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Polyfluorinated cycloalkoxyphenyl ether systems as dopants for liquid crystal display applications

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ABSTRACT

The effects on response times of representative commercially available dielectrically negative (−LC) and positive (+LC) type liquid crystal mixtures doped with small quantities of various tetrafluorinated cycloalkoxyphenyl ether derivatives were investigated. The synthesis of various polyfluoroaryl systems by nucleophilic aromatic substitution processes is described and electro-optical measurements concerning the rise and decay times and voltage holding ratios of each doped LC formulation are reported. The response time of the dielectrically negative LC host was improved upon doping with each of the tetrafluorinated cycloalkoxyphenyl ether derivatives, although no improvement in the performance of the dielectrically positive LC host was achieved.

1. Introduction

Liquid crystal (LC) materials are used extensively in electronic devices as the major components of the majority of modern TV, laptop and computer display screens. Consumer demand for larger screens that have ever sharper image quality continues to drive innovation in the design and application of novel liquid crystal materials that can deliver higher performance display systems. In particular, the response times of modern LCD devices need to be improved to prevent moving-image blurring, or ‘ghosting’, which becomes increasingly apparent for fast moving objects on large displays. The response time of an LCD device is defined as the length of time required to switch a constituent LC cell ‘on’ and then ‘off’ again, and this parameter is formulated from two components, the time taken to switch the LC cell ‘on’, referred to as the rise time, and the time taken to switch the cell ‘off’, the decay time.

Commercial LC mixtures generally consist of 10–15 molecular components, each of which contributes to the overall performance of the LC display image. The addition of small quantities of chemical additives, or ‘dopants’, into a commercial LC blend in order to enhance a particular electro-optical property for a specific application has been adopted by a number of research groups and, to this end, there have been several recent reports concerning the addition of small quantities of nanoparticulate dopant materials into various commercial nematic hosts to improve their respective performance [1–7].

In previous publications, we have demonstrated that the introduction of small quantities (2–4 wt%) of polyfluorinated biphenyl ether derivatives into various commercial LC formulations is highly effective at improving their response times by reducing the rotational viscosity of the host mixture [8,9]. In particular, several tetrafluorinated biphenyl ether derivatives were found to be especially effective at reducing the rise times of dielectrically negative and positive type LC mixtures.

In order to further assess some of the factors that contribute to dopant/LC performance, we aimed to design dopant systems that maintained the strongly polar aromatic ‘head group’ and ether functionality but increased the steric demand of the hydrocarbon moiety which would, in principle, further reduce the viscosity of the host LC system and, therefore, potentially improve response times. In this paper, we describe the synthesis of several tetrafluorophenyl cycloalkoxyl ether derivatives, which meet these molecular design characteristics, and assess their use as dopants in representative commercial LC host mixtures.

2. Results and discussion

2.1. Synthesis of tetrafluorophenyl cycloalkoxyl ether derivatives

Reaction of pentafluorobenzene **1** with appropriate cycloalkoxide salts **2** upon reflux in THF overnight gave access to a range of

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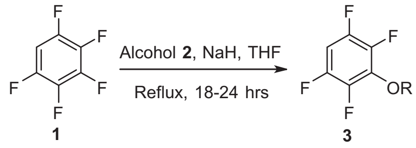
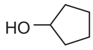
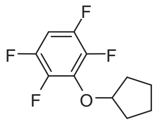
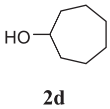
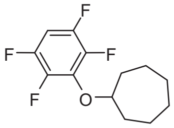
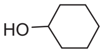
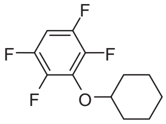
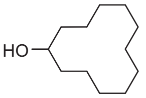
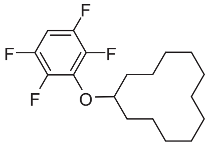
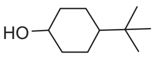
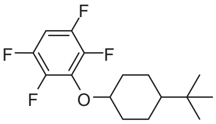
Table 1
Synthesis of tetrafluorophenyl cycloalkoxyl ether derivatives.



