[TECHNOLOGY BRIEF]

Rapid Analysis of Carcinogenic Aromatic Amines Using the ACQUITY UPLC H-Class SQD System with Empower 3 Software

GOAL

To rapidly analyze 24 carcinogenic aromatic amines at legislated levels using Waters® ACQUITY UPLC® H-Class System, coupled with the ACQUITY® SQ Detector, and Empower™ 3 Software.

BACKGROUND

Azo dyes are used in a wide variety of consumer goods, including leather, clothing, food, toys, medical devices, plastics, and cosmetics. There are more than 3,000 azo dyes that are available in a broad spectrum of colors, and these represent more than 65% of the global dye market.

Some azo dyes can degrade and release the carcinogenic aromatic amines listed in Table 1. The potential health risk of carcinogenic aromatic amine exposure to consumers has led to stricter government regulations worldwide. U.S. FDA regulations 21 CFR 74.705 and 21 CFR 74.706 restrict the use of azo food dyes that could degrade into carcinogenic aromatic amines. EU Directive (2002/72/EC) prohibits the use of food contact materials that release carcinogenic amines. EU Directive (2002/61/EC) bans the use of azo dyes in textile and leather articles, which, upon reduction, form carcinogenic aromatic amines.

Screen samples for carcinogenic aromatic amines 7 times faster than conventional HPLC methods.

THE SOLUTION

Screen samples for carcinogenic aromatic amines 7 times faster than conventional HPLC methods. The ACQUITY UPLC H-Class System, coupled with the ACQUITY SQ Detector and Empower 3 Software, is an LC/MS system that is specifically designed to comply with governmental regulations and QC protocols required by manufacturers and retailers. Using mass spectrometry to improve selectivity, the system is able to monitor multiple SIR channels, thereby tracking peak position, even with co-eluting peaks. This enables a reduction in method development time and provides confidence in the detection of specific analytes. The system performance is automatically monitored, insuring high data quality and more efficient use of the analyst's time.

Empower 3 Software chromatography data system (CDS) provides advanced data storage and security management, and other enhanced features, including audit trails to manage compliance with 21 CFR Part 11 and other GxP requirements.

In compliance with global regulations, and in order to offer consumers safer products, major textile and leather product manufacturers and retailers have issued product certifications and QC protocols that limit the maximum amount of carcinogenic aromatic amines to no more than 20 mg/kg. To be more cost effective, there are growing demands for faster and more accurate analytical methods to test for these aromatic amines in consumer products.

Using the ACQUITY UPLC H-Class System with the ACQUITY SQD, 24 carcinogenic aromatic amines were analyzed in 10 minutes using SIR mode. The SIR chromatograms shown in Figure 1 indicate that the carcinogenic aromatic amines were all easily detected at 20 times below the legislative limit. The structural isomers 2,6-xylidine (G) and 2,4-xylidine (J) were well separated, with retention times of 4.8 and 5.0, respectively.

Compared with the conventional HPLC/PDA method[1], which typically requires 70 minutes for the separation of carcinogenic aromatic amines, this solution requires only 10 minutes, greatly increasing sample throughput. The ACQUITY UPLC H-Class/SQD System can be easily added to laboratories that already use Empower Software, circumventing the requirement of dedicated MS software. Empower users can thereby reap the benefits of MS without the need for additional training.

Label	Compound Name	Cas No.	m/z	RT (min)
A	2,4-Diaminoanisole	615-05-4	139	1.2
B	2,4-Diaminotoluene	95-80-7	123	1.3
C	o-Anisidine	90-04-0	124	3.3
D	Benzidine	92-87-5	185	3.6
E	4,4'-Diaminobiphenylether	101-80-4	201	3.8
F	4-Chloroaniline	106-47-8	128	4.0
G	2-Amino-4-nitrololuene	99-55-8	153	4.3
H	2,6-Xylidine	87-62-7	122	4.8
I	p-Cresidine	120-71-8	138	4.8
J	4,4'-Diaminobiphenylmethane	101-77-9	199	5.0
K	2,4-Xylidine	95-68-1	122	5.0
L	o-Dianisidine	119-90-4	245	5.4
M	2-Naphthylamine	91-59-8	144	5.4
N	4,4'-Diaminobiphenylsulfide	139-65-1	217	5.4
O	Orthotolidine	119-93-7	213	5.4
P	4-Chloro-o-toluidine	95-69-2	142	5.7
Q	2,4,5-Trimethylaniline	137-17-7	136	6.4
R	4,4'- Diamino-3,3'-dimethylbiphenylmethane	838-88-0	227	7.0
S	4-Biphenylamine	92-67-1	170	7.2
T	3,3'-Dichlorobenzidine	91-94-1	253	7.5
U	4-Aminoazobenzene	60-09-3	198	7.6
V	4,4'- Diamino-3,3'-dichlorobiphenylmethane	101-14-4	267	7.7
W	o-Aminoazotoluene	97-56-3	226	8.2
X	o-Toludine	95-53-4	108	8.2

