FIELD IONISATION / DESORPTION
TIME OF FLIGHT MASS SPECTROMETRY

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HIGHLIGHTS
High Mass Resolution: R=30,000
Wide Mass Range: 6000 M/Z
High Mass Accuracy
Soft Ionisation Ideal for MW Determination
C10-C120 Analysis

DEGREE OF SATURATION

All organic compounds have non-integer molecular weights, including the mono-isotopic ions seen in mass spectra:

For example, the following compounds all have the same nominal integer mass of 380:

But accurate masses would be:

380.4382 $C_{27}H_{56}$ (Z = +2)

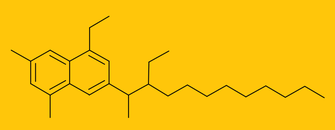
380.3443 $C_{28}H_{44}$ (Z = -12)

380.2504 $C_{29}H_{32}$ (Z = -26)

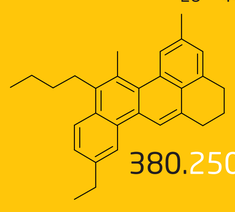
380.2538 $C_{26}H_{36}S$ (Z = -16S)

380.1565 $C_{30}H_{20}$ (Z = -40)

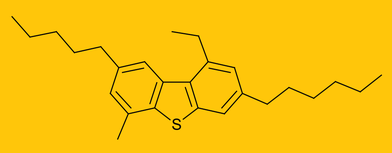
380.4382 $C_{27}H_{56}$ (Z = +2)



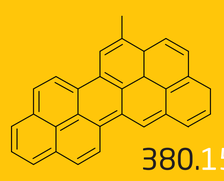
380.3443 $C_{28}H_{44}$ (Z = -12)



380.2504 $C_{29}H_{32}$ (Z = -26)



380.2538 $C_{26}H_{36}S$ (Z = -16S)



380.1565 $C_{30}H_{20}$ (Z = -40)

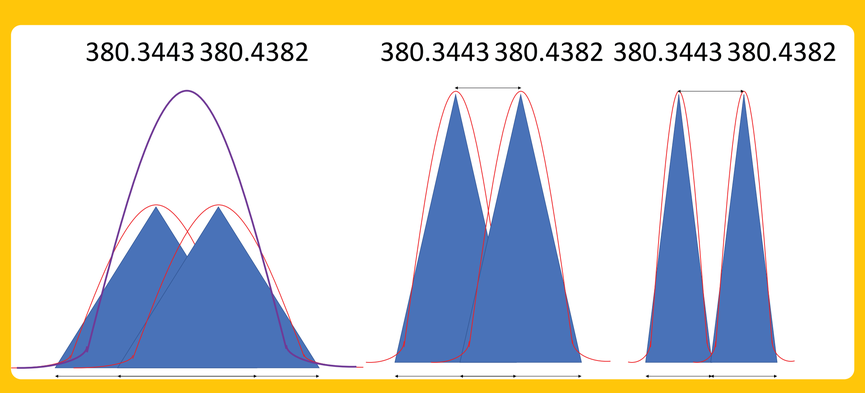
The general formula for hydrocarbons is presented as: C_nH_{2n+Z}

Z represents the degree of unsaturation i.e. the number of rings and double bonds in a structure

$$Z = 2 - (2*RDBE)$$

$$Z = 2 - (2*7) = 2-14 = -12$$

Mass Resolution

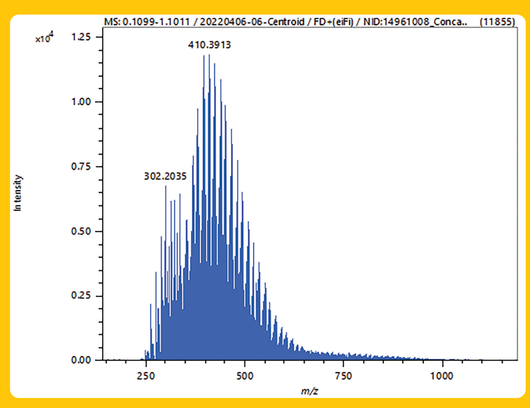


RESULTS

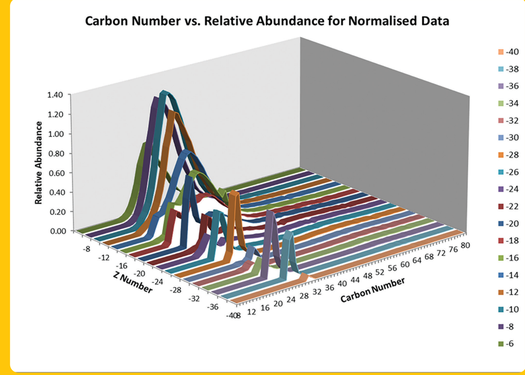
SATURATES	
HYDROCARBON TYPE	**Z NUMBER**
Total Alkanes	2
Cycloalkanes	0
Bicycloalkanes	-2
Tricycloalkanes	-4
Tetracycloalkanes	-6
Pentacycloalkanes	-8
Hexacycloalkanes	-10
Heptacycloalkanes	-12
Octacycloalkanes	-14
Nonacycloalkanes	-16
Decacycloalkanes	-18
Undecacycloalkanes	-20
Dodecacycloalkanes	-22
Tridecacycloalkanes	-24

