2′-azobis[5,6-didodecyloxyquinoxaline] with $(CH_3COO)_2Pd$ in hot DMF under oxygen-free argon atmosphere. An orange crystalline material was obtained. In principle, the palladation of asymmetrical substituted azobenzenes [17] can take place on either of the phenyl rings and therefore an isomer mixture is expected as a product. However, in practice the symmetrical binuclear cyclopalladated 2,2′-azobis[5,6-didodecyloxyquinoxaline] complex (**6**) forms as the main product and can be easily isolated without use of any further purification techniques.

The new compounds were characterized by UV–vis, IR, and NMR spectroscopies, mass spectrometry, and elemental analysis. The results are consistent with the predicted structures as shown in Section 2. In the IR spectra, 5,6-didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**) showed characteristic stretching vibrations due to the OH and NH groups at 3419 and 3330 cm^{-1}. The vibration bands due to the N–O stretches occurred at 952 cm^{-1}. After conversion into 2,2′-azobis[5,6-didodecyloxyquinoxaline] (**5**), the characteristic N=N stretches occurred at 1503 cm^{-1} and the bands at 3419 and 3330 cm^{-1} due to the OH and NH groups disappeared, indicative of azo formation. Aromatic CH stretching appears at ca. 3040 cm^{-1} and aliphatic CH stretching at ca. 2950 and 2850 cm^{-1} for all the compounds. The vibration bands between 1612 and 1622 cm^{-1} are assigned to C=N and between 1514 and 1538 cm^{-1} to C–C aromatic vibrations. The compounds showed characteristic vibrations due to ether groups (C–O–C) between 1281 and 1210 cm^{-1}.

The ^1H NMR spectra of **4** showed two types of D_2O exchangeable protons at 9.98 and 9.70 ppm as singlets for the OH and NH protons. Integration of peak areas yielded one proton each as expected. The azomethine (N=CH) proton was observed at 7.45 ppm as a singlet (peak area equivalent to one proton). The aromatic protons were observed at

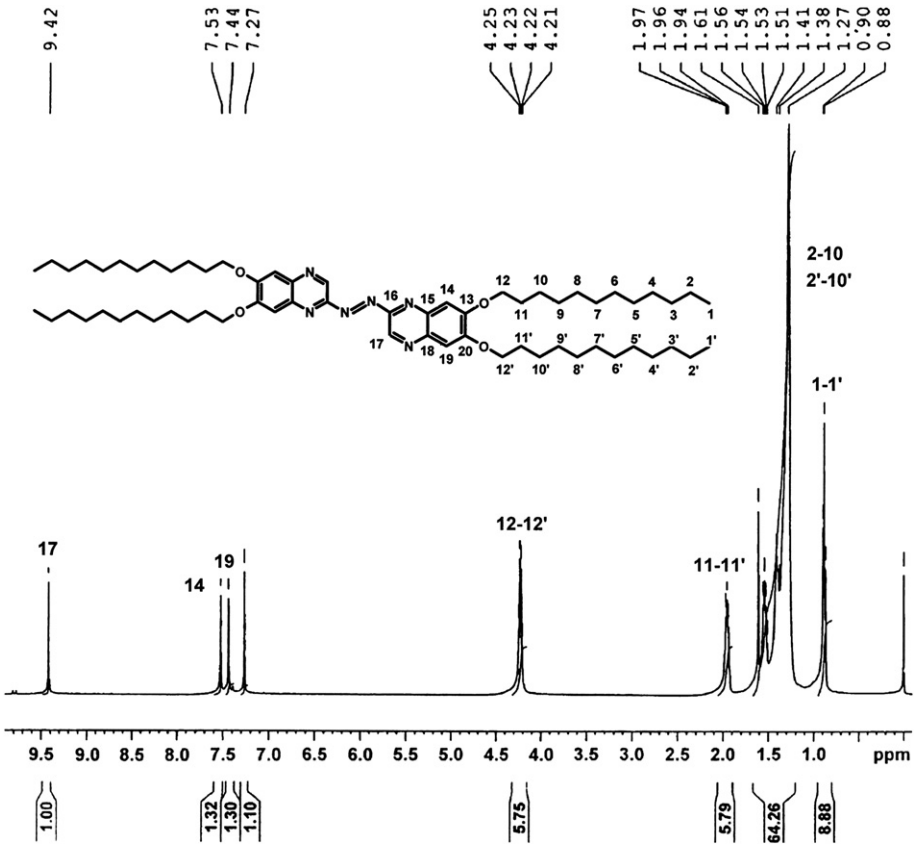


Fig. 1. ^1H NMR spectrum of compound **5** in $CDCl_3$.

