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New Applications for Comprehensive Two Dimensional Gas Chromatography

Ronald D. Snelling, Clifford M. Taylor,
Zhuangzhi ‘Max’ Wang, Richard R. Whitney,
Shimadzu Scientific Instruments

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Comprehensive 2D GC

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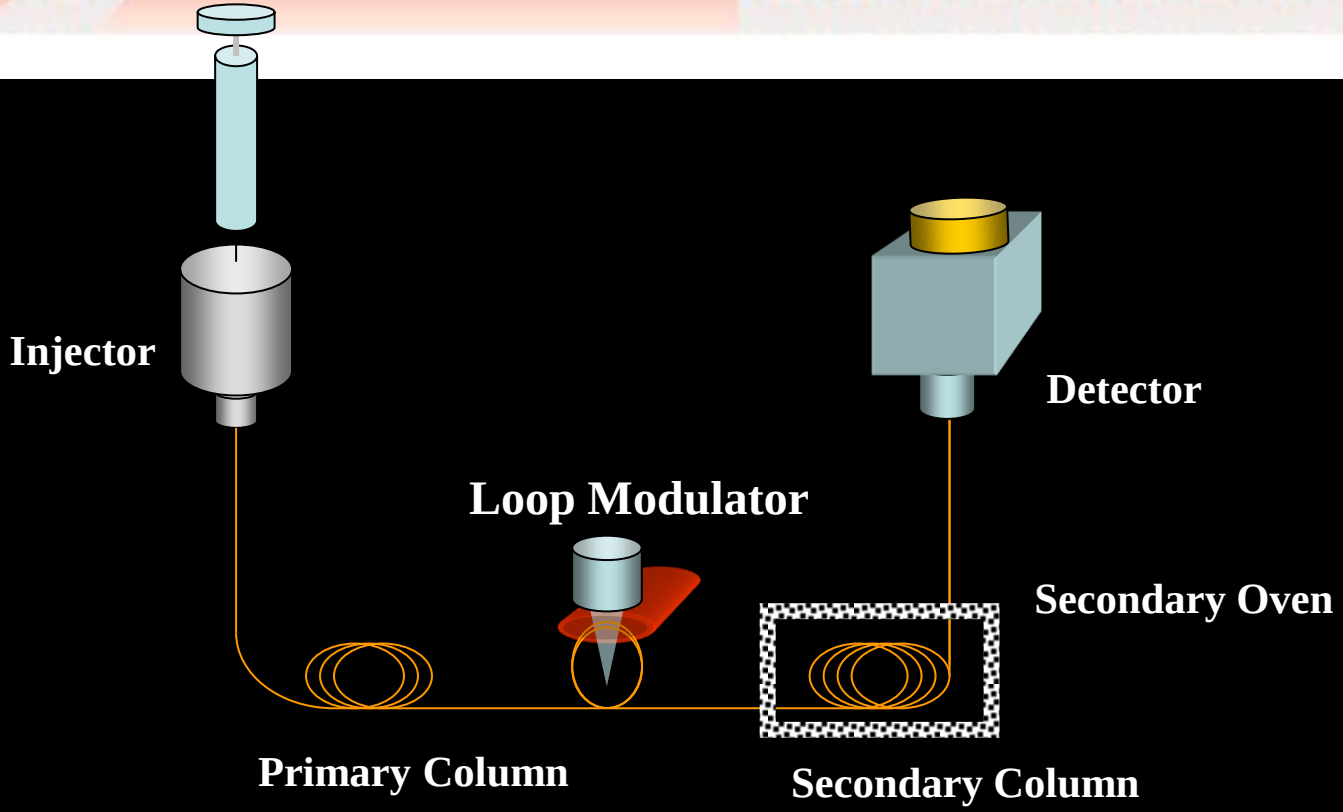
Comprehensive two dimensional gas chromatography is a technique where all the analytes eluting from one chromatographic column are subsequently run on a second, dissimilar column to improve chromatographic resolution. The first column is usually a long 0.25 mm id column, and the second column is a shorter, smaller inner diameter column of a different phase. The transfer to the second column is through a modulator, usually cryogenically cooled. Analytes exiting the first column are cryogenically trapped, then released every 2 to 6 seconds onto the second column.

GC x GC Schematic



GC x GC Loop Modulator

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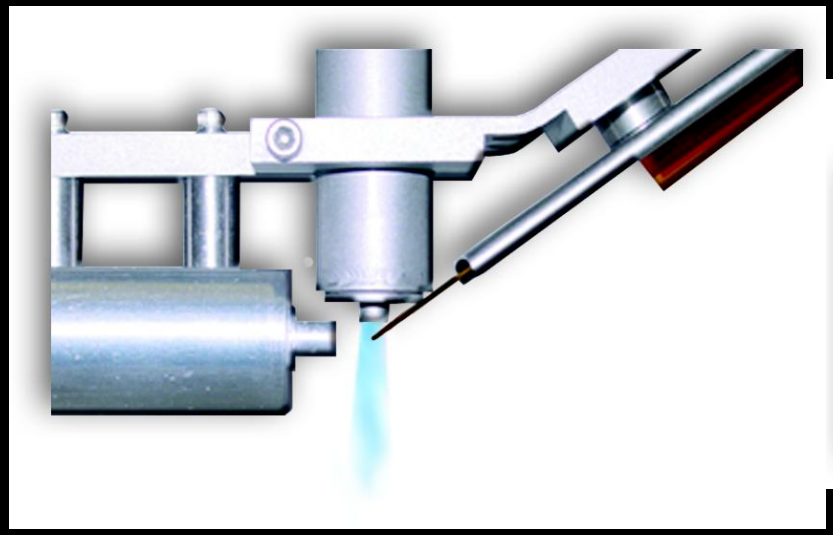

