Chromatography Technical Note No AS131

Determination of Acetaldehyde in water from plastic bottles using Hot Injection Trapping (HIT) technique on GC-MS

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Introduction

Acetaldehyde is a volatile, colourless liquid with a high fruity odour. It is commonly used as intermediate in the manufacture of some industrial chemicals. It is used as solvent in the rubber, tanning and paper industry, and as a preservative for fruits and fish. It is also used as flavouring.
Acetaldehyde is known to react with many parts of the human body. It causes irritation of the respiratory tract and has been classified as possibly carcinogenic to humans by the International Agency for Research on Cancer.

Within this application note, we show how Hot Injection Trapping (HIT) can be fully automated to enrich acetaldehyde from water samples using a static headspace technique. The HIT technique enables the trapping of multiple headspace injections from the same sample on a cold inlet and consequently improves limits of detection (LODs).

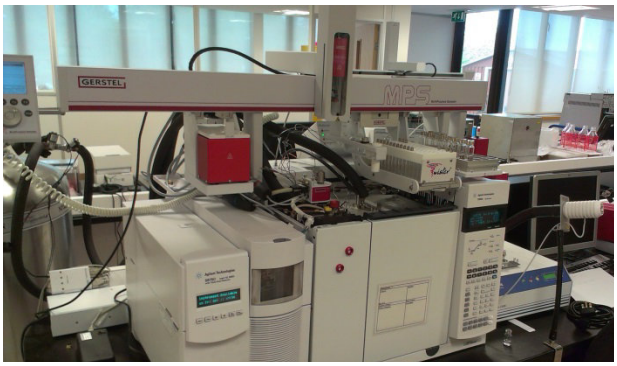

Figure 1: Gerstel Single Head MPS with GC/MS Single Quadrupole

Figure 2 shows a more detailed photograph of the inlet and the headspace unit. The head is configured with a 2.5 ml headspace syringe and injects in a TDU tube with a transport adaptor for liquid injection.

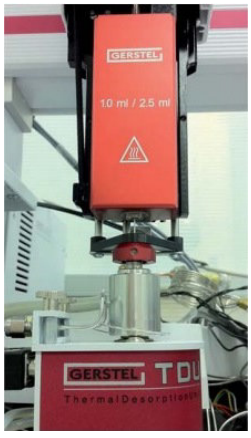

Figure 2: Detail photograph of a HIT injection

Instrumentation

Agilent GC 7890A and Agilent MSD 5975C inert with EI source
Gerstel MPS 2 XL-*xt*
Gerstel CIS4, TDU & Headspace kit
Agilent MSD Chemstation software (version E.02.02.1431)
Maestro software integrated (version 1.4.18.25/3.5)

Method

Headspace parameters:

Incubation at 70°C for 30 minutes
Sampling 5 x 2 ml of headspace

GC-MS parameters:

TDU hold at 150°C
CIS 4 (Tenax liner) : -40 °C ramped to 250 °C
Column: DB-WAX 30 m x 0.25 mm x 0.5 µm
Thermal gradient: 40°C (4 minutes); 10°C/min to 100°C
MS: EI source, SIM performed using ions 29 (Acetaldehyde) and 43 (Ethyl Acetate, used as internal standard)

Hot Injection Trapping (HIT) procedure:

The Headspace syringe, containing the headspace extract, is inserted into the TDU which is kept heated at 150°C to prevent cold spots where the analytes might be adsorbed. The analytes are trapped in the Tenax packed CIS4 liner at -40°C, enabling the use of multiple injections: 5 injections of 2 ml headspace extracts.

An eight point calibration for Acetaldehyde was prepared in water at concentrations ranging from 1 µg/l to 100 µg/l, keeping Ethyl Acetate (used as an internal standard) consistent at 50 µg/l.

Results

Below (Figure 3) shows the calibration plot for Acetaldehyde in water, corrected by internal standard. A correlation coefficient of 0.999 was achieved.

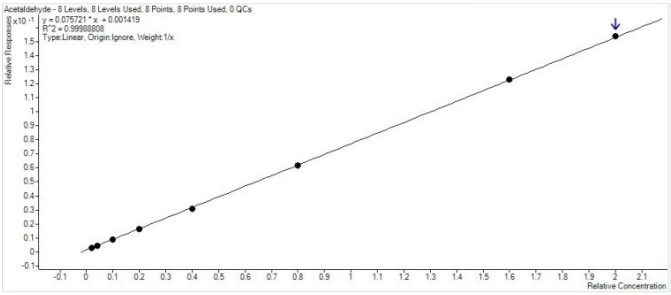

Figure 3: Linearity plot of Acetaldehyde in water

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Figure 4 shows a comparison between a blank and Acetaldehyde at 10 µg/l.

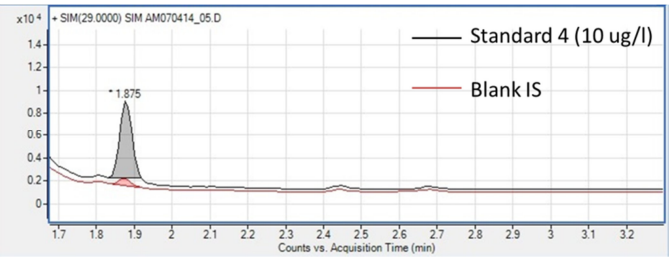

Figure 4: SIM chromatogram (ion 29) comparison of a Blank and the calibration level 4 in water (Acetaldehyde 10 µg/l)

Figure 5 shows a comparison between a HIT performed once (one injection of 2ml headspace extract) and a HIT performed 4 times (4 x 2ml headspace extract). The sensitivity is proportionally increased.

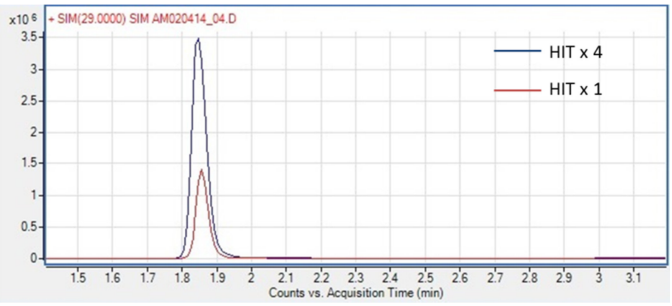

Figure 5: SIM chromatogram (ion 29) comparison of HIT performed once and HIT performed 4 times

Reproducibility experiments have been carried out on 4 aliquots of water spiked at 5 µg/l and the correlation coefficient (corrected with internal standard) has been calculated to be 2%.

Water samples from plastic bottles were analysed using this method and acetaldehyde levels were recorded and results were given to our customer.

Table 2 shows this reproducibility data.

	Acetaldehyde
Amount spiked (µg/l)	5.0000
Amount detected (µg/l)	4.8338
	4.8827
	4.8476
	5.0706
Mean (µg/l)	4.9087
SD	0.1099
%CV	2.24

Table 2: Reproducibility for Acetaldehyde in water using HIT-Headspace technique

Discussion

This application note shows how the Hot Injection Trapping (HIT) technique enables the quantification of Acetaldehyde present in water down to 1 µg/l.

Further work is due to be completed on real samples from a variety of sources, to establish method robustness and variability. Additionally recovery and precision in these "real" samples will be determined via standard addition.

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