

THE USE OF HEADSPACE WITH SAMPLE CONCENTRATION TO MEET ALBERTA TIER 1 VOC GUIDELINES

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Abstract

This application note describes the use of a sample concentration device in conjunction with a headspace sampling system to achieve the detection limits for VOC analysis as listed in the Alberta Tier 1 Soil and Groundwater Remediation Guidelines. This analysis has traditionally been done using Purge&Trap (P&T) sampling to meet the low detection limits required. Headspace sampling has been thought to be not sensitive enough for this analysis. Some compounds such as Vinyl Chloride and Carbon Tetrachloride have especially challenging detection limits (0.34 ppb and 0.56 ppb respectively) and have been looked upon as to the measure of how good your analytical method is. We will concentrate on these two compounds in this application note. This application note shows that using a CTC CombiPAL system with ITEX accessory allows the use of headspace sampling to meet the guidelines for Alberta Tier 1 VOC analysis.

Introduction

VOC analysis has been done typically using a Purge and Trap device coupled to a GC/MS to achieve required detection limits. Purge and Trap systems are somewhat difficult to use, have the potential for carryover contamination issues which can cause downtime, they tend to be the least reliable component in the system and they are expensive to purchase. Headspace sampling devices are relatively easy to use, have minimal potential for carryover contamination, have been shown to be reliable and cost less than a comparable Purge and Trap sampling system. It is these features of headspace sampling which are pushing people to explore the technique for traditional P&T methods.

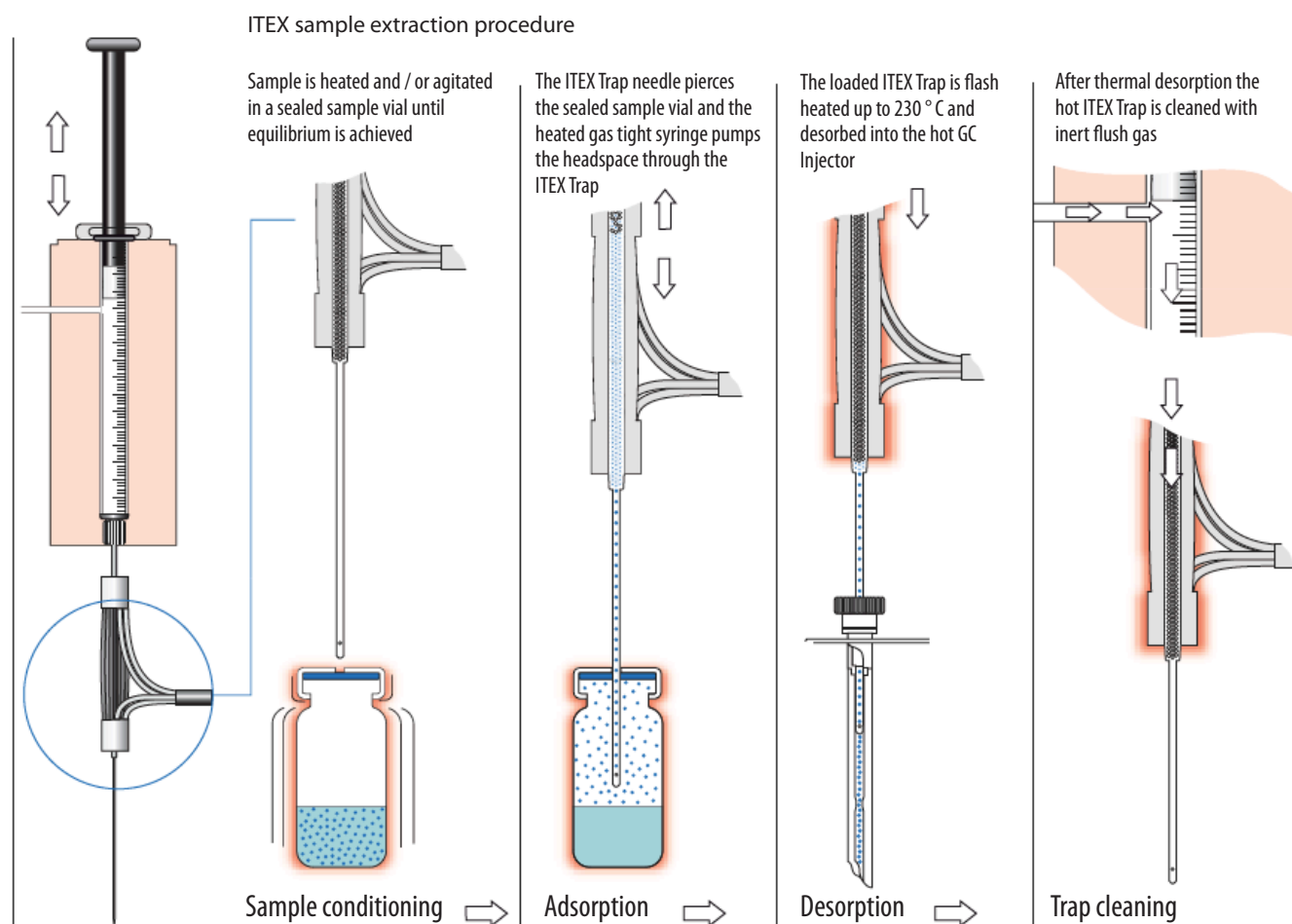
This application note describes how K'Prime has successfully met the Alberta Tier 1 Soil and Groundwater Remediation VOC Guidelines using a modified headspace sampling device.



