

Improving Sensitivity and Selectivity for Primary Aromatic Amines Analysis with the SQ Detector 2

GOAL

To demonstrate the use of the SQ Detector 2 for the analysis of Primary Aromatic Amines with improved sensitivity, selectivity, and robustness, compared with existing analog detection methodologies.

BACKGROUND

The inks and dyes industry is highly legislated and manufacturers who use these materials must monitor and quantify various regulated parameters, such as the presence or absence of Primary Aromatic Amines (PAAs).

PAAs can be used to produce many commodities, such as pharmaceuticals, pesticides, explosives, epoxy polymers, rubber, aromatic polyurethane products, and azo-dyes. They can be found in final products due to incomplete reactions, as impurities, by-products, or as degradation products.

Many PAAs are suspected carcinogens and therefore have a range of potential health risks, which have lead to strict worldwide regulations. U.S. FDA regulations (21 CFR 74.705 and 21 CFR 74.706) restrict the use of azo-dyes that could degrade to PAAs; whereas EU regulations (commission directive 2002/72/EC and directive 19/2007/EC) set legislative limits.

Analytical laboratories require accurate and robust techniques to ensure confidence and versatility in meeting these legislative requirements. The SQ Detector 2 offers a flexible solution for this industry.

SQ Detector 2 linked to ACQUITY UPLC H-Class provides improved confidence in the identification and quantification of Primary Aromatic Amines.

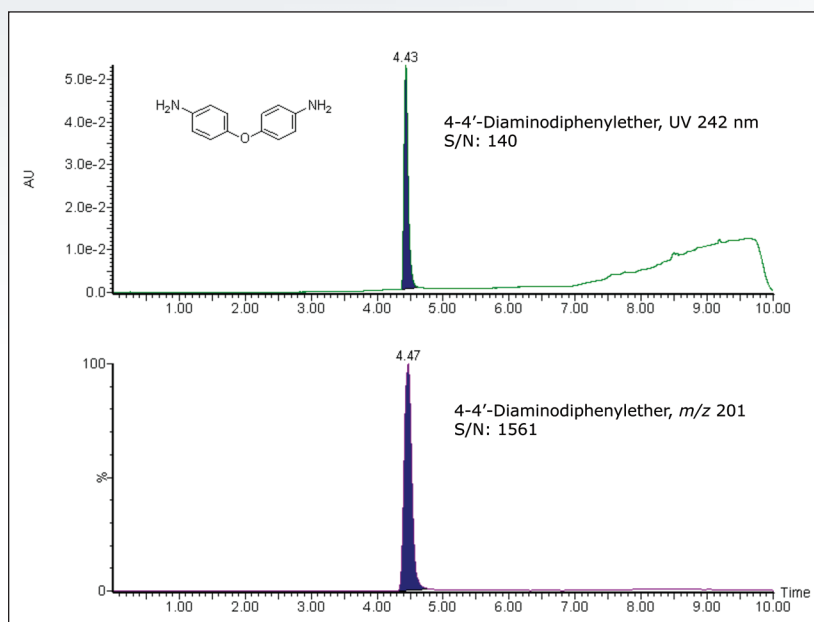
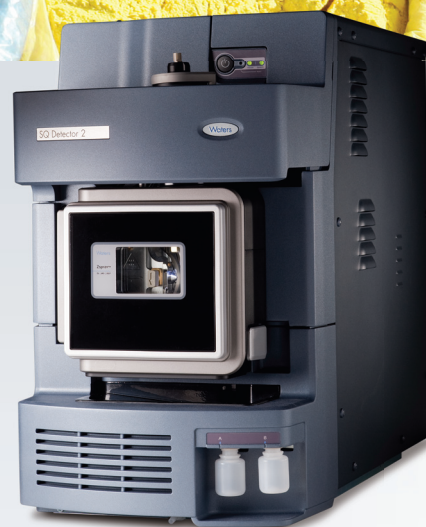


Figure 1. UV and extracted ion chromatograms for 4-4'-Diaminodiphenylether.

