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Synthesis and liquid crystalline properties of biphenyl esters of monothio- and dithiotrimellitimides†

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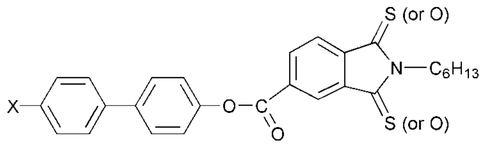
A series of 4′-substituted biphenyl esters of 5-carboxy-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dione (*N*-hexyltrimellitimide) has been synthesised. The ester imides obtained were treated with Lawesson's reagent, and the resulting monothio- and dithiotrimellitimides were purified by column chromatography. A majority of the products showed interesting thermotropic properties. For instance, 5-(biphenyl-4-yloxycarbonyl)-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dithione showed a monotropic transition at 70 °C, whereas its dioxygen analogue 5-(biphenyl-4-yloxycarbonyl)-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dione was not a mesomorphic compound.

A variety of liquid crystal compounds containing a sulfur–carbon bond have been reported in the literature. Typical examples are thioether, thioester and isothiocyanate derivatives of differing mesogenic structures.[1–4]

Properties of sulfur-containing liquid crystal molecules differ to some extent from those of the respective oxygen analogues; for instance, the terminal thiomethyl group is more smectogenic as compared with methoxy compounds.[5] The substituted 1,3-dithianes and 1,3-oxathianes have been also reported as an interesting class of mesomorphic materials.[6,7] The isotropisation temperatures of 1,3-dithiane-like compounds differ to some extent from those of the corresponding 1,3-dioxanes. The magnitude of this effect depends on the actual substituent(s) in the dithiane ring.[8–10] The mesomorphic phase appears less readily in the compounds of the oxathiane class. This is probably because of a slight shape irregularity of the 1,3-oxathiane ring resulting from the different sizes of the sulfur and oxygen atoms.

Trimellitic anhydride is a very useful reagent in the synthesis of many ester imides and poly(ester imide)s.[11] It was found that aromatic esters of N-substituted trimellitimides induced liquid crystalline properties in such compounds.[12]

During the last few years we have been studying the mesomorphic properties of some cholesteryl and biphenyl ester imides.[13,14] Below, we will demonstrate that these compounds can be easily transformed into liquid crystalline ester thioimides of the following general formula:



X= H, CN or $C_{10}H_{21}O$

The key step in the synthesis is the reaction of an appropriate carbonyl compound with 2,4-bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide (Lawesson's reagent, LR) which exchanges the oxygen atom of the carbonyl group for a sulfur atom.[15] Imides can be easily converted into thioimides with LR.[15,16] Thioimides, in contrast to other types of thiocarbonyl compounds, are generally very stable materials. However, according to the best of our knowledge, there have been no reports of mesogenic structures based on this group. We intend to show in the present paper some perspectives that have been opened by using LR for the synthesis of liquid crystal materials.

Experimental

Instrumentation

All product structures were confirmed by FT-IR, ^{1}H NMR, and in some cases ^{13}C NMR spectroscopy. In this chapter only data essential for product identification or that are discussed below are presented. The infrared spectra [in CH_2Cl_2] were recorded on a Perkin-Elmer 2000 apparatus equipped with Pegrams 2000 software, and the NMR spectra [in $CDCl_3$] were recorded using a Varian Gemini 200 MHz spectrometer. UV spectra [in $CHCl_3$] were acquired with a Perkin-Elmer λ11 apparatus. DSC studies were performed at a rate of 10 °C min^{-1} using a Perkin-Elmer 7 calorimeter. The textures of the mesophases were studied using a polarising, hot-stage-equipped microscope Biolar PI.

Synthesis

All chemicals used were analytical grade commercial products [Aldrich] and were used without further purification. 4-Decyloxy-4′-hydroxybiphenyl (**1c**) was obtained as described previously;[13] mp 151 °C. 5-Carboxy-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dione (*N*-hexyltrimellitimide, **2**) was synthesised according to the method described earlier,[12] mp 142 °C.