Experimental

The CTC CombiPAL headspace system was used. The system was modified with the ITEX Option from CTC Analytics. The ITEX option consists of an add-on module which can be used with any existing or new CombiPAL system. It performs enrichment of volatile or semi-volatile compounds during headspace analysis. A microtrap filled with adsorbent material, such as Tenax or activated charcoal is placed between the CombiPAL Headspace syringe and the syringe

needle. Using the HS syringe as a pump, a part of the gaseous phase of the samples is pumped repeatedly through the microtrap. To gain sensitivity the number of pumping strokes can be increased or several different vials containing the sample can be extracted. During thermal desorption into the GC injector the microtrap is rapidly flash heated. Compounds reach the GC column as a narrow band no cryofocussing is needed to obtain sharp peaks.



* An industry standard Agilent 7890 Gas chromatograph coupled to a Agilent 5975 VL MSD with Triple Axis Detector was used with the CombiPAL headspace sampling system.

** After several different column selections, it was found that the Agilent VF-624ms 30m x 0.25 diameter x 1.4 um film thickness (p/n CP910215) produced the best results for this application.

*** The K'Prime Solutions Group has developed a custom macro for this ITEX application. Without this macro you may experience split peaks or poor chromatography. This macro is available as part of the K'Prime Solution.

Operating Conditions

ITEX

Syringe Temp 65°C
Incubation Temp 60°C
Incubation Time 5min
Extraction Volume 1000ul
Extraction Strokes 20
Desorption Temp 200°C
Injection Volume 250ul

