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Two-Dimensional Chromatography (2D-GC) for CSIA of Difficult Samples

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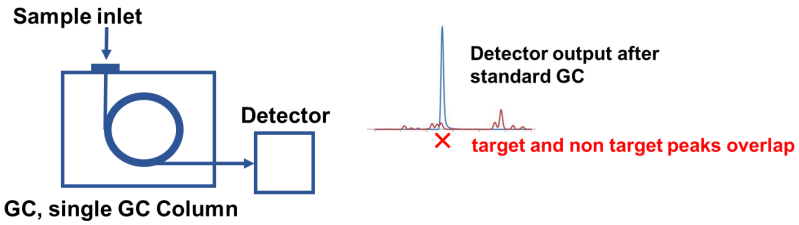
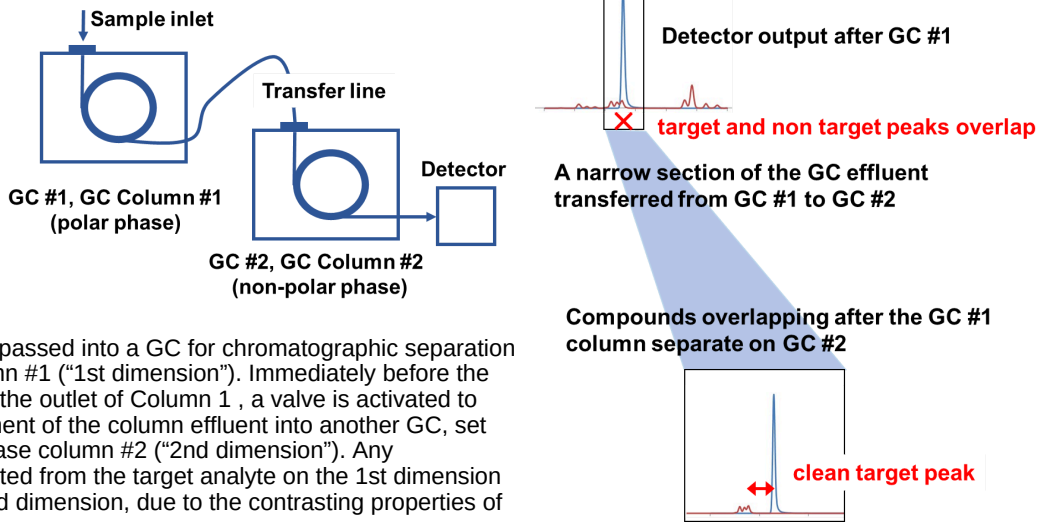
Carbon and hydrogen CSIA require high quality separation of the target compound peak(s) to permit accurate determination of the target's isotope ratio(s). While conventional gas chromatography (using a single GC column) is appropriate for most environmental samples, there are several categories of samples (listed below) with predictable GC interferents that decrease the quality of CSIA results or even prevent obtaining meaningful results.

1. Chlorinated ethene samples from sites with commingled hydrocarbon plumes (the interferents are the individual hydrocarbon compounds and volatile metabolites from fuel biodegradation).
2. Chlorinated ethene samples downgradient from EVO treatment areas (the interferents are volatile metabolites from degradation of EVO).
3. Samples from sites with alkyl halides, such as chloroform, 1,2-DCA, etc. in sulfide-rich groundwater (the interferents are volatile sulfides forming in reactions of the halides with sulfide ion).
4. Samples with target compound(s) in a complex organic matrix, such as minor hydrocarbons in gasoline matrix or individual VOCs in indoor air samples.

The principle of operation of the 2D-GC method



In standard GC, target compounds are resolved from the non-target compounds using a single GC column. Given the complexity of organic matrix of environmental samples, it is relatively common that more than one compound elute from the GC column at the same time (compounds with similar volatility, polarity etc).