Synthesis of 3a–c

To 1 mmol of *N*-hexyltrimellitimide (**2**) (275 mg) dissolved in 25 cm^3 of dry CH_2Cl_2, 1 mmol of **1a–c**, 1.1 mmol (230 mg) of dicyclohexylcarbodiimide (DCC) and a catalytic amount of dimethylaminopyridine (DMAP) were added. The reaction mixtures were stirred for 6 h, the side product *N,N*-dicyclohexylurea was filtered off and the crude products of interest were crystallised from hexane–benzene (3 : 2) after removing methylene chloride. The yield was approximately 65% for all compounds. Phase transition temperatures are given in

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†Full experimental details, micrographs showing textures of nematic phases of **3b**, **4b** and **6b** and the smectic phase of **6b**, and a colour version of Fig. 2 are available as supplementary data from the RSC web site, see http://www.rsc.org/suppdata/jm/b0/b000686f/

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Table 1 Phase transition temperatures (°C) for imides, monothioimides and dithioimides

Compound	Cr		Sm		N		Iso
3a	●	160[a]	—		—		●
4a	●	99	—		—		●
5a	●	132	—		—		●
6a	●	105	—		—		●
	—		—		●	[70][b,c]	●
3b	●	113	●	132	●	204	●
	●	[89]	●	[110]	●	[201]	●
4b	●	146	●	151	●	180	●
	●	[86]	●	[109]	●	[178]	●
5b	●	128	●	145	●	178	●
	●	[82]	●	[135]	●	[176]	●
6b	●	128	●	155	●	186	●
	●	[89]	●	[139]	●	[185]	●
3c	●	148	—		●	203	●
	●	[114]	—		●	[198]	●
4c	●	108	—		●	173	●
	●	[74]	—		●	[164]	●
5c	●	140	—		●	188	●
	●	[96]	—		●	[184]	●
6c	●	82	—		●	171	●
	●	[49]	—		●	[169]	●

[a] Transitions on heating run. [b] Monotropic transition. [c] Square brackets indicate transitions on cooling run.

Table 1. The purity of the products was verified by silica gel TLC using hexane–benzene (3 : 2) as eluent.

5-(Biphenyl-4-yloxycarbonyl)-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dione 3a. FT-IR (cm^{-1}) 1775 (C=O$_{\text{imide}}$), 1742 (C=O$_{\text{ester}}$), 1716 (C=O$_{\text{imide}}$); ^{1}H NMR (ppm) 3.71 (t, 2H), 7.95 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.53 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.62 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); ^{13}C NMR (ppm) 13.8, 22.3, 26.4, 28.3, 31.157, 38.2, 121.5, 123.2, 124.5, 126.9, 127.3, 128.1, 128.7, 132.4, 134.7, 135.6, 135.8, 139.2, 139.9, 149.8, 163.1, 167.0, 167.1; $R_f=0.08$.

5-(4′-Cyanobiphenyl-4-yloxycarbonyl)-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dione 3b. FT-IR (cm^{-1}) 2230 (CN), 1776 (C=O$_{\text{imide}}$), 1745 (C=O$_{\text{ester}}$), 1718 (C=O$_{\text{imide}}$); ^{1}H NMR (ppm) 3.73 (t, 2H), 8.00 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.57 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.65 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); $R_f=0.07$.

5-(4′-Decyloxybiphenyl-4-yloxycarbonyl)-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dione 3c. FT-IR (cm^{-1}) 1772 (C=O$_{\text{imide}}$), 1743 (C=O$_{\text{ester}}$), 1719 (C=O$_{\text{imide}}$); ^{1}H NMR (ppm) 3.73 (t, 2H, N-CH$_2$), 4.00 (t, 2H, O-CH$_2$-), 7.99 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.58 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.67 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); $R_f=0.09$.

Synthesis of 4a–c, 5a–c and 6a–c

A mixture of 1 mmol of **3a–c** and 1 mmol of Lawesson's reagent in 10 cm^3 of toluene was refluxed for 1 h. The solvent was then evaporated to dryness and the crude products were purified and separated (each product mixture contained three thioimide derivatives) by column chromatography (SiO$_2$) using hexane–benzene (1 : 1) as an eluent. The yields for series **a** were approximately 10–30%, for series **b** 10–35% and for series **c** 10–25%.

