

Workflow for Migrating OLED Impurity Profiling from R&D to QC Setting with Solvent Compatible Mass Detector System

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TECHNOLOGY BENEFITS

Waters offers analytical solutions and techniques that effectively address the chemical diversity found in samples so that you can:

- Maximize separation for complex samples reliably and routinely in both the reversed-phase and normal-phase separation space.
- Obtain the highest level of structural information that reveals even trace amounts of compounds.
- Visualize, process, and report data more effectively to support batch release.
- Make critical decisions in less time with greater confidence using tools and techniques to improve workflow and increase testing throughput.

WATERS SOLUTIONS

Impurity discovery/profiling:

[ACQUITY™ UPLC™ I-Class System](#)

with hexane/THF compatibility kit

[Xevo™ G2-XS QToF Mass Spectrometer](#)

[UNIFI™ Scientific Information System](#)

Impurity quantitation:

[ACQUITY Arc™ System](#)

with hexane/THF compatibility kit

[ACQUITY QDa™ Mass Detector](#)

with extended solvent compatibility kit

[Empower™ 3 Chromatography Data Software](#)

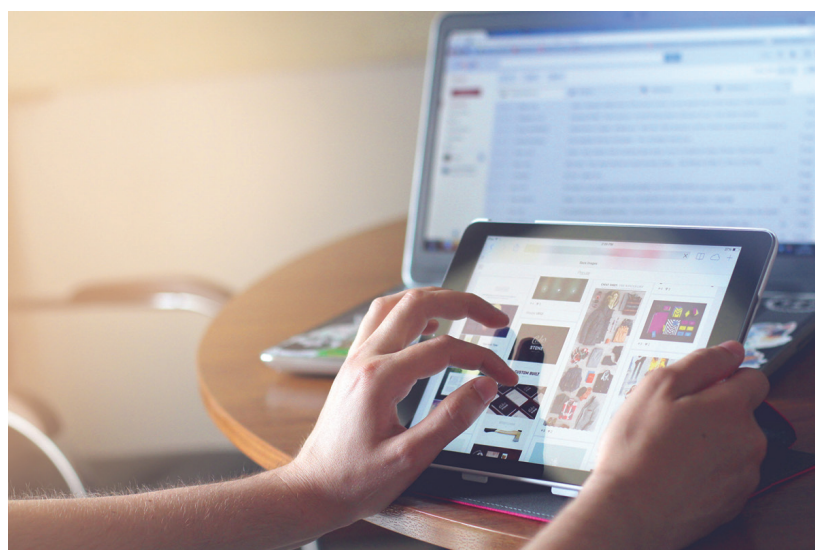
KEYWORDS

OLED, impurity profiling, quantitation, THF, MS^E, QuanTof, UPLC, solvent compatibility, display technology

INTRODUCTION

Organic light emitting diode (OLED) color dopants are high-value organic and organometallic chemicals that make up important components of OLED display technologies. Chemical impurities in the OLED materials can significantly impact product degradation and hence shorten device lifetimes to commercially unacceptable levels. Controlling the types and amounts of trace impurities in the materials is critical for manufacturers to provide high-quality OLED display technologies.

Potential impurities can enter the finished material via pathways such as impurities present in raw materials, synthetic by-products, degradation products, or external contamination during final product manufacturing processes. Due to this variability across raw materials, synthetic and purification processes, and batch lots, it is necessary to utilize analytical technology that can detect and identify unknown organic impurities with high sensitivity over a broad chemical space.



DISCUSSION

Identification and control of impurities is a critical task in manufacturing process development for quality and performance of OLED materials. The most commonly used analytical technique for impurity analysis in chemical materials is via chromatographic method, namely High Performance liquid chromatography (HPLC). When coupled to an orthogonal detection technique such as mass spectrometry, an additional dimension of chemical information can be obtained that can increase analytical sensitivity, and also offer INSIGHT into the chemical nature and potential source of any impurities detected over information offered from optical detection alone.

In the case of “discovery,” or unknown impurity profiling, essential experimental parameters required for successful impurity elucidation include, but are not limited to:

Acquisition mode: To detect unknown impurities, it is necessary to use a full scan data acquisition mode (i.e., covering a broad mass range or wavelength) rather than monitoring only at a specific wavelength or m/z . And, since the chemical components are likely unknown, the acquisition mode of a mass spectrometer will benefit from providing a means of identifying chemical components.

