

Analysis of VOC Gases and TO-1/TO-2 Standards via Dynatherm 9300 TDA Using VOCGAS Focussing Trap

A novel analysis for VOC gases and TO1, TO2 standards without cryogen has been developed. The analysis involves the use of a newly developed 2mm focussing trap without the use of either cryogen or Peltier cooling. The standard desorption protocol has been modified by adding a predesorption step to the Dynatherm 9300 thermal desorption system.

The concentrations of the VOC gases as well as the TO-1/TO-2 standards was 100ng/ul. One microliter of each analyte was spiked onto a 6mm sorbent tube containing Tenax TA, Carboxen 1000, and Carbosieve SIII. The spiked sample tube was placed on the Dynatherm 9300 TDA Autosampler. They were desorbed at 325°C/3min to the newly designed 2mm focussing trap (idle at 35°C). The trap was desorbed at 300°C/5 min at a helium flow of 48mls/min. The CDS 9300 TDA was interfaced to a Varian 3800 Gas Chromatograph and the detector was the Varian Saturn 2000 Ion Trap. The column used was a CP Select 624 (30m x 0.25mm x 1.4µm). The GC program was 30°C/3.2min, 11°C/min to 160°C (Figure 1) Another identically spiked tube was run with the same program, but through a GC Cryo oven starting at -50°C (Figure 2).

Figure 1 shows the VOC gases as well as the TO1/TO2 standards captured by the 2mm focussing trap. It should be noted, that ions created by the ion trap from the methanol, hide and does not permit detection of chloroethane or bromomethane.

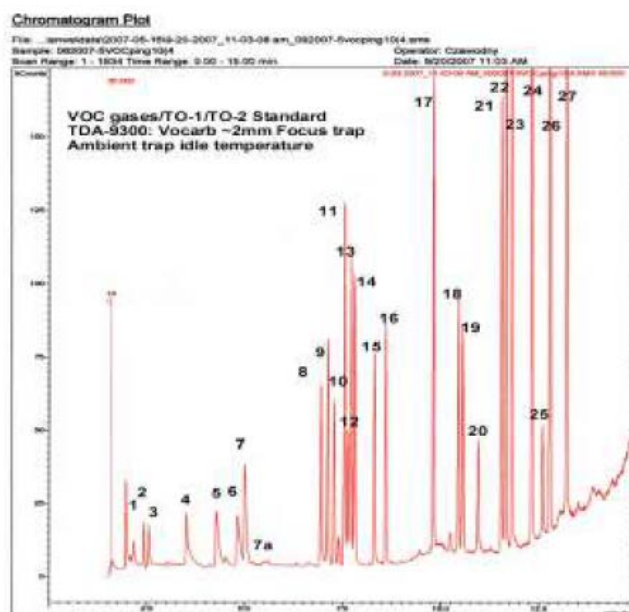


Figure 1 - VOC Analysis (Std conditions)

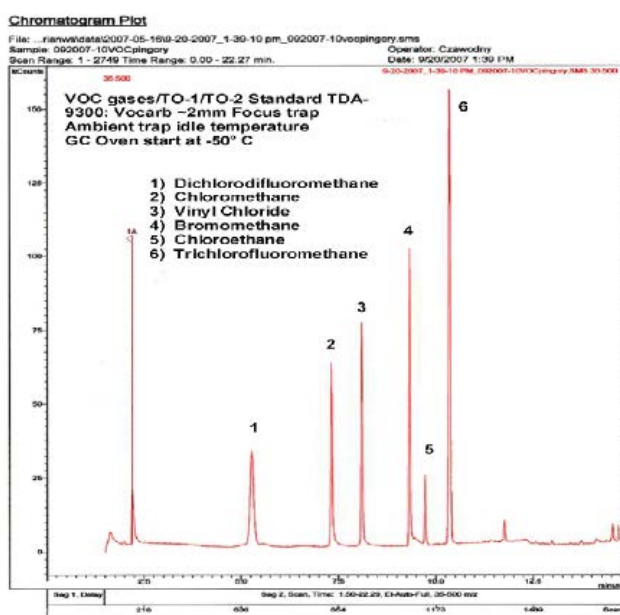


Figure 2 - VOC Gas (-50°C oven start)

Figure 2 shows the same VOC completely resolved using the same 2 mm focussing trap except with an initial column temperature of -50°C. Under these conditions the Chloroethane and Bromomethane are sufficiently separated from the Methanol, and no interference is seen.

CDS 9300 TDA Conditions

Valve Oven: 130°C
Transfer Line: 130°C
Tube Idle: 35°C
Dry Tube: 35°C
Tube Heat: 325°C
Tube Cool: 0.5min
Trap Idle: 35°C
Trap Heat: 300°C
Interconnect Line: 130°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, 11°C/min to 160°C

Figure 1 Identifications

- 1.
2. Chloromethane
3. Vinyl Chloride
4. Trichlorofluoromethane
5. 1,2-Dichloroethene
6. Allyl Chloride
7. Methylene Chloride
8. Chloroform
9. 1,1,1-Trichloroethane
10. Carbon Tetrachloride
11. Benzene
12. 1,2-Dichloroethane
13. Heptene
14. Heptane
15. Trichloroethene
16. 1,2-Dichloropropane
17. Toluene
18. Tetrachloroethylene
19. 1,3-Dichloropropane
20. 1,2-Dibromomethane
21. Chlorobenzene
22. Ethyl Benzene
23. o,m-Xylene
24. p-Xylene
25. Bromoform
26. Isopropyl Benzene
27. Bromobenzene

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