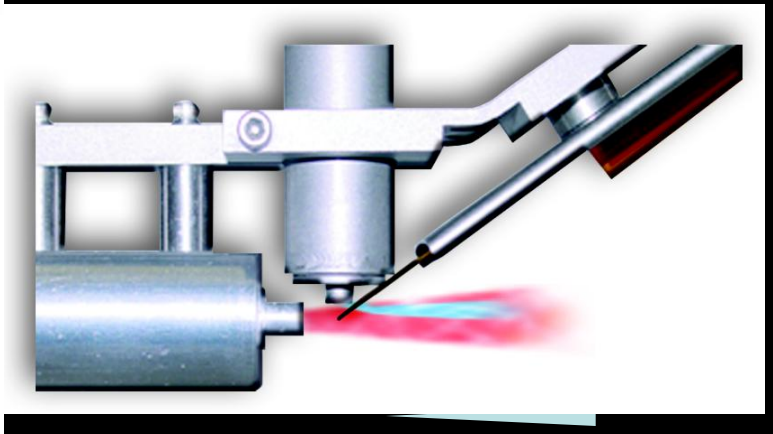

Cold Jet **Hot Jet**

In one commonly used modulator there are two jets of nitrogen gas, a hot jet and a cold jet. The cold jet operates continuously, and at a regular interval the hot jet is activated, releasing the compounds that have been trapped. The modulator used in this work is a double focusing modulator, which improves resolution in the second dimension by providing a very narrow injection band.

In the ideal case, there are two orthogonal separations, one based on carbon number or boiling point, the second based on polarity. This allows improved chromatographic resolution for very complex mixtures.