Sensitivity: In the case of trying to identify 0.01% concentration of impurities relative to the material, if the material is dissolved in a solvent at a concentration of 100 ppm (0.01%), the impurity concentration will be 10 ppb. For identifying unknown impurities, it is necessary to have a sufficiently sensitive detection limit that enables detecting and identifying peaks with concentrations in the range of several tens ppb in full scan mode.

Data processing: Due to the large data volume acquired during impurity profiling, efficient workflows will be facilitated by a solution that can extract impurity peaks from enormous amounts of full scan data, provide elemental compositions, and possess the ability for database searching to assist with structural elucidation studies.

In the case of impurity quantitation, experimental focus shifts from comprehensive full scan data capture to more routine targeted analyses workflows. Here, ease-of-use, system robustness, and the ability to operate separation technology across both reversed- and normal-phase conditions become key drivers. The workflow employed in impurity characterization and quantitation, and where different types of MS technology are employed, is summarized in Figure 1.

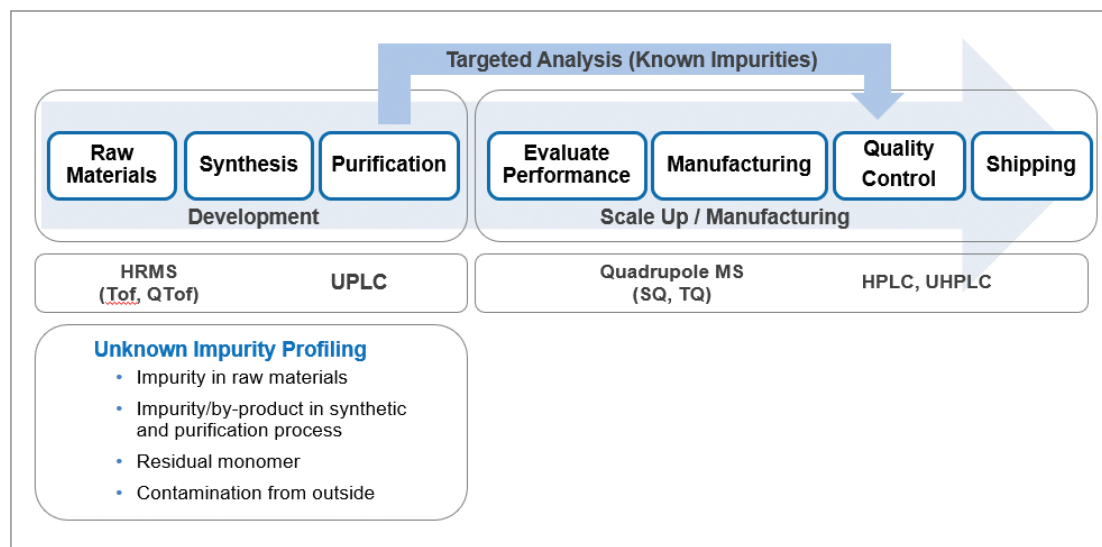


Figure 1. Workflow for impurity characterization and quantitation.

Outlined below is an example of the combination of HRMS and quadrupole MS technology that has been successfully deployed in the impurity profiling of commercialized OLED material E709:

To determine the identity and target analysis for the presence of those impurities, OLED material analysis was moved to a HRMS system.

Three kinds of sample solutions (Figure 3) have been analyzed with UPLC-MS. The QToF MS was operated in MS^E mode, whereby the MS/MS acquisition mode alternates between a low-energy scan to detect the precursor ions and then switches to a high-energy scan to produce the product ions – all accomplished within a single analysis.

As the data set was acquired with MS^E mode which can provide precursor and fragment ion spectrum, information regarding the main component can provide clues to aid in the discovery and identification of impurities (Figure 4).

UNIFI Scientific Information System was used to process the data set. There are three main approaches outlined below to extract impurity peaks.

