Hyphenating Convergence Chromatography with UV and MS Detection for Compositional, Impurity and Degradation Analysis of High Performance Electronic Materials

Jane A Cooper*

Waters Corporation, Stamford Avenue, Altrincham Road, Wilmslow, SK9 4AX, UK

jane_cooper@waters.com, +44 (0) 161 435 4117

INTRODUCTION

Liquid crystals combine the physical and optical properties of both liquids and solids. They flow and pour like liquids, but they have some of the optical properties of solids, such as birefringence. They also react predictably to an electric current, which enables the control of light passage.

Due to these properties, liquid crystals are used in many high performance electronic materials, for example: mobile phones, desktop monitors, and TVs. Liquid crystal intermediate compounds are the building blocks used to prepare liquid crystals. In order to achieve the material properties required between 10 and 20 individual intermediate compounds are in a typical liquid crystal mix. The composition, purity and degradation of the liquid crystal compounds used is critical to ensuring optimum optical quality, performance, and lifetime of the electronic display device.

Typical techniques used for the impurity profiling, impurity and degradation analysis of liquid crystal intermediate compounds include: HPLC with UV detection [1], HPLC with MS detection [2], and GC with MS detection [3]. However these techniques have some limitations: the compounds might not be thermally stable and / or volatile; there might be limited sample availability; the sample solubility might be incompatible with the mobile phase; long analysis times with insufficient selectivity and sensitivity.

Convergence Chromatography (CC) is a separation technique that uses carbon dioxide as the primary mobile phase, with a co-solvent such as acetonitrile to give similar selectivity as normal phase LC. Various detection methods can be used including UV and Evaporative Light Scattering Detection (ELSD). But there is also the option of interfacing CC with Mass Spectrometry (MS) detection, with the addition of a MS splitter, which introduces a controlled leak to the system and enables the maintenance of the CO_2 pressure.

The option to add a solvent via a makeup pump to the flow prior to MS detection can be used to provide greater solvating powers, to enhance the selectivity and sensitivity of MS detection, and also to influence ionization.

RESULTS

IMPURITY AND DEGRADATION ANALYSIS

The UPC^2 conditions were optimized for the analysis of a select group of liquid crystal intermediate compounds. Retention times, UV optimum absorbances were established by analysing single component standards [4]. The UV chromatograms in a mixed 0.1 mg/mL calibration standard, are shown in Figure 1.

