

Automated Liquid Liquid Extraction of Epichlorohydrin using GC/MS

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Introduction

Epichlorohydrin is a widely used raw material used in the manufacturing process of plastics and other polymers, but also water treatment resins. It has the potential to leach from epoxy resin coatings on water pipes, or via flocculation processes within water treatment. The World Health Organisation (WHO) has a provisional guideline value for drinking water 0.4 ug/L (2004). Figure 1 shows the structure of Epichlorohydrin.

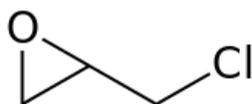


Figure 1 – Structure of epichlorohydrin

The current method for epichlorohydrin at ALS Environmental is a manual method whereby 1 mL of DCM is added to 10 mL of water shaken. The extract is then dried with sodium sulphate. The reproducibility at AQC level is at 13% RSD.

An automated Liquid Liquid method has been developed for epichlorohydrin using the MultiPurpose Sampler with mixing and centrifugation capability by John Quick at ALS Environmental.

Figure 2 shows the extract of the aqueous-organic mixture after it has been centrifuged at 4500 rpm. The vial is a high recovery vial enabling withdrawal of a low volume of the dichloromethane from the bottom of the vial. In this instance, 300 µL was used.



Dichloromethane after centrifugation

Figure 2 – High recovery vial with low volume of the dichloromethane present at the bottom of the vial

Instrumentation

Dual Head GERSTEL MPS
GERSTEL mVORX
Agilent GC/MS 6890/5975
Anatune CF-200 Centrifuge
High recovery 9.5 mL vial Anatune

Method

9 mL of deionised water was added to a high recovery vial, followed by 300 µL of DCM. This was shaken using a GERSTEL mVorx for 60 seconds. The vials were then centrifuged for 90 seconds to separate the layers. 100 µL was then removed from the DCM layer and added to an empty GC vial ready for analysis. The deionized water was spiked with an epichlorohydrin standard, giving a calibration range of 0.2 to 10 µg/L. In total, the procedure took slightly more than 20 minutes to prepare six samples on the MPS. It is important to note that the centrifuging was done in batch, to save time. The MPS prepsequence is shown in Figure 3.

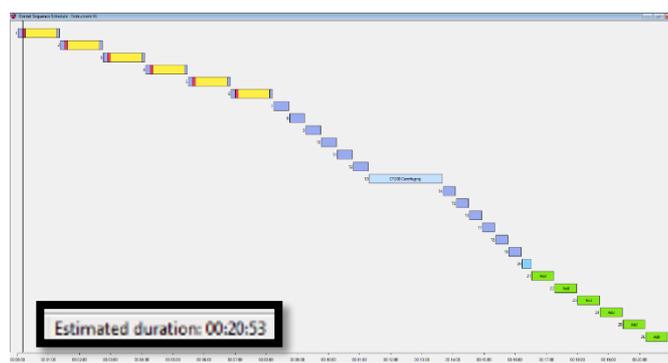


Figure 3 – The MPS prepsequence schedule for 6 samples. The total prep runtime is shown as 20:53.

Results

Figure 4 shows linearity plot for epichlorohydrin in water for concentrations between 0.2 and 10 µg/L. The calibration shows a linear response across the measured range, with an R² of 0.9997.

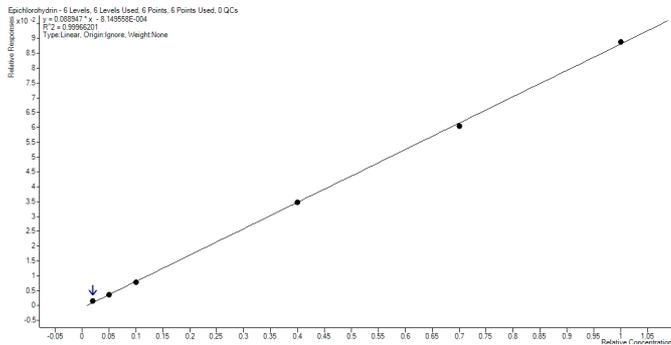


Figure 4 – Linearity for epichlorohydrin

Figure 5 shows extracted ion chromatogram for epichlorohydrin (Retention time 2.39 minutes) for a blank, the lowest, and mid calibration levels respectively.

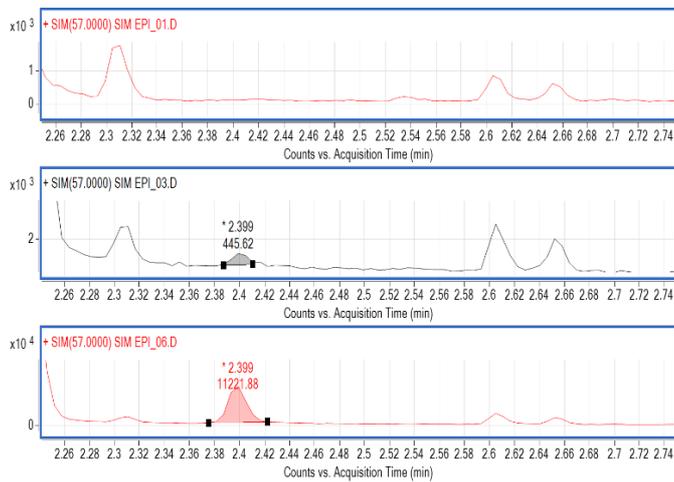


Figure 5 – Extracted ion chromatogram epichlorohydrin (spiked into water at 0.0, 0.2 and 1 µg/L respectively).

The method has been validated to NS30 for drinking water. The drinking water recovery at 8 µg/L has been calculated as 103.6%, with an RSD of 4.7%. This represents a large improvement on the current method.

Discussion

This application note has demonstrated a new approach to measuring epichlorohydrin. The advantages of using this approach are threefold. The automation of manual tasks leads to greater productivity, and sample turnaround. The addition of automated steps also leads to increased data quality, as there is no chance for human error, or analyst variability. Finally, with the use of small volumes of DCM, greater sensitivities can be achieved, again due to the precision of automating the method. Additionally, the analysts' safety is improved due to the much lower levels of DCM used.