THE SOLUTION

Waters® SQ Detector 2 with an ElectroSpray Ionization (ESI) source, coupled to an ACQUITY UPLC® H-Class System, were used to monitor PAAs in a 10-minute run. MassLynx™ Software was used to acquire and process the data.

Developing analytical methods and validation with enhanced sensitivity, selectivity, and method robustness to meet legislative requirements can be costly and time consuming. By utilizing the many features of ACQUITY UPLC H-Class and the SQ Detector 2, several business benefits can be achieved. Financial savings can be realized with much faster run times, the use of less solvent, and increased sample throughput. A method with a run time of 10 minutes has been developed for PAAs, which is up to seven times faster than some existing methods. In addition, no time-consuming derivatization stages were used, affording further savings in the sample preparation stages.

Improvements in sensitivity can be demonstrated by considering the signal-to-noise (S/N), when comparing the UV and the mass spectral data for the PAA 4-4'-Diaminodiphenylether. This PAA has a precursor ion at m/z 201 and a maximum UV absorbance at 242 nm. The increase in S/N when using mass spectral data is illustrated in Figure 1.

In order to observe improvement in selectivity, the abilities to measure analytes of interest accurately and specifically in the presence of a complex matrix need to be considered. This is demonstrated by considering the PAA 2,4,5-Trimethylaniline, which when spiked in ink cannot be distinguished due to other UV absorbing compounds present, as shown in Figure 2. However, mass detection is sufficiently sensitive and selective to enable confident detection and quantification of 2,4,5-Trimethylaniline in an ink matrix.

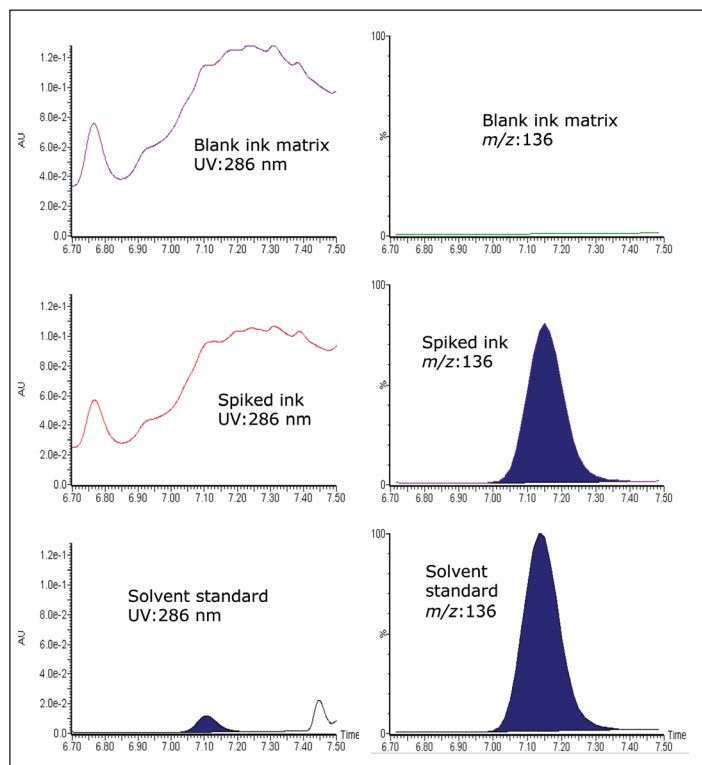


Figure 2. UV and extracted ion chromatograms for 2,4,5-Trimethylaniline spiked in ink (4.6 $\mu\text{g/mL}$), solvent standard (5.0 $\mu\text{g/mL}$), and blank ink matrix. UV and MS data were acquired simultaneously.

SUMMARY

The SQ Detector 2 coupled to an ACQUITY UPLC H-Class System can provide increased confidence in the identification and quantification of PAAs in ink, compared to techniques typically used for this analysis.

Due to reduced run time, UPLC® analysis for PAAs affords increased sample throughput and a reduction of solvent usage, offering businesses valuable time and money savings.

The SQ Detector 2 offers the ability to acquire data in Selected Ion Recording mode (SIR), which provides enhanced selectivity and sensitivity, delivering increased confidence when reporting quantitative results.

Waters

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