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PARALLEL SYNTHESIS OF 4-ALKYL-4′-CYANOBIPHENYL LIQUID CRYSTALS

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In the search for novel nematic liquid crystal materials for display applications, it is common to investigate several different alkyl chain homologues of a particular core structure. It is sometimes possible to develop a synthetic pathway that allows homologous variation in the ultimate step, but the majority of published methods for liquid crystal synthesis begin from an alkyl chain containing intermediate. This results in a separate pathway for each homologue.

A "one-pot" technique has been developed for the parallel synthesis of up to five homologues of a given core structure, exemplified by "the 4-alkyl-4′-cyanobiphenyl series, together with their isolation in gram quantities and high purity by preparative high performance liquid chromatography. The technique demonstrates that it is possible to identify and monitor each intermediate throughout the synthetic pathway, using gas chromatography/mass spectrometry. It is also shown that there are no substantive losses in yield for any homologue throughout the pathway. It is concluded that this technique is a viable method for the synthesis of novel liquid crystal materials.

Keywords: chromatography; cyanobiphenyl; Nematic Liquid Crystal Mixtures; parallel synthesis

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INTRODUCTION

Calamitic nematic liquid crystals (LC) of which 4-n-alkyl-4′-cyanobiphenyls are a good example (as shown in Figure 1 below) usually, but not exclusively conform to the general design principle shown in Figure 2 below:-

Many structure-property relationships for designing specific properties into nematic liquid crystals are established and published [1–3]. Variation of the parts of the core group, linking groups and lateral substituents are all ways of tailoring the properties and a systematic variation of the alkyl chain length is known to produce a rich variety of mesophases and property trends [4].

In the search for improved nematic LC's, it is common to investigate several different alkyl chain homologues of a particular core structure. The majority of published methods for liquid crystal synthesis begin from an alkyl chain containing intermediate. This results in a separate pathway for each homologue, which is both time consuming and expensive.

In the last 10 years, the fields of combinatorial chemistry and parallel synthetic methods have developed rapidly and are responsible for many new molecules and materials in many chemical and pharmaceutical areas, such as catalysis [5], natural product synthesis [6], drug discovery [7], agrochemicals [8], fluorescent materials [9] & conjugated electronic polymers [10]

Although it is possible to synthesise many hundreds or thousands of new molecules by the combinatorial approach, materials are commonly only

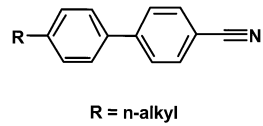

FIGURE 1 4-n-alkyl-4′-cyanobiphenyls.

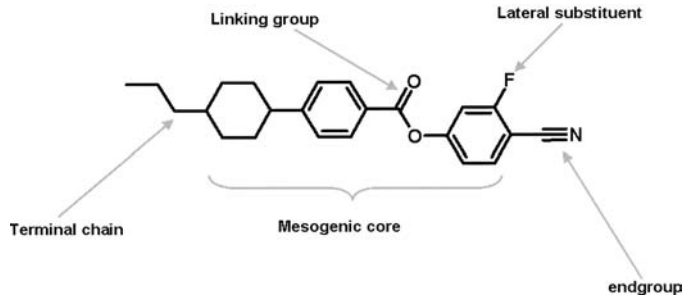

FIGURE 2 Molecular components of a typical calamitic nematic liquid crystal.

prepared in very small amounts and in an impure form. For initial pre-screening and lead generation in drug discovery, such an approach is adequate, but for the examination of a new nematic LC, a gram quantity of material with a chemical purity of 99% + is required. This allows an accurate evaluation of the properties such as the mesophase sequence and transition temperatures, together with extrapolated properties such as birefringence, dielectric anisotropy and rotational viscosity [11].

There are some documented examples of a parallel synthesis approach to produce gram quantities of pure materials, notably dyestuffs [12]. There are also some documented examples of one pot syntheses of LC mixtures of homologues, by groups at BASF [13] & Merck [14], but these concentrate on production of simple mixtures of reactive mesogens for photocurable coating applications. There is also some recently reported work by Bäuerle *et al.* on the combinatorial parallel synthesis of liquid crystal quaterphenyls [15]. In this paper, a "one-pot" technique is described for the parallel synthesis of up to five homologues of a given core structure, exemplified by the 4-alkyl-4′-cyanobiphenyl series, together with their isolation in gram quantities and high purity by preparative high performance liquid chromatography. 4-*n*-alkyl-4′-cyanobiphenyls were chosen as model compounds for the study as they are well known and characterised, and have an established synthetic pathway.

