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Synthesis of 4-Alkyl-2", 3" difluoro terphenyl nitrile using coupling reactions

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ABSTRACT

Study of chirality in liquid crystals is one of the most interesting areas of liquid crystal science. Liquid crystalline terphenyls with two lateral fluoro substituent and alkyl substituent in the 4- have been synthesised. Convergent approach was applied which involved the use of arylboronic acids and aryl halides in palladium-catalysed cross-coupling reactions. All the diifluoroterphenyls generate the smectic C phase. Mixture of 4-Alkyl-2'', 3difluoro terphenyl nitrile in achiral host mixture HM1 generates ferroelectric liquid crystal properties. Ferroelectric properties were found to be highly dependent upon the relative lengths of the alkyl chain attached to chiral dopant. The compounds were studied using optical polarisation microscopy, differential scanning calorimetric, nuclear magnetic resonance, and Instec elecro-optical device to find ferroelectric liquid crystal properties.

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1. Introduction

Dopants ferroelectric properties could be increased by using a terphenyl structure [1,2] and ferroelectric properties can be improved by increasing the length of the chain attached to nitrile [3]. The increased chain length increases the spontaneous polarisation Ps, viscosity by making them bulky and decreases the pitch length. The decrease in pitch length results from the restricted rotation produced by lengthening the chain. Position of fluorine on terphenyl plays important role in chiral dopant ferroelectric properties [4]. The SmC¹ phase behaviour is higher when the difluoro unit is located on the end ring.

The chiral 'dopant' needs not be mesogenic but it should not cause a large reduction of the SmC phase stability or to raise the melting point. FLC^2 is not a single compound but mixture of many components i.e. mixture of chiral dopant and achiral host mixture. Major component is achiral host mixture which has SmC phase at room temperature, low viscosity and excellent stability. Small percentage of achiral host mixture is doped with chiral dopant. The resulting FLC³ should have an $I^3-N^{4*}-SmC^*$ cooling phase sequence. Isotropic phase is liquid phase when temperature is lowered its phase changes to chiral mematic phase. Further cooling transforms chiral nematic to chiral smectic

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⁴ Nematic.

0167-7322/\$ - see front matter © 2012 Elsevier B.V. All rights reserved. http://dx.doi.org/10.1016/j.molliq.2012.08.012 A phase and then to chiral Smectic C phase. The FLC is enclosed in cell to measure its ferroelectric properties. For ideal FLC the N* pitch length should be>4× the cell gap. The SmC* pitch length should be greater than the cell gap. Spontaneous polarisation Ps should be moderately large, around 15–30 nC cm⁻² and ideally, the tilt angle would remain constant over the operating temperature of the device at 22.5°.

Two achiral host mixtures are used in this research work. One of them is HM1 which is mixture of three components.



25 %

4-pentyl-4"-heptyl-2', 3'-difluoroterphenyl



4-pentyl-4"-heptyl-2, 3-difluoroterphenyl



4-pentyl-4"-heptyl-2", 3"-difluoroterphenyl

Another achiral host mixture is LM3 which is also consisted of three components.

¹ Smectic C.

² Ferroelectric Liquid Crystal.

³ Isotropic.

⁵ SmecticA.



Ternary host mixture was prepared in weight ratios of 1:1:1. The actual measured percentage of compound 1, 2 and 3 were 33.1%, 33.7% and 33.12% respectively. This mixture melts below the room temperature. Mixture has thermally stable SmC phase with a transition of 66.5 °C to the SmA phase. This achiral host mixture is called LM3.

2. Synthesis of 4-Alkyl-2", 3" difluoro terphenyl nitrile

Ortho difluoro substituted materials were synthesised that possess a central terphenyl core and to incorporate chiral centre in the terminal chain. Two fluorine rings were attached to terminal ring. Length of alkyl chain was changed from pentyl-heptyl-nonyl. Synthesis routes of these chiral dopants can be found in some other paper [3].

Research paper Cyanoalkyl difluoro-terphenyl-carboxylate chiral dopants Z. N. Kayani. et.al [3] provides synthesis details of biphenyl cyanohydrin ester which is initial compound for synthesis of 4-Alkyl-2'', 3'' difluoro terphenyl nitrile. Same research paper [3] provides Synthesis steps of 4-(pentyl, heptyl)-2'', 3'' difluoro terphenyl nitrile using coupling reactions [1]. Biphenyl cynohydrin ester is used as starting material in this synthesis. [3] Shows synthesis steps of 4-nonyl-2'', 3'' difluoro terphenyl nitrile using coupling reactions. For this chiral dopant synthesis route described for 4-(pentyl, heptyl)-2'', 3'' difluoro terphenyl nitrile were not adopted.

2.1. Synthesis details of all intermediates and final products

Synthesis of Biphenyl cynohydrin ester 4-bromo-4'-acetyl biphenyl



Quantities and procedure:

Dichloromethane (500 ml) was added to Aluminium chloride (26.66 g, 199.7 mmol, 133.5 g/mol). The mixture was stirred and cooled to a 5 °C in a salt/ice bath. Acetyl chloride (26.83 g, 343.97 mmol, 78 g/mol) was quickly added through a dropping funnel and cooled to -3 °C to give a transparent yellow solution. Solid 4-bromobiphenyl (40 g, 171.67 mmol, 233 g/mol) was added in small amounts and stirred, wait for ½ an hour then add more. The mixture was left stirring for ten hours with the temperature rising to 15 °C. The mixture was poured into bucket of ice /37% HCl to destroy the aluminium chloride, then transferred to a separatory funnel and extracted into DCM. Then the mixture was washed twice with 200 ml of water. The collected DCM layer was dried over Magnesium Sulfate, the most of it filtered by gravity. The solvent was removed in vacuo and residue was re-crystallised through ethanol.

