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Synthesis of novel liquid crystals having the perfluoropyrrolidino group

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Novel liquid crystals which have the perfluoropyrrolidino group, a heterocyclic perfluoroalkyl group, and which show smectic phases have been synthesized with perfluoro-(2-pyrrolidinopropionyl fluoride) as one of the starting materials. Analogues with a polymerizable functional group also have been prepared and polymerized to give side chain liquid crystal polymers.

Introduction of fluoro-substituents to mesogens often affords new series of liquid crystals with modified physical properties [1]. Fluorinated ferroelectric liquid crystals have recently attracted considerable interest because they have large spontaneous polarization and low viscosity, which can improve the response time of the liquid crystal for switching [2-8].

Although a number of liquid crystals that have fluorinated aromatic rings [9-15], or perfluoro-*n*-alkyl groups [16-24], have been synthesized, no liquid crystals with a bulky cyclic perfluoroalkyl group have yet been prepared. Perfluorocarboxylic acids with a cyclic dialkylamino group, such as the pyrrolidine ring, can be used as the source of such a bulky perfluoroalkyl group. We report upon the synthesis of several compounds in a new class of rod-shaped liquid crystals that have a cyclic perfluoroalkyl group on one end and an ordinary *n*-alkyl tail on the other using perfluoro-(2-pyrrolidinopropionyl fluoride) as one of the starting materials. The phase transition behaviour of these liquid crystals was also investigated. Analogues that had an acryloyl or methacryloyl group at the ω -end of the *n*-alkyl group were also prepared and polymerized yielding side chain liquid crystal polymers.

Compounds possessing an alkyl chain, benzene rings, and a perfluoropyrrolidine ring were prepared as shown in the scheme. Intermediate esters with a phenolic hydroxy group were obtained from perfluoro-(2-pyrrolidinopropionyl fluoride) and excess

amounts of hydroquinone or biphenyldiol. The esters were fairly stable for moisture in comparison with those prepared from perfluoro-(3-pyrrolidinopropionyl fluoride) and hydroquinone that were readily hydrolysed in methanol. They were allowed to react with 4-alkoxybenzoic acid yielding final compounds of rod-shaped molecules. The phase transition temperatures of the compounds were measured by differential scanning calorimetry (see table 1). All the compounds showed the liquid crystalline S_C phase exhibiting fan-shaped textures through a polarizing microscope. In the series of compounds with two benzene rings, the temperature of the phase transition from smectic to isotropic tended to decrease with the increase in alkyl chain length. Hex-

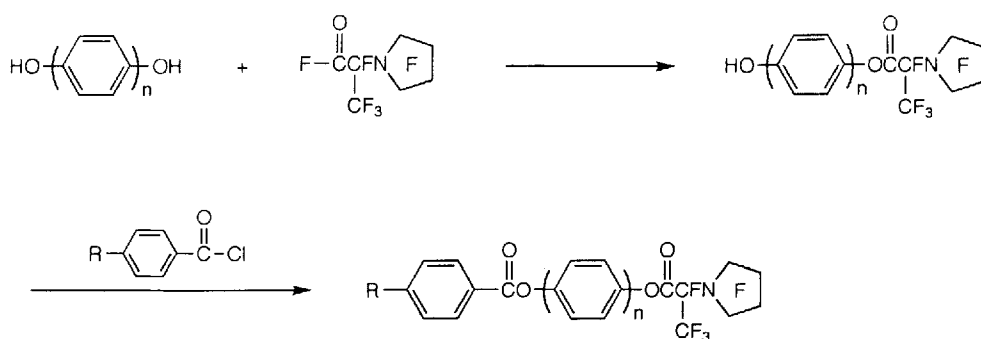
Table 1. Phase transition temperatures of the perfluoropyrrolidine LCs^a.

Compound	Phase transition/°C ^b				
Bu-Hq-Py	Cr	74 (42)	S _C	111 (109)	I
Hex-Hq-Py	Cr	45 (10)	S _C	82 (82)	I
Oct-Hq-Py	Cr	49 (29)	S _C	64 (64)	I
Hex-Bp-Py	Cr	105 (66)	S _C	186 (186)	I

^a Measured by DSC at heating and cooling rates of 5°C min⁻¹; Cr, crystalline; S, smectic; I, isotropic.

^b Values of the second heating. Values in parentheses are of the first cooling.

