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### 응용 자료

# Rapid Screening of 36 Synthetic Dyes using the ACQUITY UPLC H-Class System with the ACQUITY QDa Detector

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This is an Application Brief and does not contain a detailed Experimental section.

### Abstract

This application brief demonstrates to selectively analyze 36 disperse, basic, acid, solvent and direct synthetic dyes to below EU legislative limits.

#### **Benefits**

Reduce analysis times from 17 min to <5 min.

## Introduction

Synthetic dyes are classified according to how they are used in the dyeing process. For example, disperse dyes which are mostly azo- or anthraquinone compounds are generally used for dyeing synthetic textile materials such as polyester, nylon, and PVC. The type of bonds formed between the dyes and the fabric, determine the properties of the dyes. For example, disperse dyes are not chemically bonded to the fibers of the textile, thus they can easily migrate onto the skin of the person wearing the garment, especially if the textile fastness is poor.

A number of synthetic dyes are known to be allergenic when they come into contact with human skin or if they are are classified as potentially sensitizing, carcinogenic, mutagenic, or toxic to reproduction. 20 known allergenic dyes are listed by the Oeko-tek Standard 100 and the permitted limit is <50 mg/kg. Other legislation such as European Union (EU) 2009/567/EC has banned the use of these sensitizing dyes.

The standard method for the analysis of disperse dyes in textile products and components is DIN 54231-2005 using HPLC-UV-MS with an analysis time of 17 minutes.

For synthetic dyes that have very similar UV olvent, and direct dyes. absorbance maxima, it can be challenging to correctly identify their presence. Whereas, for the dyes considered that have different molecular weights, utilizing mass spectrometry can provide more selectivity and confidence in their identification.

# Results and Discussion

The Waters ACQUITY UPLC H-Class System with the ACQUITY QDa Detector was used to monitor a total of 36 disperse, basic, acid, solvent, and direct dyes including 28 dyes listed in the Oeko-Tex Standard 100 and 2009/567/EC. The list of the synthetic dyes considered are provided in Table 1.

	Compounds	CAS no.	Retention time	SIR	Polarity
1	Basic Red 9	569-61-9	0.70	288	+
2	Basic Violet 14	632-99-5	0.83	302	+
3	Disperse Blue 7	3179-90-6	1.22	359	+
4	Acid Violet 49*	1694-09-3	1.36	712	+
5	Disperse Red 11	2872-48-2	1.60	269	+
6	Disperse Blue 3	2475-46-9	1.66	297	+
7	Disperse Blue 102	69766-79-6	2.03	366	+
8	Disperse Red 17	3179-89-3	2.32	345	+
9	Solvent Yellow 1*	60-09-3	2.41	198	+
10	Disperse Blue 106	68516-81-4	2.49	336	+
11	Disperse Orange 3	730-40-5	2.57	243	+
12	Disperse Yellow 3	2832-40-8	2.72	270	+
13	Disperse Yellow 39	12236-29-2	2.83	291	+
14	Basic Violet 1*	8004-87-3	2.88	358	+
15	Disperse Blue 1	2475-45-8	2.89	268	+
16	Disperse Brown 1	23355-64-8	2.92	433	+
17	Disperse Red 1	2872-52-8	3.05	315	+
18	Disperse Blue 35A	56524-77-7	3.16	285	+
19	Basic Violet 3*	548-62-9	3.33	372	+
20	Disperse Orange 11	82-28-0	3.35	238	+
21	Disperse Yellow 49	54824-37-2	3.35	375	+
22	Solvent Yellow 2*	60-11-7	3.37	226	+
23	Disperse Blue 26	3860-63-7	3.40	299	+
24	Disperse Blue 124	61951-51-7	3.43	378	+
25	Solvent Yellow 3*	97-56-3	3.92	226	+
26	Basic Blue 26*	2580-56-5	4.00	470	+
27	Disperse Orange 37/76	13301-61-6	4.08	392	+
28	Disperse Blue 35B	56524-76-6	4.16	299	+
29	Disperse Orange 1	2581-69-3	4.33	319	+
30	Disperse Yellow 23	6250-23-3	4.36	303	+
31	Disperse Orange 149	85136-74-9	5.05	459	+
32	Direct Blue 6	2602-46-2	1.22	421	-
33	Acid Red 26	3761-53-3	1.86	435	
34	Direct Red 28	573-58-0	2.30	651	-
35	Direct Brown 95*	16071-86-6	2.53	357	-
36	Direct Black 38	1937-37-7	3.23	736	-

