

KORY'S GC TECH – TIPS

Environmental Volatiles Analysis

Synonyms: EPA Method 8260B, EPA Method 524.2, EPA Method 624, Volatiles

These are all examples of volatiles screening methods. Analytes are solvents or volatiles with boiling points less than 200 °C and commonly toxic. The large number of analytes, typically >80, leads to chromatographic co-elutions so MS detection is required for verification and quantitation.

Purge and trap sample preparation is often used and greatly concentrates analytes for very low levels of detection. The sample is then introduced onto a column like a ZB-624, whose phase was specifically designed for EPA method 624. Both the column and the detector are used to quantitate analytes with the requirement that co-eluting compounds have different masses.

Before analyzing samples, a mix of System Performance Check Compounds (SPCC) is run to verify system performance and includes: chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. The purpose is to test system activity and purge efficiency and requires a minimum response factors be met for each compound.

Once the system passes, a five point calibration curve can be run for quantitation using the aid of internal standards. RSD values for all compounds are calculated from response factors and must be below 15% with the exception of Calibration Check Compounds (CCC) which includes poorer performing compounds: 1,1-Dichloroethene, Toluene, Chloroform, Ethylbenzene, 1,2-Dichloropropane, Vinyl chloride. These require RSD values to be below 30%.

Verification of system performance is also required every 12 hours by re-running and passing the requirements for the System Performance Check Compounds (SPCC). The middle calibration mix is then re-run and no response factor may be off by more than 20%.

The use of the purge and trap reduces the amount of any higher molecular weight contaminants that may be introduced onto the column, so column lifetimes tend to be long. However, there can still be problems with this method and some common ones are summarized below.

- Some compounds do not purge well – Purge and trap works well for volatile compounds but will still have poor response for compounds that are not volatile
- Sample carryover if conditions are not optimized - May require more conditioning time between runs to remove or minimize carryover
- Irreversible trapping - Some compounds may not desorb from the trap. This can cause the trap to become saturated as well as actually change the polarity of the trap.
- Purge and Trap may be the limiting step (~20 min) – Fast GC analysis may still have to wait on sample preparation. Some labs use two purge and traps per instrument, but this is expensive though and not common.
- Purge flow hard to optimize - Bromoform is lost if the purge flow is too slow. Chloromethane is lost if the purge flow is too fast.

Recommended Applications:

App 14783 – EPA Method 8260B on ZB-624 – 60 m x 0.25 mm x 1.4 µm

App 15866 – EPA Method 8260B on ZB-624 – 20 m x 0.18 mm x 1.0 µm

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