Yield (40.11 g, 84.98%): M.P. 133.3 [5]; $\delta_{H}(400 \text{ MHz; CDCl}_{3})$ 2.64 (3H,s,<u>C</u>H₃),8.03 (2H, d, J 8.25),7.64 (2H,dJ 8.61), 7.59 (2H,dJ 8.80), 7.49 (2H, d, J 8.43); δ_{C} (100.5;CDCl₃)26.67 (<u>C</u>HMe),127.03, 128.81, 129.01, 132.09, 136.12, 138.76, 144.49, 197.62 (CO);

4-bromo-biphenyl carboxylic acid



Quantities and procedure:

A solution of sodium hypobromite prepared at 0 °C (by keeping flask in ice) by dissolving bromine (79.6 g, 497.5 mmol, 160 g/mol) in a solution of sodium hydroxide (71.4 g, 1785 mmol, 40 g/mol) in a 350 ml water, was added with stirring to a solution of the 4- bromo-4''acetylbiphenyl (20 g, 72.73 mmol, 275 g/mol) in dioxan (300 ml). The oil bath was used and the temperature was maintained at 35–40 °C throughout the addition and for 15 minutes afterwards. The suspension was treated with enough aqueous sodium thiosulfate pentahydrate to remove the excess of hypobromite till solution was white. Water (500 ml) was added and liquid was distilled off. The residual suspension or solution was acidified with concentrated hydrochloric acid and cooled. The free acid was filtered off and crystallised from propanol and ethanol. It was then filtered off and left in 60 °C oven overnight.

Yield (15.81 g, 78.18%); M.P.306 °C [6] m/z 278 (M⁺, 100%), 261, 230, 196, 152, 76; $\delta_{\rm H}(400 \text{ MHz};{\rm CDCl}_3)$ 7.53 (2H, d, J 8.43),7.6 (2H,d,J 8.61),7.65 (2H,d,J 8.06),8.09 (2H,d,J 8.25); ; $\delta_{\rm C}(100.5;{\rm CDCl}_3)$ 122.22, 126.64, 128.8, 130.28, 131.93, 138.88, 143.66, 168.05 (CO);

Bromobiphenyl carbonyl chloride



Quantities and procedure:

4- Bromobiphenyl carboxylic acid (3 g, 10.79 mmol, 278 g/mol) was put in a 100 ml round bottom flask with (30 ml, 36.5 g/mol) DCM, placed it on a mechanical stirrer. A nitrogen bubbler was attached to it. Syringed out (1.12 ml, 12.95 mmol, 126 g/mol) of oxalyl chloride and few drops of DMF. After adding few drops of DMF switched off the Nitrogen. Bubbles will be coming out. Left it to stir for the time bubbles stopped coming off. The solvent was removed in vacuo.

bromobiphenyl carbonyl amide



Quantities and procedure:

The s-(-)-lactamide (0.19 g, 10.25 mmol, 89 g/mol) was dissolved in (8.94 ml) pyridine with magnetic stirrer. The bromobiphenyl carbonyl chloride (3.19 g, 10.79 mmol, 296 g/mol) in 15 ml ether and 15 ml THF was added to the above solution drop wise with the help of dropping funnel while stirring. It was left stirring for ten hours. Ice was put in 40 ml of diluted HCl solution; over it suspension in round bottom flask was poured. It was filtered and dry product, re-crystallised through hexane and toluene.

Yield (1.51 g, 40%); M.P. 171.1 °C (from hexane and toluene). **Elemental analysis:** calc. for (Found: C, 55.49; H, 3.94 ; N, 3.86 . $C_{16}H_{14}BrNO_3$ requires C, 55.19; H, 4.05; N, 4.02%). *m/z* 349 (M⁺), 276, 261 (100%), 230, 152, 76; IR(KBr) v_{max}/cm^{-1} 3419 and 3199 (CONH₂), 1716 vs, (CO), 1636, 1608, 1481, 1393, 1294, 1276, 1119, 1076, 1003, 817, 767; $\delta_{H}(400 \text{ MHz}; \text{CDCl}_3)$ 1.65 (3H,t, J6.78), 5.52 (1H,q, J6.96, C<u>H</u>Me), 5.59 and 6.15 (1H, brs, N<u>H</u>),7.50 (2H,d,J 8.80),7.61 (2H,d,J 8.61), 7.66 (2H,d,J 8.61), 8.14 (2H,d,J 8.61); δ_{C} (100.5; CDCl₃) 17.7, 53.42, 122.85, 127.09, 128.89, 130.41, 132.16, 138.59, 145.17, 164.97 (CO), 172.41 (CONH₂); (S)-1-cynoethyl 4'-bromobiphenyl -4-carboxylate



Quantities and procedure:

Dry DMF (40 ml) was cooled to 0 $^{\circ}$ C. POCl₃ (1.85 ml) was added slowly by syringe under nitrogen keeping the temperature below 5 $^{\circ}$ C. It was stirred for 20 minutes and turned coloured. Bromobiphenyl amide (1.39 g, 3.98 mmol, 349 g/mol) was added in portion and stirred for twenty hours at room temperature. Solution was poured in ice and water mixture. Swirled around and was placed to settle down. A precipitate was obtained. It was filtered and washed with water. The dry product was re-crystallised through toluene.

Yield (1.39 g, 100%); M.P. 167 °C (from toluene). **Elemental analysis:** (Found: C, 58.45; H, 3.57; N, 4.11. C16H12BrNO2 requires C, 58.2; H, 3.66; N, 4.24%); IR (KBr) v_{max}/cm^{-1} 1718 s(CO),1653, 1605, 1585, 1558, 1478, 1388, 1277, 1253, 1203, 1178, 1077, 997, 816, 757, 685; $\delta_{H}(400 \text{ MHz;CDCl}_3)$ 1.80 (3H, d, J 6.96, Me), 5.68 (1H, q, J 6.96, C<u>H</u>Me), 7.50 (2H, d, J 8.80), 7.61 (2H, d, J 8.61), 7.66 (2H, d, J 8.61), 8.12 (2H, d, J 8.61), δ_{C} (100.5;CDCl₃) 18.99, 57.80, 117.54 (CN), 123.02, 127.51, 128.86, 130.63, 132.16, 138.53, 145.42, 164.41 (CO);

Synthesis of 4-(pentyl, heptyl)-2'', 3'' difluoro terphenyl nitrile 4-pentyl-2'', 3''-difluoro terphenyl



Quantities and procedure:

1, 2-dimethoxyethene (150 ml) and sodium carbonate (2 M, 150 ml) were added to diflouroboronic acid (4.79 g, 30.33 mmol, 158 g/mol) and 4-bromo-4''pentylbiphenyl (7.07 g, 23.33 mmol, 303 g/mol) under dry nitrogen with continuous stirring. Tetrakis (triphenylphosphine) palladium (0) (0.1348 g, 0.11 mmol, 1156 g/mol) was added. The stirred mixture was heated under reflux (ca. 120 °C) for 2 h (i.e., until g. L.c/ t. l. c. analysis revealed a complete reaction). Reaction mixture was cooled to room temperature and 100 ml water was added. The product was extracted into 200 ml ether (twice) and the combined ethereal extracts were washed with brine and dried (MgSO₄), the most of it filtered off by pressure. The solvent was removed in vacuo. The dry product was purified by addition to a packed chromatography column of silica gel [silica gel, Hexane] to yield white product. Product was re-crystallised through ethanol. It was put in the desiccators overnight.