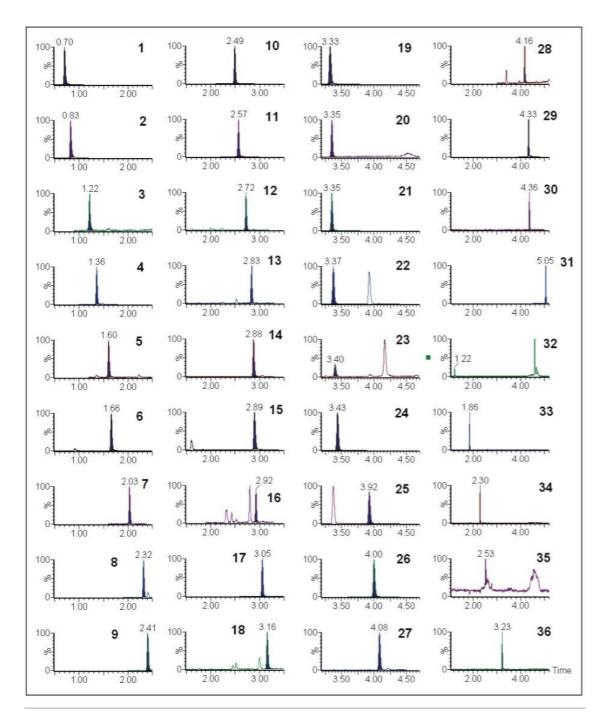
Table 1. Retention times, SIR m/z, and polarity for 36 disperse, basic,

acid, solvent, and direct dyes.

\* Compounds not listed in the Oeko-tek Standard 100 and EU 2009/567/EC

The time required for method development was greatly reduced using the pre-optimized source parameters in the ACQUITY QDa Detector, where the required sensitivity were achieved in both polarities for both positive and negative ionizing dyes. The low system dispersion in the ACQUITY UPLC H-Class System and the use of sub-2-µm particle columns also greatly increased peak resolution and enhanced sensitivity. Here, two methods were developed for both positive and negative ionizing dyes with analysis time of 5 and 4 minutes respectively.

Dyes were monitored according to their respective retention time, ionizing polarity, and Single Ion Recording (SIR) mass-to-charge ratio (m/z), as described in Table 1. In the highly regulated inks and dyes industry, SIR provides more selectivity and sensitivity compared to HPLC-UV analysis. The SIR chromatograms, shown in Figure 1, indicate that synthetic dyes can be easily and confidently detected at low levels.



*Figure 1. SIR chromatogram of 36 disperse, basic, acid, solvent and direct dye standards. SIR 1 to 31 were acquired in positive polarity, and 32 to 36 were acquired in negative polarity.* 

Current EU legislation prohibits the use of sensitizing dyes in textiles, while allowing 5 ppm limit on the other dyes. Quantitation was also carried out with concentrations ranging from 0.3 to 2.0 ppm, and linearity of >0.997 was achieved for all the dyes. The calibration curve of Disperse Blue 3 is shown in Figure 2.

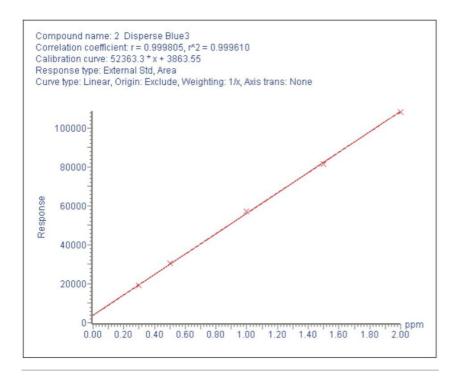


Figure 2. Calibration curve of Disperse Blue 3.

## Conclusion

The ACQUITY UPLC H-Class System with the ACQUITY QDa Detector provides a faster and more reliable analytical tool for the identification and quantification of synthetic dyes. Two methods of less than 5 minutes have been developed for both positive and negative ionizing dyes that provide increased throughput and reduced solvent usage.

The ACQUITY QDa Detector can also be easily integrated into current LC-UV analysis methods which allows unsurpassed sensitivity and selectivity, with limits of detection achieved well below the EU regulatory limit of 5 ppm.

## Featured Products

ACQUITY UPLC H-Class PLUS System <https://www.waters.com/10138533>

ACQUITY QDa Mass Detector <https://www.waters.com/134761404>

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