5-(Biphenyl-4-yloxycarbonyl)-2-hexyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one 4a. FT-IR (cm^{-1}) 1743 (C=O$_{\text{ester}}$), 1742 (C=O$_{\text{imide}}$), 1344 (C=S); ^{1}H NMR (ppm) 4.08 (t, 2H), 7.92 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.55 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.76 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); ^{13}C NMR (ppm) 13.9, 22.4, 26.5, 27.8, 31.3, 41.3, 121.7, 122.9, 125.2, 127.1, 127.4, 128.2, 128.8, 131.1, 134.7, 134.8, 137.1, 139.4, 140.1, 149.9, 163.5, 168.9, 195.4; $R_f=0.17$.

5-(Biphenyl-4-yloxycarbonyl)-2-hexyl-1-thioxo-2,3-dihydro-1*H*-isoindol-3-one 5a. FT-IR (cm^{-1}) 1747 (C=O$_{\text{imide}}$), 1742 (C=O$_{\text{ester}}$), 1344 (C=S); ^{1}H NMR (ppm) 4.08 (t, 2H), 8.07 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.53 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.61 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); ^{13}C NMR (ppm) 13.9, 22.5, 26.5, 27.8, 31.3, 41.4, 121.6, 123.8, 124.5, 127.0, 127.4, 127.6, 128.2, 128.8, 133.8, 135.7, 139.4, 140.1, 149.9, 163.5, 168.7, 195.2; $R_f=0.29$.

5-(Biphenyl-4-yloxycarbonyl)-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dithione 6a. FT-IR (cm^{-1}) 1741 (C=O$_{\text{ester}}$), 1361 (C=S); ^{1}H NMR (ppm) 4.57 (t, 2H), 7.96 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.50 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.66 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); ^{13}C NMR (ppm) 14.0, 22.5, 26.5, 27.6, 31.3, 44.3, 121.7, 123.3, 125.0, 127.1, 127.4, 128.2, 128.8, 133.6, 134.4, 134.8, 137.6, 139.3, 140.1, 150.0, 163.7, 195.6, 195.7; $R_f=0.58$.

5-(4′-Cyanobiphenyl-4-yloxycarbonyl)-2-hexyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one 4b. FT-IR (cm^{-1}) 2230 (CN), 1741 (C=O$_{\text{imide,ester}}$), 1345 C=S; ^{1}H NMR (ppm) 4.09 (t, 2H), 7.94 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.56 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.76 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); $R_f=0.31$.

5-(4′-Cyanobiphenyl-4-yloxycarbonyl)-2-hexyl-1-thioxo-2,3-dihydro-1*H*-isoindol-3-one 5b. FT-IR (cm^{-1}) 2230 (CN), 1746 (C=O$_{\text{imide}}$), 1741 (C=O$_{\text{ester}}$), 1345 (C=S); ^{1}H NMR (ppm) 4.08 (t, 2H), 8.10 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.54 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.62 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); $R_f=0.21$.

5-(4′-Cyanobiphenyl-4-yloxycarbonyl)-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dithione 6b. FT-IR (cm^{-1}) 1740 (C=O$_{\text{ester}}$), 1344 (C=S); ^{1}H NMR (ppm) 4.46 (t, 2H), 7.99 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.51 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.67 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); $R_f=0.31$.