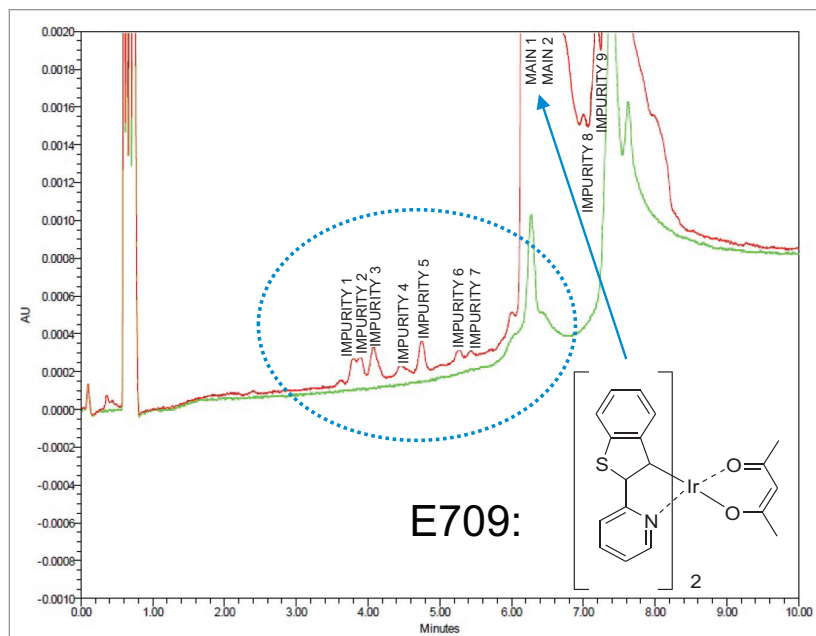


Figure 2. HPLC trace of E709 showing unknown impurities.

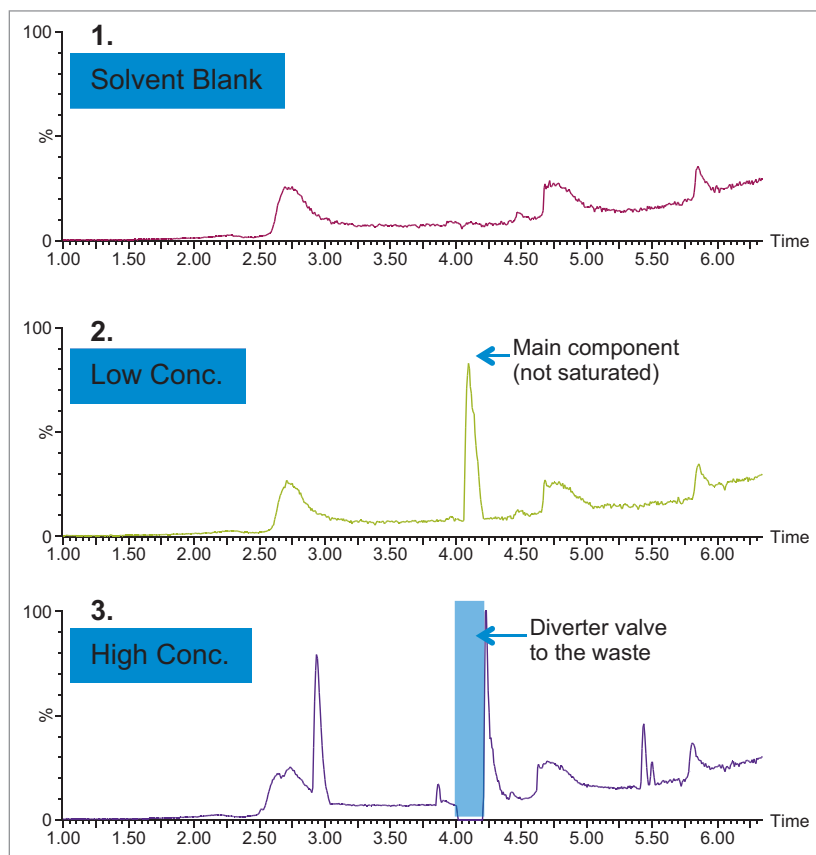


Figure 3. (1) Solvent blank that is used as reference for comparing with sample solution, (2) low concentration sample that is used as reference for calculating relative concentration of impurity, and (3) high concentration sample that is used for identification of impurity and calculating its concentration (saturated main component is diverted to the waste).