Alcohol **2**	Product **3**, (Yield / %)	Alcohol **2**	Product **3**, (Yield / %)
 **2a**	 **3a**, (61)	 **2d**	 **3d**, (52)
 **2b**	 **3b**, (50)	 **2e**	 **3e**, (43)
 **2c**	 **3c**, (50)		

tetrafluorophenyl cycloalkoxylether derivatives **3** (Table 1). All ethers **3** were characterised by elemental analysis and NMR and, in particular, the ^{19}F NMR spectra, which showed two resonances in the ranges −140.89 to −140.97 and −156.40 to −156.81 ppm in a 1:1 ratio, were consistent with the structures proposed. In all cases, nucleophilic aromatic substitution occurs at the site that is most activated by the presence of two *ortho* and two *meta* fluorine atoms and *para* to ring hydrogen, consistent with reported reactions of oxygen nucleophiles with pentafluorobenzene and well established rules governing the regioselectivity of S_NAr reactions involving highly fluorinated aromatic systems [10].

2.2. Electro-optical analysis

A prerequisite for modern active matrix addressing technology, used to control individual picture elements (pixels) of an LCD screen, is that the component LC mixture must have an excellent electrical resistivity so that a consistent voltage may be maintained across the electrodes of each LC cell. This parameter is determined by voltage holding ratio (VHR) measurements and, gratifyingly, each of our new −LC formulations were measured to have VHR's matching those of the commercial host and so are suitable for use in display applications (Chart 1).

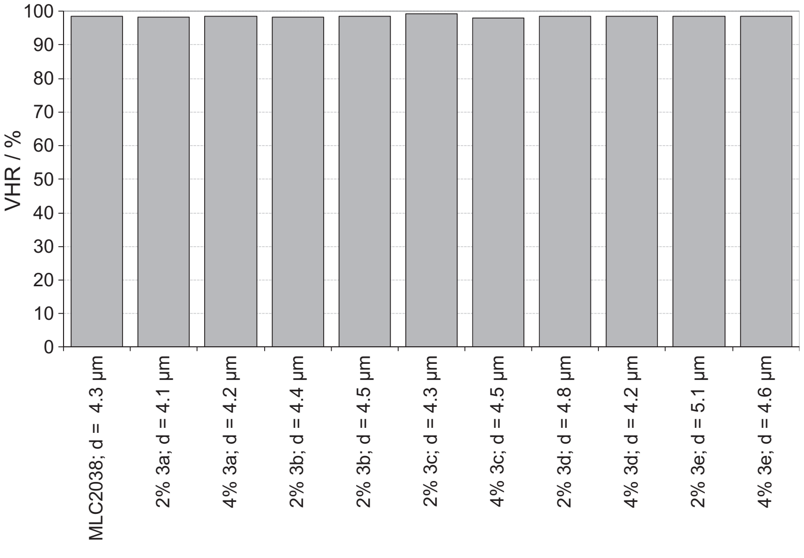


Chart 1. VHR data for MLC2038 doped with 2 and 4 wt% **3a–e**.

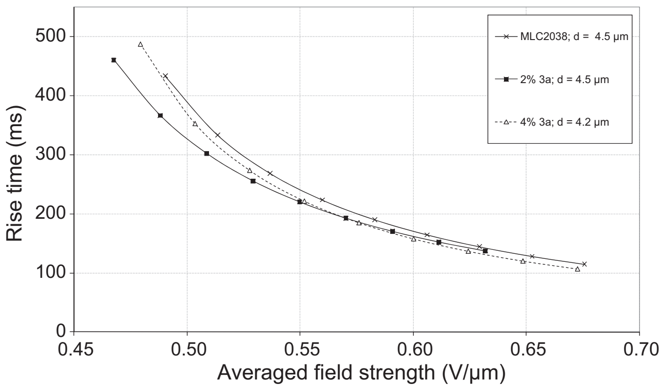


Chart 2. Rise time data for MLC2038 doped with 2 and 4 wt% **3a**.