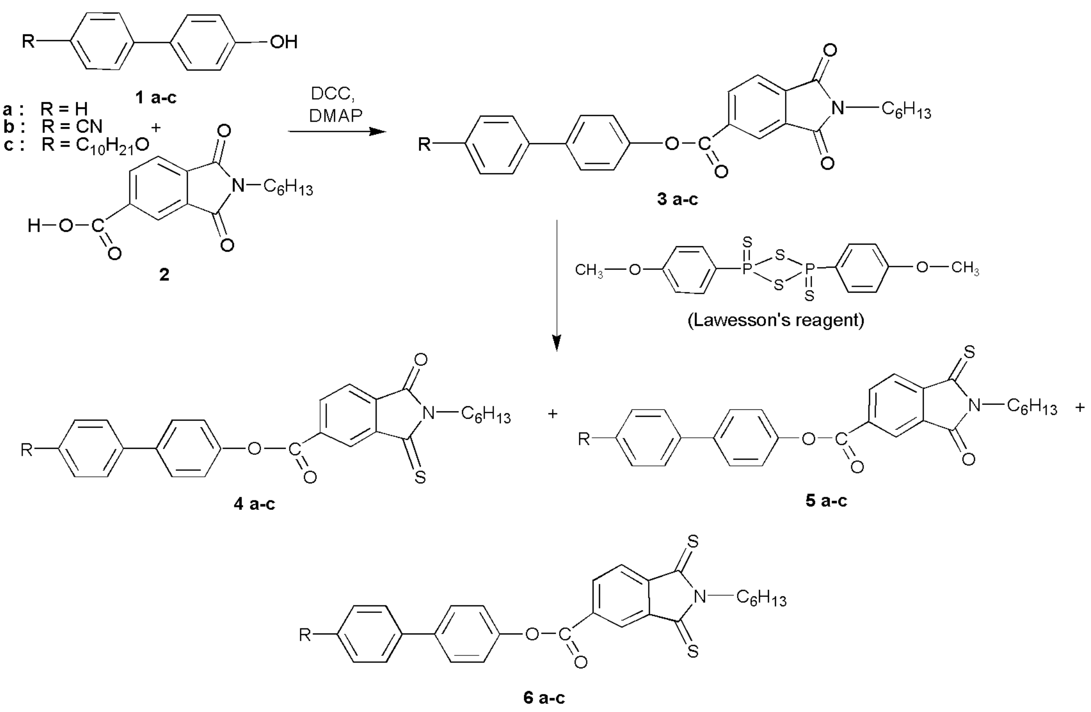
5-(4′-Decyloxybiphenyl-4-yloxycarbonyl)-2-hexyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one 4c. FT-IR (cm^{-1}) 2230 (CN), 1743 (C=O$_{\text{imide,ester}}$), 1361 (C=S); ^{1}H NMR (ppm) 4.0 (t, 2H, O-CH$_2$-), 4.08 (t, 2H), 7.92 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.55 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.76 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); $R_f=0.13$.

5-(4′-Decyloxybiphenyl-4-yloxycarbonyl)-2-hexyl-1-thioxo-2,3-dihydro-1*H*-isoindol-3-one 5c. FT-IR (cm^{-1}) 1742 (C=O$_{\text{imide,ester}}$),1344 (C=S); ^{1}H NMR (ppm)) 4.00 (t, 2H, O-CH$_2$-), 4.08 (t, 2H), 8.09 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.54 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.62 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); $R_f=0.26$.

5-(4′-Decyloxybiphenyl-4-yloxycarbonyl)-2-hexyl-2,3-dihydro-1*H*-isoindole-1,3-dithione 6c. FT-IR (cm^{-1}) 1740 (C=O$_{\text{ester}}$), 1361 (C=S); ^{1}H NMR (ppm)) 4.0 (t, 2H, O-CH$_2$), 4.46 (t, 2H), 7.98 (dd, 1H, $J_{7,6}=7.8$ Hz, $J_{7,4}=0.6$ Hz), 8.51 (dd, 1H, $J_{6,7}=7.8$ Hz, $J_{6,4}=1.4$ Hz), 8.67 (dd, 1H, $J_{4,6}=1.4$ Hz, $J_{4,7}=0.6$ Hz); $R_f=0.54$.

Results and discussion

The synthetic pathway employed is outlined in Scheme 1. We have found that the treatment of ester imides **3a–c** with an



Scheme 1 Synthesis of monothio- and dithioimides.

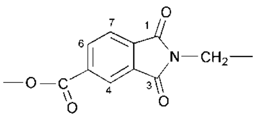
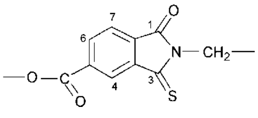
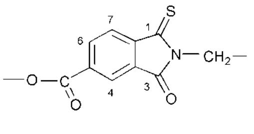
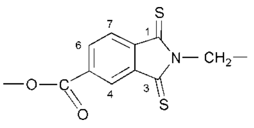
equimolar amount of LR led to a mixture of three products. These products formed three distinct colour spots on TLC and could be easily separated by column chromatography. Analysis of the respective FT-IR spectra revealed all the products [one dithionated and two monothionated compounds, see Scheme 1] were formed by imide group thionation, whereas the ester group remained unchanged under the given reaction conditions. Although the absorption bands of the C=O stretching vibrations of the imide, thioimide and ester groups strongly interfered with each other, we succeeded in separating these signals. All these products and the starting ester imides **3a–c** exhibited strong ester carbonyl absorptions at 1740–1750 cm^{-1}. Absorption bands at 1780 and 1720 cm^{-1} that are characteristic of imide carbonyl groups were absent from the spectra of the gold–brown compounds corresponding to the TLC spots with the highest R_f values. Instead, absorption bands in the range of 1340–1360 cm^{-1} were seen that are characteristic of the C=S group.[17] Based on these results we proposed the dithioimide structures of **6a–c**.