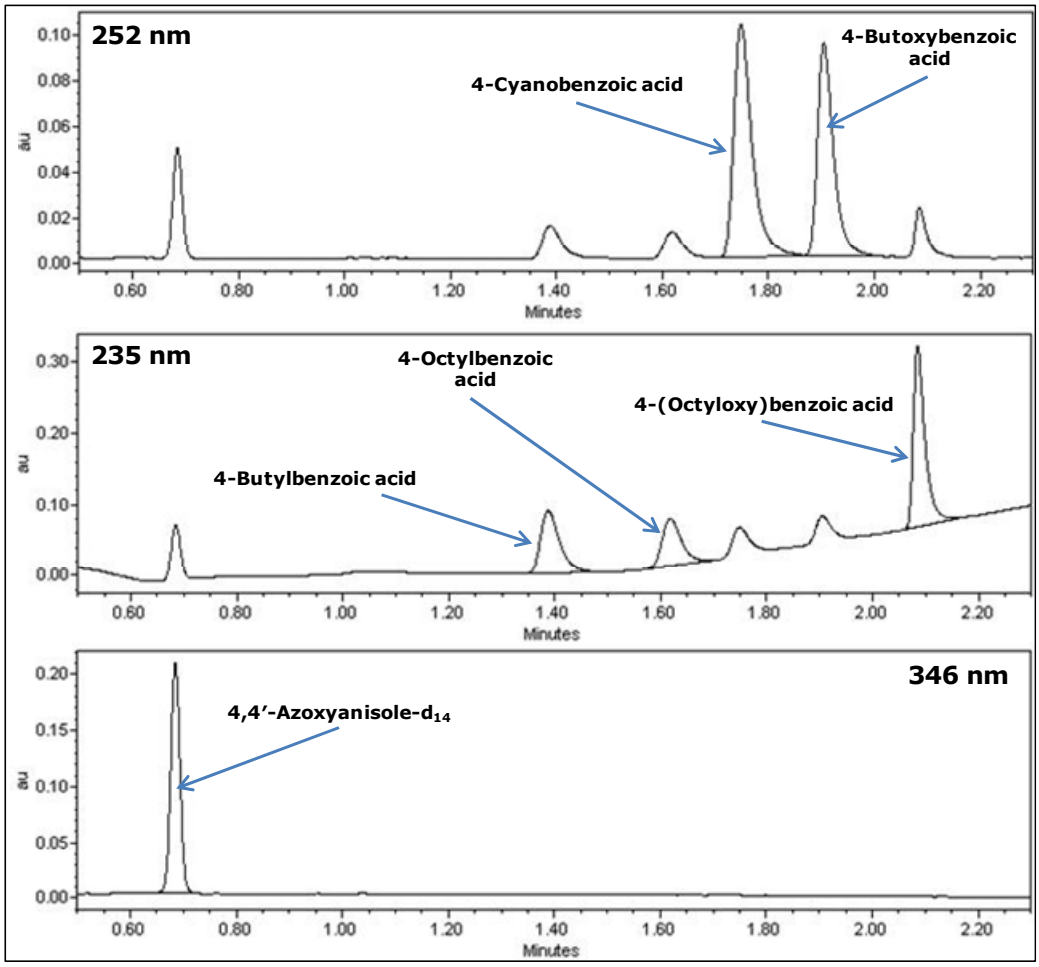

Figure 1: UV chromatograms in a mixed liquid crystal 0.1 mg/mL calibration standard.

In order to demonstrate impurity profiling analysis, 4-Butyl benzoic acid was spiked at 0.1% with three other liquid crystal intermediate compounds and analyzed using the developed UPC^2 conditions with PDA detection [4]. The resulting UV chromatograms achieved are shown in Figure 2, which illustrate that the identification of an impurity at 0.1% can be achieved for the liquid crystal intermediate compounds considered.