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code	R	n
Bu-Hq-Py	C ₄ H ₉ O	1
Hex-Hq-Py	C ₆ H ₁₃ O	1
Oct-Hq-Py	C ₈ H ₁₇ O	1
Hex-Bp-Py	C ₆ H ₁₃ O	2
Aa-Hq-Py	CH ₂ =CHCO ₂ C ₆ H ₁₂ O	1
Aa-Bp-Py	CH ₂ =CHCO ₂ C ₆ H ₁₂ O	2
Ma-Hq-Py	CH ₂ =C(CH ₃)CO ₂ C ₆ H ₁₂ O	1
Ma-Bp-Py	CH ₂ =C(CH ₃)CO ₂ C ₆ H ₁₂ O	2

Scheme 1.

Hq-Py gave the most stable smectic phase. Hex-Bp-Py, a compound with three benzene rings, showed transitions at high temperatures, and its smectic phase existed over a wider temperature range than that of Hex-Hq-Py.

Compounds prepared as monomers that had an acryloyl or methacryloyl group in the ω -position of the n -alkyl group did not possess a liquid crystalline phase. These monomers were radically polymerized to yield white powdery polymers that had mesogenic groups in the side chains with a terminal perfluoropyrrolidine ring and a hexyloxy spacer. Polymerization results are shown in table 2. Poly(Aa-Bp-Py) and poly(Ma-Bp-Py), with mesogenic groups consisting of three benzene rings, possessed mesomorphic phases, whereas poly(Aa-Hq-Py) and poly(Ma-Hq-Py) exhibited only a glass tran-

sition (see table 3). Broken fan-shaped textures were seen in the smectic phases of poly(Aa-Bp-Py) and poly(Ma-Bp-Py).

Preparative liquid chromatography showed that the racemic liquid crystals obtained could be optically resolved. Further investigations are in progress.

The materials used in this study were prepared as follows:

4-(Perfluoro-2-pyrrolidinopropionyloxy)phenol (Hq-Py). Perfluoro-2-pyrrolidinopropionyl fluoride (5.00 g, 1.38×10^{-2} mol) [25] was added dropwise to a tetrahydrofuran solution (10 ml) of hydroquinone (1.67 g, 1.52×10^{-2} mol) and pyridine (1.5 ml) at 0°C under an argon atmosphere. The mixture was stirred for 0.5 h at room temperature, the solvent and excess pyr-

Table 2. Radical polymerization of methacrylates carrying mesogens with a perfluoropyrrolidine ring^a.

Monomer/g	AIBN/mol %	THF/ml	Time/h	Yield ^b /g	$\overline{M}_n^c/10^4$	$\overline{M}_w/\overline{M}_n$
Aa-Hq-Py 0.140	1.0	0.40	48	0.113	0.99	1.3
Aa-Bp-Py 0.300	1.0	0.80	48	0.240	1.1	1.4
Ma-Hq-Py 1.00	0.50	1.0	24	0.820	11	1.9
Ma-Bp-Py 0.150	1.0	0.40	48	0.130	8.3	2.2

^a Polymerizations were carried out at 60°C *in vacuo*.

^b Methanol-insoluble fraction.

^c Determined by GPC.

Table 3. Phase transitions of polymeric acrylates and methacrylates carrying mesogens with a perfluoropyrrolidine ring^a.

Compound ($\overline{M}_n \times 10^{-4}$)	Transition scheme/ $^{\circ}\text{C}^b$						
poly(Ma-Hq-Py) (11)	G				95		I
poly(Aa-Hq-Py) (0.99)	G				60		I
poly(Ma-Bp-Py) (8.3)	G	167	S _C		196	S _A	209
poly(Aa-Bp-Py) (1.1)	G	58	S _C		148	S _A	169

^a Measured by DSC at a heating rate of $5^{\circ}\text{C min}^{-1}$; S, smectic; I, isotropic; G, glassy.

