

Routine High Resolution Mass Spectrometry (HRMS) for the Screening of Extractables and Leachables (E&Ls) Using the Waters™ ACQUITY™ RDa™ Mass Detector

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Abstract

This technical brief highlights a targeted workflow for routine HRMS screening using the ACQUITY RDa mass detector with UNIFI™ Software for extractables and leachables analysis. To demonstrate this, a standard mix of eighteen common polymer additives (Extractables and Leachables Screening Standard SKU p/n: [186008063 < https://www.waters.com/nextgen/global/shop/standards--reagents/186008063-extractables--leachables-screening-standard.html>](https://www.waters.com/nextgen/global/shop/standards--reagents/186008063-extractables--leachables-screening-standard.html)) was analyzed.

- Combining the ACQUITY RDa with the UNIFI screening workflow identification and characterization of the sample components and the associated fragments generated, was carried out automatically using libraries within the processing method
- All components were detected and identified with mass measurements less than or equal to 3.1 ppm

accuracy without the need for manual interpretation

- This demonstrates a platform that is capable of providing robust E&L screening characterization for a wide range of analytical expertise.

Benefits

- Routine acquisition of sub 5 ppm mass accuracy for E&L compounds
- Simultaneous acquisition of fragmentation data for additional compound identification confidence
- Access to HRMS data for non-expert users
- Compliant ready UNIFI software as part of the waters_connect informatics platform
- The ACQUITY™ Bridged Ethylene Hybrid™ (BEH™) Column technology enabling chromatographic separation at 70 °C with no column degradation

Introduction

With increasing regulation around the monitoring and characterization of extractables and leachables across numerous areas of industry *e.g.* pharmaceuticals, medical devices, food contact materials (FCM's), and electronic nicotine delivery systems (ENDS), there is in turn a concomitant increased need for access to instrumentation that provides the necessary selectivity to confidently identify these compounds.^{1,2,3,4} HRMS provides this compound characterization capability but can be expensive and require high levels of expertise to operate. With the ACQUITY RDa Mass Detector, HRMS analysis is accessible to both experts and non-experts alike. In combination with the compliant ready UNIFI screening application within the waters_connect™ Software platform this provides a robust platform for routine screening of extractable and leachable compounds.

Results and Discussion

The ACQUITY RDa Detector was set up automatically, including detector, auto-tune, and mass calibration with no requirement for manual intervention enabling the analyst to focus solely on sample analysis, and result generation. Following this routine set up, full scan accurate mass data were acquired in positive mode

incorporating the 'Scheduled Lockmass' function to mitigate any potential mass accuracy shifts due to *e.g.* temperature changes in the laboratory environment. This feature runs a lockmass correction once per hour providing stable mass accuracy with a significant reduction in lockmass solution usage. Using the *Full Scan with Fragmentation* function allows cone voltage ramping to simultaneously acquire high and low energy spectra. The high energy data function, containing fragment ion information, was assigned automatically providing further confidence for compound identification. The E&L standard was injected directly with no sample preparation. Using a BEH C₁₈ Column technology enabled the use of elevated column temperature (70 °C) to aid with separation of the components of the E&L mix without degradation of the column Figure 1.⁵