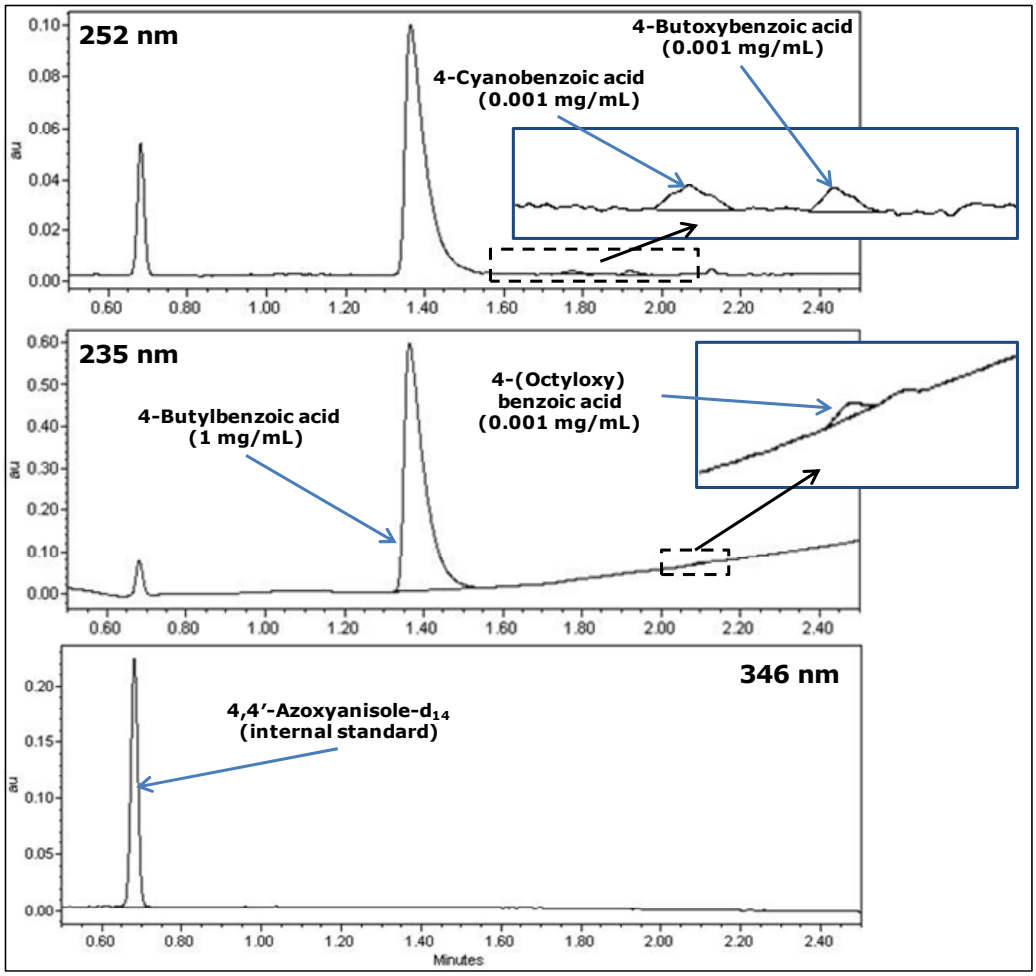

Figure 2: Impurity profiling UV chromatograms. 4-Butylbenzoic acid at 1 mg/mL, spiked with 4-Cyanobenzoic acid, 4-Butoxybenzoic acid, and 4-(Octyloxy)benzoic acid all at 0.001 mg/mL (equivalent to 0.1% impurity in the product).

COMPOSITIONAL ANALYSIS

UPC^2 conditions were optimized for the analysis of four Merck E7 liquid crystal compounds, 4-cyano-4'-n-puntyl-biphenyl (5CB), 4-cyano-4'-n-heptyl-biphenyl (7CB), 4-cyano-4'-n-oxyoctyl-biphenyl (8OCB) and 4-cyano-4"-n-pentyl-p-terphenyl (5CT) [5]. Retention times, UV optimum absorbances were established by analyzing single component standards. Mixed calibration standards were analyzed for all compounds. In order to demonstrate compositional analysis, a mix containing the correct ratio and one at an incorrect ratio, were both analyzed using the developed UPC^2 conditions with PDA detection. The resulting QC custom reports are shown in Figure 3.