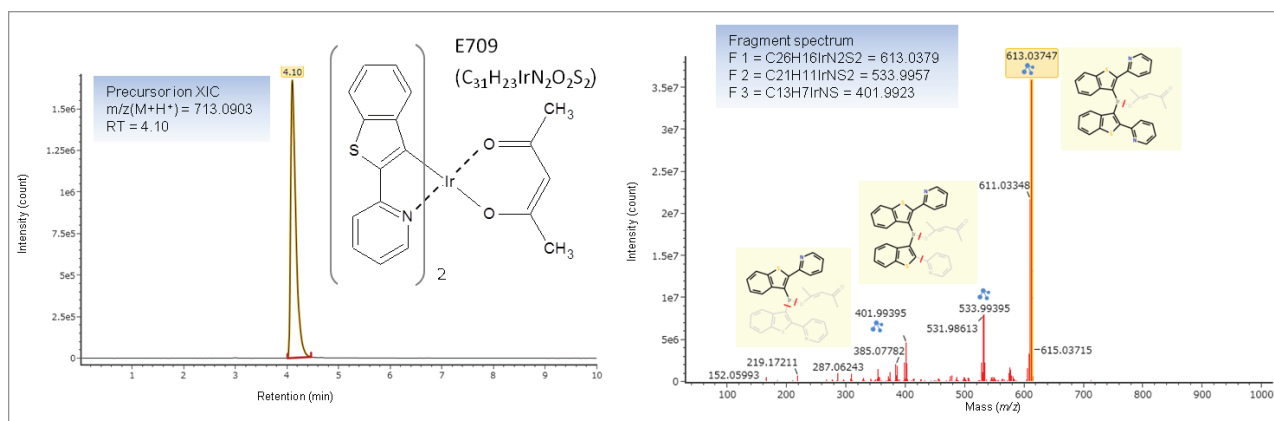


Figure 4. Precursor and fragment ion spectrum of E709.

Extract by software: Extract peaks with same fragment/neutral loss in common with the main component. Since the experiment was conducted in MS^E mode, all precursor and product ion fragments were collected in one analysis. The information content then enabled UNIFI to search impurity peaks with fragment/neutral loss in common with the main component. Figure 5 shows the impurity with fragment in common with main component.

Compare by software – Binary compare:

Often when comparing a total ion chromatogram of sample with another sample, it is difficult to extract peaks that are unique to only one of the samples. A graphic comparison of one chromatogram against a second is called “binary comparer” and can often highlight unique peaks in one sample over a second. To illustrate this principle, Figure 5 shows unique peaks detected from the sample by comparing sample and solvent blank.

Search by software – Search by synthetic pathway:

If the synthetic pathway is known (or can be proposed) based on prior knowledge, UNIFI can automatically search the related impurities by looking for transformations of main component structure.

UNIFI can not only extract impurity peaks by these approaches, but also perform a database search using the elemental composition, and generate structural elucidation using fragment match.

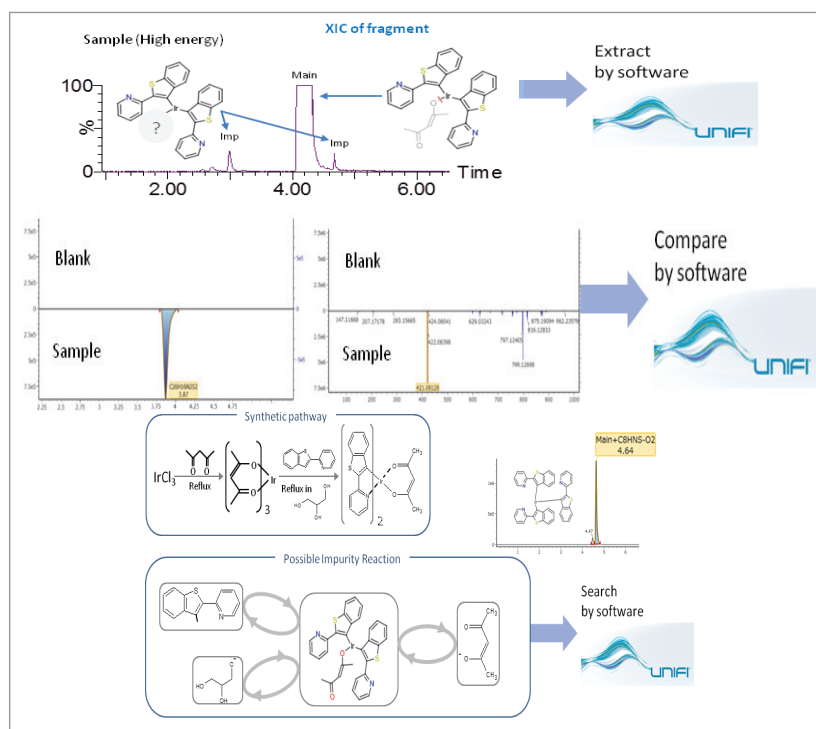


Figure 5. UNIFI has three main approaches to extract impurity peaks.

In this way, several types of impurities were detected, and structures elucidated. Some examples are shown in Figure 6. The lowest concentration of impurity was 0.005% (Imp05), and the highest of that was 0.127% (Imp06).

Having successfully profiled the main impurities present in E709 using HRMS technology, the analysis method was transferred to quadrupole MS detection for deployment in QC monitoring for the presence of the most abundant impurity.