GC x GC Loop Modulator

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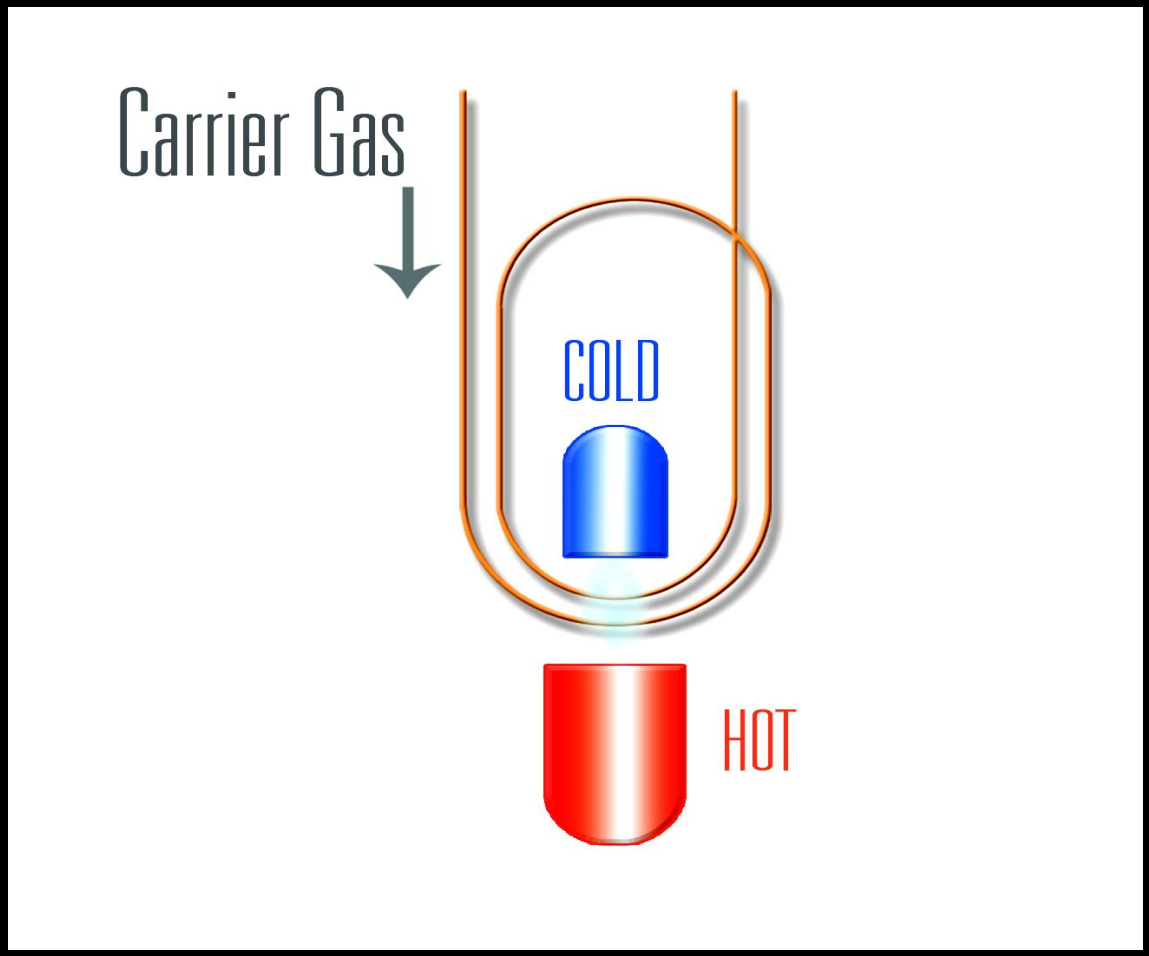