Yield (6.47 g, 82.53%).M.P. 97.5 °C (from EtOH). **Elemental analysis:** (Found: C, 82.40; H, 6.74 .Calc. for $C_{23}H_{22}F_2$: C, 82.11; H, 6.59%);m/z 336 (M⁺), 292, 279 (100%),165, 151; transitions / °C K 95.8 (25.79 J/g) SmA 108.8 (18.98 J/g) I;IR (KBr) υ_{max}/cm^{-1} 2927 (CH), 1473, 1398, 1264, 1216, 1100, 897, 817, 785, 721; δ_H (400 MHz;CDCl₃) 0.80 (3H,tJ 7, C**H**₃CH₂), 1.19–.27 (8H, m), 1.57 (2H, quint, J7.7, ArCH₂C**H**₂), 2.57 (2H, t, J7.9, ArCH₂), 7.02 (1H, ddd, J 6.8, 6.6, 1.5, 20H), 7.16 (1H, ddd, J 6.8, 6.4, 1.6, 19H),7.47 (2H,dJ 8.1), 7.52 (2H, dd, J 8.2, 1.5), 7.59 (2H, d, J8.2),;(δ_F (376; CDCl₃)) – 143.30 (1F, dd, J_{F,F}20.81, J_{F,H}6.9), – 143.88 (1F, dd, J_{F,F}18.5, J_{F,H}6.9); δ_C (100.5;CDCl₃) 14.10, 22.68, 29.20, 29.34, 31.50, 31.82, 35.64, 116.0 (d, J 16.9), 124.08 (dd, J_{C,F} 5.4, 4.6), 125.20 (dd, J_{C,F} 3.8, 1.5), 126.93, 127.11, 128.92, 129.27 (d J_{C,F} 3.1), 130.83, 131.00, 133.26 (d, J_{C,F} 4.6), 137.75, 141.00, 142.51;





Quantities and procedure:

A solution of n-butylithium (8.27 ml, 22.39 mmol in hexanes) was added drop wise to a stirred, cooled (-78 °C) solution of 4-pentyl-2'', 3''-difluoro terphenyl (6.27 g, 18.66 mmol, 336 g/mol) in(150 ml) THF under dry nitrogen..The reaction mixture was maintained at

- 78 °C for 1 hour, poured on to mixture of dry ice and dry ether and allowed to warm at room temperature. 50 ml of diluted hydrochloric acid was added. The THF was evaporated through rotary evaporator till 50 ml solution was left. It was cooled by ice and after an hour filtered by pressure. It was washed by water several times to remove excess acid. It was re-crystallised through isopropanol. The dry product was placed in oven overnight.

Yield (6.40 g, 85.46%). M.P. 270 °C (from isopropanol); **Elemental analysis**: (Found: C, 77.94; H, 5.83. calc. for C₄₇H₄₄F₄O₂: C, 75.77; H, 5.83%); m/z 380 (M⁺).336, 323 (100%), 310, 277, 165, 153; IR(KBr) v_{max}/cm^{-1} 2925 (CH),1700 (COvs), 1653, 1623, 1507, 1464, 1419, 1399, 1303, 1220, 1105, 809, 781, 765; δ_{H} (400 MHz;CDCl₃) 0.90 (3H,t, J 7.0, CH₃CH₂),1.28–1.39 (4H,m),1.63 (2H, quint, J 7.7, ArCH₂CH₂), 2.64 (2H,t, J 7.9, ArCH₂), 7.34 (2H,d,J 8.2), 7.53 (1H, ddd, J 7, 6.8, 1.6), 7.68 (2 H, d, J 8.2), 7.73 (2 H, dd, J 8.4, 1.5), 7.77 (1 H, ddd, J 7.0, 6.8, 1.6),7.84 (2H,d,J 8.6),; δ_{F} (376 MHz;CDCl₃), -135.16 (1F, dd, J_{FF} 20.81, J_{FH} 6.9), -142.43 (1F, dd, J_{FF} 18.5, J_{FH} 6.9); δ_{C} (100.5;CDCl₃) 13.93, 21.97, 30.69, 30.91, 34.70, 120.43 (dd, J_{CF} 3.8, 2.3), 124.68 (dd, J_{CF} 3.8, 2.3),125.35.126.41 (d, J_{CF} 3.8), 126.61, 126.85, 128.99, 129.37 (d, J_{CF} 3.1), 131.92, 133.35, 133.44, 136.59, 140.65, 142.23, 164.04 (dd, J_{CF} 3.1, 2.3, CO);

^{*} 4-pentyl-2", 3"-difluoro terphenyl carbonyl chloride



Quantities and procedure:

4- pentyl-2",3"-difluoro terphenyl carboxylic acid (3 g, 7.89 mmol, 380 g/mol), DCM (30 ml, 36.5 g/mol), oxalyl chloride (1.1 ml, 12.98 mmol, 126 g/mol) and few drops of DMF.

The experimental procedure was as described for the preparation of compound Bromobiphenyl carbonyl chloride.

4-pentyl-2", 3"-difluoro terphenyl carbonyl amide



Quantities and Procedure:

4-pentyl-2", 3"-difluoro terphenyl carbonyl chloride (3 g, 7.54 mmol, 398 g/mol), s-(-)-lactamide 0.86 g, 9.66 mmol, 89 g/mol, pyridine (8.4 ml, 106.33 mmol, 79 g/mol) and dry THF (60 ml). The experimental procedure was as described for the preparation of compound bromobiphenyl carbonyl amide.