SYNTHESIS

The parallel synthesis was performed as a conventional procedure, according to the route shown in Figure 3, using only one reaction vessel to produce the 5 homologues.

Modifications to the originally reported pathway [4] were made to improve yields, reduce the usage of toxic or harmful materials, and use milder reaction conditions. Purification was performed at each stage, both as a test of the feasibility of using conventional techniques for purifying homologous mixtures and to determine changes in the homologous composition through the pathway. The reported yields are after a purification step.

Friedel-Crafts acylation was performed on biphenyl, according to a standard procedure [16], using an equimolar mixture of n-alkanoyl chlorides. Reduction of the acyl carbonyl to methylene was performed using polymethylhydrosiloxane [17]. Use of this reagent was preferable to the toxic and aggressive conditions used in the Huang-Minlon reaction (diethylene glycol & hydrazine hydrate at elevated temperature). Functionalisation of the biphenyl 4′-position was effected cleanly using an oxidative iodination procedure [18].

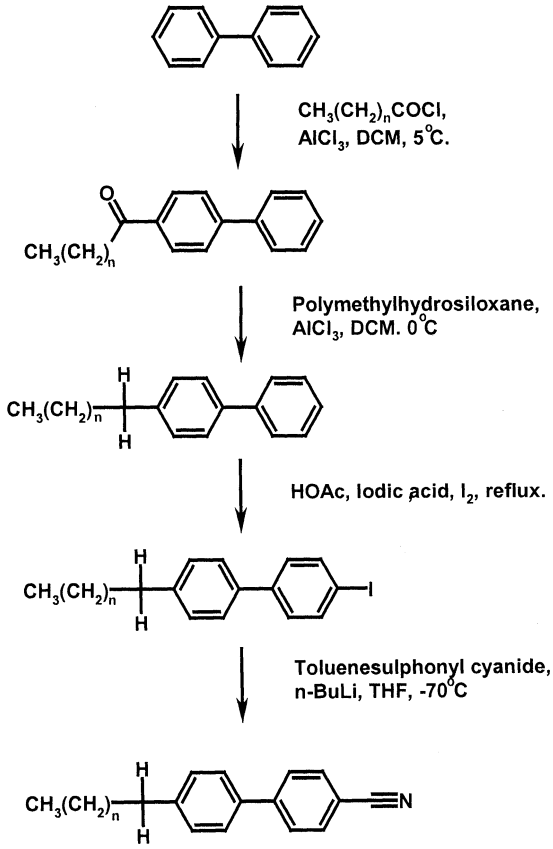

FIGURE 3 Synthetic pathway for 4-n-alkyl-4′-cyanobiphenyls, using a parallel synthetic approach.

The final stage of the synthesis was an iodine – cyanide exchange at deep temperature (−70°C) using n-butylithium to generate the lithium anion and quenching with toluene sulphonyl cyanide [19]. After work up of the final stage, the crude mixture of cyanobiphenyls was a yellow isotropic liquid, indicating that further purification was necessary. After flash chromatography over silica gel, eluting with toluene. The product of this procedure was a clear isotropic liquid, indicating that there were still impurities present which depressed the nematic liquid crystal phase, which would be present if equivalent percentages of the pure and individual compounds were mixed together. As a final stage of the purification, the clear isotropic liquid was Kugelrohr distilled to remove both low boiling impurities and non-volatile impurities. Upon cooling to room temperature,

the distilled product produced a nematic liquid crystal phase at room temperature.

RESULTS AND DISCUSSION

Parallel Synthesis

The parallel synthesis was successful as gram quantities of pure materials were obtained at the end of the experiment. Using HPLC, GC & GC-MS, it was possible at all stages in the synthesis to separate and identify all homologues. Table 1 shows mass and percentage yields, together with combined purities for the target homologues. The individual step yields and cumulative yields through the process are comparable with expected yields for liquid crystal syntheses, but are not optimised and could be improved.

Examination of Chart 1 shows that the different steps in the synthetic pathway show different trends for the percentage yields for each homologue. There are several possible reasons for the trends, which can either be inherent to the reaction mechanism or a function of the purification procedure. In step 3, the oxidative iodination; the trend is for lower yield with shorter chain homologue. The mixture at step 3 was finally purified by recrystallisation. Differential solubility of homologues with greater solubility exhibited by the shorter chain homologues may be responsible for the yield trend. Another example is in step 1, the Friedel Crafts acylation, where the trend is for a lower yield with increasing chain length. As there are no aspects of the work up procedure likely to cause a fractionation of the products, such as recrystallisation or chromatography, it must be presumed that differential reactivity of acid chlorides, decreasing with increasing chain length is responsible. Consideration of intermediate purification procedures is therefore necessary in the design of a "one pot" parallel synthetic pathway.

