Military University of Technology

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Soft Matter

SYNTHESIS, PROPERTIES AND ENANTIOSEPARATION OF FOUR-RING RACEMIC SMECTICS

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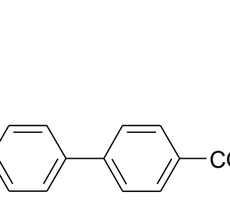
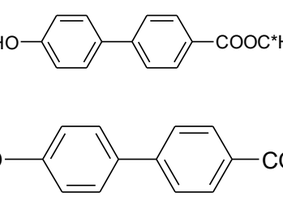
Introduction

Liquid crystals (LCs) represent a unique state of matter combining fluidity with long-range molecular ordering. Among thermotropic liquid crystals, smectic phases are characterised by both orientational and positional order, resulting in layered structures. Smectic A (SmA) phases exhibit the director parallel to the normal layer, whereas more complex arrangements such as smectic C_A (SmC_A) involve tilted ordering and anticlinic packing in neighbouring layers. In this work, two racemic, four-ring aromatic smectics differing in the number of methylene groups in the flexible spacer were synthesised and systematically characterised. Their mesomorphic properties were investigated by polarising optical microscopy and differential scanning calorimetry, confirming the formation of smectic phases SmA and SmC_A within distinct temperature ranges, as determined by dielectric spectroscopy. Since the synthesised materials occur as racemic mixtures, chiral separation was undertaken to obtain optically pure enantiomers without the need for independent asymmetric synthesis.

Smectic A phase

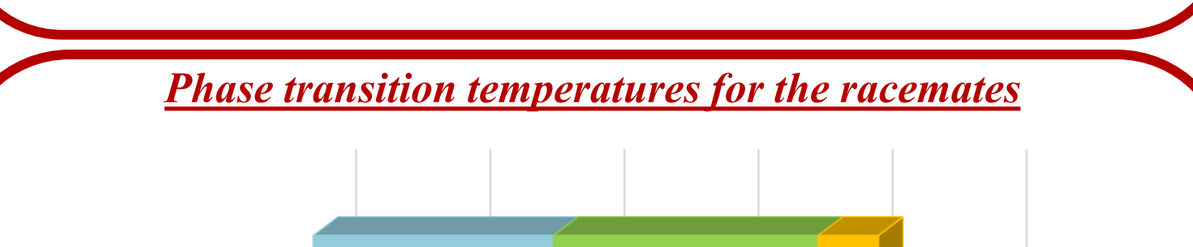




Smectic C_A phase

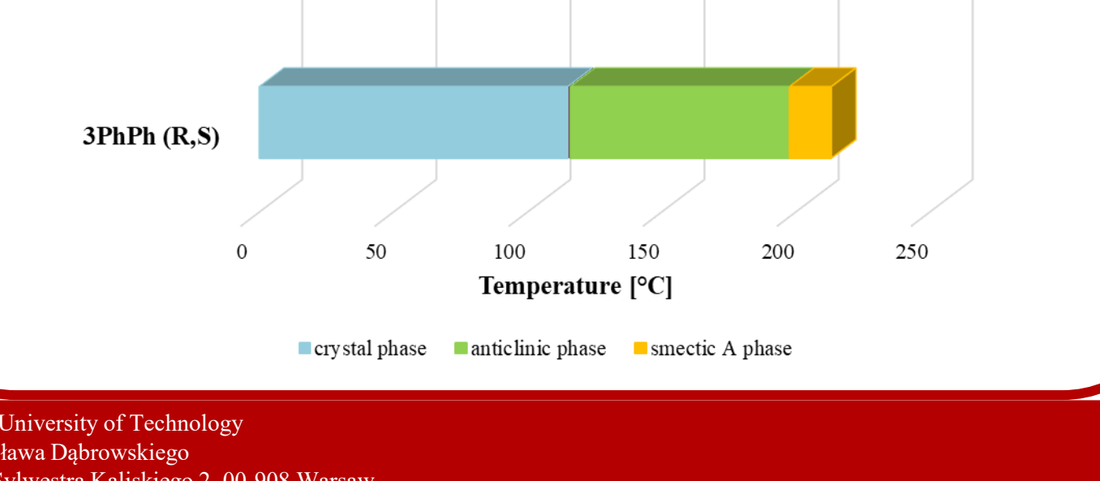
The structures and acronyms of smectic racemates

3PhPh (R,S)



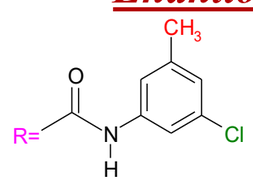
7PhPh (R,S)

Synthesis of the racemates



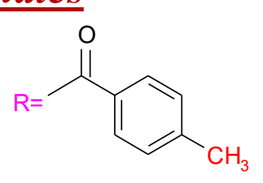
Phase transition temperatures for the racemates