6.70 ppm as a singlet. Peak integration yielded two protons. The aliphatic CH_2 protons were observed between 4.01 and 3.98 ppm as a triplet, 1.85 and 1.81 ppm as a multiplet and 1.46 and 1.26 ppm as a broad peak with areas of 4, 4 and 36 protons, a total of 44 protons as expected for the aliphatic CH_2 protons. The methyl protons were observed at 0.88 ppm as a triplet (6 protons). Fig. 1 shows the ^{1}H NMR spectra of **5** in $CDCl_3$. The azomethine (N=CH) protons were observed at 9.42 ppm as a singlet (2 protons). The aromatic protons were observed at 7.53 and 7.44 ppm as singlets (2 protons each). A total of four is expected for the aromatic protons. The aliphatic CH_2 protons were observed between 4.25 and 4.21 ppm as a triplet, 1.97 and 1.94 ppm as a multiplet and 1.61 and 1.27 ppm as a broad peak (8, 8, 72 protons, respectively, as expected for the aliphatic CH_2 protons). The methyl protons were observed at 0.88 ppm as a triplet (12 protons). The azomethine (N=CH) protons of the cyclopalladated complex **6** were observed at 8.28 ppm as singlet (2 protons). The aromatic protons were observed at 7.45 and 7.35 ppm as singlets (4 protons each). Eight protons are expected for the aromatic protons. The aliphatic CH_2 protons were observed at 4.17 ppm as a triplet and between 2.42 and 0.92 ppm as a broad peak (16 and 160 protons, respectively). A total of 176 is expected for the aliphatic CH_2 protons. The methyl protons of the acetate group were also observed between 2.42 and 0.92 ppm (6 protons as expected for the acetate methyl protons). The chain methyl protons were observed at 0.88 ppm as a triplet (24 protons).

The electronic absorption spectra of the compounds **4**–**6** in chloroform are depicted in Fig. 2. The absorption bands were observed at 249 and 367 nm for compound **4**, 238, 288 and 430 nm for compound **5** and 241 and 549 nm for complex **6**.

3.2. Mesomorphic properties

The phase transition behaviour of the substituted quinoxaline-2-(1*H*)-one oxime (**4**) was determined by DSC and polarizing microscopic observations. Phase transition temperatures and enthalpy changes of this compound are presented in Table 1. Compound **4** exhibits mesomorphic properties at room temperature after cooling down from the isotropic melt. The thermograms of compound **4** recorded at increasing temperature showed clearly two endothermic peaks in the first heating cycle. The first one appearing mostly around 66 °C showed a very large enthalpy in comparison to the second one. We assume that the first peak is related to the transition from one crystal phase (Cr1) to another crystal phase (Cr), and the second one at 90 °C to the transition from crystal phase (Cr) to an isotropic liquid phase. Compound **4** showed only one peak at the second heating cycle around 71 °C when heating up from the liquid crystal phase to the isotropic melt and around 62 °C when cooling down from the isotropic melt to liquid crystal phase. These investigations indicate that the compound **4** exhibits a monotropic liquid crystalline behaviour at room temperature.

Table 1
Phase transition[a] temperatures (°C) by DSC (heating and cooling rates are 10 °C min^{-1}) for compound **4**

Compound		Heating	Cooling
4	First	Cr1 $\xrightarrow{61.64}$ Cr $\xrightarrow{90.00}$ I	I $\xrightarrow{61.56}$ Col_{ho}
	Second	Col_{ho} $\xrightarrow{71.77}$ I	I $\xrightarrow{62.39}$ Col_{ho}

[a] Cr: solid phase, Col_{ho}: hexagonally ordered discotic mesophase, I: isotropic phase.