TABLE 1 Overall Yields from Parallel Synthesis Procedure

Reaction	Mass yield/g	Step yield/%	Overall Yield/%	Purity/% (HPLC) [Based on sum of homologue peaks]
Step 1. Friedel-Crafts acylation	69.98	85	85	88.90
Step 2. PMHS reduction	32.20	55	47	99.83
Step 3. Oxidative iodination	30.10	58	27	98.38
Step 4. Tosylcyanide cyanation	8.0	38	10	99.32

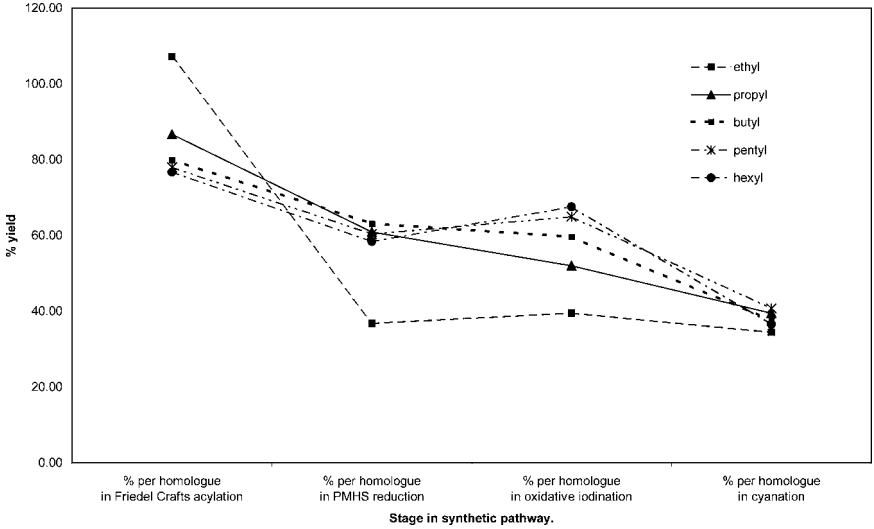

CHART 1 Comparative yields for homologues in parallel synthesis procedure.

Analysis of Intermediate Stages in Parallel Synthetic Pathway

Figures 4–9 show that by using gradient elution methods in HPLC and temperature ramp methods in GC-MS, it is possible to achieve good separation of homologues. Examination of the various chromatograms shows that it is possible to gain the same levels of separation in both polar materials, such as the 4-n-alkyl-4′-cyanobiphenyls and the non-polar materials such as the 4-n-alkyl-4′-biphenyls. Compared to many liquid crystal materials, 4-n-alkyl-4′-cyanobiphenyls have a comparatively low molecular weight.

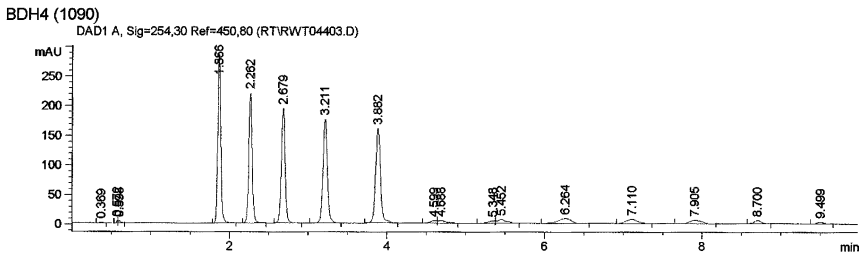

FIGURE 4 HPLC chromatogram of the Friedel Crafts acylation crude product (4-n-alkanoylbiphenyls).

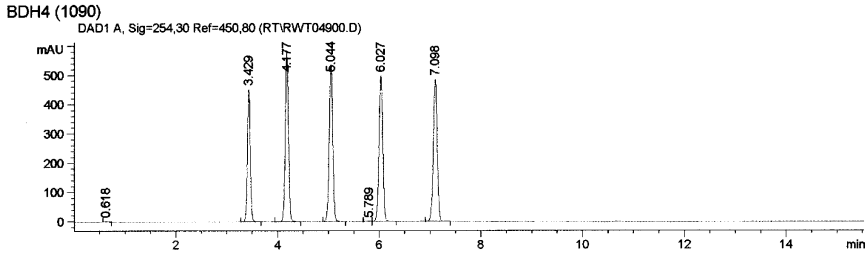

FIGURE 5 HPLC chromatogram of the PMHS reduction crude product. (4-n-alkylbiphenyls).