^b Values in the second heating.

idine were evaporated under reduced pressure. The product was isolated by column chromatography and recrystallized from hexane (3.43 g, 55 per cent yield). IR (KBr), 3340 cm^{-1} (O-H), 1790 (C=O) ; $^1\text{H NMR}$ (CDCl_3), 7.04 ppm (d, 2H, aromatic H, $J=9.2\text{ Hz}$), 6.87 (d, 2H, aromatic H, $J=9.2$), 4.94 (broad s, 1H, OH); $^{19}\text{F NMR}$ (CDCl_3), -75.5 ppm (m, 3F), $-87.6\text{ (AB m, 2F, } J=174\text{ Hz)}$, $-93.1\text{ (AB m, 2F, } J=174)$, $-131.4\text{ (AB m, 2F, } J=251)$, $-135.8\text{ (AB m, 2F, } J=251)$, -137.3 (m, 1F) ; m.p. 76°C .

The $^{19}\text{F NMR}$ spectra of the following compounds were the same as the spectrum for Hq-Py within experimental error.

4'-(Perfluoro-2-pyrrolidinopropionyloxy)-4-biphenylol (Bp-Py). Perfluoro-2-pyrrolidinopropionyl fluoride ($5.00\text{ g, } 1.38 \times 10^{-2}\text{ mol}$) was added dropwise to a tetrahydrofuran solution (30 ml) of 4,4'-biphenyldiol ($7.71\text{ g, } 4.14 \times 10^{-2}\text{ mol}$) and pyridine (1.5 ml) at 0°C under an argon atmosphere. The mixture was stirred for 0.5 h at room temperature, the solvent and excess pyridine were evaporated under reduced pressure. The product was isolated by column chromatography and recrystallized from hexane (3.73 g, 51 per cent yield). IR (KBr), 3560 cm^{-1} (O-H), 1780 (C=O) ; $^1\text{H NMR}$ (CDCl_3), 7.60 ppm (d, 2H, aromatic H, $J=8.8\text{ Hz}$), 7.44 (d, 2H, aromatic H, $J=8.6$), 7.21 (d, 2H, aromatic H, $J=8.8$), 6.91 (d, 2H, aromatic H, $J=8.6$), 4.90 (broad s, 1H, OH); m.p. 129°C .

4-(4-Hexyloxybenzoyloxy)phenyl perfluoro-2-pyrrolidinopropionate (Hex-Hq-Py). Thionyl chloride (2.8 g, $2.4 \times 10^{-2}\text{ mol}$) and a catalytic amount of *N,N*-dimethylformamide were added to a solution of 4-hexyloxybenzoic acid (1.08 g, $4.86 \times 10^{-3}\text{ mol}$) in tetrahydrofuran (10 ml), after which the mixture was refluxed and stirred for 1 h at room temperature under an argon atmosphere. Evaporation of the solvent and excess thionyl chloride under reduced pressure afforded the acid chloride as a viscous liquid. The acid chloride was dissolved in hexane (10 ml), and the resulting solution added dropwise to Hq-Py (2.00 g, $4.43 \times 10^{-3}\text{ mol}$) and pyridine (0.5 ml) in ether (10 ml) at 0°C . After the mixture was stirred for 0.5 h at room temperature, the solvent and excess pyridine were evaporated under

reduced pressure. The product was isolated by column chromatography and recrystallized from methanol (2.30 g, 79 per cent yield). IR (KBr), 2930 cm^{-1} (C-H), $1785, 1735\text{ (C=O)}$, 1065 (C-O-C) ; $^1\text{H NMR}$ (CDCl_3), 8.13 ppm (d, 2H, aromatic H, $J=9.1\text{ Hz}$), 7.26 (m, 4H, aromatic H), 6.98 (d, 2H, aromatic H, $J=9.1$), 4.05 (t, 2H, OCH_2 , $J=6.4$), 2.00–1.15 (m, 8H, $(\text{CH}_2)_4$), 0.92 (t, 3H, CH_3 , $J=5.9$).

The next three compounds were prepared by a procedure similar to that described above.

4-(4-Butoxybenzoyloxy)phenyl perfluoro-2-pyrrolidinopropionate (Bu-Hq-Py). Yield 45 per cent; IR (KBr), 2950 cm^{-1} (C-H), $1785, 1735\text{ (C=O)}$, 1065 (C-O-C) ; $^1\text{H NMR}$ (CDCl_3) 8.13 ppm (d, 2H, aromatic H, $J=8.8\text{ Hz}$), 7.26 (m, 4H, aromatic H), 6.97 (d, 2H, aromatic H, $J=8.8$), 4.06 (t, 2H, OCH_2 , $J=6.1$), 2.07–1.20 (m, 4H, $(\text{CH}_2)_2$), 0.99 (t, 3H, CH_3 , $J=5.7$).