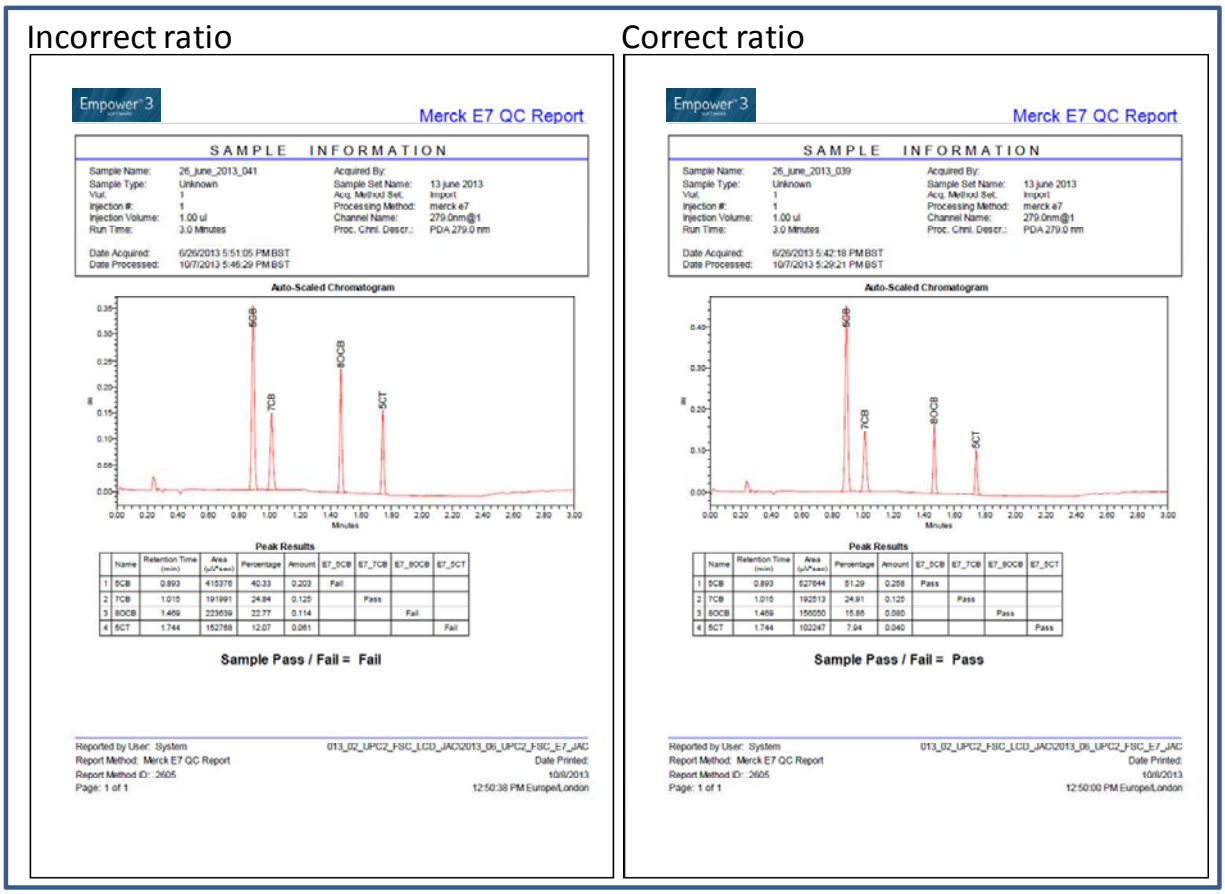

Figure 3: Merck E7 liquid crystal compositional QC custom reports.

UPC^2 WITH MS DETECTION USING THREE DIFFERENT IONIZATION TEHCNIQUES

When greater selectivity and specificity are required for the analysis of liquid crystals, it is possible to combine UPC^2 with MS detection [6]. In order to demonstrate the MS ionization options available when combine UPC^2 with MS detection, a selected groups of liquid crystal intermediate compounds were considered. First the UPC^2 conditions were optimized using PDA detection. Then using the on-board fluidics system on the Xevo TQD, individual standards were infused into the source using atmospheric pressure chemical ionization (APCI), in order to establish the MS and MRM conditions. In this example the established MRM conditions were also used for atmospheric pressure photo ionization (APPI) and electrospray ionization (ESI).

Mixed standards were analyzed using the optimized APPI, APCI and ESI conditions. When considering the MS splitter conditions, the makeup solvent and flow required for each ionization mode were optimized. When using ESI, formic acid was added to the makeup solvent to aid protonation, enhance ionization and increase sensitivity. In APPI, the addition of the dopant toluene to the makeup solvent was used to enable and enhance ionization. Whereas when using APCI, the solvent present, from both the co-solvent and the makeup solvent act as a chemical

ionization reagent gas in order to ionize the sample. The resulting MRM chromatograms using APCI, APPI and ESI ionization modes are shown in Figure 4.