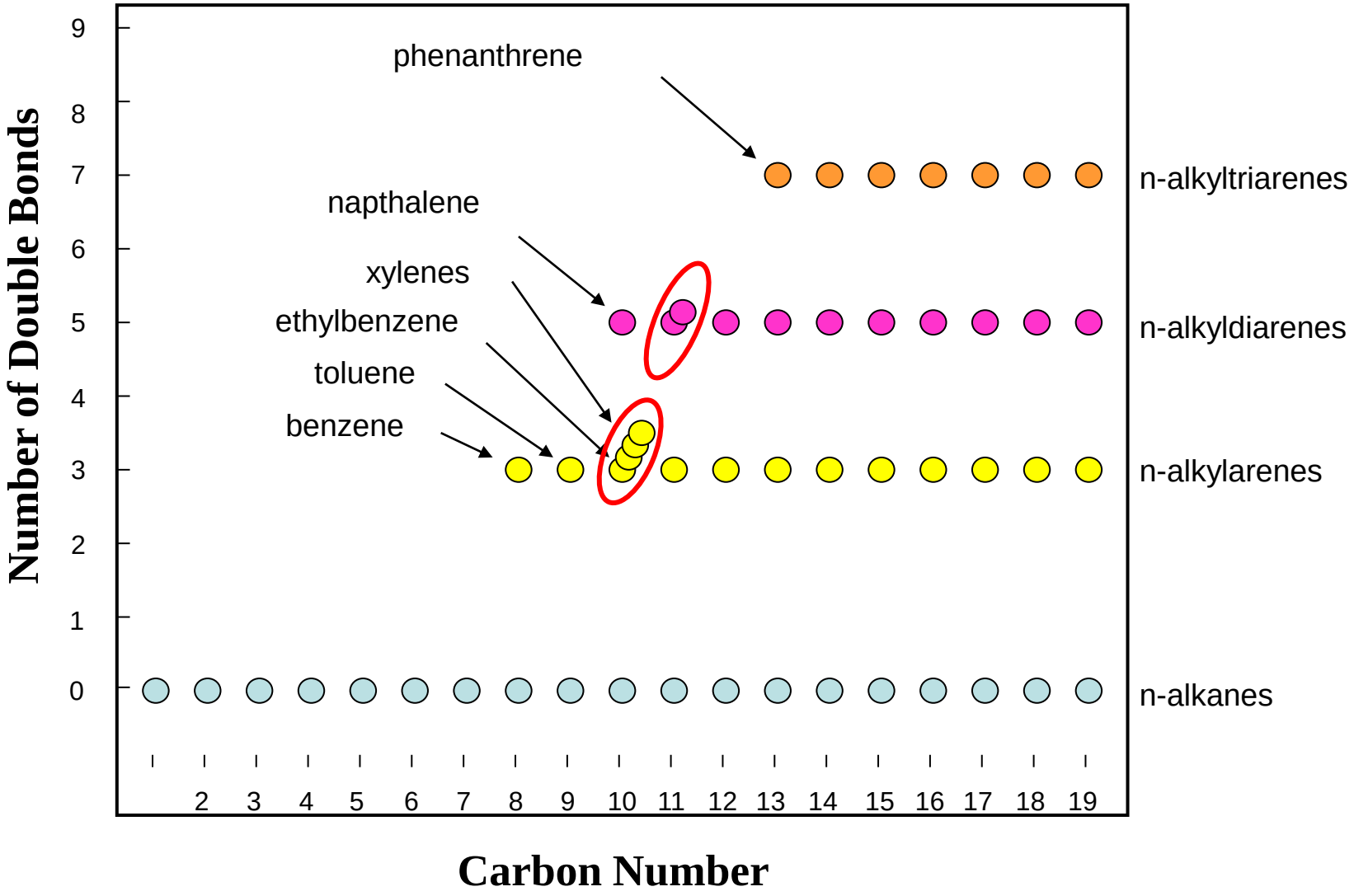
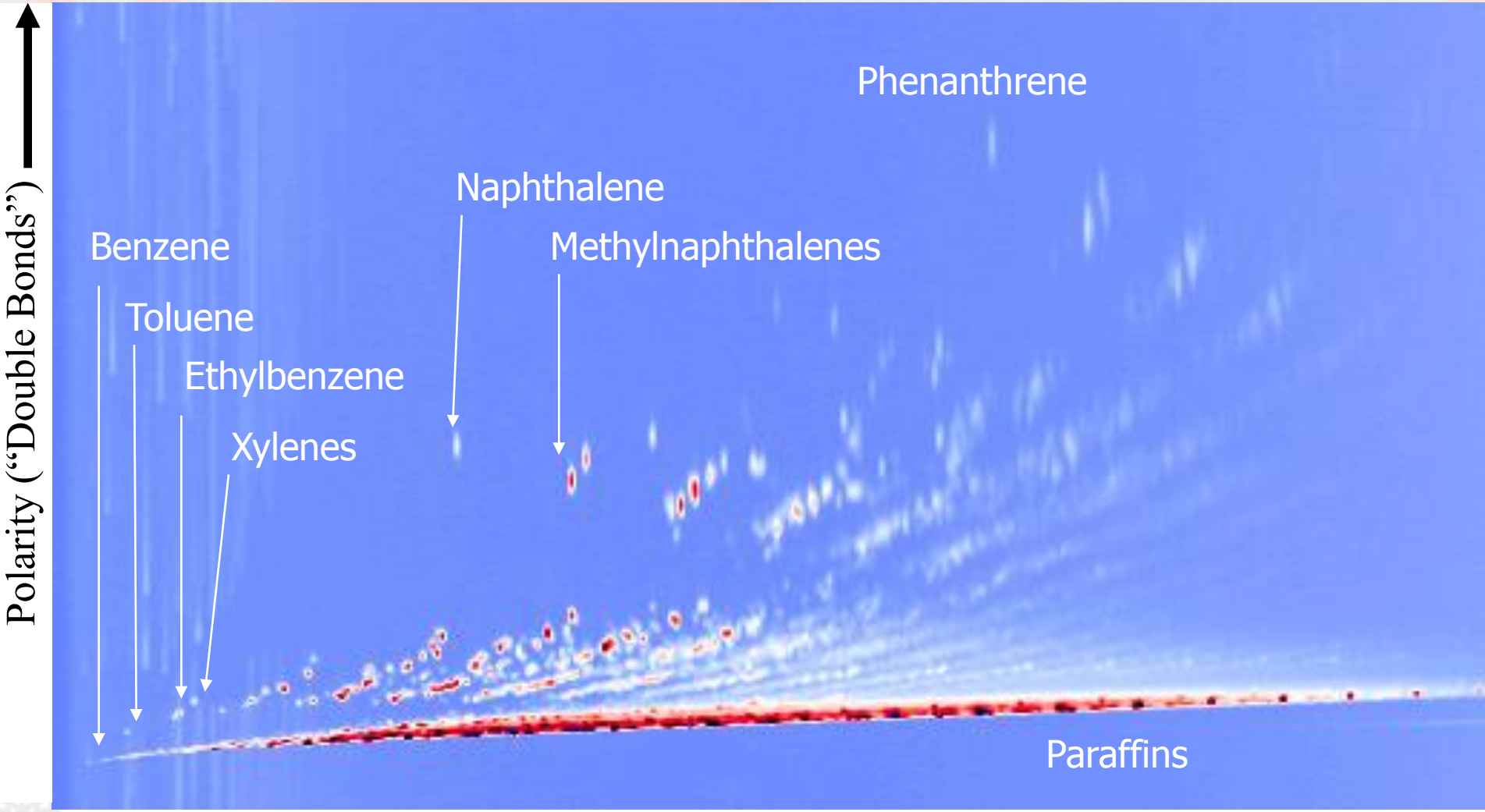