4-(4-Octyloxybenzoyloxy)phenyl perfluoro-2-pyrrolidinopropionate (Oct-Hq-Py). Yield 69 per cent; IR (KBr), 2920 cm^{-1} (C-H), $1790, 1735\text{ (C=O)}$, 1070 (C-O-C) ; $^1\text{H NMR}$ (CDCl_3) 8.13 ppm (d, 2H, aromatic H, $J=9.0\text{ Hz}$), 7.26 (m, 4H, aromatic H), 6.97 (d, 2H, aromatic H, $J=9.0$), 4.05 (t, 2H, OCH_2 , $J=6.3$), 2.05–1.10 (m, 12H, $(\text{CH}_2)_6$), 0.89 (t, 3H, CH_3 , $J=5.7$).

4-(4-Hexyloxybenzoyloxy)-4-biphenyl perfluoro-2-pyrrolidinopropionate (Hex-Bp-Py). Yield 48 per cent; IR (KBr), 2950 cm^{-1} (C-H), $1810, 1735\text{ (C=O)}$; $^1\text{H NMR}$ (CDCl_3), 8.16 ppm (d, 2H, aromatic H, $J=8.9\text{ Hz}$), 7.64 (d, 2H, aromatic H, $J=8.8$), 7.59 (d, 2H, aromatic H, $J=8.8$), 7.30 (d, 2H, aromatic H, $J=8.8$), 7.26 (d, 2H, aromatic H, $J=8.8$), 6.98 (d, 2H, aromatic H, $J=8.9$), 4.05 (t, 2H, OCH_2 , $J=6.3$), 2.07–1.16 (m, 8H, $(\text{CH}_2)_4$), 0.92 (t, 3H, CH_3 , $J=5.9$).

4-[4-(6-Methacryloyloxyhexyloxy)benzoyloxy]phenyl perfluoro-2-pyrrolidinopropionate (Ma-Hq-Py). Thionyl chloride (9.0 g, $7.6 \times 10^{-2}\text{ mol}$) and a catalytic amount of *N,N*-dimethylformamide were added to a mixture of 4-(6-methacryloyloxyhexyloxy)benzoic acid ($2.23\text{ g, } 7.28 \times 10^{-3}\text{ mol}$) [26, 27] and a trace of 2,6-di-*tert*-butyl-*p*-cresol, after which the mixture was stirred for 1 h at room temperature under an argon atmosphere. Evaporation of the solvent and excess thionyl chloride under reduced pressure afforded the acid chloride as a

viscous liquid. The acid chloride was dissolved in hexane (22 ml), and the resulting solution added dropwise to Hq-Py (3.00 g, 6.65×10^{-3} mol) and pyridine (1.0 ml) in ether (15 ml) at 0°C. After the mixture was stirred for 0.5 h at room temperature, the solvent and excess pyridine were evaporated under reduced pressure. The product was isolated by column chromatography and recrystallized from methanol (3.00 g, 60 per cent yield). IR (KBr) 2950 cm^{-1} (C-H), 1790, 1730, 1710 (C=O), 1645 (C=C); $^1\text{H NMR}$ (CDCl_3) 8.11 ppm (d, 2 H, aromatic H, $J=8.9$ Hz), 7.24 (m, 4 H, aromatic H), 6.96 (d, 2 H, aromatic H, $J=8.9$), 6.08 (s, 1 H, $\text{CH}_2=$), 5.53 (s, 1 H, $\text{CH}_2=$), 4.16 (t, 2 H, CO_2CH_2 , $J=6.4$), 4.04 (t, 2 H, OCH_2 , $J=5.8$), 1.93 (m, 3 H, CH_3), 2.08–1.31 (m, 8 H, $(\text{CH}_2)_4$); m.p. 54°C.

The following compounds were prepared by a procedure similar to that described above.