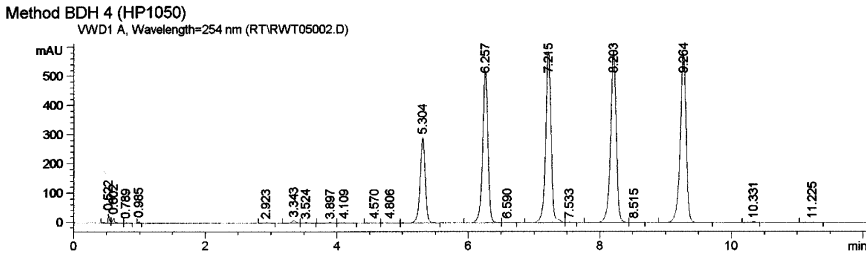

FIGURE 6 HPLC chromatogram of the oxidative iodination recrystallised product. (4-n-alkyl-4′-iodobiphenyls).

For this class of structures, homologous variation makes a substantial difference to the separation characteristics. For homologues of liquid crystal structures with substantially greater molecular weights, homologous variation may not make such a large difference. This is also a consideration when planning a one pot parallel synthesis procedure.

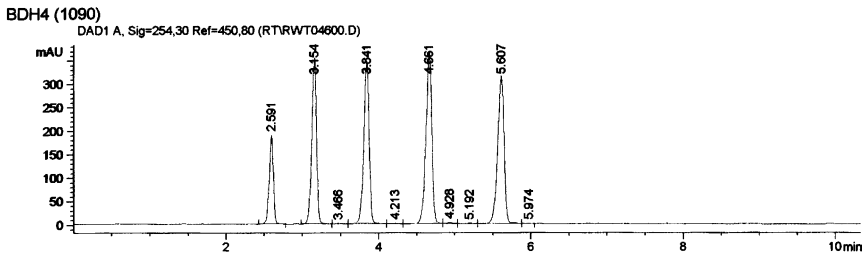

FIGURE 7 HPLC chromatogram of the cyanation final product after distillation. (4-n-alkyl-4′-cyanobiphenyls).

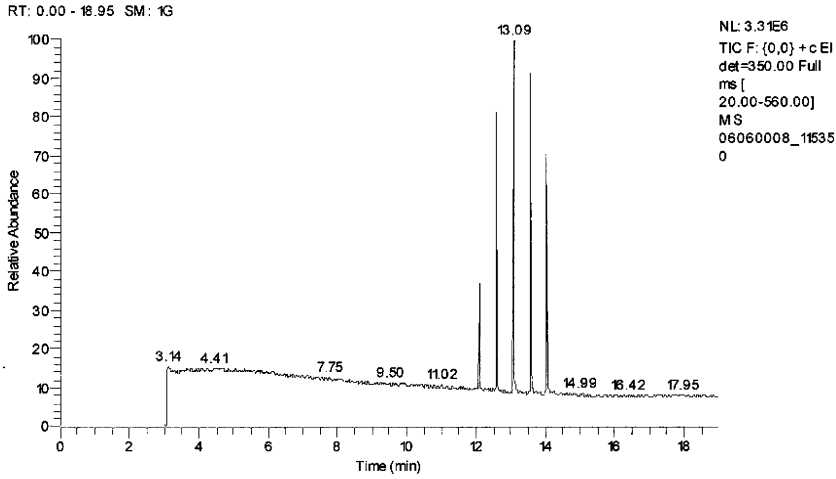

FIGURE 8 GC-MS chromatogram of the oxidative iodination recrystallised product. (4-n-alkyl-4′-iodobiphenyls).