Table 1. 24 Banned carcinogenic aromatic amines listed with their CAS number, mass-to-charge ratios, and retention times.

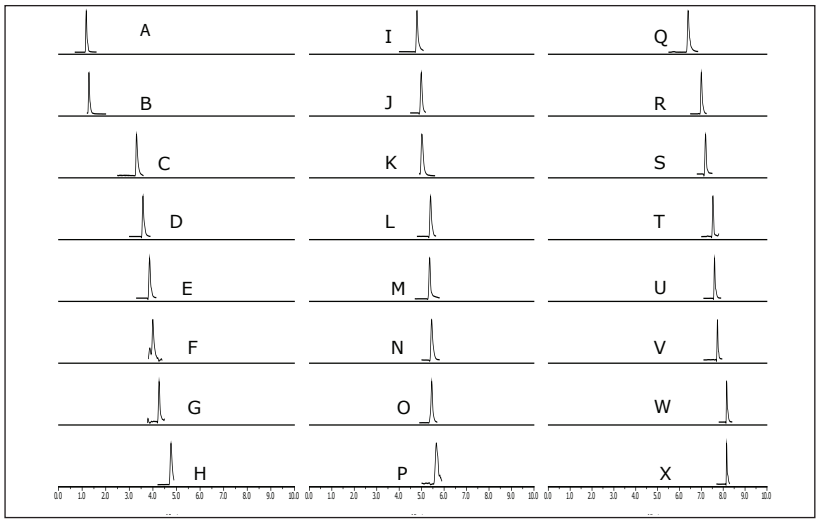

Figure 1. TIC chromatograms of 24 carcinogenic aromatic amines at 1 mg/kg obtained using the following conditions: ACQUITY UPLC H-Class/SQD, ESI+, capillary voltage 3.5 kV, source temp 150 °C, desolvation temperature 450 °C, desolvation gas 900 L/hr, cone gas 10 L/hr, selected ion recording mode (SIR); Column: ACQUITY UPLC BEH C_{18} Column, 2.1 x 50 mm, 0.5 mL/min; column temp 50 °C, sample temp 4 °C. Mobile phase A (10 mM ammonium acetate in water/ methanol: 98/2), mobile phase B (10 mM ammonium acetate in methanol), injection volume 10 µL. Linear gradient method: 5% B for 1 min, increased to 25% B over 2.1 min; then increased to 41% over another 3 min, and finally to 100%, over an additional 1.9 min. Column flushed for 2 min at 100% B. Total gradient time 10 min.

SUMMARY

This work illustrates that the ACQUITY UPLC H-Class System, combined with ACQUITY SQD and Empower 3 Software enables rapid, selective, sensitive, and reproducible analysis of 24 carcinogenic aromatic amines. This 10-minute LC/MS method can be used to screen for the presence of carcinogenic aromatic amines in samples at the regulated limits. The ability to quickly and unambiguously screen samples for carcinogenic aromatic amines can facilitate quality control and regulatory compliance in textile and leather related industries. In addition, with a separation that is seven times faster than the conventional HPLC method, solvent consumption is reduced, and less hazardous waste is generated, resulting in cost and safety benefits. Other industries that have vested interests in the analysis of carcinogenic aromatic amines can also benefit from this methodology. Examples include the cosmetics, personal care products, food, and food packaging industries.

Reference

1. The European Standard EN 14362-1 and 2: 2003.

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