Yield (1.3 g, 37.25%); M.P.193.4 °C (from ethyl acetate); **Elemental analysis:** calc. for (Found: C, 71.93; H,6.25; N, 3.14 . $C_{27}H_{27}F_2NO_3$ requires C, 71.82; H, 6.03; N, 3.10%); *m/z* 451 (M⁺), 394, 379, 363, 335, 322, 278, 165, 153, 44 (100%); transitions/°C K 177.8 (79.86 J/g)SmA* 206.4 (20.11 J/g)I; IR (KBr) v_{max}/cm^{-1} 3391 (CONH₂), 2925 (CH), 1723 vs (CO), 1652 vs(CONH₂), 1559, 1506, 1458, 1419, 1300, 1165, 1105800, 779; $\delta_{\rm H}$ (400 MHz;CDCI₃) 0.91 (3H,tJ 6.9, C<u>H</u>₃C<u>H</u>₂), 1.34– 1.38 (4H,m), 1.63 (3H, d, J 7, Me), 1.82 (2H, quint, J 7.9, ArC<u>H</u>₂CH₂), 2.66 (2H,t, J 7.9, ArC<u>H</u>₂), 5.4 (1H, q, J 6.78, C<u>H</u>Me), 6, 8 and 6.92 (1H, brs, NH), 7.3 (2H,dJ 8.2), 7.39 (1H, ddd, J 6. 8,6.6, 1.5), 7.58 (2H, d, J 8.4),7.66 (2H, dd, J 7, 1.5), 7.73 (2H, d, J 8.4), 7.85 (1H, ddd, J 6.6, 6.4, 1.8); $\delta_{\rm F}$ (376 MHz;CDCI₃), -134.55 (1F, dd, J_{F,F} 20.81, J_{F,H} 6.9), -142.26 (1F, dd, J_{F,F} 20.81, J_{F,H} 6.9); $\delta_{\rm C}$ (100.5;CDCI₃) 13.99, 17.79, 22.36, 30.99, 31.35, 35.37, 71.18 (<u>C</u>HMe), 121.5, 124.4, 126.46, 126.73, 127.04, 128.94, 129.21, 135.09, 137.05, 142.59, 146.58, 160.07, 172.41;

Quantities, procedure, yield and elemental analysis of 4-pentyl-2", 3"-difluoro terphenyl carbonyl nitrile can be found in some other paper [3].

4-heptyl-2", 3"-difluoro terphenyl



Quantities and procedure:

4-bromo-4"-heptyl biphenyl (7.07 g, 21.36 mmol, 331 g/mol), diflouroboronic acid (5.69 g, 27.77 mmol, 158 g/mol), Tetrakis (triphenylphosphine) palladium (0)(.2 g, 0.11 mmol, 1156 g/mol),1, 2-dimethoxyethene (150 ml) and sodium carbonate (2 M, 150 ml).

The experimental procedure was as described for the preparation of compound 4-pentyl-2", 3"-difluoro terphenyl except that reaction completed in 23 h.

Yield (6.5 g, 83.55%); M.P. 47.6 °C (from EtOH). **Elemental analysis**: (Found: C, 82.56; H, 7.47. calc. for $C_{25}H_{26}F_2$: C, 82.38; H, 7.19%);m/z 364 (M⁺), 292, 279 (100%),165, 151; transitions/°C K 34.5 (11.67 J/g) cryst E 61.1 (6.15 J/g) SmB 73.7 (1.3 J/g) SmA 104.6 (18.81 J/g) I ; IR (KBr) υ_{max}/cm^{-1} 2925 (CH),1472, 1398, 1265, 1216, 1099, 896, 816, 701, 718; $\delta_{H}(400 \text{ MHz}; \text{CDCl}_3)$ 0.80 (3H,tJ7.1,C $\underline{H}_3\text{CH}_2$),1.19–.27 (8H, m),1.57 (2H, quint, J 7.7, ArCH₂C \underline{H}_2), 2.57 (2H, t, J 7.9, ArCH₂), 7.02 (1H, ddd, J 6.8, 6.6, 1.5, 20H),7.16 (1H, ddd, J 6.8, 6.4, 1.6, 19H),7.47 (2H, d, J 8.1), 7.52 (2H, dd, J 8.2, 1.5),7.59 (2H,dJ 8.2); ($\delta_F(376; \text{CDCl}_3)$) – 143.30(1F, dd, J_{FF} 20.81, J_{FH} 6.9), –143.88 (1F, dd, J_{FF} 18.5, J_{FH} 6.9); δ_C (100.5; CDCl₃) 14.10, 22.68, 29.20, 29.34, 31.50, 31.82, 35.64, 115.91, 116.08, 124.06 (dd, J_{CF} 5.4, 3.5), 125.20 (dd, J_{CF} 3.8, 3.5), 126.93, 127.11, 128.92, 129.27 (d, J_{CF} 3.1), 130.90, 131.00, 133.26 (d, J_{CF} 4.6), 137.75, 141.00, 142.51;

4-heptyl-2", 3"-difluoro terphenyl carboxylic acid



Quantities and procedure:

4-heptyl-2", 3"-difluoro terphenyl (6.25 g, 17.17 mmol, 364 g/mol), n-butylithium (8.24 ml, 20.60 mmol in hexanes) and THF.

The experimental procedure was as described for the preparation of compound 4-pentyl-2", 3"-difluoro terphenyl carboxylic acid.

Yield (4.13 g, 61.4%); M.P. 247.7 °C (from isopropanol); **Elemental analysis:** (Found: C, 76.22; H, 6.21. calc. for $C_{26}H_{26}F_2O_2$: C, 76.45; H, 6.42%);m/z 408 (M⁺), 364, 336, 323 (100%),310, 279, 165, 153; IR(KBr) v_{max}/cm^{-1} 2924 (CH), 1684 (COvs), 1623, 1506, 1465, 1419, 1306, 1103, 956, 894, 806, 777; δ_{H} (400 MHz;CDCl₃) 0.89 (3H,tJ 7.0, C**H**₃C**H**₂), 1.24–1.39 (9H, m),1.65 (2H, quint, J 7.7, ArC**H**₂C**H**₂), 2.65 (2H,t, J 7.9, ArC**H**₂), 7.29 (2H,dJ 8.2), 7.35 (1H, ddd, J 6.8, 6.6, 1.6), 7.57 (2H,dJ 8.1),7.65 (2H, dd, J 7, 1.5), 7.72 (2H,dJ 8.6),7.78(1H, ddd, J 6.8, 6.6, 1.6); (δ_{F} (376; CDCl₃)) – 135.8 (1F, dd, J_{F,F} 18.5, J_{F,H} 6.9), – 143.111 (1F, dd, J_{F,F} 20.8, J_{F,H} 6.9); δ_{C} (100.5;CDCl₃) 14.06, 22.5, 29.00, 29.11, 31.34, 31.64, 35.44, 119.82 (dd, J_{C,F} 3.8, 3.1), 124.09 (d, J_{C,F} 3.8), 126.44 (d, J_{C,F} 4.6), 126.73, 127.00, 128.93, 129.22 (d, J_{C,F} 3.1), 132, 28, 134.15, 134.24 (d, J_{C,F}2.3), 137.17, 141.36, 142.57, 146.93, 164.86 (dd, I_{C,F} 3.8, 3.1),

4-heptyl-2", 3"-difluoro terphenyl carbonyl chloride



Quantities and procedure:

4-heptyl-2'', 3''-difluoro terphenyl carboxylic acid (2 g, 4.9 mmol, 408 g/mol), DCM (20 ml, 36.5 g/mol), oxalyl chloride (0.75 ml, 8.65 mmol, 126 g/mol) and few drops of DMF.