4'-[4-(6-Methacryloyloxyhexyloxy)benzoyloxy]-4-biphenyl perfluoro-2-pyrrolidinopropionate (Ma-Bp-Py). Yield 41 per cent; IR (KBr), 2940 cm^{-1} (C-H), 1790, 1710 (C=O), 1640 (C=C); $^1\text{H NMR}$ (CDCl_3), 8.16 ppm (d, 2 H, aromatic H, $J=8.9$ Hz), 7.65 (d, 2 H, aromatic H, $J=8.6$), 7.60 (d, 2 H, aromatic H, $J=8.6$), 7.30 (d, 2 H, aromatic H, $J=8.6$), 7.26 (d, 2 H, aromatic H, $J=8.9$), 6.10 (s, 1 H, $\text{CH}_2=$), 5.55 (s, 1 H, $\text{CH}_2=$), 4.17 (t, 2 H, CO_2CH_2 , $J=6.5$), 4.06 (t, 2 H, OCH_2 , $J=6.1$), 1.95 (m, 3 H, CH_3), 2.04–1.34 (m, 8 H, $(\text{CH}_2)_4$); m.p. 83°C.

4-[4-(6-Acryloyloxyhexyloxy)benzoyloxy]phenyl perfluoro-2-pyrrolidinopropionate (Aa-Hq-Py). Yield 56 per cent; IR (KBr), 2930 cm^{-1} (C-H), 1790, 1725 (C=O); $^1\text{H NMR}$ (CDCl_3), 8.13 ppm (d, 2 H, aromatic H, $J=9.0$ Hz), 7.25 (m, 4 H, aromatic H), 6.97 (d, 2 H, aromatic H, $J=9.0$), 6.40 (dd, 1 H, $\text{CH}_2=$, $J_1=17.4$, $J_2=1.5$), 6.12 (dd, 1 H, $\text{CH}_2=$, $J_1=17.4$, $J_2=10.5$), 5.81 (dd, 1 H, $=\text{CH}-$, $J_1=10.5$, $J_2=1.5$), 4.18 (t, 2 H, CO_2CH_2 , $J=6.4$), 4.06 (t, 2 H, OCH_2 , $J=6.2$), 2.20–1.30 (m, 8 H, $(\text{CH}_2)_4$); m.p. 47°C.

4'-[4-(6-Acryloyloxyhexyloxy)benzoyloxy]-4-biphenyl perfluoro-2-pyrrolidinopropionate (Aa-Bp-Py). Yield 64 per cent IR (KBr), 2945 cm^{-1} (C-H), 1795, 1725 (C=O); $^1\text{H NMR}$ (CDCl_3), 8.16 ppm (d, 2 H, aromatic H, $J=8.8$ Hz), 7.64 (d, 2 H, aromatic H, $J=8.5$), 7.60 (d, 2 H, aromatic H, $J=8.6$), 7.30 (d, 2 H, aromatic H, $J=8.5$), 7.25 (d, 2 H, aromatic H, $J=8.6$), 6.98 (d, 2 H, aromatic H, $J=8.8$), 6.40 (dd, 1 H, $\text{CH}_2=$, $J_1=17.4$, $J_2=1.4$), 6.13 (dd, 1 H, $\text{CH}_2=$, $J_1=17.4$, $J_2=10.6$), 5.81 (dd, 1 H, $=\text{CH}-$, $J_1=10.6$, $J_2=1.4$), 4.19 (t, 2 H, CO_2CH_2 , $J=6.3$), 4.06 (t, 2 H, OCH_2 , $J=6.3$), 2.20–1.30 (m, 8 H, $(\text{CH}_2)_4$); m.p. 80°C.

Polymerization was performed as follows. An acrylic or methacrylic monomer, 2,2'-azobis(isobutyronitrile) as the radical initiator and tetrahydrofuran as the solvent were placed in an ampoule. Then the mixture

was degassed by successive freeze-thaw cycles. Polymerization was carried out at 60°C *in vacuo*. After polymerization, the mixture was diluted with tetrahydrofuran and poured into methanol. The precipitated polymer was collected and dried *in vacuo* then purified by reprecipitation from methanol.

IR spectra were obtained with a JASCO IR-810 infrared spectrophotometer. $^1\text{H NMR}$ (90 MHz) and $^{19}\text{F NMR}$ (84.68 MHz) measurements were recorded on a Hitachi R-90F, chemical shifts being defined as their respective δ values relative to TMS and CFCl_3 . Molecular weights of the polymers were correlated to standard polystyrenes using Toso GMH6 columns with tetrahydrofuran as the eluent. Differential scanning calorimetry was performed with a Perkin-Elmer DSC7 at heating and cooling rates of 5°C. The textures of the liquid crystals were observed with a Nikon SMZ-1B microscope under cross nicols.

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