Two-Dimensional Sort of Selected Hydrocarbons

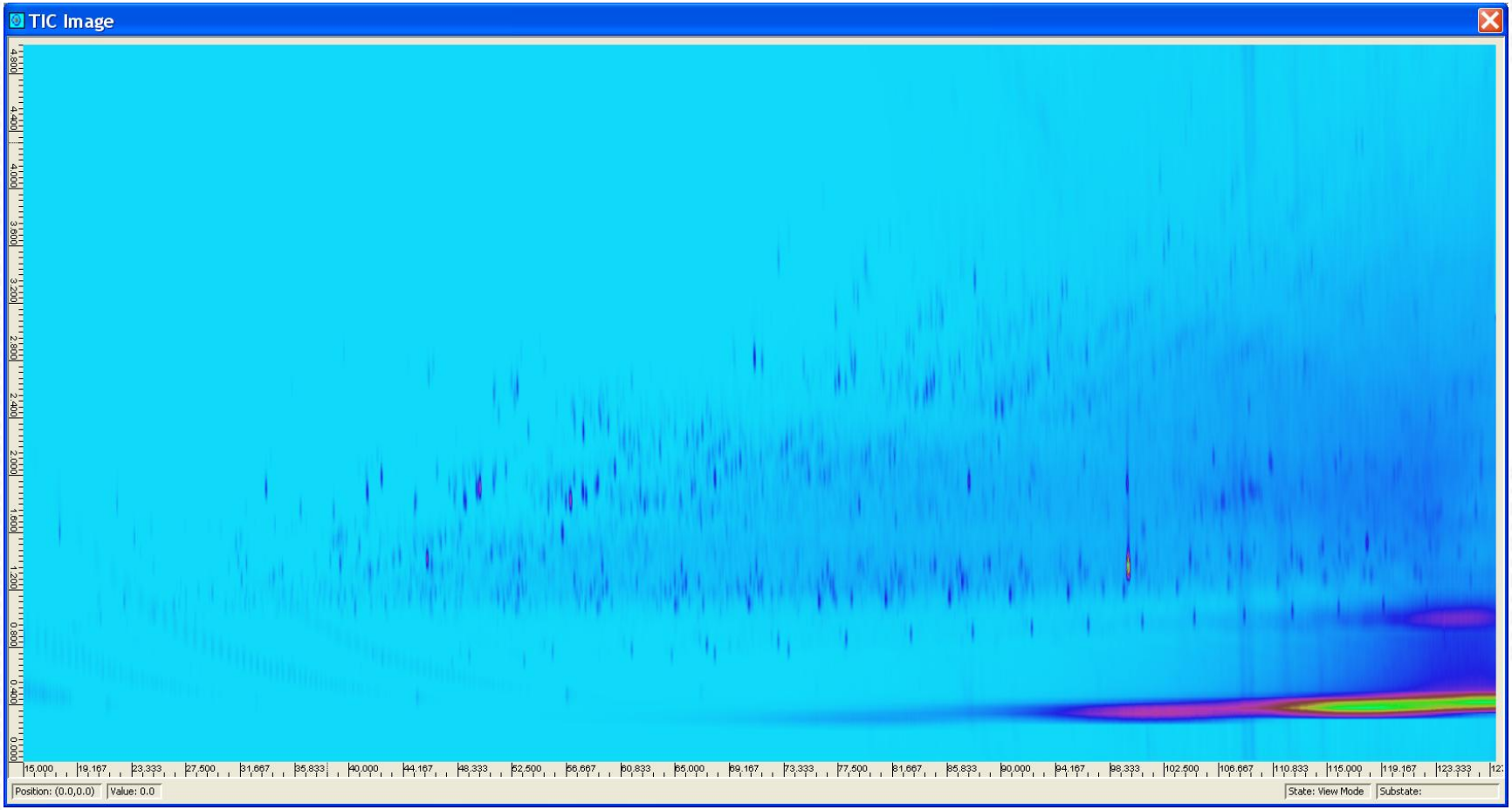


2D-GC Image of Diesel Fuel

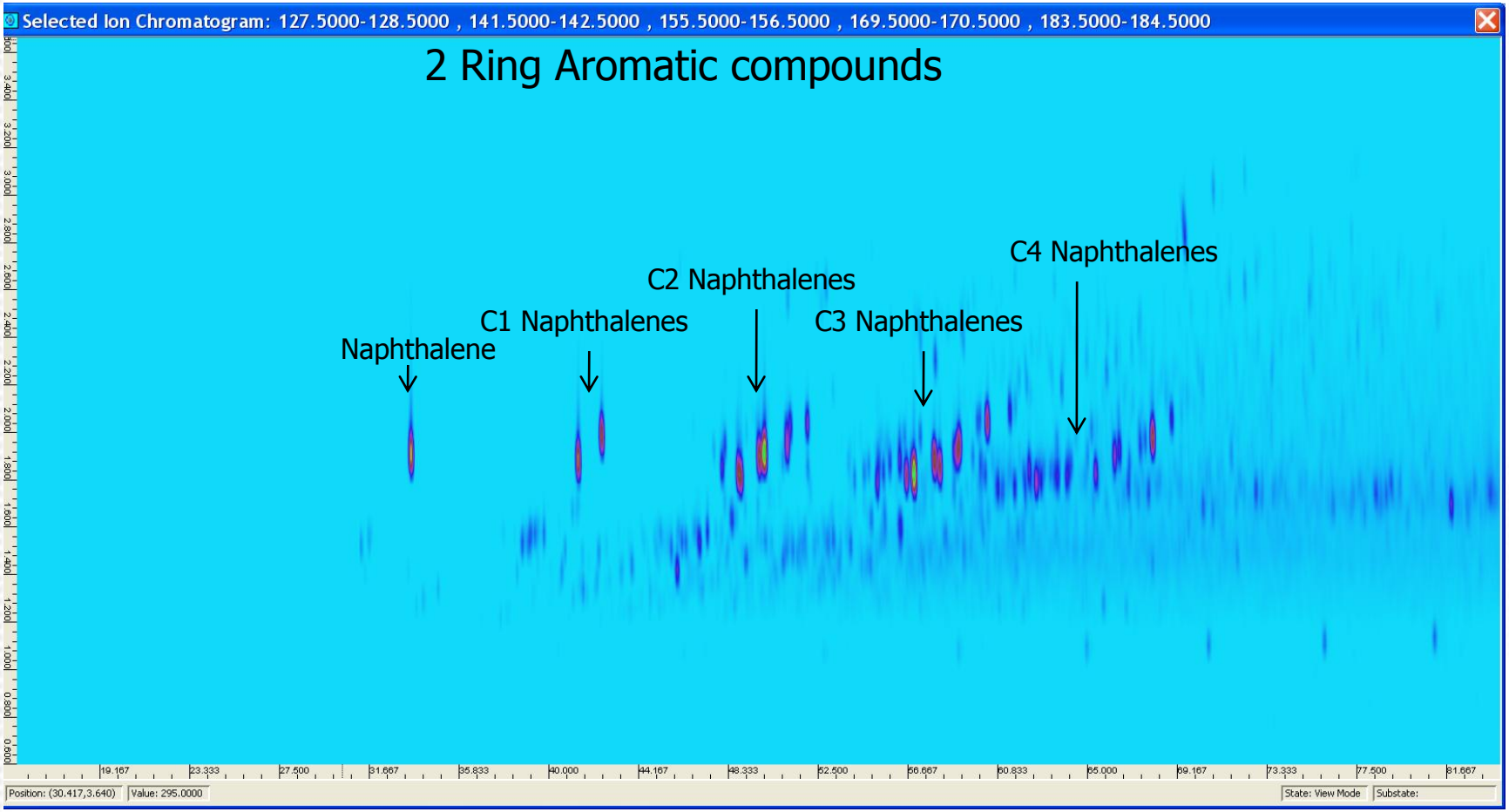


Comprehensive two dimensional chromatography can be used with a mass spectrometer as the detector, which gives an additional dimension of information for positive compound identification. The GC Image analysis software allows the use of selected ions instead of the TIC. This simplifies the identification of compounds.