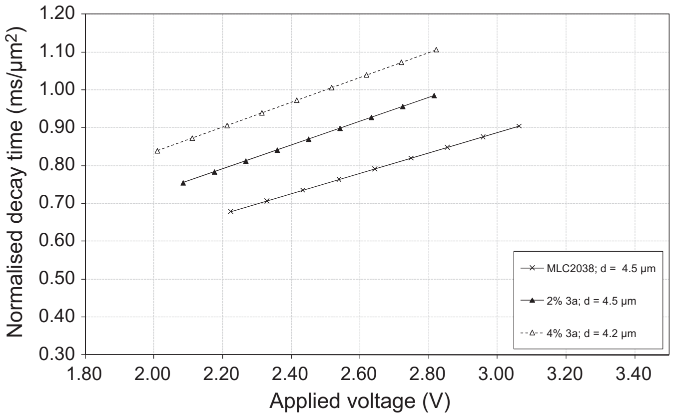


Chart 3. Decay time data for MLC2038 doped with 2 and 4 wt% **3a**.

To evaluate the performance of **3a**–**e** as dopants for commercial LC host mixtures, the rise and decay times of display cells fabricated from each new formulation were measured by optical microscopy, following procedures that we have described in detail previously [8,9]. In this paper, rise times are plotted against averaged field strength, which corresponds to the applied voltage divided by the thickness of the LC cell gap (d) whereas normalised decay times are plotted against the applied voltage. Improvements in rise times are indicated by curved plots which approach the origin whilst faster decay times are indicated by linear plots closer to the x-axis.

For example, Chart 2 shows a plot of rise time versus averaged field strength for a liquid crystal host phase, MLC2038, doped with 2 and 4 wt% of tetrafluorophenyl cyclopentyl ether **3a**, along with undoped material as a reference. All of the doped LC systems were observed to show faster rise times than the undoped material for a particular averaged field strength, indicating an improvement in device performance. The data presented in Chart 2 also suggests that increasing the level of doping does not significantly change the performance of the LC cell, as all plots obtained for both 2 and 4 wt% of dopant **3a** appear in the same region. Similar results in rise times were obtained for MLC2038 doped with **3b**–**e** (Charts 2a–d, Supporting Information).

Chart 3 shows a plot of normalised decay time versus applied voltage for MLC2038 doped with 2 and 4 wt% of dopant **3a**, alongside data for the standard undoped system. Doping was observed to lengthen LC decay times relative to the commercial host, however, no significant correlation between dopant concentration and decay time was measured. Similar results for decay times of MLC2038 doped with small quantities of **3b**–**e** were also observed (Charts 3a–d, Supporting Information).

Given the improved performance in rise times for the various LC formulations, a further parameter for use in LCD technology is that they must remain in the nematic mesophase ($T_{\mathrm{Cr-N}}$ to $T_{\mathrm{N-I}}$) over the operating temperature range of typical display devices. Particularly important is the nematic to isotropic transition temperature, or clearing point of an LC mixture ($T_{\mathrm{N-I}}$), which, for our purposes, must remain greater than a target temperature of 70 °C. The clearing points of each of the new −LC formulations were measured by DSC and experimental data is summarised in Table 2.

The data in Table 2 indicates that the introduction of 4 wt% of dopants **3a**–**e** into MLC2038 is not practical as, with the exception of **3b**, the clearing points of each new formulation are below the minimum acceptable temperature of 70 °C. Nevertheless, the clearing points of −LC systems doped at 2 wt% were found to satisfy this constraint and may be considered for practical applications.

Dopants **3a**–**e** were also blended with a commercial dielectrically positive LC host (SY5524) and the voltage holding ratios of these new LC systems were found to be excellent (Chart 4).