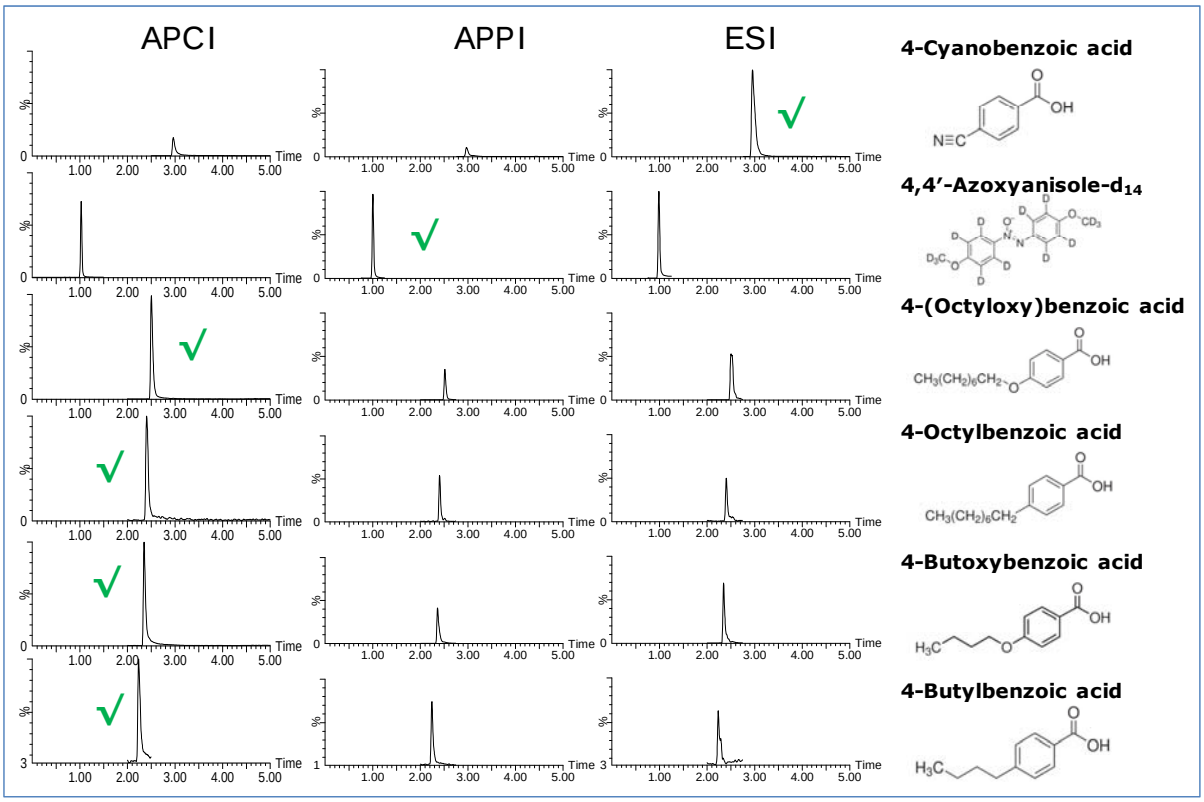

Figure 4: MRM chromatograms using APCI, APPI and ESI ionization modes for the five liquid crystal intermediate compounds and one internal standard in a mixed 0.1 mg/mL calibration standard (√ refers to ionization mode which gave the largest response for each compound).

CONCLUSIONS

Many liquid crystal intermediate compounds are not very stable at high temperatures, have low volatility, and have similar UV spectra. Therefore, separation by UPC^2 with CO_2 as the mobile phase is an ideal alternative to both HPLC and GC analysis. By utilizing Waters® ACQUITY UPC^2 with PDA detection a cost effective, efficient impurity profiling and compositional analysis can be achieved. When greater selectivity and specificity are required for the analysis of liquid crystals, it is possible to combine UPC^2 with MS detection. The efficiency of ACQUITY UPC^2, hyphenated with PDA and MS detection can be used as an orthogonal technique to ensure full characterization of liquid crystal intermediate compounds. The described approaches offers many business and analytical benefits, when compared HPLC for the analysis of liquid crystal intermediate compounds, with typically greater than 13 fold increase in sample thought put and greater than 110 fold reduction in the volume of toxic solvent required.

REFERENCES

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