The system chosen for QC monitoring was the ACQUITY Arc System fitted with hexane/THF compatibility kit and ACQUITY QDa with extended solvent compatibility kit that allows for successful deployment of low aqueous reversed-phase methods (LARP) in the QC environment. Shown below are overlaid, single ion monitoring (SIM) chromatograms for E709 material and most abundant impurity, impurity 6.

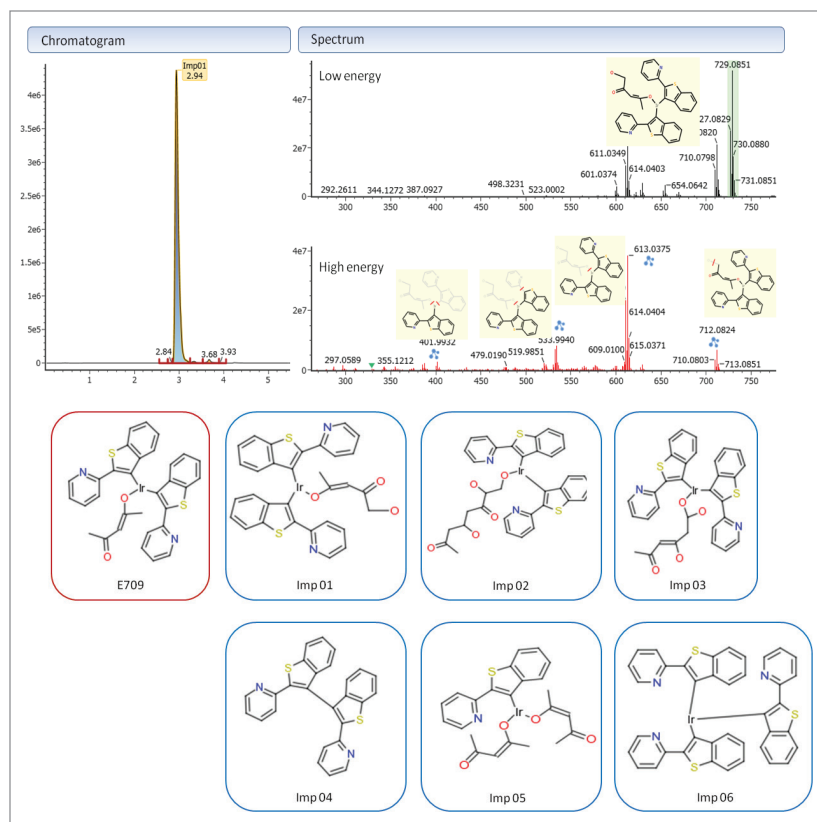


Figure 6. Impurity structural elucidation with UNIFI.

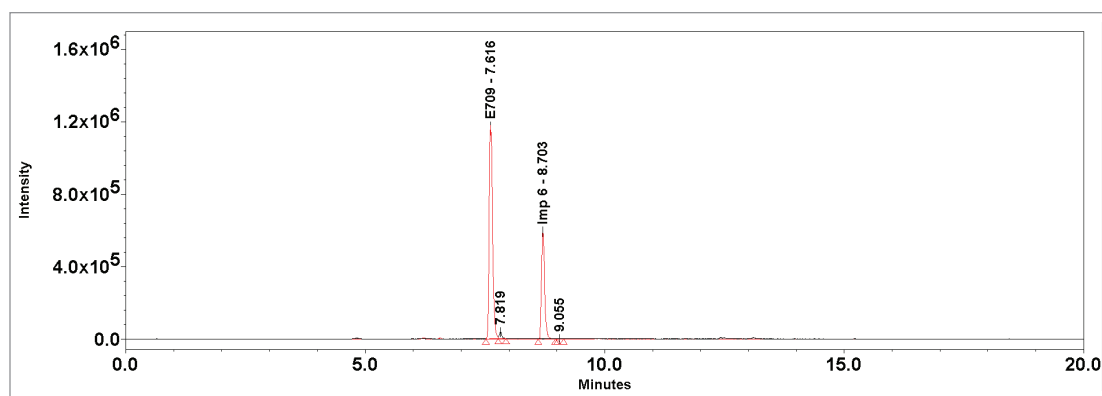


Figure 7. Single ion monitoring (SIM) chromatograms for E709 material and impurity 6.

CONCLUSIONS

Impurity profiling and quantitation of chemical components is a critical, quality control strategy for OLED material manufacturers. Waters offers high-resolution and tandem quadrupole MS technologies, coupled with streamlined informatics solutions, to aid in every step of the workflow.

This allows OLED material manufacturers to successfully produce high-value, specialty chemical components for consumer products.

Waters

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