

# Detection of Unexpected Contaminants During Routine Chemical Industry QC Monitoring Using Tandem Quadrupole with RADAR Functionality

## GOAL

To carry out QC monitoring for the manufacture of Diethylhexyl Phthalate (DEHP) using tandem quadrupole MS with RADAR™ functionality to scan for unexpected process contamination.

## BACKGROUND

Bulk chemical manufacturing processes, such as DEHP, need to have quick and robust QC checks in place to ensure the purity of both the starting materials and the final product. Producing materials that are out of specification is not only expensive in terms of raw materials, but the potential cost of a plant shutdown and start up can run into millions of dollars. Early detection of contamination while still at a low level may allow corrective action to be taken rather than requiring a full shutdown.

Waters® Xevo® TQD with the ACQUITY UPLC® System allows the monitoring of known compounds using Multiple Reaction Monitoring (MRM), which delivers high sensitivity and selectivity to eliminate the risk of misidentification. Waters RADAR functionality simultaneously acquires full scan data while maintaining the quality of MRM data and allowing post analysis interrogation to identify unexpected contamination if required. This aids in complete understanding of the manufacturing process and sample purity.

The Xevo TQD with RADAR functionality provides MS full scan acquisition without compromising MRM data quality.

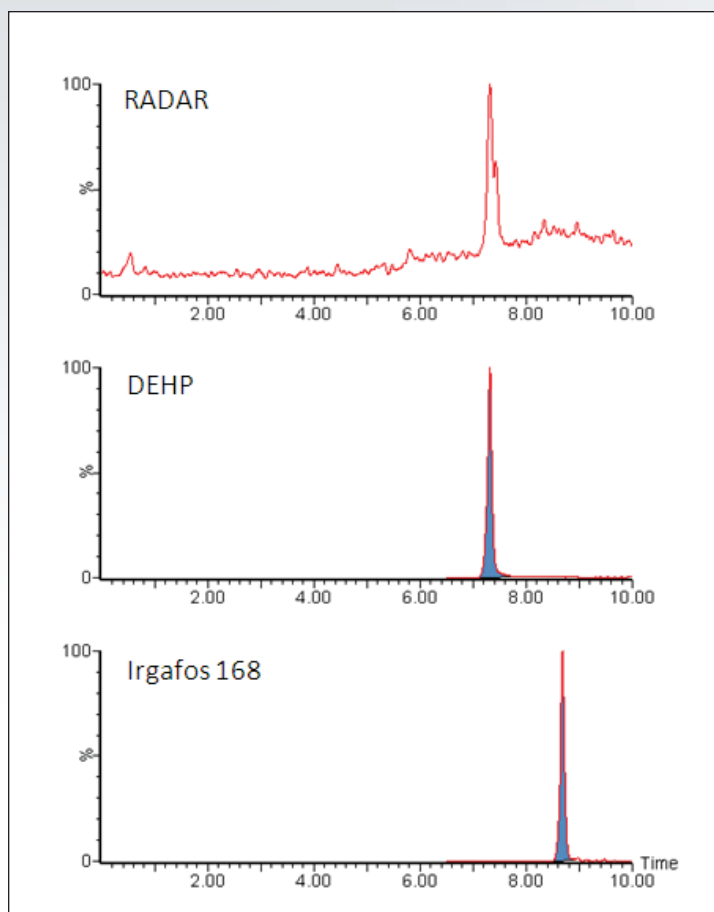


Figure 1. Xevo TQD RADAR full scan Total Ion Chromatogram (TIC) with MRM chromatograms for DEHP (plasticizer) and Irgafos 168 (stabilizer).

## THE SOLUTION

Waters Xevo TQD MS utilizing Atmospheric Pressure Photo Ionization (APPI), coupled to an ACQUITY UPLC System were used to monitor the plasticizer DEHP and the stabilizer Irgafos 168. Compounds of interest can be identified with complete confidence by operating the tandem quadrupole instrument in MRM mode. Many plasticizers, antistatic agents, UV absorbers, stabilizers, and optical brighteners commonly used in the polymer industry can be monitored in this way.

During the monitoring of DEHP and Irgafos 168, RADAR scan functionality was used. RADAR completes a full mass spectral scan every duty cycle during the analytical run without affecting the quality of the MRM data, as shown in Figure 1.

RADAR full scan data were interrogated post analysis and a low level of contamination was found. Figure 2 shows RADAR and the extracted ion chromatogram of mass  $m/z$  587, with the mass spectra for the peak as an insert. Investigation into the mass  $m/z$  587 shows it is likely to be the precursor ion of Irganox 245, a stabilizer commonly used in the polymer industry.

The added information that RADAR provides allows plant managers to make knowledgeable decisions if the product falls out of specification. Contamination that is considered to be "benign" and have no implication on the use of the product could allow an out of specification batch to be blended with other batches. There are clear financial and environmental advantages where this is possible.

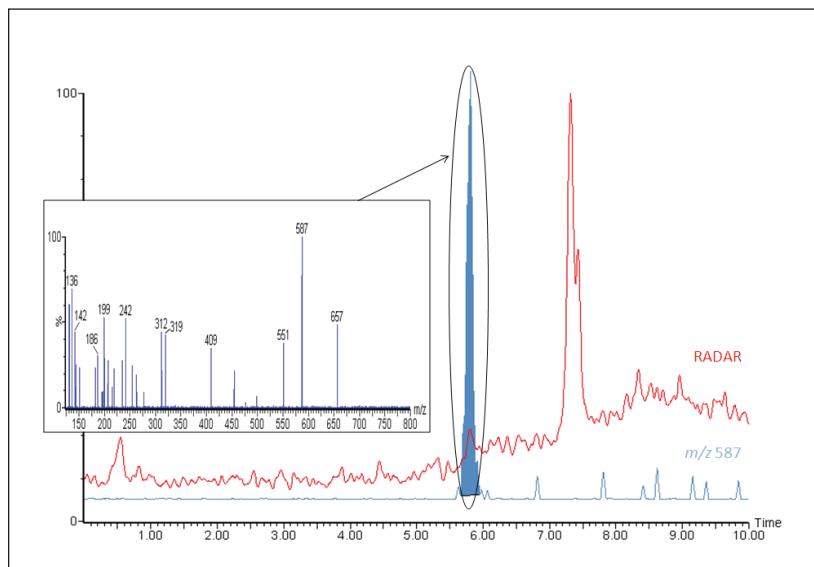


Figure 2. RADAR scan showing DEHP, Irgafos 168, and Irganox 245 (contamination), extracted ion chromatogram of  $m/z$  587 with an insert of the full mass spectrum of the peak. (Chromatograms are not to the same scale).

## SUMMARY

Waters Xevo TQD provides high-quality quantitative MRM data while simultaneously acquiring full mass spectral scans without compromising data quality. Quantification by MRM allows high sensitivity and selectivity, which eliminates the risk of misidentification.

The extra information provided by RADAR functionality offers a better understanding of how the manufacturing process is running. The identification of unknown contamination allows better business and process decisions to be made. If contamination is detected, action can be taken to address the problem and possibly save the batch, which offers significant cost savings to the business.

# Waters

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