Agilent 7890 Conditions

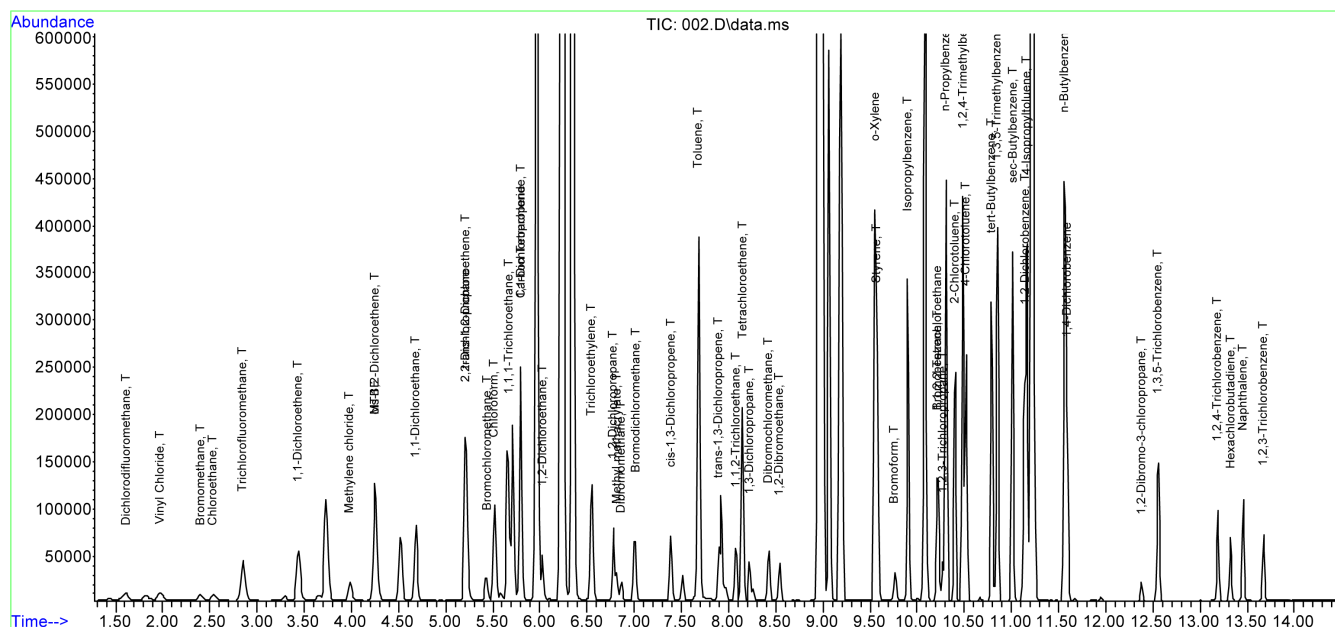
Initial 30 °C with hold of 2min.
Ramp 15 °C/min to 225 °C
Oven Final hold time of 3min for bake out at 225 °C