Chart 5 shows representative plots of rise time versus averaged field strength for a dielectrically positive LC host (SY5524) doped with 2 wt% of **3a**–**e** and Chart 6 contains the corresponding decay time data for these systems. Electro-optical data for SY5524 doped

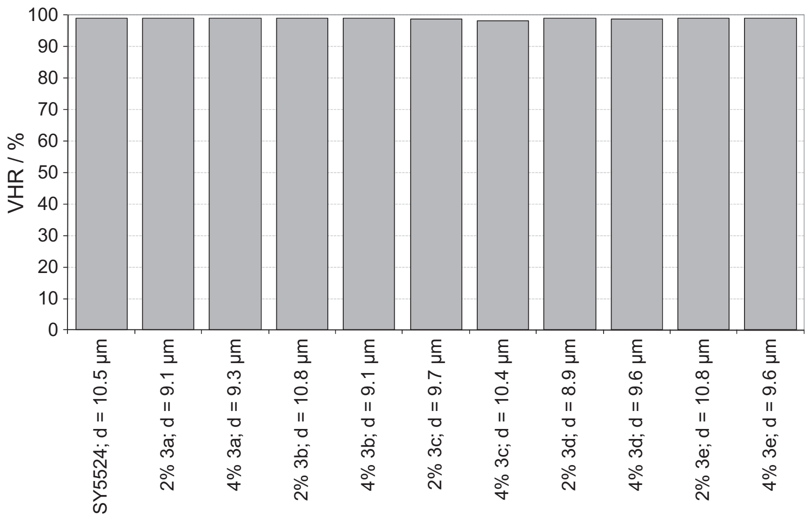


Chart 4. Voltage holding ratio data for SY5524 doped with 2 and 4 wt% **3a**–**e**.

Table 2
Nematic phase range of MLC2038 doped with 2 and 4 wt% **3a–e**.

Material	$T_{Cr–N}$ (°C)	$T_{N–I}$ (°C)
MLC2038	−66.70	80.00
2% **3a**	−68.40	74.50
4% **3a**	−69.65	69.40
2% **3b**	−67.30	74.60
4% **3b**	−67.00	70.10
2% **3c**	−67.15	73.40
4% **3c**	−66.30	67.65
2% **3d**	−67.10	74.25
4% **3d**	−98.95	68.30
2% **3e**	−66.30	74.40
4% **3e**	−66.35	69.55

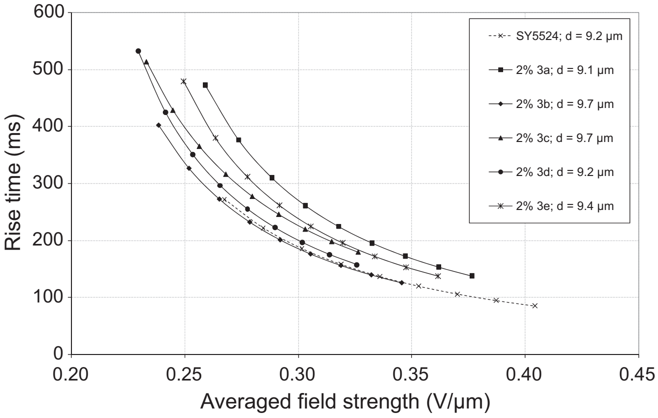


Chart 5. Rise time data for SY5524 doped with 2 wt% **3a–e**.

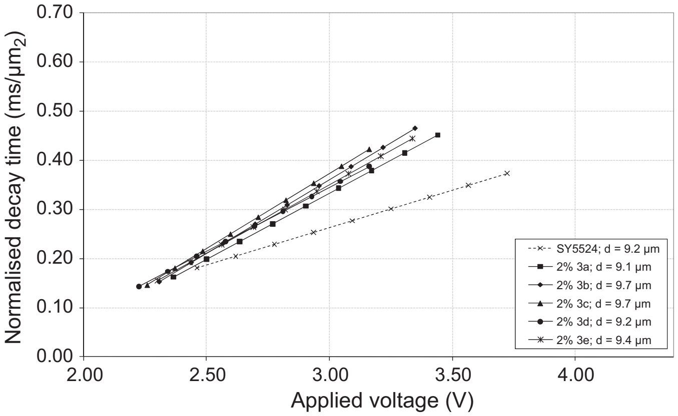


Chart 6. Decay time data for SY5524 doped with 2 wt% **3a–e**.

with 4% **3a–e** is collated in Supporting Information (Charts 5a and 6a).