In 2D-GC, a sample is passed into a GC for chromatographic separation on a polar phase column #1 ("1st dimension"). Immediately before the target analyte reaches the outlet of Column 1 , a valve is activated to transfer 1-2 min increment of the column effluent into another GC, set up with a non-polar phase column #2 ("2nd dimension"). Any compounds not separated from the target analyte on the 1st dimension will separate on the 2nd dimension, due to the contrasting properties of the two GC columns.

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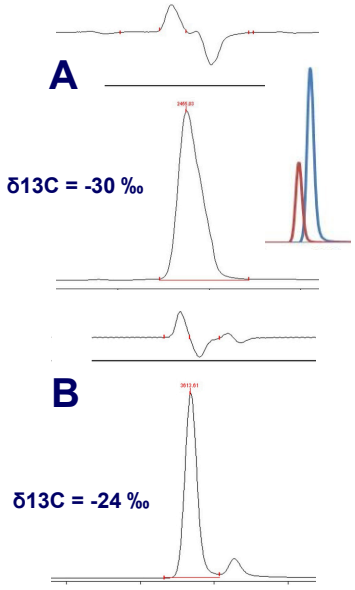
Case 1: Separation of TCE from Diethyl Sulfide (DES)

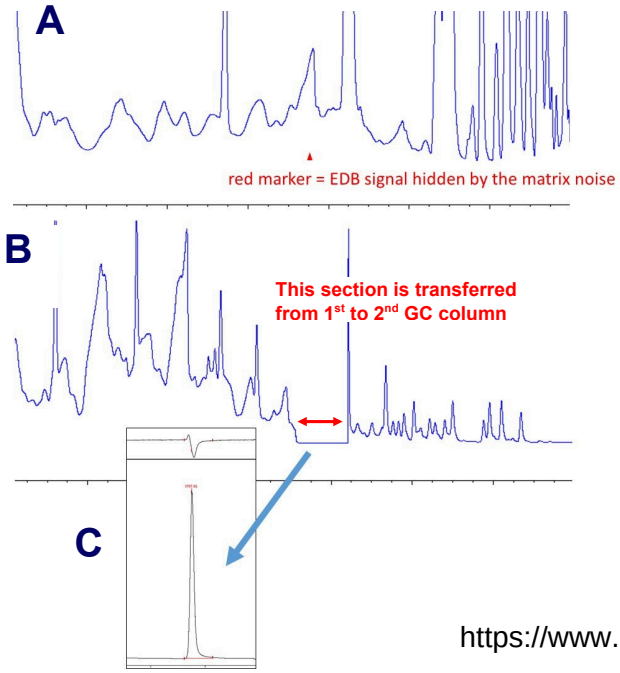
CSIA utilizing a standard GC configuration produced a peak at the re- tention time of TCE. The peak geometry was slightly deformed (apparent for the 45/44 ratio trace in particular). Verification by GCMS confirmed that the observed peak represents two overlapping com- pounds, TCE and DES . DES was probably formed by degradation of co-occurring chlorinated ethanes, under sulfate re- duction environment. After 2D-GC, the TCE peak is completely free from the DES interference.



A. Standard GC chromatogram and mass 45/44 ratio trace of a sample containing Trichloroethene and Diethyl Sulfide. B. The same sample, analyzed by GCMS (TCE = mass 132 plotted in blue, DES = mass 90 plotted in red.

B. 2D-GC chromatogram and mass 45/44 ratio trace of the same sample, showing a clean TCE peak.

Case 2: Separation of EDB from Concentrated Hydrocarbon Matrix



A. In groundwater, ethylene dibromide (EDB) often occurs together with significant concentrations of gasoline-range hydrocarbons. Standard GC is unable to separate EDB from the hydrocarbon matrix (GC with a FID detector).

B and C. 2DGC chromatograms of a sample containing ~ 200 ng of EDB (the target analyte) and several mg of gasoline-range hydrocarbons. Panel B shows a FID detector signal for the after separation on the 1st column. Panel C shows IRMS detector signal after completed 2D separation..

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