The experimental procedure was as described for the preparation of compound Bromobiphenyl carbonyl chloride. 4-heptyl-2",3"-difluoro terphenyl carbonyl amide





Quantities and procedure:

4-heptyl-2", 3"-difluoro terphenyl carbonyl chloride (2 g, 4.69 mmol, 426 g/mol), s-(-)-lactamide 0.57 g, 6.4 mmol, 89 g/mol, pyridine (5.6 ml, 79.89 mmol, 79 g/mol) and dry THF (40 ml).

The experimental procedure was as described for the preparation of compound bromobiphenyl carbonyl amide.

Yield (0.9 g, 40.9%); M.P. 195.9 °C (from ethyl acetate); **Elemental analysis:** calc. for (Found: C, 72.68; H, 6.81; N, 2.85 . $C_{29}H_{31}F_2NO_3$ requires C, 72.63; H,6.52; N,2.92%);m/z 479 (M⁺, 100%),407, 391, 380, 323, 307, 278, 165, 153; transitions/°C K 174.5 (65.57 J/g) SmA* 198.6 (98.26 J/g) I; IR (KBr) υ_{max}/cm^{-1} 3392 (CONH₂), 2924 (CH), 1723 vs

Table 1

Transition temperatures (°C) and enthalpy change taken by optical polarising microscope and differential scanning calorimeter for the 4-alkyl-2", 3"-difluoro terphenyl nitrile and their intermediate compounds.

Structure	Mesomorphic behaviour
	Melting point: 97.5°C Transitions/°C K 95.8 (25.79J/g) SmA 108.8 (18.98J/g) 1
	Melting point: 191.45°C transitions/° C K 177.8 (79.86J/g) SmA* 206.4 (20.11J/g) l
	Melting point: 103.2°C Transitions/°C SmC* 102.8 (15.85J/g)SmA* 164.9 (16.45J/g) I[3]
C7H15	Melting point: 47.61°C Transitions/°C K 34.5 (11.67J/g) cryst E 61.1 (6.15J/g) SmB 73.7 (1.3J/g) SmA 104.6 (18.81J/g) I
	Melting point: 195.93°C transitions/°C K 174.5 (65.57J/g) SmA* 198.6 (98.26J/g) I
	Melting point: 95.78°C transitions/°C SmC* 95.2 (14.61 J/g)SmA* 145.5 (14.85J/g) I I[3]
C ₉ H ₁₉	Melting point: 66.66°C Transitions temperature/°C K 52.4 (28.58 J/g) Cryst E 61.6 (4.4 J/g) SmB 71.6 (64.26 J/g) SmA 103.1 (18.97 J/g) I;
	Melting point:193.60 ° C (97.52 J/g) Transitions/°C K 182.9 (94.19 J/g) SmA 190.3 (97.52 J/g)
	Melting point: 85.43°C (13.93 J/g) Transition/°C K 82.9 (13.93 2 J/g) SmY 94.9 (0.40 J/g) SmX 100.99 (0.44 J/g) SmA 131.6 (11.42 J/g) J[3]

(CO), 1663.1652 (CONH₂),1635, 1558, 1507, 1464, 1300, 1232, 1166, 1105804, 780; $\delta_{H}(400 \text{ MHz};\text{CDCI}_3)$ 0.89 (3H, t, J7.0, CH₃CH₂), 1.24–1.42 (8H, m),1.65(3H, d, J 6.8, Me), 1.69 (2H, quint, J 7.3, ArCH₂CH₂), 2.56–2.74 (8H, m), 5.45 (1H, q, J 6.8, CHMe), 6.49 and 6.73(1H, brs, NH), 7.29 (2H,dJ 8.1) 7.38 (1H, ddd, J 7, 6.6, 1.3), 7.57 (2H, d, J 8.1),7.65 (2H, dd, J 7.1, 1.0), 7.72 (2H,dJ 8.4), 7.84 (1H, ddd, J 7, 6.6, 1.6); ($\delta_{F}(376; \text{CDCI}_3)$) – 139.92 (1F, dd, J_{FF} 20.81, J_{FH} 6.9), – 142.69 (1F, dd, J_{FF} 20.81, J_{FH} 4.6); δ_{C} (100.5;CDCl₃) 14.01, 17.71, 22.38.28.92 (d, J10), 31.23, 31.52, 35.2971.02, 118.37, 124.32, 126.44, 126.63, 126.93, 128.90, 129.20 (d, J_{C,F} 3.1), 131.90, 136.92, 141.37, 142.5, 144.09, 161.87, 172.22;

Quantities, procedure, yield and elemental analysis of **4-heptyl-2**", **3"-difluoro terphenyl carbonyl nitrile** can be found in some other paper [3]. Synthesis of 4-nonyl-2'', 3''difluoro terphenyl nitrile 4-nonanoyl-4'-bromo biphenyl



Quantities and procedure:

4'-bromobiphenyl (50 g, 214.59 mmol, 233 g/mol), aluminium chloride (33.36 g, 249.91 mmol, 133.5 g/mol), nonanoyl chloride (52.23 ml, 277.9 mmol, 176.69 g/mol) and DCM (650 ml, 36.5 g/mol).



Fig. 1. (a) Mixture of 7% D9R in HM1, Isotropic phase to nematic phase transition; shearing induces homogeneous alignment and produces birefrigent, (b) Mixture of 14% D5R in HM1; fingerprint texture of chiral nematic phase, (c) Mixture of 7% D9R in HM1; focal conic texture of SmA* phase and (d) Mixture of 7% D9R in HM1; the scheliring texture of SmC* phase (e). The scheliring texture of nematic phase of 3% D5R in pyrimidine (LM3) mixture, (f) The scheliring texture of SmC* phase of 3% D5R in pyrimidine (LM3) mixture.

The experimental procedure was as described for the preparation of compound 4-bromo-4'-acetyl biphenyl.

