

CDSolutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Purge and Trap - The Gases

Although purge and trap analyses can include compounds ranging from dichlorodifluoromethane all the way to trichlorobenzene, perhaps the most difficult from a chromatographic standpoint are the first six compounds - the gases - which include vinyl chloride and bromomethane. Figure 1 shows these six compounds purged from water at a concentration of 0.5 PPB. Water samples were purged with helium for 11 minutes at 40 ml/minute to a multi-bed sorbent trap, which was then desorbed to the GC/MS. The figure shows total ion chromatograms, and seven runs overlaid to indicate reproducibility. For replicate runs, standard deviations average about 6%.

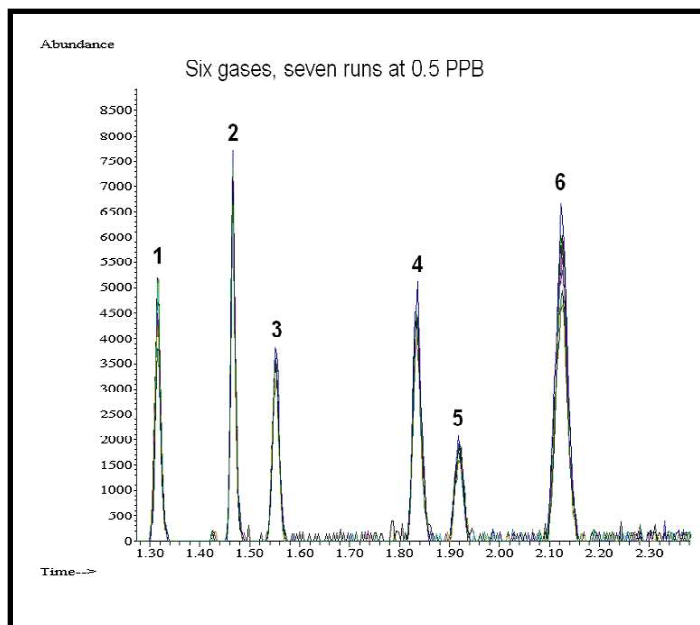


Figure 1. Multiple runs of the six gases at 0.5 PPB. Peaks identified in the tables on the back.

The CDS Model 7000 Purge & Trap includes a method step called trap preheat which begins the trap desorption process before gas flow from the GC is introduced. This permits rapid transfer of the very early eluters in a way that increases resolution and peak height, making peaks for the gases, even at sub-PPB levels, sharp and well separated. Cryogenics are not used or needed, either for focusing the analytes before chromatography, or for the GC oven.

As shown in Figure 2, linearity for these compounds is also excellent. This figure plots the peak area ratio of the vinyl chloride peak to the internal standard fluorene over the range from 0.5 to 50 PPB. Details of reproducibility, linearity and detectability, as well as full operating parameters for both the CDS 7000 and the GC/MS may be found in the tables on the reverse side of this sheet.

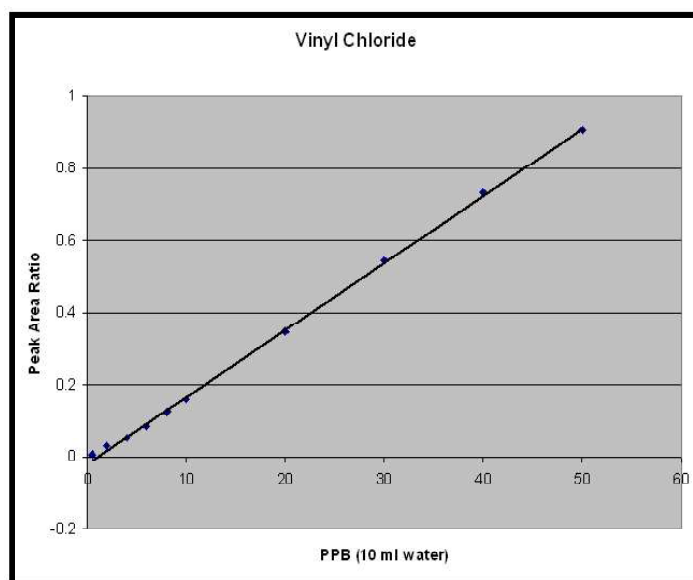


Figure 2. Linearity for Vinyl Chloride from 0.5 to 50 PPB.