Agilent 5975 Conditions

Interface 250°C
SIM mode
GAIN 1

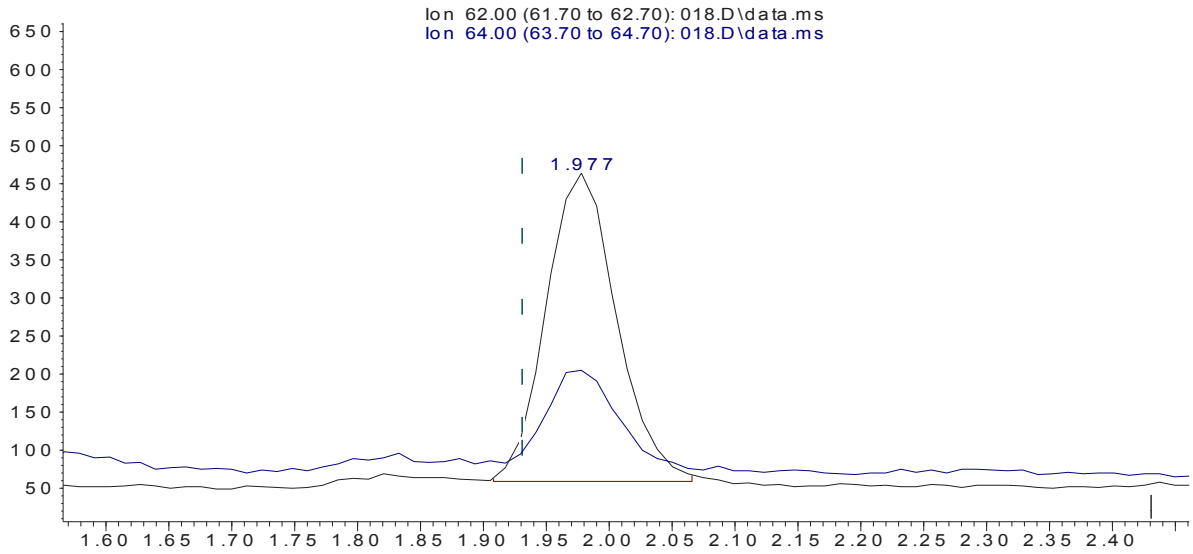
Results and Discussion

As mentioned in the Abstract, the detection limits for Vinyl Chloride and Carbon Tetrachloride are particularly challenging for analysts. The described system with ITEX sample concentration option was able to detect these compounds at limits lower than described in the Alberta Tier 1 regulations.