Preparative HPLC Separation

By adaptation of the analytical HPLC method, which used a gradient elution of acetonitrile and water, to a method which used a constant solvent composition (20% water and 80% acetonitrile) a preparative HPLC method was eveloped that allowed the solvent to be recycled in the

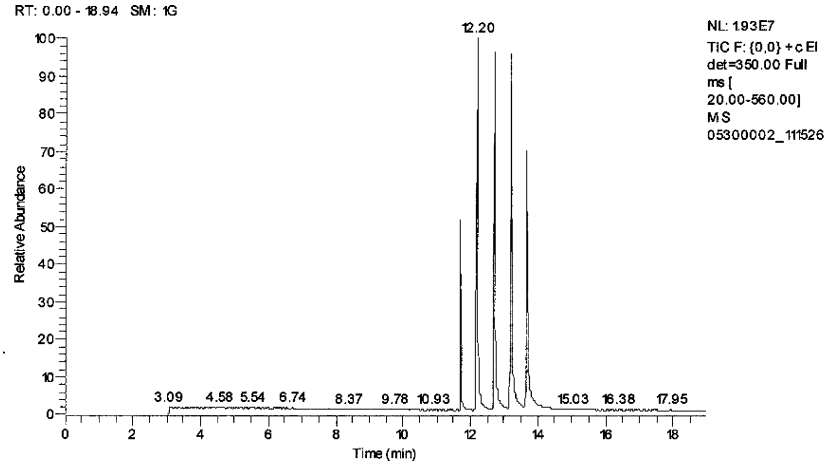

FIGURE 9 GC-MS chromatogram of the cyanation final product after distillation. (4-n-alkyl-4′-cyanobiphenyls).

TABLE 2 Isolated Yields and HPLC Purities for Separated Homologues

Homologue	Amount/g	Purity (HPLC)/%
4-ethyl-4′-cyanobiphenyl	0.30	99.9
4-propyl-4′-cyanobiphenyl	1.03	99.9
4-butyl-4′-cyanobiphenyl	1.09	99.8
4-pentyl-4′-cyanobiphenyl	1.16	99.3
4-hexyl-4′-cyanobiphenyl	1.47	99.7

process. In the analytical HPLC method, the solvent system was not of constant composition because of the gradient elution method, thus preventing it from being recycled in the process. From the data in Table 2, it can be seen that the separation was excellent and that highly pure single substances were isolated. Additionally, the recovery yield for preparative HPLC separation is good, with 7 g of purified homologous mixture being used and 5.05 g of pure single substances returned, a yield of 72%.

Comparison of Parallel Synthesised Pure Materials with Classically Synthesised Pure Materials

As an additional test of the efficiency of the parallel synthesis procedure as a viable method for liquid crystal material synthesis, an examination by DSC was made of the transition temperatures for the materials produced in the parallel synthesis procedure against materials made by conventional synthetic techniques [20]. Liquid crystal transition temperatures are very sensitive to impurities or variations in chemical structure. If the data in Table 3 is examined, it can be seen that there is excellent agreement between the two sets of transition temperatures, showing the authenticity of the parallel synthesised materials.

TABLE 3 Comparison of Liquid Crystal Transition Temperatures for Parallel and Conventionally Synthesised 4-n-alkyl-4′-cyanobiphenyls

Homologue	Transition temperatures /°C for parallel synthesised homologues	Transition temperatures /°C for conventionally synthesised homologues
4-ethyl-4′-cyanobiphenyl	K 76 (N 24) I	K 74 (N 22) I
4-n-propyl-4′-cyanobiphenyl	K 65 I	K 67 I
4-n-butyl-4′-cyanobiphenyl	K 48.2 (N 16) I	K 49 (N 17) I
4-n-pentyl-4′-cyanobiphenyl	K 24 N 35 I	K23 N 35 I
4-n-hexyl-4′-cyanobiphenyl	K 16 N 29 I	K 15 N 29 I

TABLE 4 Compositions of Final Distilled Mixture of 4-n-alkyl-4′-cyanobiphenyls Ex Parallel Synthesis Procedure and Mixture Formulated from Conventionally Synthesised 4-n-alkyl-4′-cyanobiphenyls

Homologue	Composition of distilled parallel synthesis mixture/%	Composition of formulated mixture/%
4-ethyl-4′-cyanobiphenyl	7.46	7.70
4-n-propyl-4′-cyanobiphenyl	18.37	18.56
4-n-butyl-4′-cyanobiphenyl	21.53	21.68
4-n-pentyl-4′-cyanobiphenyl	26.46	26.86
4-n-hexyl-4′-cyanobiphenyl	25.64	25.86

Comparison of One Pot Mixture with Formulated Mixture

As a final test of the efficiency of the parallel synthesis procedure, a mixture of the conventionally synthesised homologues was formulated to match to amounts of each homologue in the parallel synthesised mixture as analysed by HPLC. The nematic to isotropic transition temperature was measured by DSC. Table 4 shows the composition of the mixtures. The nematic to isotropic transition for the distilled parallel synthesis mixture was measured at 26.4 C and at 28.3 C for the conventionally formulated mixture. This is a fairly good agreement and within experimental error.

