

Waters Alliance® LC/MS System



Analysis of Low Level of Surfactant Residues: Manufacturing Process Monitoring

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Key Words

Contamination
Detergent, non-ionic
Electrospray ionization, ESI
Flow injection analysis, FIA
Process monitoring
Sensitivity
Surfactant
ZMD mass detector

Background

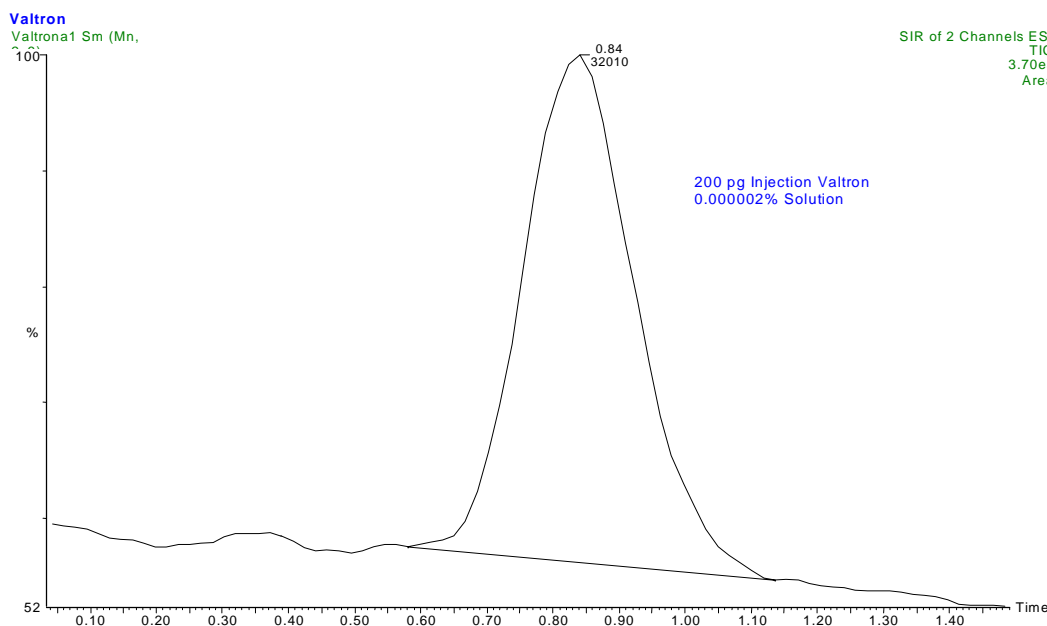
During the production of electronic components, it is very important to remove all traces of chemicals used in manufacturing and cleaning process. Failure to do so can lead to poor performance and component failure. An example of this is the removal of surfactant residues from the surfaces of hard disk drives.

This application was designed to perform flow injection analysis (FIA) of wash solutions to establish the contamination level of a non-ionic detergent. The normal method for this analysis would be to use a wet chemical method with a limit of detection of about 0.2%. This LC/MS method is a much more sensitive (0.00002%) and specific method where a unique mass of surfactant is monitored.

Analytical Conditions

Flow injection analysis (FIA) without a chromatographic separation was used as a rapid monitoring method. The Waters Alliance® HT LC/MS system (2790 Separations Module and ZMD). The mobile phase was 50:50 methanol-water at a flow rate of 0.2 mL/min. Electrospray positive mode was used. A 10 µL injection was made. Full scan mode was used to identify unique ions. SIR mode was then used for maximum sensitivity of detection.

Figure 1: TIC Chromatogram of a 10 µL Flow Injection Peak

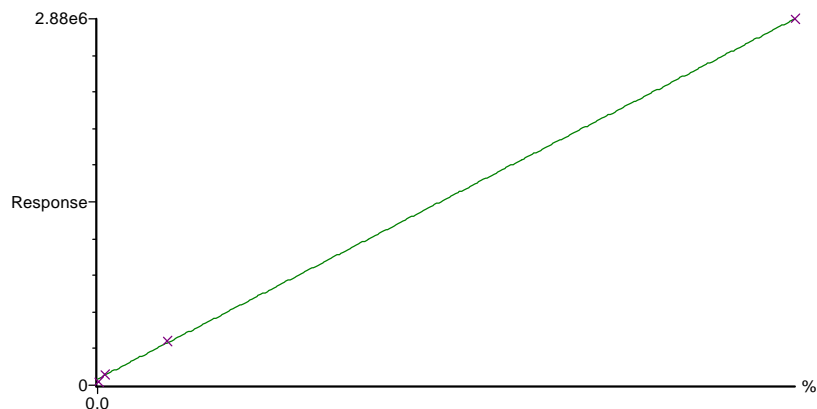


This is the response of a 10 µL flow injection analysis (FIA) of a 0.00002% solution (200pg) of Valtron

Flow injection analysis provides a rapid analytical tool by eliminating the more time consuming chromatographic step. FIA useful for monitoring known compound where the sample matrix is simple. It is not appropriate for all analyses where other compounds may interfere with the analysis, e.g. ion suppression, or where physical separation of the analytes by chromatography is required before mass spectra are obtained.

Figure 2: Calibration Curve

Compound 1 name: Valtron
Correlation coefficient: $r = 0.999928$, $r^2 = 0.999855$
Calibration curve: $1.41709e9 * x + 49577.3$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: Null, Axis trans: None



A linear response can be obtained over several orders of magnitude by SIR monitoring on one of the major peaks unique to the surfactant (see Figure 3). A correlation coefficient r^2 of 0.999 was obtained.

Figure 3: Full Scan Spectrum of a Non-ionic Detergent

VAL1 64 (1.257) Cm (62:71-39:55)

Scan ES+
5.55e5