2D Image of Shale Oil, TIC



2D Image of Shale Oil, Extracted Ions



Citrus Oil

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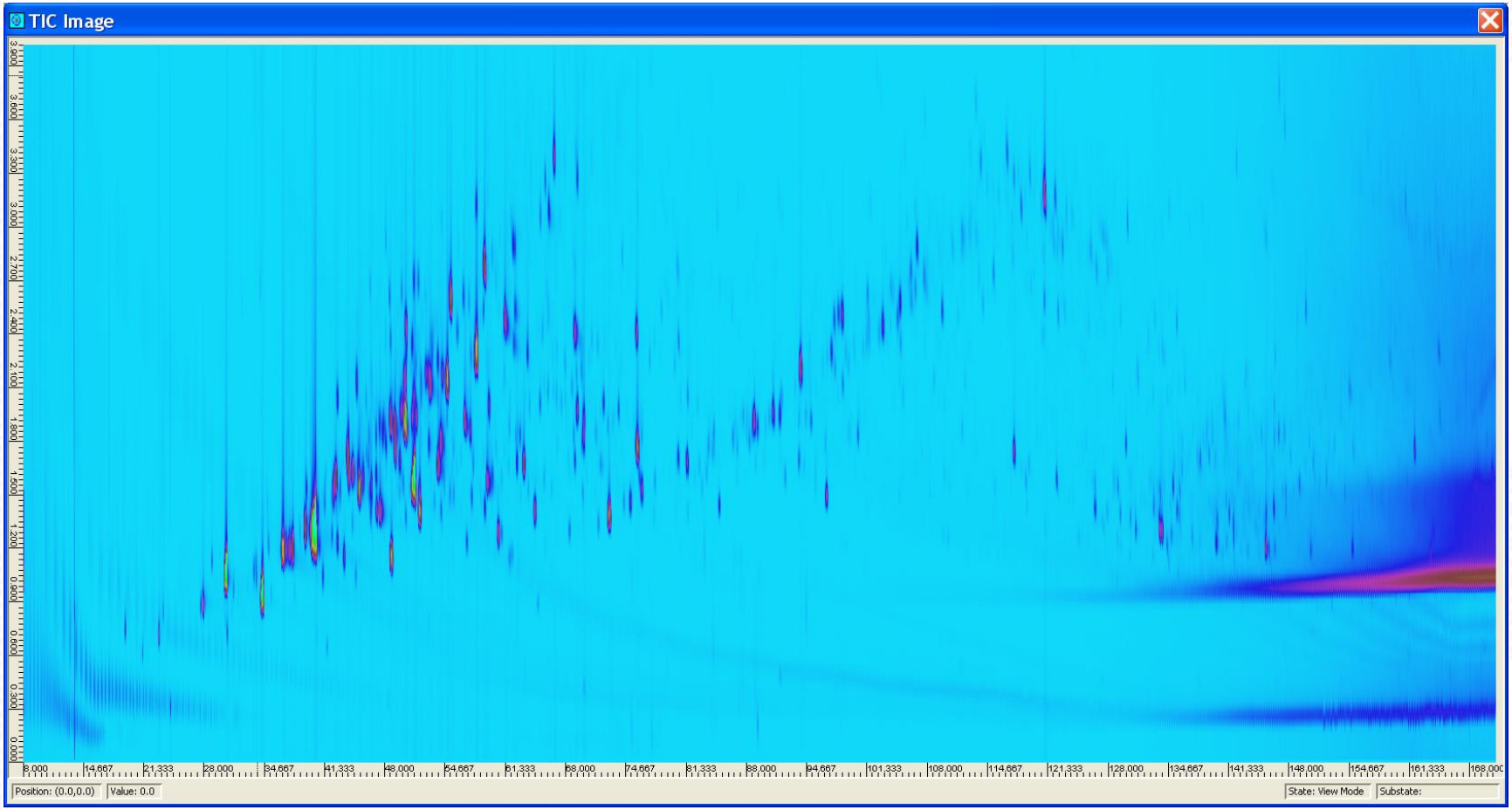
- Citrus oil is a very complex mixture
- Pesticides and off-flavor and off-odor components may be difficult to detect due to matrix interferences
- Comprehensive two dimensional GCMS allows more definite identification of compounds
- A citrus oil image was collected
- A split of the citrus oil was spiked with a pesticide mixture