The other products which showed both imide and thiocarbonyl absorption bands were the isomeric monothioimides **4a–c** and **5a–c**. They can be differentiated on the basis of the strong deshielding effect of the C=S group in the ^{1}H and ^{13}C NMR spectra. Table 2 presents selected data for the biphenyl-substituted compounds (series **a**). The key information for the identification of isomeric monothioimides of the **4** and **5** series were the chemical shifts of the 4H and 7H protons, which strongly suggested the presence of a sulfur atom at position 3 in **4a** and position 1 in **5a**. To verify the above conclusions, ^{1}H NMR spectra were simulated with ACD/HNMR 1.0 software for compounds **3a–6a** [Table 2, in parentheses]. One can see that the trend in the changes of the calculated chemical shift was in a good agreement with the authentic ^{1}H NMR data [see Table 2]. Similar assignments were made by other authors for thionated 4-alkoxyphthalimides.[18] Considering the above, the salmon-coloured compounds [intermediate mobility spots] were assigned structures **5a–c**, whereas structures **4a–c** were postulated for the pink-coloured compounds [low mobility spots]. Notably, substitution of the carbonyl oxygen atoms for sulfur atoms resulted in coloured thioimides, whereas their parent imides were colourless. UV-VIS spectra of ester imides **3a–c** revealed weak C=O electron bands [n→π*] in the near UV range overlapping with π→π* lines. On the other hand, a marked bathochromic shift of the [n→π*] band of the C=S absorption (maximum at about 510 nm as compared to about 300 nm of the corresponding C=O absorption) was observed in all thio- and dithiotrimellitimides. Representative absorbances in the visible range for the **a** series compounds are shown in Fig. 1.

Another important characteristic of the newly synthesised thioimides is their liquid crystal behaviour. The biphenyl moiety induces liquid crystalline properties in a wide variety of molecules. In our previous papers the synthesis and thermotropic properties of some biphenyl ester imides were described.[12,13] It has been found using DSC analysis and polarised optical microscopy that all the newly synthesised 4-cyano and 4-decyloxy compounds showed nematic [series **c**] or nematic and smectic mesophases [series **b**, see Table 1]. Among the series **a** compounds [unsubstituted biphenyls], only the dithioimide **6a**, the sulfur analogue of **3a**, was a liquid crystal showing monotropic transition. Phase transition temperatures for all imides and thioimides investigated are given in Table 1. Our optical microscopy observations indicated that the compounds of series **b** exhibit a smectic A phase texture (focal conic fan texture).

Calculated molecular geometry [Fig. 2] indicated that the introduction of sulfur atom(s) resulted in only minor variations in the molecule shape. The difference in mesomorphic behaviour among these compounds may be explained in terms of polar interactions. One should expect that the position of the sulfur atom in the monothionated compounds **4a** and **5a** would result in different polarities. The calculated dipole moments [semi-empirical AM1, HyperChem] of the newly synthesised compounds are in good agreement with their predicted structures [Fig. 2]. The shape of the molecules suggested that the lateral dipole moment might be stronger in **5a** than in **4a** because of the presence of the C=O group at position 3 instead of 1. More essential, however, are the lateral interactions between large and polarisable sulfur atoms inducing molecular ordering. Besides, it should be noticed that abatement of polarity in sulfur-containing compounds involved a decrease in clarification temperature of the cyano-