CONCLUSIONS

It has been shown that it is possible to produce gram quantities of liquid crystal homologues in high purity and reasonable yield, by the use of a one pot parallel synthesis procedure and final step separation by preparative HPLC. It is possible to analyse and identify each homologue at every intermediate stage in the synthetic pathway. A well known and characterised set of homologous materials, with an established chemistry were chosen for the study, in order to maximise the chances of a successful synthesis. In order to evaluate the technique fully as a viable synthetic method, synthesis of homologous series of novel liquid crystal materials should be made.

EXPERIMENTAL TECHNIQUES

Synthesis procedures such as reactions, workups, chromatography and recrystallisations were performed according to Vogel [21]

Thin Layer Chromatography (TLC)

TLC was performed according to Vogel [22] using Merck silica gel 60 F_{254} TLC plates with petrol/ethyl acetate mixtures or Merck RP-18 F_{254} reverse phase TLC plates with acetonitrile/water mixtures. Visualisation of the plates was achieved by UV irradiation and viewing at 254 nm or 366 nm.

Differential Scanning Calorimetry (DSC)

Mesophase transition temperatures were determined by differential scanning calorimetry using a Perkin Elmer DSC-7. Samples were run in a nitrogen atmosphere with a scanning rate of 5°C/min against an empty cell reference.

Fourier Transform Nuclear Magnetic Resonance (FT-NMR)

Proton FT-NMR samples were analysed on a Bruker AC300 300 MHz instrument.

High Performance Liquid Chromatography (HPLC)

Chemical purity of compounds and mixtures by reverse phase HPLC was performed using a Hewlett-Packard 1090 diode-array instrument, fitted with a Merck 250 mm RP-18 5 μm Lichrospher column. Samples were gradient eluted with water/acetonitrile mixtures at 2 ml per minute at 40 C.

Gas Chromatography – Mass Spectrometry (GC-MS)

Mass ions and mass fragmentation patterns of compounds and mixtures were determined using a ThermoQuest trace GC – MS with Excalibur software. The oven temperature was ramped at 10 C per minute from 100 C to 280 C.

Gas Chromatography (GC)

GC analysis of compounds and mixtures was performed on a Perkin-Elmer 8500GC using a flame ionisation detector (FID) with nitrogen carrier gas flow rate of 20 mL/min with a pressure of 10 psi. The injector temperature is 260°C and a detector temperature of 280°C. The oven temperature was ramped at 10 °C per minute from 100 °C to 280 °C.

Preparative High Performance Liquid Chromatography (prep. HPLC)

Prep. HPLC of the final homologous mixture to isolate individual compounds was performed on a Novaprep Instrument in the Central Process Development department of Merck KGaA

The instrument was equipped with a 50 mm bore 300 mm RP-18 5 μm Lichrospher column. Elution was performed with Acetonitrile at 100 ml min at 22°C

Synthetic Procedures

4-n-alkanoylbiphenyls

Aluminium chloride (48.06 g, 0.36 mol) was dissolved in dichloromethane (400 cm^3) under nitrogen. The solution was stirred and cooled to −10°C. Biphenyl (50 g, 0.33 mol) was added and stirred for 30 min. Acetyl chloride (5.11 g, 0.065 mol), propionyl chloride (6.02 g, 0.065 mol), butanoyl chloride (6.93 g, 0.065 mol), pentanoyl chloride (7.84 g, 0.065 mol) and hexanoyl chloride (8.75 g, 0.065 mol) were combined and diluted with dichloromethane (200 cm^3). The mixed acid chloride solution was added drop-wise, maintaining the solution at −10°C. The solution was allowed to warm to room temperature over 18 hours.

The reaction mixture was poured carefully onto ice (1000 cm^3) and the phases were separated. The aqueous phase was washed with dichloromethane (2×100 cm^3) and the combined organic layers were washed with dilute hydrochloric acid (3×300 cm^3), water (2×300 cm^3) and calcium carbonate solution (300 cm^3). The combined organic layers were filtered through Hiflo, dried (Na_2SO_4) and the solvent removed on a rotary evaporator under reduced pressure to yield a yellow solid. Yield: 69.58 g. Analytical data for the homologous series of products is shown below in Table 5.