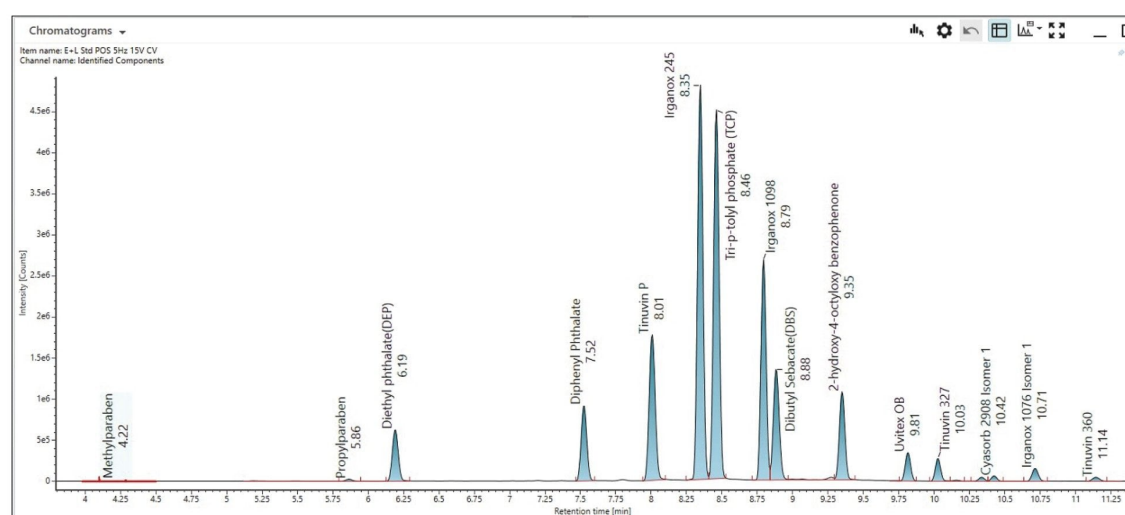


Figure 1. Chromatogram of the Extractables and Leachables Screening Standard.

All compounds were detected and identified with mass accuracy measurements ≤ 3.1 ppm Table 1.

Component	Concentration (µg/mL)	Neutral mass	Measured mass (m/z)	Mass accuracy (ppm)	Retention time (minutes)
2-hydroxy-4-octyloxy benzophenone	0.1	326.1882	327.1966	3.0	9.35
Cyasorb 2908 isomer 1	0.1	474.4073	475.4151	-1.4	10.42
Cyasorb 2908 isomer 2	0.1	474.4062	475.4135	-2.2	10.72
Dibutyl sebacate (DBS)	0.1	314.2455	332.2793	-0.6	8.88
Diethyl phthalate (DEP)	0.1	222.0898	223.0971	2.9	6.91
Diphenyl phthalate	0.1	318.0885	336.1223	-2.1	7.52
Ethanol 330 (Irganox 1330)	1.0	774.5951	792.6288	-0.2	10.25
Irganox 1010	1.0	1176.7803	1194.8141	-3.1	10.16
Irganox 1076	1.0	530.4699	548.5038	3.0	10.71
Irganox 1098	0.1	636.4866	637.4941	-0.7	8.79
Irganox245	0.1	586.3502	604.3841	-0.5	8.35
Methylparaben	0.1	152.0470	153.0543	-0.7	4.22
Propylparaben	0.1	180.0792	181.0865	3.0	5.86
Tinuvin 327	1.0	357.1608	358.1679	-0.4	10.03
Tinuvin 360	1.0	658.3993	659.4065	-0.4	11.14
Tinuvin P	1.0	225.0910	226.0986	2.5	8.01
Tri-p-tolyl phosphate (TCP)	0.1	368.1176	369.1249	-0.3	8.46
Uvinul 3030	0.1	1060.3444	1078.3783	-2.6	8.90
Uvitex OB	0.1	430.1713	431.1786	-0.4	9.81

Table 1. Compounds detected with mass accuracy and retention times listed.

Conclusion

- The ACQUITY RDa has demonstrated the ability to detect and identify all the components of the screening standard. All components were identified with mass accuracy measurements of less than or equal to 3.1 ppm
- With the RDa's and UNIFI as part of waters_connect providing simple intuitive workflows, HRMS measurements for extractables and leachables are achievable to experts and non-experts alike

References

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[ACQUITY RDa Detector <https://www.waters.com/waters/nav.htm?cid=135077027>](https://www.waters.com/waters/nav.htm?cid=135077027)

[UNIFI Scientific Information System <https://www.waters.com/waters/nav.htm?cid=134801359>](https://www.waters.com/waters/nav.htm?cid=134801359)

[waters_connect Software Solutions <https://www.waters.com/waters/nav.htm?cid=135040165>](https://www.waters.com/waters/nav.htm?cid=135040165)

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