

TO-17 Analysis using the CDS TDA 9300 Autosampler

TO-17 is the EPA method for the determination of VOC's in ambient air by pulling of a known volume of air through a tube packed with a variety of sorbent materials. The sorbent tube is then thermally desorbed and analyzed using gas chromatography and mass spectrometry. The detection limits for all VOC's in air range from 0.5ppb to 2ppb, with a linear range up to 200 ppb. The list of compounds includes the gases (dichlorofluoromethane, vinyl chloride), heavier alkyl halides (chloroform, trichloroethylene), as well as the aromatics (benzene through trichlorobenzene). A multibed sorbent tube is used to collect these compounds, with the higher molecular weight compounds retained on the front sorbent and the gases retained on the later, stronger adsorbents.

A cylinder containing 65 compounds at 1 ppm concentration was purchased from Restek. A one liter Tedlar bag was filled to capacity with the compound mixture. One end (non frit or exit end) of a standard six inch Tenax/Carboxen 1000/Carbosieve SIII packed thermal desorption tube was attached to a portable vacuum pump. The other end was attached to the one liter Tedlar bag. The flow rate was monitored using an accurate flow meter integrated within the vacuum system, set for 200ml/min for 5 minutes. The tube was then thermally desorbed using the CDS 9300 TDA which was interfaced to a GC/Ion Trap. Figure 1 shows the overlay of a calibration from 5 ppbv to 100ppbv of the TO17 standard.