Yield (68.23 g, 85.24%); M.P. 106.7 °C (from ethanol, ethyl acetate); δ_H (400 MHz;CDCl₃) 0.88 (3H,t,J7.1,CH₃CH₂), 1.23–1.43 (10H, m), 1.75 (2H, quint, [7.5, ArCH₂CH₂), 2.98 (2H, t, [7.5), 7.49 (2H,d,[8.8), 7.59 (2H, d, J 8.8), 7.64 (2H, d, J 8.6), 8.03 (2H, d, J 8.8); δ_C(100.5;CDCl₃) 14.11, 22.67, 24.45, 29.18, 29.43 (d, J 5.4), 31.85, 38.73, 122.59, 127.02, 128.79 (d, J 3.8), 132.09, 136.09, 138.85, 144.24, 200.13; m/z 374 (M⁺), 276, 261, 152 (100%);

4- nonyl-4'-bromo biphenyl

NH2NH2.H2C KOH,DEG

Quantities and procedure: 4- onanoyl-4'-bromo biphenyl (50 g, 139.16 mmol, 359.30 g/mol) and potassium hydroxide (23.38 g, 147.48 mmol, 56 g/mol)were



Hull Liquid Crystal Group: dapreut

STAR[®] SW 8.10



Fig. 2. Heating and cooling of 3%, 7% and 14% 4-pentyl-2", 3"-difluoroterphenyl nitrile and 3%, 7% 4-(heptyl, nonyl)-2", 3"-difluoroterphenyl nitrile in achiral host mixture HM1 showing SmC* phase, SmA* phase, nematic phase and isotropic phase. 14 % 4-pentyl-2'', 3''-difluoroterphenyl nitrile in HM1 does not possess ferroelectroic properties as sown in curve c.







added to a 1 litre three necked flask. Diethylene glycol was added and the mixture heated to 80 °C, then hydrazine monohydrate (23 ml, 416.7 mmol, 50 g/mol) was added slowly. Then the mixture was heated at 130–140 °C for 3 hours continuous stirring under reflux to give a light brown liquid, before distilling off the hydrozone monohydrate and some vapours at 120 °C. The mixture was heated to 180–190 °C for 3 hours to give a dark brown liquid that was then cooled to 80 °C and poured into ice/37% HCl to remove KOH. The crude was extracted through DCM (250 ml).The organic extracts was then washed with water, dried over MgSO₄ and then solvent removed in vacuo. It was

re-crystallised through ethanol. It still has impurity. So the crude was purified by addition to a packed chromatography column of silica gel [silica gel, Hexane] to yield white product.

Yield (24.15 g, 48.3%); M.P. 87.6 °C (from ethanol); $\delta_{\rm H}$ (400 MHz; CDCl₃) 0.88 (3H, t, J 7.1, C**H**₃CH₂), 1.23–1.41 (13H, m), 1.64 (2H, quint, J7.7, ArCH₂C**H**₂), 2.63(2H, t, J 7.9), 7.23 (2H, d, J 8.2), 7.44 (2H, d, J 8.8), 7.46, (2H, d, J 8.6), 7.54 (2H, d, J 8.6); $\delta_{\rm C}$ (100.5; CDCl₃) 14.11, 22.68, 29.34 (d, J 2.3), 29.54 (d, J 3.1), 31.48, 31.9, 35.61, 121.15, 126.76, 128.57, 128.96, 131.79, 137.28, 140.10, 142.62; *m/z* 360 (M⁺), 247 (100%), 178, 165, 152, 41





Fig. 2 (continued).





Quantities and procedure:

4- nonyl-4'-bromo biphenyl (10 g, 27.8 mmol, 359 g/mol), difluoroboronic acid (7.03 g, 36.21 mmol, 158 g/mol), dimethoxyethane (220 ml), 2 M sodium carbonate (220 ml) and tetrakis (triphenylphosphine) palladium(0)(.241 g, 0.208 mmol, 1156 g/mol). The experimental procedure was as described for the preparation of compound 4-(pentyl, heptyl)-2", 3"-difluoro terphenyl.

Yield (7.54 g, 69.17%); M. P. 66.66 °C Transitions temperature / °C K 52.4 (28.58 J/g) Cryst E 61.6 (4.4 J/g) SmB 71.6 (64.26 J/g) SmA 103.1 (18.97 J/g) I; **Elemental analysis:** calc. for (Found: C; H; N. $C_{27}H_{30}F_2$ requires C, 82.62; H, 7.7; N%); δ_{H} (400 MHz;CDCl₃) 0.88 (3H, t, J7, C<u>H</u>₃CH₂),1.23–1.41 (13H, m), 1.66 (2H, quint, J7.9, ArCH₂C<u>H</u>₂), 2.66 (2H, t, J 7.9, ArCH₂), 7.02 (1H, ddd, J 6.8, 6.6, 1.5, 20H), 7.16 (1H, ddd, J 6.8, 6.4, 1.6, 19H), 7.56 (2H, d, J8.2), 7.61 (2H, dd, J8.6, 1.6), 7.6 8(2H, d, J8.6); (δ_{F} (376; CDCl₃)) – 143.30(1F, dd, J_{F.F} 20.81, J_{F.H} 6.9), – 143.88(1F, dd, J_{F.F} 18.5, J_{F.H} 6.9); δ_{C} (100.5;CDCl₃) 14.13, 22.7,



Fig. 2 (continued).

29.37 (d, J 4.6), 29.56 (d, J 2.3), 31.51, 31.91, 35.65, 116.02 (d, J 16.9), 124.09(dd, $J_{C,F}$ 5.8, 4.6), 125.21 (dd, $J_{C,F}$ 3.1, 2.3), 126.95, 127.12, 128.94, 129.29 (d $J_{C,F}$ 3.1), 130.92, 131.02, 133.29 (d, $J_{C,F}$ 2.3),137.77, 141.02, 142.53; *m/z* 392 (M⁺), 292, 279 (100%), 165; 4- nonyl-2', 3'-difluoroterphenyl carboxylic acid



Quantities and procedure:

4- nonyl-2', 3'-difluoroterphenylphenyl (6.25 g, 15.94 mmol, 392 g/mol), n-butylithium (7.65 ml, 19.13 mmol in hexanes) and THF (150 ml).

The experimental procedure was as described for the preparation of compound 4-(pentyl, heptyl)-2", 3"-difluoro terphenyl carboxylic acid.