The textures observed by polarizing optical microscopy for the compound **4** are very similar to those described in the literature [3,4]. Excellent textures of samples of the mesophases were obtained by slowly cooling (5 °C min^{-1}) from the isotropic melt. In this case fan-like textures appear. The texture of compound **4** under polarizing optical microscopy at room temperature is presented in Fig. 3.

The mesophases were identified by microscopic observation and X-ray diffraction measurements at 30 °C. The X-ray data are summarized in Table 2. Powder diffraction patterns of **4** contain the typical reflections of a columnar mesophase [3,4]. The low angle of the X-ray diffraction diagrams of the compound **4** show up to five sharp Bragg reflections with *d*-spacing ratios 1:1/√3:1/√4:1/√7:1/√9 (Table 2). The lattice constant is

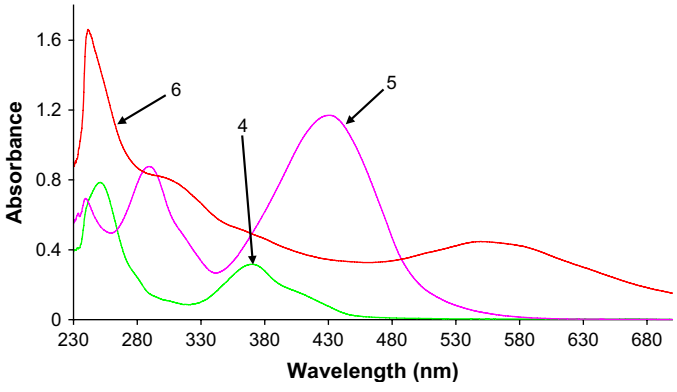


Fig. 2. Absorption spectra of 5,6-didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**), 2,2′-azobis[5,6-didodecyloxyquinoxaline] (**5**) and di-μ-acetato-2,2′-azobis[5,6-didodecyloxyquinoxaline] dipalladium(II) (**6**) in chloroform.

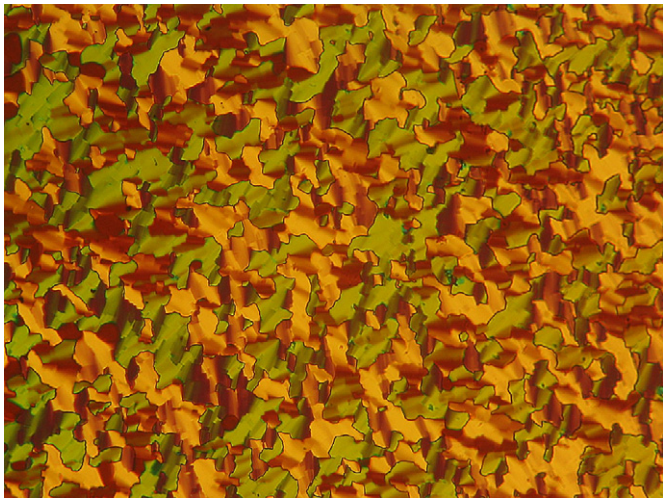


Fig. 3. Optical texture of **4** observed at 25 °C (magnification 25×).

Table 2
X-ray diffraction data of the compound **4** at room temperature

Compound	Spacing (Å)		Ratio	Miller Indices	Lattice constant
	$d_{observed}$	$d_{calculated}$			
	38.92	38.92	1	100	
	23.70	22.47	$\sqrt{3}$	110	
4	19.43	19.46	$\sqrt{4}$	200	$a = 44.9$ Å
	15.54	14.71	$\sqrt{7}$	210	
	12.74	12.97	$\sqrt{9}$	300	

44.9 Å for compound **4**. This result suggests a two-dimensional hexagonal lattice with disk-like molecules stacked in columns in the hexagonal arrangement.

4. Conclusions

We have prepared and characterized the compounds 5,6-didodecyloxyquinoxaline-2-(1*H*)-one oxime (**4**), 2,2′-azobis [5,6-didodecyloxyquinoxaline] (**5**), and the cyclopalladated complex di-μ-acetato-2,2′-azobis[5,6-didodecyloxyquinoxaline] dipalladium(II) (**6**) for the first time. The liquid crystalline properties of 5,6-didodecyloxyquinoxalin-2-(1*H*)-one oxime (**4**) were investigated. This compound shows a thermotropic liquid crystalline behaviour in a large temperature interval including room temperature.

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