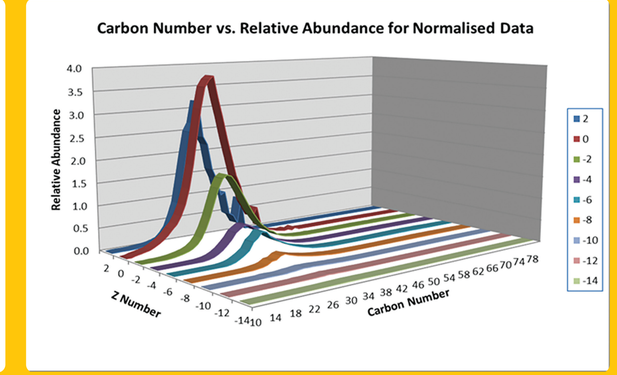
Sample data is tabulated into a matrix of Hydrocarbon type/z number versus carbon number with a limit of detection of 100 ppm. Yield data from the initial separation can be used to factor up the analysis into the whole sample.

AROMATICS	
HYDROCARBON TYPE	**Z NUMBER**
Alkyl Benzenes	-6
Indanes	-8
Indenes	-10
Naphthalenes	-12
Acenaphthenes	-14
Acenaphthylenes/ Fluorenes	-16
Phenanthrenes	-18
C_nH_{2n-20}	-20 (-10S)
C_nH_{2n-22}	-22
C_nH_{2n-24}	-24
C_nH_{2n-26}	-26 (-16S)
C_nH_{2n-28}	-28
C_nH_{2n-30}	-30
C_nH_{2n-32}	-32 (-22S)
C_nH_{2n-34}	-34
C_nH_{2n-36}	-36
C_nH_{2n-38}	-38
C_nH_{2n-40}	-40
C_nH_{2n-42}	-42
C_nH_{2n-44}	-44
C_nH_{2n-46}	-46



Alternatively, the data can be presented graphically



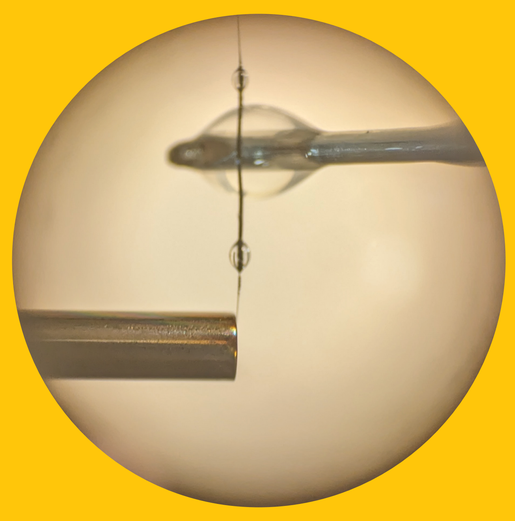


SAMPLE PREPARATION

In order to analyse samples should be separated into Saturates and Aromatics. This is typically conducted using Liquid Chromatography. A modified version of the method IP 368 allows good separation of most sample types in the analysis range.

Importantly the methodology uses Heptane as a mobile phase which needs removal prior to introduction to the FDMS. This is usually conducted by blowing down with a stream of nitrogen, therefore the sample should have a carbon distribution significantly away from C7, hence the analysis starts at C10.

Other separation techniques can be used and is dependent on the sample undertest. Trials are underway using Chromarods as a medium of separation of Bitumen and heavy residues.



INSTRUMENT

Field Desorption

As per the figure above the sample fraction is applied directly to the filament, a current is passed across this filament. Molecules in the condensed phase are ionised to produce molecular ions largely M+. and [M+H]+ ions

Field Ionisation

The field ionization source uses the same architecture but allows a GC column to be terminated in the proximity of the filament, Molecules in the gas phase are ionised to produce molecular ions largely M+. and [M+H]+ ions

After ionisation the ions pass into the time of flight mass spectrometer.

TARGETED ANALYSIS

GCMS/MS

We have the capability of performing targeted analysis for analytes such as PAHs to low levels with a GC-MS/MS. The instrument allows for multiple reaction monitoring (MRM) which leads to increased sensitivity for low level compounds in complex mixutures. If the sample is ameanable to direct injection via a split/splitless GC injector the sample preparation can be minimal, but the power of the MS/MS allows for potential sub-ppm level detection limits for targeted compounds.

FUTURE DEVELOPMENTS

The instrument is also capable of coupling to a GC, however Field Desorption cannot be used, Instead the instrument operates in Field Ionisation mode. Work is underway to produce a GCxGC-FI-TOF-MS methodology. Initial data using Kerosene type samples compares well with data taken from GCxGC-FID methods using internationally recognized standards.

The eventual goal is produce a method in which a sample can be tested from C10-C120 without separation

SCAN ME
FOR MORE INFORMATION