Yield (6.2 g, 89.08%),M.P. 255 °C(Dec); **Elemental analysis:** calc. for (Found: C, H, N, $C_{28}H_{30}F_2O_2$ requires C,77.04; H, 6.93%); δ_H (400 MHz; CDCl₃) 0.88 (3H, t, J 7.1, CH₃CH₂),1.24–1.34 (6H, m), 1.65 (2H, quint, J 7.5, ArCH₂CH₂), 2.66(2H, t, J 7.9, ArCH₂), 7.29 (2H, d, J 8.1),7.32(1H, ddd, J 8.2, 6.6, 1.8), 7.57 (2H, d, J 8.2),7.65(2H, dd, J 7.7, 1.5),7.71 (2H,d,J 8.6), 7.79 (1H, ddd, J 8.4, 6.6, 1.8); (δ_F (376; CDCl₃)) –135.4(1F, dd, J_{F,F} 18.5, J_{F,H} 6.9), –142.66 (1F, dd, J_{F,F} 20.81, J_{F,H} 6.9); δ_C (100.5; CDCl₃) 14.09, 22.57, 29.23 (d, J 2.3), 29.43 (d, J 3.1), 31.41, 31.79, 35.53, 119.81 (d, J7.7), 124.08 (dd, J_{C,F} 4.6, 2.3), 126.4 4(dd, J_{C,F} 4.6, 3.1), 126.82, 127.09, 128.95, 129.23 (d J_{C,F} 3.1), 132.35, 137.33, 141.5, 142.64, 165.05; *m/z* 436 (M⁺), 392, 323 (100%), 279, 44;

4-nonyl-2'-3'-difluoroterphenyl carbonyl chloride



Quantities and procedure:

4-nonyl-2'-3'-difluoroterphenyl carboxylic acid (3 g, 6.86 mmol, 437 g/mol), oxyl chloride (1.05 ml, 12.18 mmol, 126 g/mol), DCM (30 ml) and catalyst DMF (one drop).The experimental procedure as described by compound 4-(pentyl, heptyl)-2", 3"-difluoro terphenyl carbonyl chloride.

4-nonyl-2'-3'-difluoroterphenyl carbonyl amide



Quantities and procedure:

4-nonyl-2'-3'-difluoroterphenyl carbonyl chloride (3 g, 6.59 mmol, 455 g/mol), S-(-)-lactamide (0.8 g, 8.99 mmol, 89 g/mol), pyridine (8.4 ml, 106.33 mmol, 79 g/mol) and dry THF (60 ml).The experimental procedure as described by compound 4-(pentyl, heptyl) -2", 3"-difluoro terphenyl carbonyl amide.

Yield (3.15 g, 94%); M. P 193.60 °C (85.43 J/g) (from ethyl acetate); Transitions temperature /°C K 182.9 (94.19 J/g) SmX 190.3 (97.52 J/g) I; **Elemental analysis:** calc. for (Found: C; H; N. C₃₁H₃₅F₂NO₃ requires C, 73.35; H, 6.95; N, 2.76%); δ_{H} (400 MHz; CDCl₃) 0.87 (3H,tJ 7.1,C**H**₃CH₂), 1.24–1.34 (13H, m), 1.65 (2H, quint, J 8.1, ArCH₂C**H**₂), 2.63 (2H, t, J 8.1, ArCH₂), 5.19 (1H, q, J 6.8), 7.29 (2H, d, J 8.2), 7.32 (1H, ddd, J 8.2, 6.6, 1.8), 7.62 (2H, d, J8.2), 7.7 (2H, dd, J 8.2, 1.3), 7.79 (2H, d, J8.6), 7.86 (1H, ddd, J 8.5, 6.8, 1.8); (δ_{F} (376; CDCl₃)) – 136.03 (1F, dd, J_{F.F}20.81, J_{F.H}6.9), – 143.52 (1F, dd, J_{F.F} 25.43, J_{F.H} 4.6); δ_{C} (100.5; CDCl₃) 13.78, 17.40, 22.01, 28.61, 28.84 (d, J6.9), 30.88, 31.19, 34.10, 78.87, 116.85 (d, J6.9), 117.17, 124.50 (dd, J_{C.F} 4.6, 2.3), 126.36 (dd, J_{C.F} 4.6, 3.1), 126.94, 127.30,

4- pentyl-2",3" difluoro terphenyl nitrile in HM1 Spontaneous plorisation Vs Reduced Temperature



4- pentyl-2",3" difluoro terphenyl nitrile in HM1 Spontaneous plorisation Vs Reduced Temperature



4-pentyl-2",3" difluoro terphenyl nitrile in HM1 Spontaneous plorisation Vs Reduced Temperature



3 % 4-Alkyl-2', 3' diflouoroterphenyl nitrile in HM1







4- heptyl-2",3" difluoro terphenyl nitrile in HM1 Viscosity Vs Reduced Temperature



4- heptyl-2",3" difluoro terphenyl nitrile in HM1 Viscosity Vs Reduced Temperature







7 % 4-Alkyl-2',3' difluoroterphenyl nitrile in HM1



Reduced temperature Tc-T °C

7 % 4-Alkyl-2', 3' diflouoroterphenyl nitrile



Fig. 3. Variation of polarisation and viscosity with T_c-T °C of mixture of 4-(pentyl, heptyl, nonyl)-2'', 3'' difluoro terphenyl nitrile in HM1.

128.98, 129.27 (d $J_{\rm C,F}$ 3.1), 131.92, 136.13, 136.24, 137.38, 142.09, 142.85, 152.78, 161.67, 171.45;

Quantities, procedure, yield and elemental analysis of **4-nonyl-2'-3'-difluoroterphenyl carbonyl nitrile** can be found in some other paper [3].

3. Results and discussions

Transition temperatures of helical phases of 4-Alkyl-2", 3" difluoro terphenyl nitrile and all intermediates (which has liquid crystalline properties) were studied by optical polarising microscope and further confirmed by Differential Scanning Calorimeter. This mesomorphic behaviour is illustrated in Table 1.

From Table 1 it is clear that as the length of the alkyl chain to the nitriles increases, the melting point decreases. Increase in alkyl chain length makes nitrile unstable. All nitriles are smectic materials exhibiting SmC*phase at high temperature. 4- nonyl-2'', 3'' difluoro terphenyl nitrile has shown some unknown smectic phases when it was cooled from isotropic phase to smectic phases.