4-n-alkylbiphenyls

Aluminium chloride (22.03 g, 0.018 mol) was dissolved in dichloromethane (200 cm^3) with stirring under a nitrogen atmosphere. The mixture of

TABLE 5 HPLC, GC & MS Analyses for the Homologous Mixture of 4-n-alkanoylbiphenyls

HPLC elution time	HPLC purity	GC	M/S: m/z
1.87 min	19.65%	11.09 min	196 (M+•), 181, 152, 127, 91, 76, 63, 43
2.26 min	17.03%	11.61 min	210 (M+•), 181, 152, 127, 76, 51
2.69 min	16.76%	12.06 min	224 (M+•), 196, 181, 152, 127, 98, 76, 41
3.21 min	17.37%	12.57 min	238 (M+•), 196, 181, 152, 127, 102, 76, 41
3.88 min	18.09%	13.07 min	252 (M+•), 233, 196, 181, 152, 127, 102, 76, 55

TABLE 6 HPLC, GC & MS Analyses for the Homologous Mixture of 4-n-alkylbiphenyls

HPLC elution time	HPLC purity	GC	M/S: m/z
3.42 min	14.52%	9.80 min	182 (M+•), 166, 164, 151, 114, 76,
4.17 min	20.95%	10.36 min	196 (M+•), 166, 164, 151, 114, 76.
5.04 min	21.45%	10.94 min	210 (M+•), 166, 164, 151, 114, 76.
6.02 min	21.33%	11.48 min	224 (M+•), 166, 164, 151, 114, 76.
7.10 min	21.58%	12.02 min	238 (M+•), 166, 164, 151, 114, 76.

4-n-alkanoylbiphenyls(69.58 g) was dissolved in dichloromethane (400 cm^3) and added drop-wise to the aluminium chloride solution. The reaction mixture was cooled to 0°C then polymethylhydrosiloxane solution (500 cm^3 50% w/w in DCM) was added drop-wise and allowed to stir for 20 hours.

The reaction mixture was poured carefully onto ice (1000 cm^3) and water (1000 cm^3) with stirring. Sodium hydroxide (25%, 1200 cm^3) was added with agitation and left for 18 hours, to effect good separation of phases. The phases were separated and the aqueous layer was washed with dichloromethane (2×300 cm^3). The combined organic layers were washed with sodium hydroxide solution (25%, 2×400 cm^3), water (3×500 cm^3) and 2M hydrochloric acid (300 cm^3). The dichloromethane solution was dried (Na_2SO_4) and the solvent removed on a rotary evaporator under reduced pressure to leave a yellow solid. Yield; 40.1 g.

The crude solid was purified by flash chromatography over silica gel, eluting with 80–100 petrol to yield a colourless solid. Yield: 32.2 g. Analytical data for the homologous series of products is shown above in Table 6.

4-n-alkyl-4′-iodobiphenyls

The purified alkylbiphenyl mixture (33 g) was dissolved in acetic acid (128 cm^3, 2.12 mol) under a nitrogen atmosphere. To the acetic acid solution, water (40.4 cm^3, 2.243 mol), sulphuric acid (11.16 cm^3, 0.11 mol), iodic acid (6.68 g, 0.038 mol), iodine (38.58 g, 0.15 mol) and dichloromethane (27 cm^3) were added and heated to reflux for 20 hours.

The reaction mixture was cooled to room temperature. A solution of 10% w/v sodium metabisulphate (250 cm^3) was added, followed by methyl t-butylether (MTBE) (500 cm^3) and the phases separated. The aqueous phase was washed with MTBE (3×200 cm^3) and the combined organic layers were washed with dilute sodium metabisulphate solution (300 cm^3) water (700 cm^3), sodium hydroxide solution (500 cm^3) and finally water (3×500 cm^3). The crude solution was dried (Na_2SO_4) and the solvent was removed under reduced pressure on a rotary evaporator. The crude

TABLE 7 HPLC, GC & MS Analyses for the Homologous Mixture of 4-n-alkyl-4′-iodobiphenyls

HPLC elution time	HPLC purity	GC	M/S: m/z
5.30 min	10.37%	12.10 min	308 (M+•), 293, 165, 127, 63
6.26 min	19.13%	12.59 min	322 (M+•), 293, 165, 127, 63
7.21 min	21.88%	13.10 min	336 (M+•), 293, 165, 127, 63
8.20 min	23.15%	13.57 min	350 (M+•), 293, 165, 127, 63
9.26 min	23.85%	14.04 min	364 (M+•), 293, 165, 127, 63,

product was recrystallised from 5 volumes of ethanol, to yield a colourless solid. Yield: 30.10 g. Analytical data for the homologous series of products is shown above in Table 7.