Equipment Parameters

CDS 7000

Purge Volume: 10 ml
 Purge time: 11 minutes
 Purge Flow: 40 ml/minute
 Desorb pre-heat: 245°C
 Desorb: 250°C for 2 minutes
 Trap Bake: 260°C for 10 minutes
 Valve oven: 130°C
 Transfer Lines: 130°C
 Wet Trap Ready: 50°C
 Wet Trap Bake: 260°C

GC/MS

GC/MS: Agilent 6890 with 5975B
 Column: Varian CP 624 CB
 30 m, 0.25mm, 1.4 um
 Flow Rate: 1.3 ml/min
 Split ratio: 40:1
 Program: 40°C for 5 minutes
 Ramp: 10°C/minute
 Final: 180°C

Linearity:

| Compound | 0.5 | 1 | 2 | 4 | 10 | 20 | AVG | %RSD | Linearity | |
|--------------------------|-------------------|-------|-------|-------|-------|-------|-------|------|-----------|--|
| FluoroBenzene (IS) | Internal Standard | | | | | | | | | |
| 1 Dichlorofluoromethane | 0.260 | 0.225 | 0.363 | 0.331 | 0.304 | 0.295 | 0.296 | 16.6 | 0.9984 | |
| 2 Methane, chloro- | 0.404 | 0.370 | 0.467 | 0.426 | 0.419 | 0.422 | 0.418 | 7.6 | 0.9998 | |
| 3 Vinyl Chloride | 0.388 | 0.318 | 0.387 | 0.355 | 0.335 | 0.319 | 0.350 | 9.0 | 0.9990 | |
| 4 Methane, bromo- | 0.281 | 0.237 | 0.279 | 0.254 | 0.261 | 0.257 | 0.262 | 6.3 | 0.9998 | |
| 5 Chloroethane | 0.202 | 0.185 | 0.188 | 0.181 | 0.176 | 0.169 | 0.184 | 6.3 | 0.9996 | |
| 6 Trichlorofluoromethane | 0.622 | 0.569 | 0.597 | 0.537 | 0.502 | 0.482 | 0.551 | 9.8 | 0.9994 | |
| 1,2-Dichlorobenzene (S) | 0.242 | 0.264 | 0.243 | 0.244 | 0.265 | 0.276 | 0.256 | 5.8 | --- | |

Reproducibility

| Compound | S-80 | S-81 | S-82 | S-83 | S-84 | S-87 | S-88 | SDEV | MDL |
|--------------------------|------|------|------|------|------|------|------|------|------|
| FluoroBenzene (IS) | 6214 | 6037 | 5965 | 6043 | 5808 | 5835 | 5789 | --- | --- |
| 1 Dichlorofluoromethane | 0.66 | 0.66 | 0.66 | 0.58 | 0.58 | 0.54 | 0.43 | 0.08 | 0.25 |
| 2 Methane, chloro- | 0.49 | 0.48 | 0.50 | 0.51 | 0.48 | 0.49 | 0.48 | 0.01 | 0.03 |
| 3 Vinyl Chloride | 0.53 | 0.58 | 0.53 | 0.55 | 0.54 | 0.52 | 0.51 | 0.02 | 0.07 |
| 4 Methane, bromo- | 0.54 | 0.57 | 0.55 | 0.58 | 0.53 | 0.56 | 0.54 | 0.02 | 0.05 |
| 5 Chloroethane | 0.61 | 0.59 | 0.58 | 0.56 | 0.55 | 0.57 | 0.50 | 0.04 | 0.11 |
| 6 Trichlorofluoromethane | 0.76 | 0.80 | 0.74 | 0.74 | 0.67 | 0.64 | 0.57 | 0.08 | 0.24 |

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