Integration of Vinyl Chloride peak at 0.100ppb

Abundance



Time-->

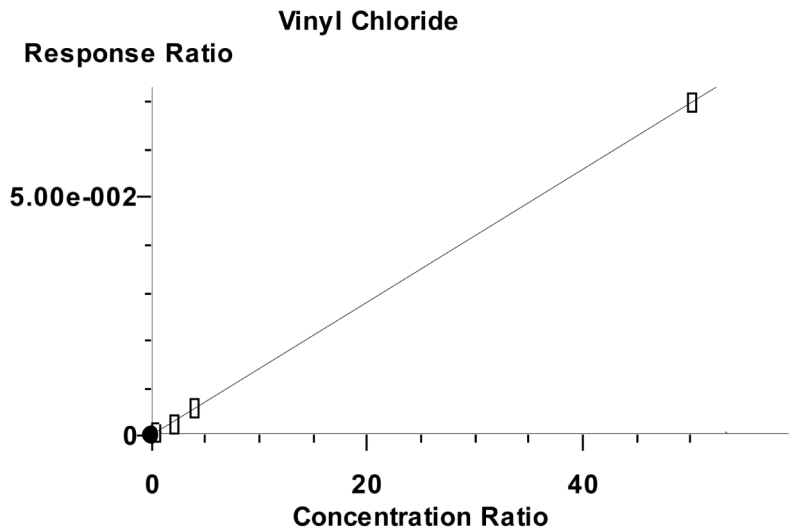
Vinyl Chloride Curve using ITEX system

4 point calibration for vinyl chloride

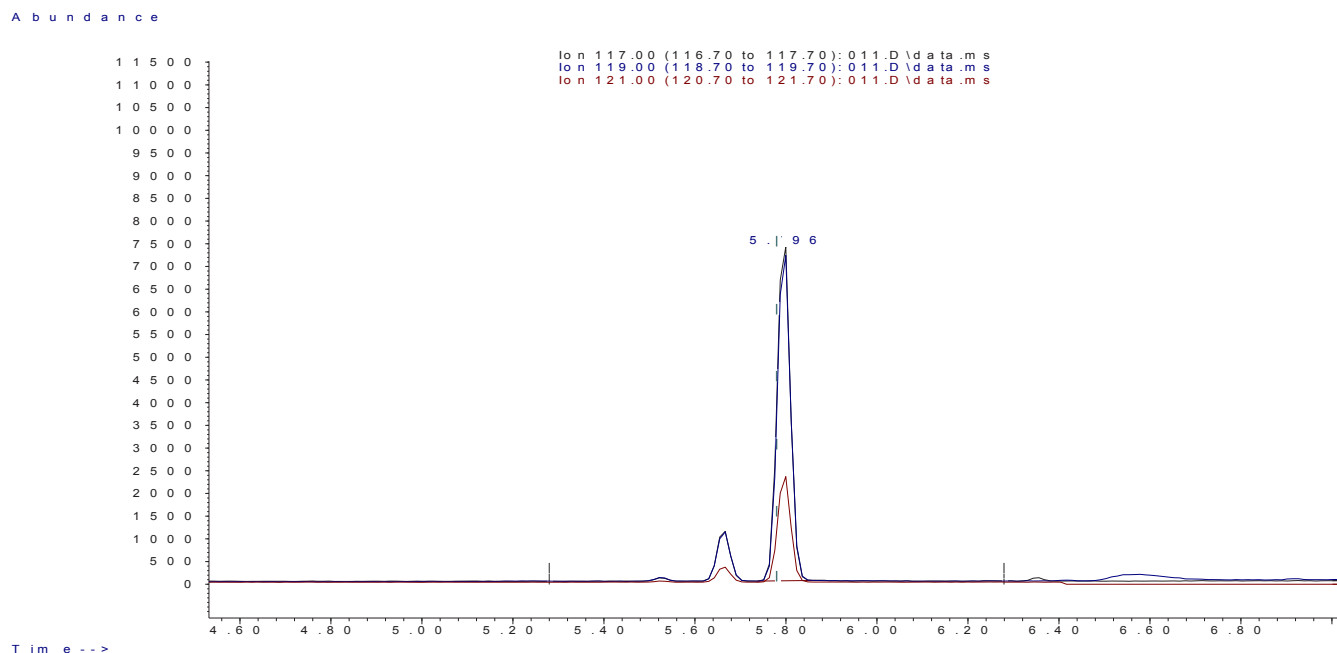
Linear Regression = 0.999371

Curve included levels from 50ppb to 0.4ppb

The check standard was calculated to be 0.110ppb and 0.090ppb respectively.



Integration of Carbon Tetrachloride peak at 0.400ppb



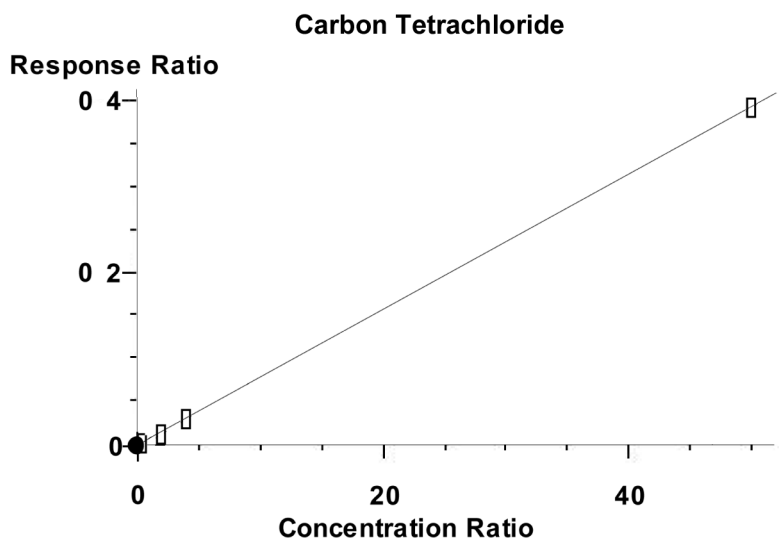
Carbon Tetrachloride Curve using ITEX system

4 point calibration for Carbon Tetrachloride

Linear Regression = 0.999884

Curve included levels from 4.00ppb to 0.4ppb

The check standard was calculated to be 0.410ppb and 0.410ppb respectively.



Conclusion

We have successfully shown that a CTC CombiPAL headspace system with the ITEX sample concentration option coupled with an Agilent 7890/5975 GC/MS can be used to successfully meet the Alberta Tier1 Soil and Groundwater Remediation VOC Guidelines. With the introduction of the Agilent 5977 MSD system which has up to a 10X sensitivity gain over the system used in this note, we are even more confident that the ITEX option will prove to be a robust easy to use solution for VOC analysis.

Acknowledgments

K'(Prime) Technologies would like to thank the local environmental laboratory community who provided us with standards and method advice to help make this possible.

References

1. Alberta Tier 1 Soil and Groundwater Remediation Guidelines, December 2010.
2. ITEX Brochure - CTC Analytics AE; Industriestrasse 20 CH-4222; Zwingen Switzerland

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