4-n-alkyl-4′-cyanobiphenyls

The 4-n-alkyl-4′-iodobiphenyl mixture (30.10 g) was dissolved in tetrahydrofuran (150 cm^3) and cooled to −80°C. n-Butyllithium (58 cm^3, 0.094 mol) was added dropwise over 20 min and stirred at −80°C for 2 hours. Toluene sulphonylcyanide (20.00 g, 0.108 mol) was dissolved in tetrahydrofuran (150 cm^3) and added to the mixture dropwise, maintaining the reaction mixture at −80°C. The solution was then allowed to warm to room temperature slowly over 20 hours. Dilute hydrochloric acid (200 cm^3) was added and the phases separated. The aqueous phase was washed with MTBE (2×200 cm^3) and the combined organic layers were washed with sodium thiosulphate solution (300 cm^3), water (3×300 cm^3) and then dried (Na_2SO_4) and the solvent removed under reduced pressure on a rotary evaporator, to yield a yellow oil. The oil was purified by column chromatography over silica gel eluting with a petrol/DCM mixture to yield a light yellow oil. Yield: 11 g. Final purification of the light yellow oil was accomplished by Kugelrohr distillation. The distillation results are shown in Table 8 below. Fraction 2 produced a room temperature nematic liquid crystal. Yield: 8 g.

TABLE 8 Distillation Conditions for the Homologous Mixture of 4-n-alkyl-4′-cyanobiphenyls

Fraction	Temperature/°C	Pressure/mbar	Amount/g
1	150	0.25	0.5
2	245	0.25	8.0
3	pot	0.25	1.5

TABLE 9 HPLC, GC & MS Analyses for the Homologous Mixture of 4-n-alkyl-4′-cyanobiphenyls

HPLC elution time	HPLC purity	GC	M/S: m/z
2.59 min	9.05%	11.70 min	207 (M + •), 192, 165, 140, 115
3.15 min	19.56%	12.20 min	221 (M + •), 192, 165, 140, 115
3.84 min	21.69%	12.72 min	235 (M + •), 192, 165, 140, 115
4.66 min	25.28%	13.20 min	249 (M + •), 192, 165, 140, 115
5.60 min	23.74%	13.66 min	263 (M + •), 192, 165, 152, 115

4-cyano-4′-ethylbiphenyl
Purity (HPLC) 99.9%, Yield 0.3 g
^{1}H NMR: (CDCl3) δ 1.4 (3H t), 2.8 (2H q), 7.3 (2H d), 7.6 (2H d), 7.8 (4H dd) ppm
MS: m/z 207 (M + •), 192, 165, 140, 115

4-cyano-4′-propylbiphenyl
Purity (HPLC) 99.9%, Yield 1.03 g
^{1}H NMR: (CDCl3) δ 1.0 (3H t), 1.7 (2H m), 2.7 (2H t), 7.3 (2H d), 7.6 (2H d), 7.8 (4H dd) ppm
MS: m/z 221 (M + •), 192, 165, 140, 115

4-cyano-4′-butylbiphenyl
Purity (HPLC) 99.8% Yield 1.09 g
^{1}H NMR: (CDCl3) δ 1.0 (3H t), 1.4 (2H m), 1.7 (2H m), 2.7 (2H t), 7.3 (2H d), 7.6 (2H d), 7.8 (4H dd) ppm
MS: m/z 235 (M + •), 192, 165, 140, 115

4-cyano-4′-pentylbiphenyl
Purity (HPLC) 99.3% Yield 1.16 g
^{1}H NMR: (CDCl3) δ 1.0 (3H t), 1.4 (4H m), 1.7 (2H m), 2.7 (2H t), 7.3 (2H d), 7.6 (2H d), 7.8 (4H dd) ppm
MS: m/z 249 (M + •), 192, 165, 140, 115

4-cyano-4′-hexylbiphenyl
Purity (HPLC) 99.7% Yield 1.47 g
^{1}H NMR: (CDCl3) δ 1.0 (3H t), 1.4 (6H m), 1.7 (2H m), 2.7 (2H t), 7.3 (2H d), 7.6 (2H d), 7.8 (4H dd) ppm
MS: m/z 263 (M + •), 192, 165, 152, 115

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