Unfortunately, formulations of SY5524 mixed with both 2 and 4 wt% of **3a–e** were generally measured to have longer rise and decay times and so do not offer any improvement for display applications. Once again, no significant correlation between rise/decay times and dopant concentration or structure could be discerned with any degree of certainty.

3. Conclusions

The introduction of small quantities (2–4 wt%) of tetrafluorophenyl cycloalkoxyl ether derivatives **3a–e** into commercial −LC host MLC2038 effects an overall improvement in device response time. The voltage holding ratios of all new −LC formulations are suitable for use in modern display applications at low doping levels but the clearing temperatures of the more highly doped −LC mixtures are prohibitively low. However, we found the use of tetrafluorophenyl cycloalkyl ether systems as dopants for −LC systems do not offer a significant improvement over the corresponding biphenyl ether derivatives that we reported previously. Thus, the replacement of the phenyl group by cycloalkoxyl systems does not impart any significant change in the use of these molecules as dopant materials. Conversely, the introduction of **3a–e** into +LC host SY5224 has a detrimental effect on both rise and decay times and so these systems do not offer an improvement in LCD technology.

4. Experimental

4.1. General

Unless otherwise stated, commercially available reagents and solvents were used without purification. Dielectrically negative LC host MLC2038 was purchased from Merck and dielectrically positive LC host SY5225 was purchased from Chisso. An Innovative Technology Inc. Solvent Purification System fitted with a Metrohm 831 Karl Fischer Coulometric Titrator was used to dry THF (Fisher Scientific). Flash column chromatography was carried out using Fluorochem Silicagel LC60A (40–63 μm). Proton, carbon and fluorine nuclear magnetic resonance spectra (^{1}H NMR, ^{13}C NMR and ^{19}F NMR) were recorded on a Varian Inova-500 (^{1}H NMR, 500 MHz; ^{13}C NMR, 126 MHz; ^{19}F NMR, 470 MHz) or a Varian DD-700 (^{1}H NMR, 700 MHz; ^{13}C NMR, 176 MHz; ^{19}F NMR, 658 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR, $CHCl_3$ at 7.26 ppm; ^{13}C NMR, $CDCl_3$ at 77.36 ppm; ^{19}F NMR, $CFCl_3$ at 0.00 ppm). ^{1}H, ^{13}C and ^{19}F spectroscopic data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz), and assignment. GC–MS analysis was performed using a Trace GC–MS device (Thermo-Finnigan Corporation) operating in electron impact ionisation (EI^+) mode. C, H and N analysis was calculated with an Exeter Analytical CE-440 Elemental Analyser.

4.2. Electro-optical analysis

To establish the individual performance of the fluorinated aryl-cycloalkyl ether derivatives as LC dopants, 2–4 wt% of each substrate was mixed with commercial LC mixtures MLC2038 and SY5224, stirred at 60 °C for 1 h, allowed to cool to room temperature and left under vacuum overnight to remove any residual air bubbles. The −LC mixtures were then used to fill, by capillary action, 5 μm vertically aligned (VA) cells whilst the doped +LC mixtures were used to fill 10 μm twisted nematic (TN) cells. Both VA and TN test cells comprising indium tin oxide (ITO) coated glass substrates and a rubbed polyimide alignment layer which was purchased from EHC.

The filled doped cells were annealed in an oven for 90 min by heating to a temperature approximately 10 °C above their respective clearing points which, upon slow cooling over a period of several hours, allowed each system to reach a homogeneous nematic state. After cooling was complete, the edges of the cells were sealed and electrical wires attached to the anode and cathode of each cell. Conductive silver paint was used to ensure a good contact between the cells' electrodes and output electrical wires and, finally, the faces of all cells were cleaned with optical wipes and analytical reagent grade ethanol.

Response and transmittance measurement profiles were recorded at 35 °C using an optical microscope and proprietorial

Sony-MSL LC-drive software whereby the rise and decay times of each cell were determined across a range of applied voltages (0–5 V). In each case, three measurements were made for each concentration of dopant to ensure reproducibility of results and that cells of similar cell gap thicknesses could be compared directly. Reliable results were achieved by fabricating all devices using the same host mixtures, dopant samples and batches of manufactured LC cells.