Table 1 Physico-chemical properties of compounds **3a–6a**

				^{1}H NMR			^{13}C NMR			
Compound	Ester imide core	λ/nm (ε/dm^3 mol^{-1} cm^{-1})	ν/cm^{-1}	4H	6H	7H	1C	3C	C(ester)	N-CH$_2$
3a		249 (42 800) 301 (53 500) colorless	1716_{imide} 1742_{ester} 1775_{imide}	8.62 (8.47)[a]	8.53 (8.49)	7.95 (7.96)	167.0	167.1	163.1	38.2
4a		295 (54 000) 336 (43 800) 506 (30) pink	$1344_{\text{C=S}}$ $1742_{\text{imide,ester}}$	8.76 (9.04)	8.55 (8.35)	7.92 (8.01)	168.9	195.4	163.5	41.3
5a		295 (71 000) 305 (72 300) 330 (51 200) 511 (34) salmon	$1344_{\text{C=S}}$ 1742_{ester} 1747_{imide}	8.61 (8.53)	8.53 (8.41)	8.07 (8.18)	195.2	168.8	163.5	41.4
6a		354 (60 500) 400 (58 700) 509 (108) 540 (92) golden	$1361_{\text{C=S}}$ 1741_{ester}	8.66 (9.08)	8.50 (8.28)	7.96 (8.22)	195.6	195.7	163.7	44.3

[a] Calculated by means of computer program.

substituted compounds as well as in all phase transition temperatures of the corresponding decyloxy derivatives as compared with those of their oxygen analogues.

Conclusions

The present paper shows some perspectives opened by employing LR for the synthesis of liquid crystal materials. Importantly, the difference in the reactivity of ester and imide groups toward LR enables selective thionation of the imide carbonyl group while leaving the ester linkage intact in trimellitimides. The newly synthesised monothio- and dithioimides sulfur compounds (**4b–c**, **5b–c** and **6a–c**) show absorption bands in the visible range and have liquid crystalline properties, thus forming a new class of coloured mesogenic thioimide materials.

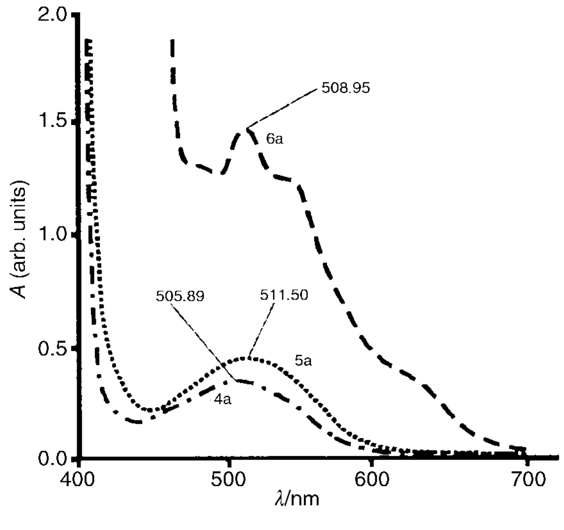


Fig. 1 UV-VIS spectra of compounds **4a–6a**. **4a** –·–·–; **5a** ···; **6a** – – –.

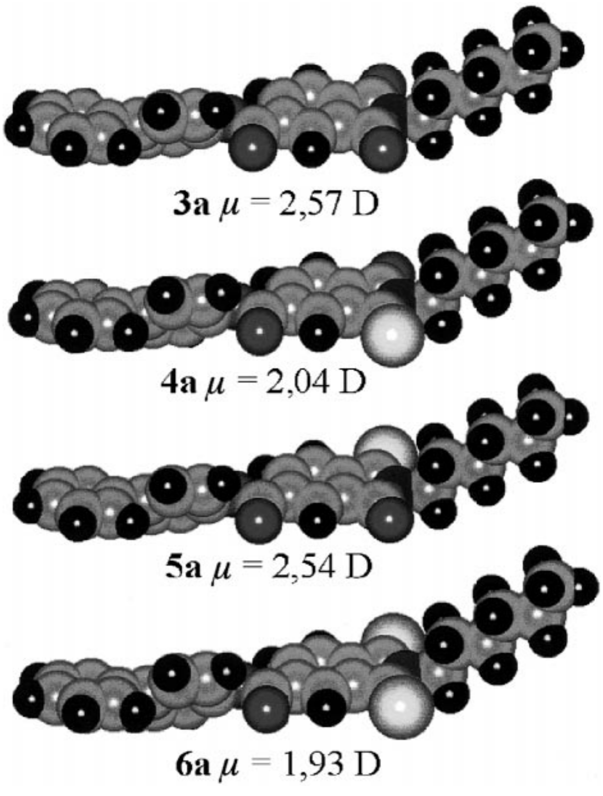


Fig. 2 Models of molecular structures of compounds **3a–6a**.

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