Liquid crystal phases were viewed through optical polarising microscope and the results are given in Fig. 1 (a–d). Fig. 1(a) shows Isotropic phase to nematic phase transition of 3% 4- heptyl-2", 3" difluoro terphenyl nitrile in achiral host mixture HM1 in which shearing induces homogeneous alignment and produces birefringent, Fig. 1(b) shows 14% D5R in HM1 unwinding of the helical texture of the N* phase. In the dark areas, the homeotropic texture of the nematic phase predominates whereas in the birefringent fingerprint region, remnants of the N* phase remain, Fig. 1(c) shows focal-conic texture of SmA* phase of 7% D9R in HM1. Fig. 1 (d) exhibit schlieren texture of the SmC* phase of mixture of 7% D9R in HM1. The scheliring texture of nematic phase and Smectic phase of 3% D5R in pyrimidine (LM3) mixture is visible in Fig. 1(e) and 1(f) respectively.

Differential scanning calorimetry (DSC) is a complementary tool to optical microscopy and reveals liquid crystal phase by detecting the enthalpy change associated with phase transition.

Liquid crystal, whose phase transition is to be evaluated, heated and cooled four times i.e., first heating, first cooling, second heating, and second cooling. This is to ensure liquid crystal phase transition temperatures. Figs. 2 shows some DSC curves obtained for mixtures prepared from 3% and 7% 4-Alkyl-2^{''}, 3^{''}-difluoroterphenyl nitrile in HM1 and one curve for 14% 4-pentyl-2^{''}, 3^{''}-difluoroterphenyl nitrile.

3.1. LM3 achiral host mixture

So far achiral host mixture HM1 was mixed with 4-Alkyl-2", 3"-difluoroterphenyl nitrile but to study effect of host mixture 3% and 7% of

3 % 4- Alkyl-2",3" difluoro terphenyl nitrile in HM1

20

Reduced Temperature Tc-T°C

40



Phase Transitions of 4-pentyl-2", 3"-difluoroterphenyl nitrile in Host Mixture LM3:-



3% chiral dopant Melting point:- 11.68 (47.16 J/g) SmC 60.8 (.444 J/g) SmA 77 N 79.8 (13.07 J/g)

4% chiral dopant Melting point: - 10.54 (49.53 J/g) SmC 48.8 $(301.26 \times 10^{-9} \text{ J/g})$ SmA 80.2 N 81.3 (14.64 J/g)

After creating achiral host mixture, the chiral dopant D5R was added in small quantities (3%, 7%) to the achiral host mixture in order to generate ferroelectric formulations. The transition temperatures/°C of the formulations and spontaneous polarisation (Ps 7.9 nC/cm^{-2}), viscosity (η 18.3 mpa-section), tilt angles (° 21) and dielectric constant (5.5) were measured as a function of temperature. In pyrimidine mixture LM3 pure and mixture with dopant (3% and 7% of D5R), the SmC* phase has transition of 2nd order. The nematic phase and the SmA phase lie very close to each other especially in the 7% mixture of D5R and HM1. From the isotropic liquid the nematic phase appears in droplets which coalance into fingerprint texture in 3% mixture of D5R while in 7% mixture of D5R the nematic droplets goes directly to the focal-conic texture of SmA. In SmA* phase both D5R mixture have focal-conic and homeo-tropic texture but in 3% mixture, the homeotropic texture dominates the focal-conic texture. In SmC* phase the 3% D5R in LM3 and 7% D5R in LM3 have schlieren texture and transition to SmC* is of 2nd order.

The Instec automatic liquid crystal tester was used to measure the ferroelectric liquid crystal properties of various mixtures prepared during this study. 3% and 7% of 4- Alkyl-2'', 3'' difluoro terphenyl nitrile was mixed with achiral host mixture HM1. Initially 14% of 4- pentyl-2'', 3'' difluoro terphenyl nitrile were mixed in HM1 but mixture did not possess ferroelectric properties. So no more 14% chiral dopants were mixed with HM1. Fig. 3 shows spontaneous polarisation and viscosity versus reduced temperature of various ferroelectric mixtures which contain 3% or 7% 4- Alkyl-2'', 3'' difluoro terphenyl nitrile in achiral host mixture HM1. The spontaneous polarisation increases as the alkyl chain length attached to the chiral dopant increases with the increase of the percentage of the chiral dopant. Viscosity also shows same trend.

Fig. 4 shows variation of dielectric constant with the reduced temperature of the FLC mixture. It shows linear behaviour. Increase in the alkyl chain length also increases dielectric constant.

Dielectric constant Vs Reduced Temperature 6 Dielectric Constant E 5.8 6.5 5.6 ۶D5B 5.4 6 5.2 ۶D7B 5 5.5 εD9R 4.8 5 4.6 4.4 45 4.2 4

60





Fig. 4. Variation of Dielectric constant with Tc-T °C of mixture of 4-(pentyl, heptyl, nonyl)-2", 3" difluoro terphenyl nitrile in HM1.



Fig. 5. Switching state of 4-Alkyl-2", 3"-difluoroterphenyl nitrile in HM1.

In mixture of achiral host material doped with 3% and 7% chiral dopant switching between two states can be produced. These mixtures are used in devices operating in the τV_{min} switching mode.

Fig. 5 shows photo micrographs of switched states of D5R and D9R in HM1 in a cell of spacing 5 μ m with an applied electric field 10 V, 25 V and 30 V.



Fig. 6. Comparison of spontaneous polarisation and viscosity of D5R in HM1 and LM3.

Fig. 6 shows comparison of ferroelectric properties of D5R in achiral host mixture HM1 and LM3. It is evident that mixture in LM3 has greater value of spontaneous polarisation, viscosity and dielectric constant than HM1. It means change in achiral host mixtures has tremendous effect on the FLC properties of materials even same chiral dopant is used.

4. Conclusions

4-Alkyl-2", 3" difluoro terphenyl nitrile are successfully synthesised. Melting point of dopants decreased as the length of alkyl chain to dopant increased. The spontaneous polarisation and viscosity increase as the alkyl chain length attached to the chiral dopant increases and % of chiral dopant in HM1 increases. Change in achiral host mixture can tremendously change FLC properties of material [7].

Abbreviations

- D5R 4-pentyl-2", 3" difluoro terphenyl nitrile
- D 7R 4-heptyl-2", 3" difluoro terphenyl nitrile
- D 9R 4-nonyl-2", 3" difluoro terphenyl nitrile
- DME 1, 2-dimethoxyethane.
- DMF N, N-Dimethylformamide.
- THF Tetra hydrofuran.
- DCM Dichloromethane.

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