The nematic–isotropic transition temperatures of all −LC formulations were measured for all systems by DSC (Netzsch, DSC-204) and voltage holding ratios were calculated using the 5 μm VA cells and a Toyo LCM-2 measurement device operating at a constant temperature of 50 °C.

4.3. Synthesis of LC dopants

4.3.1. General procedure

The cyclic alcohol derivative was added to a suspension of sodium hydride (60% dispersion in mineral oil) in THF and stirred at room temperature for 1 h. Pentafluorobenzene was added to the reaction mixture, which was heated to reflux overnight under an inert argon atmosphere. The reaction vessel was allowed to cool and the contents poured into water (100 mL) and the organic products were extracted with DCM ($3\times$ 100 mL). The organic extracts were combined, washed with water (150 mL) and dried ($MgSO_4$). Volatile material was removed *in vacuo* to give a crude product, which was purified by column chromatography using silica gel with hexane as elutant.

4.3.2. 3-(Cyclopentyloxy)-1,2,4,5-tetrafluorobenzene **3a**

NaH (0.113 g, 4.7 mmol), cyclopentanol (0.316 g, 3.7 mmol), pentafluorobenzene (0.792 g, 4.7 mmol) and THF (50 mL) gave 3-(cyclopentyloxy)-1,2,4,5-tetrafluorobenzene (0.522 g, 61%) as a colourless liquid; (Found: C, 56.19; H, 4.31. $C_{11}H_{10}F_4O$ requires: C, 56.41; H, 4.30%); δ_H 1.60–1.69 (2H, m, CH_2), 1.75–1.82 (2H, m, CH_2), 1.83–1.90 (2H, m, CH_2), 1.91–1.97 (2H, m, CH_2), 4.95–4.98 (1H, m, O–CH), 6.75 (1H, tt, $^3J_{HF}$ 10.0, $^4J_{HF}$ 7.0, H-6); δ_C 23.5 (s, CH_2), 33.2 (s, CH_2), 87.4 (s, O–CH), 94.4 (t, $^2J_{CF}$ 23.1, C-6), 137.3–137.5 (m, C-3), 141.59 (dddd, $^1J_{CF}$ 247, $^2J_{CF}$ 14.3, $^3J_{CF}$ 4.3, $^4J_{CF}$ 3.7, C–F), 146.16 (dddd, $^1J_{CF}$ 247, $^2J_{CF}$ 12.8, $^3J_{CF}$ 12.8, $^4J_{CF}$ 4.1, C–F); δ_F −140.92 (2F, m, F–Ar), −156.81 (2F, m, F–Ar); *m*/*z* (EI^+): 235 ($[M]^+$, 8%), 172 (100), 165 (9), 149 (8), 137 (43).

4.3.3. 3-(Cyclohexyloxy)-1,2,4,5-tetrafluorobenzene **3b**

NaH (0.106 g, 4.4 mmol), cyclohexanol (0.403 g, 4.0 mmol), pentafluorobenzene (0.792 g, 4.7 mmol) and THF (50 mL) gave 3-(cyclohexyloxy)-1,2,4,5-tetrafluorobenzene (0.496 g, 50%) as a colourless liquid; (Found: C, 58.08; H, 4.92. $C_{12}H_{12}F_4O$ requires: C, 58.07; H, 4.87%); δ_H 1.26–1.38 (3H, m, CH_2), 1.52–1.65 (3H, m, CH_2), 1.78–1.87 (2H, m, CH_2), 1.93–2.01 (2H, m, CH_2), 4.23–4.28 (1H, m, O–CH), 6.76 (1H, tt, $^3J_{HF}$ 10.0, $^4J_{HF}$ 7.0, H–6); δ_C 23.8 (s, CH_2), 25.6 (s, CH_2), 32.4 (s, CH_2), 83.3 (s, O–CH), 99.7 (t, $^2J_{CF}$ 23.1, C-6), 137.4 (tt, $^2J_{CF}$ 12.5, $^3J_{CF}$ 3.6, C-3), 142.1 (dddd, $^1J_{CF}$ 246, $^2J_{CF}$ 14.3, $^3J_{CF}$ 4.3, $^4J_{CF}$ 3.5, C–F), 146.5 (dddd, $^1J_{CF}$ 247, $^2J_{CF}$ 12.7, $^3J_{CF}$ 12.7, $^4J_{CF}$ 3.9, C–F); δ_F −140.97 (2F, m, F–Ar), −156.45 (2F, m, F–Ar); *m*/*z* (EI^+): 248 ($[M]^+$, 2%), 166 (55), 165 (100), 149 (11), 137 (50).