2D-GC Image of Citrus Oil



Identification of Pesticides

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- Selected ions from the pesticides were used to determine pesticide retention times in both dimensions
- Blobs on the TIC image were identified as pesticides by retention time, then the identity was confirmed by mass spectrometry
- Pesticides spiked into the citrus oil are identified with arrows on the image

Spiked Pesticides

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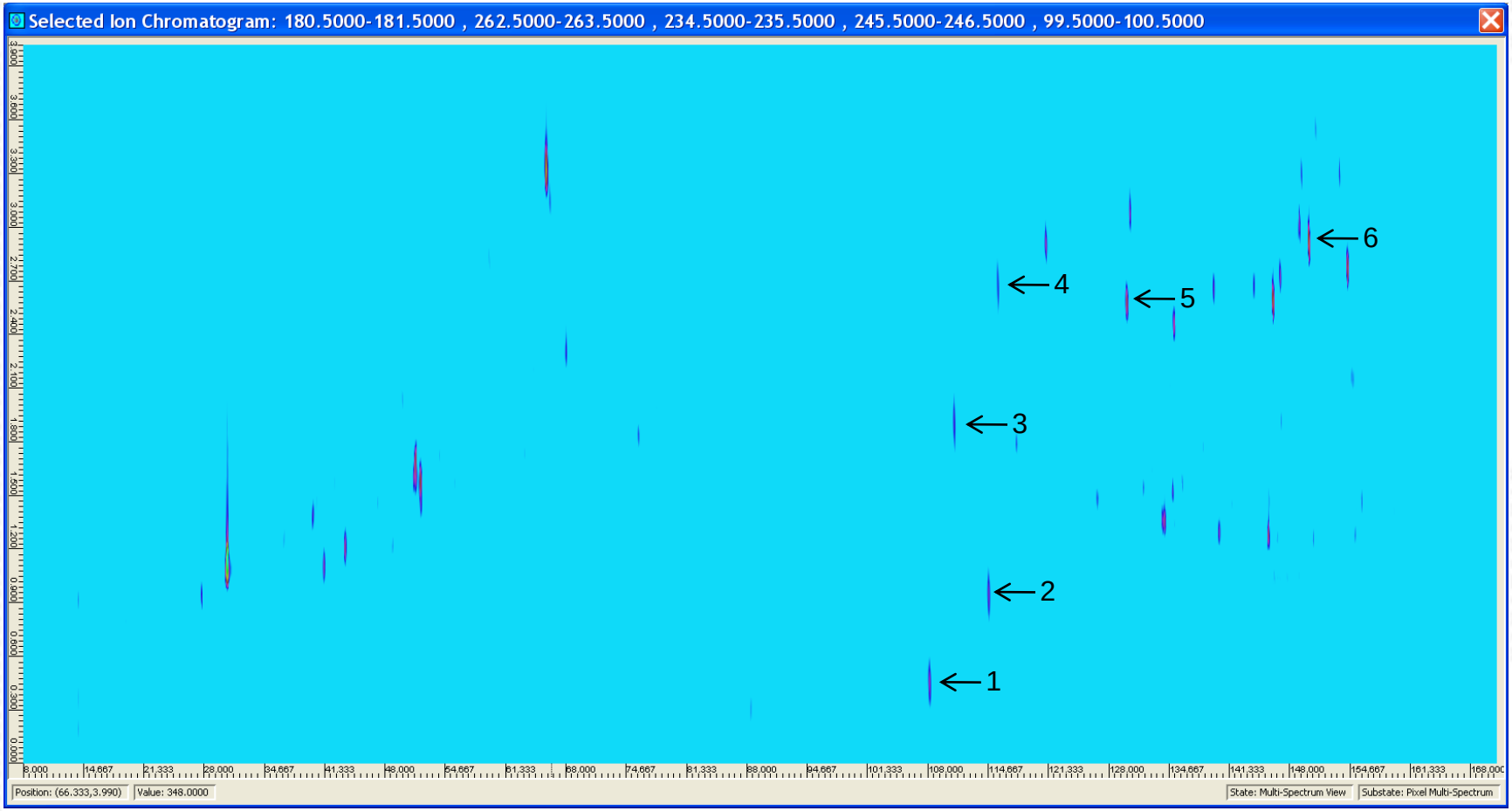
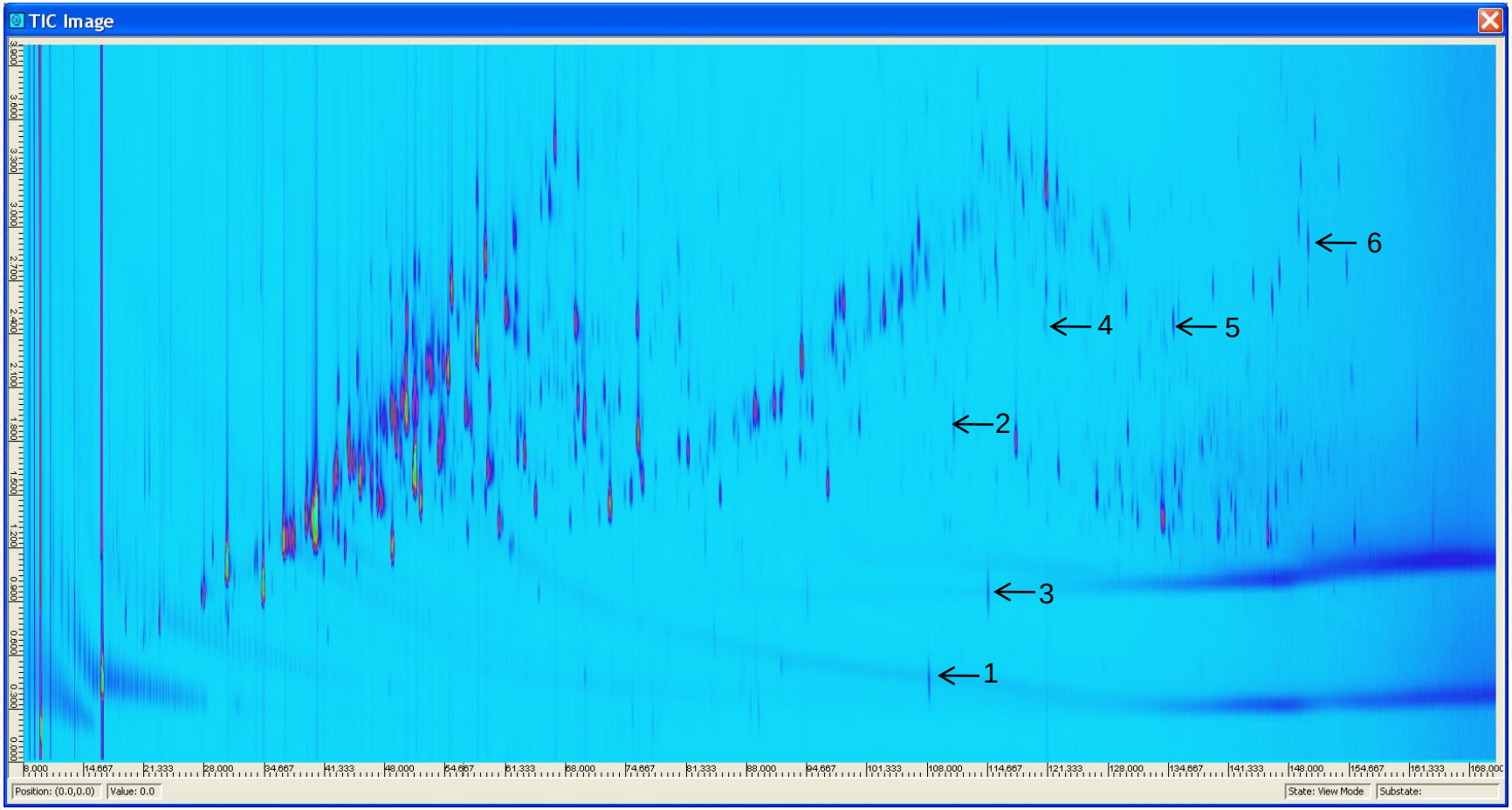
Pesticide	Mass on column
1. alpha-BHC	160 pg
2. gamma-BHC	160 pg
3. beta-BHC	160 pg
4. delta-BHC	160 pg
5. Aldrin	160 pg
6. DDE	160 pg

2D Image, Pesticide Selected Ions



2D-GC Image Spiked Citrus Oil



Conclusions

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- Comprehensive two dimensional gas chromatography can significantly increase resolution in a complex mixture
- Use of a mass spectrometer makes it possible to identify trace components in a complex matrix
- Comprehensive two dimensional gas chromatography can be used to identify target compounds as well as provide a class separation