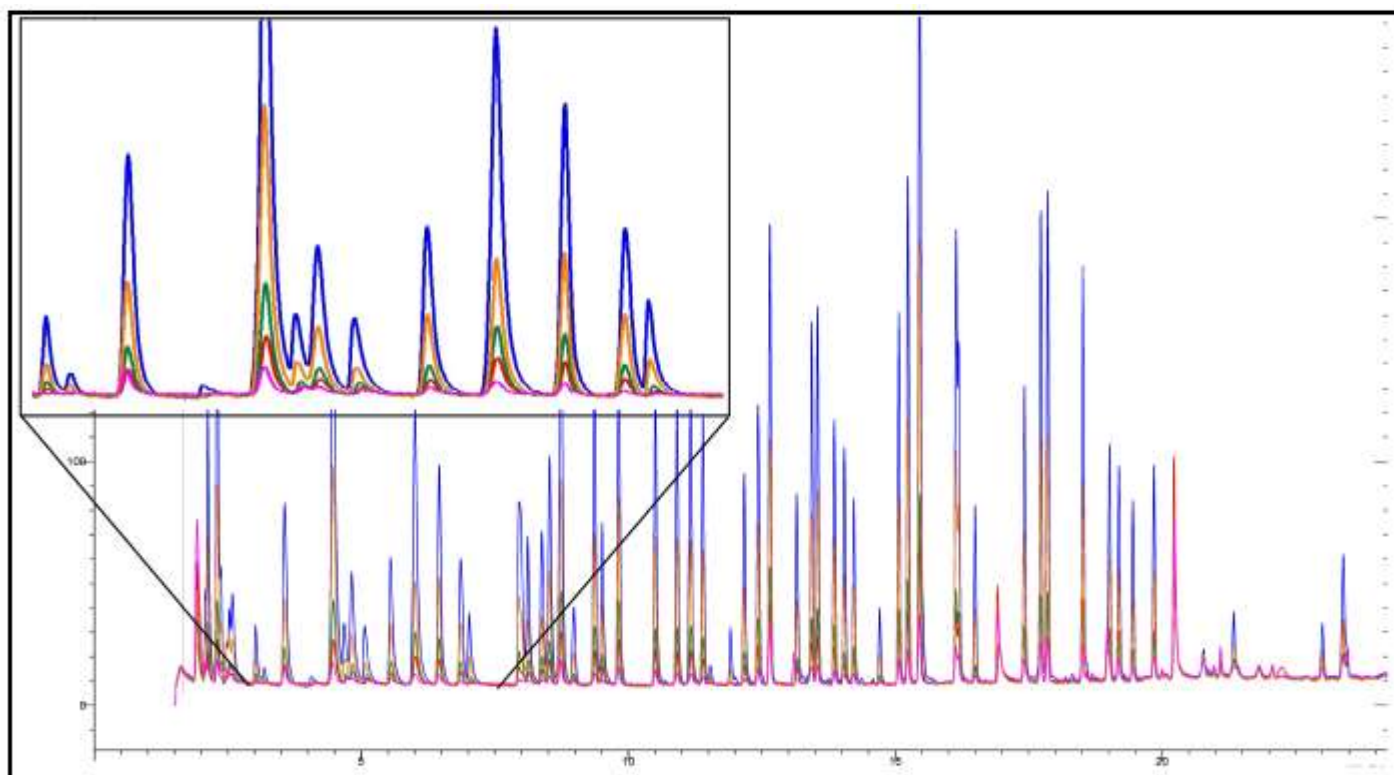


Figure 1. TO-17 standard (65 components), 5 ppbv through 100 ppbv.

Linearity

The inset in Figure 1 expands an overlay of the same compounds at various concentrations from 5 to 100 ppbv. The technique provides excellent linearity, as shown in the calibration curves for dichlorofluoromethane and toluene in Figure 2.

Equipment

These samples were analyzed using the CDS TDA 9300 interfaced to the Varian CP 3800 Gas Chromatograph. The detector used was the Varian Saturn 2000 Ion Trap.

CDS TDA 9300 Conditions

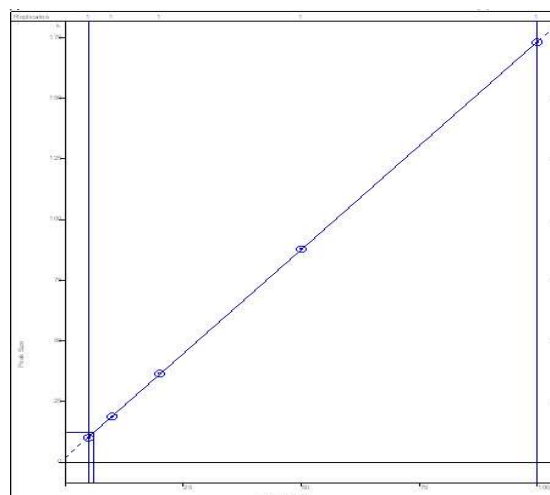
Valve Oven: 300°C
Transfer Line: 300°C
Dry Tube: 35°C/1min
Tube Heat: 350°C/5min
Tube Cool: 0.5 minutes
Trap Idle: 40°C
Trap Heat: 325°C/5min

Interconnect Line: 300°C

GC Conditions:

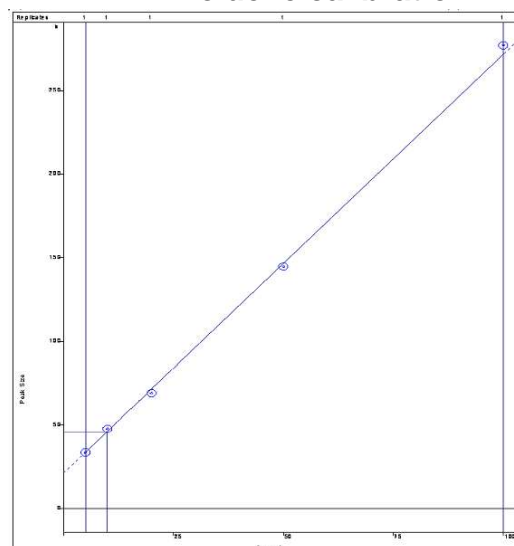
Carrier: Helium
Column: CP-Select 624
(30m x 0.25mm x 1.4µm)
Detector: Ion Trap
GC Program: 30°C/3.20min
160°C/11°C hold 1 minute
220°C/11°C/3min hold

Dichlorodifluoromethane calibration



Linearity Corr: $R^2: 0.999519$
%RSD : 6.37

Toluene calibration



Linearity Corr: $R^2: 0.999469$
%RSD : 8.24

Figure 2. Linearity for Toluene and Dichlorodifluoromethane

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