4.3.4. 3-(4-tert-Butylcyclohexyloxy)-1,2,4,5-tetrafluorobenzene **3c**

NaH (0.086 g, 3.6 mmol), 4-*tert*-butylcyclohexanol (0.513 g, 3.3 mmol), pentafluorobenzene (0.552 g, 3.3 mmol) and THF (50 ml) gave 3-(4-*tert*-butylcyclohexyloxy)-1,2,4,5-tetrafluorobenzene (0.496 g, 50%) as a colourless liquid; (Found: C, 63.05; H, 6.47. $C_{16}H_{20}F_4O$ requires: C, 63.15; H, 6.62%); δ_H 0.85 (9H, s, CH_3), 1.01–1.09 (3H, m, CH, CH_2), 1.46–1.54 (2H, m, CH_2), 1.81–1.89 (2H, m, CH_2), 2.18–2.12 (2H, m, CH_2), 4.09–4.14 (1H, m, O–CH), 6.76 (1H, tt, $^3J_{HF}$ 10.0, $^4J_{HF}$ 7.0, H-6); δ_C 25.7 (s, CH_2), 27.9 (s, CH_3), 32.5 (s, $\underline{C}(CH_3)_3$), 33.0 (s, CH_2), 47.3 (s, C–*t*Bu), 84.5 (t, $^4J_{CF}$ 2.6, O–CH), 99.8 (t, $^2J_{CF}$ 23.1, C-6), 137.3–137.5 (m, C-3), 142.1 (dddd, $^1J_{CF}$ 247, $^2J_{CF}$ 14.3, $^3J_{CF}$ 4.3, $^4J_{CF}$ 3.4, C–F), 146.5 (dddd, $^1J_{CF}$ 247, $^2J_{CF}$ 12.8, $^3J_{CF}$ 12.7, $^4J_{CF}$ 4.0, C–F); δ_F −140.94 (2F, m, F–Ar), −156.40 (2F, m, F–Ar).

4.3.5. 3-(Cycloheptyloxy)-1,2,4,5-tetrafluorobenzene **3d**

NaH (0.101 g, 4.2 mmol), cycloheptanol (0.436 g, 3.8 mmol), pentafluorobenzene (0.641 g, 3.8 mmol) and THF (50 mL) gave 3-(cycloheptyloxy)-1,2,4,5-tetrafluorobenzene (0.522 g, 52%) as a colourless liquid; (Found: C, 59.51; H, 5.42. $C_{13}H_{14}F_4O$ requires: C, 59.54; H, 5.38%); δ_H 1.36–1.46 (2H, m, CH_2), 1.53–1.64 (4H, m, CH_2), 1.71–1.80 (2H, m, CH_2), 1.81–1.89 (2H, m, CH_2), 1.97–2.05 (2H, m, CH_2), 4.41–4.47 (1H, m, O–CH), 6.75 (1H, tt, $^3J_{HF}$ 10.0, $^4J_{HF}$ 7.0, H–6); δ_C 22.5 (s, CH_2), 28.5 (s, CH_2), 34.4 (s, CH_2), 86.3 (t, $^4J_{CF}$ 2.6, O–CH), 99.6 (t, $^2J_{CF}$ 23.1, C-6), 137.4–137.6 (m, C-3), 142.0 (dddd, $^1J_{CF}$ 247, $^2J_{CF}$ 14.3, $^3J_{CF}$ 4.3, $^4J_{CF}$ 3.2, C–F), 146.56 (dddd, $^1J_{CF}$ 246, $^2J_{CF}$ 12.5, $^3J_{CF}$ 12.3, $^4J_{CF}$ 3.9, C–F); δ_F −140.95 (2F, m, F–Ar), −156.54 (2F, m, F–Ar); *m*/*z* (EI^+): 262 ($[M]^+$, 1%), 166 (15), 149 (5), 96 (100).