Enantioseparation of the racemates



ReproSil Chiral-MIG

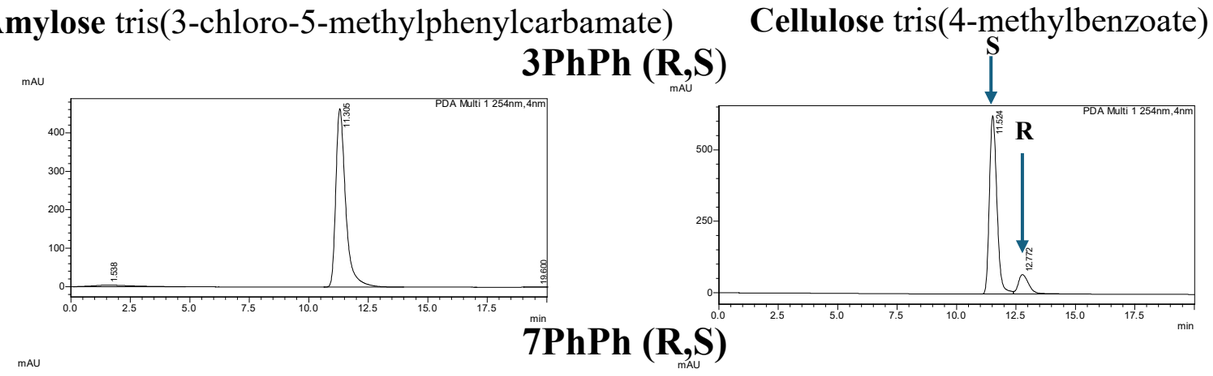
Amylose tris(3-chloro-5-methylphenylcarbamate)



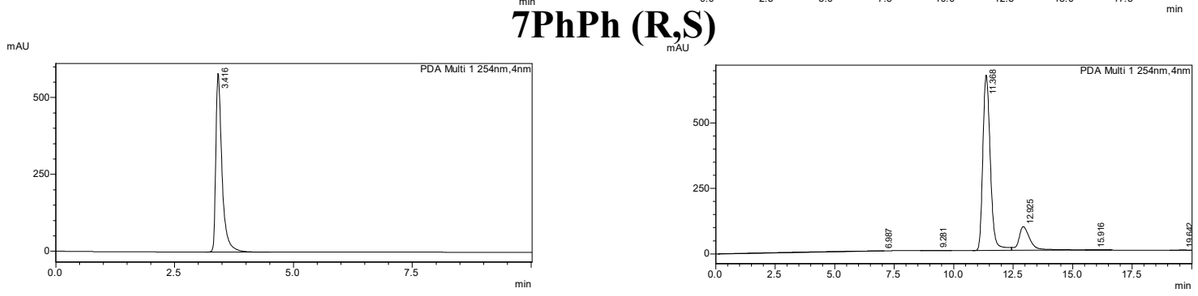
ReproSil Chiral-JM

Cellulose tris(4-methylbenzoate)

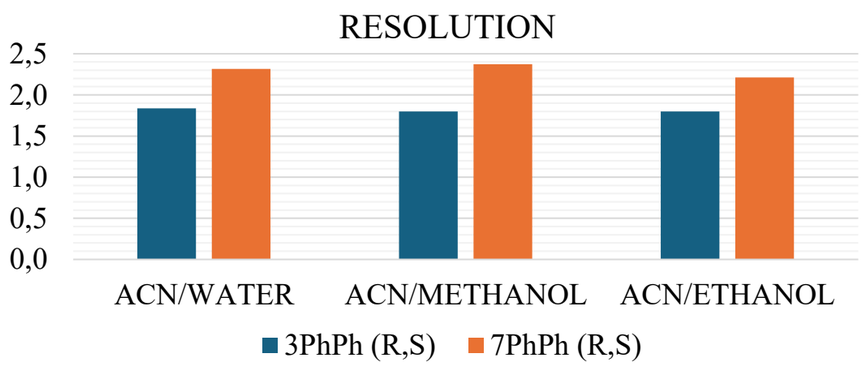
3PhPh (R,S)



7PhPh (R,S)



Chromatograms of racemates in the solvent system ACN/WATER in the ratio 99:1, at a flow rate of 1.0 or 0.3 mL/min.



Resolution of racemates from different solvent systems on the ReproSil Chiral-JM column at a flow rate of 1.0 or 0.3 mL/min.

Conclusions

This study aimed to synthesise and characterise four-ring racemic smectics. Additionally, the goal was to separate these racemates into enantiomers using chiral liquid chromatography. The studies confirmed the presence of two smectic phases in these racemates: the anticlinic phase (SmC_A) and the smectic A phase (SmA). The racemates exhibit the anticlinic phase over a very wide temperature range, making them good admixtures for antiferroelectric liquid-crystalline mixtures.

HPLC analyses revealed optimal conditions that enabled the baseline separation of both racemic mixtures into their enantiomers, with a resolution exceeding 1.5. The cellulose column provided better results than the amylose column.

This allows these conditions to be scaled up to preparative levels, enabling enantiomer separation without the need for lengthy synthesis.

References

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Acknowledgements

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