4.3.6. 3-(Cyclododecyloxy)-1,2,4,5-tetrafluorobenzene **3e**

NaH (0.191 g, 3.8 mmol), cyclododecanol (0.635 g, 3.4 mmol), pentafluorobenzene (0.579 g, 3.4 mmol) and THF (50 mL) gave 3-(cyclododecyloxy)-1,2,4,5-tetrafluorobenzene (0.496 g, 43%) as a colourless liquid; (Found: C, 65.00; H, 7.01. $C_{18}H_{24}F_4O$ requires C, 65.04; H, 7.28%); δ_H 1.29–1.41 (14H, m, CH_2), 1.41–1.53 (4H, m, CH_2), 1.61–1.68 (2H, m, CH_2), 1.80–1.88 (2H, m, CH_2), 4.45–4.50 (1H, m, O–CH), 6.75 (1H, tt, $^3J_{HF}$ 10.0, $^4J_{HF}$ 7.0, H–6); δ_C 20.9 (s, CH_2), 23.4 (s, CH_2), 23.5 (s, CH_2), 24.2 (s, CH_2), 24.6 (s, CH_2), 29.8 (s, CH_2), 83.9 (t, $^4J_{CF}$ 2.6, O–CH), 99.5 (t, $^2J_{CF}$ 23.1, C-6), 137.7–137.9 (m, C-3), 141.9 (dddd, $^1J_{CF}$ 247, $^2J_{CF}$ 14.3, $^3J_{CF}$ 4.4, $^4J_{CF}$ 3.2, C–F), 146.6 (dddd, $^1J_{CF}$ 247, $^2J_{CF}$ 12.7, $^3J_{CF}$ 12.7, $^4J_{CF}$ 3.9, C–F); δ_F −140.89 (2F, m, F–Ar), −156.64 (2F, m, F–Ar).

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jfluchem.2011.05.019.

References

[1] H. Saito, in: D.A. Dunmur, A. Fukuda, G. Luckhurst (Eds.), Physical Properties of Liquid Crystals: Nematics, London, 1999.

[2] Y. Reznikov, O. Buchnev, O. Tereshchenko, V. Reshetnyak, A. Glushchenko, J. West, Appl. Phys. Lett. 82 (2003) 1917–1919.

[3] W. Lee, C.-Y. Wang, Y.-C. Shih, Appl. Phys. Lett. 85 (2004) 513–515.

[4] Y. Sakai, N. Nishida, H. Shiraki, Y. Shiraishi, T. Miyama, N. Toshima, S. Kobayashi, Mol. Cryst. Liq. Cryst. 441 (2005) 143–152.

[5] S. Sano, T. Miyama, K. Takatoh, S. Kobayashi, Proc. SPIE – Int. Soc. Opt. Eng. 6135 (2006) 1–5.

[6] S. Sano, K. Takatoh, T. Miyama, S. Kobayashi, Dig. Tech. Pap. – SID Int. Symp. 37 (2006) 694–696.

[7] I. Keiichi, K. Takatoh, M. Akimoto, T. Miyama, Y. Sakai, S. Kobayashi, Y. Saeki, H. Hoshi, T. Kineri, N. Toshima, Dig. Tech. Pap. – SID Int. Symp. 39 (2008) 595–597.

[8] P. Kilickiran, A. Masutani, N. Hollfelder, G. Nelles, A. Yasuda, A. Tadeusiak, G. Sandford, Dig. Tech. Pap. – SID Int. Symp. 38 (2007) 999–1002.

[9] P. Kilickiran, T. Roberts, N. Hollfelder, B. Schüller, A. Masutani, G. Nelles, A. Yasuda, J. SID 16 (2008) 63–70.

[10] G.M. Brooke, J. Fluorine Chem. 86 (1997) 1–76.