The composition and analyses of oil fractions present in food

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Workshop MOCRINIS
September 11, Bologna
The analyses of oil fractions

Different from GC analyses in general: The complete matrix composition has to be determined.

Advantage: in general, contain only hydrocarbons

Disadvantage: contain enormous amounts of isomeric compounds
The number of possible hydrocarbon isomers

![Graph showing the number of possible hydrocarbon isomers as a function of carbon number. The graph includes lines for paraffins, naphthenes, mono-olefins, and aromatics.]
The composition of oil fractions

1. Saturates (MOSH)
   - Straight chain alkanes
   - Branched alkanes
   - Cyclic alkanes

2. Aromatics (MOAH)
   - Mono-aromatics
   - Higher ringsystems

(3. Sulphur compounds)
Pre-separation of MOSH and MOAH

- UV
- RI
- Fraction collection
- NH$_2$-silica column
- Injection
GC- analyses of the two fractions
1. MOSH
GC- analyses of the two fractions
2. MOAH
On-line LC-GC analyses of MOSH and MOAH
GC analysis of the different fractions
1. MOSH

The numbers indicate the n-alkanes
GC analysis of the different fractions
2. Mono-aromatics (MOAH-1)

The numbers indicate the retention time of the n-alkanes
GC analysis of the different fractions
3. Naphthalenes (MOAH-2)

1. naphthalene
2. 2-me-naphthalene
3. 1-me-naphthalene
4. biphenyl
5. C2-naphthalenes
6. C3-naphthalenes
7. C4-naphthalenes
8. C5+-naphthalenes
9. benzothiophene
10. me-benzothiophenes
11. C2-benzothiophenes
GC analysis of the different fractions
4. Other di-aromatics (MOAH-2)

1. diphenylalkanes
2. fluorene
3. me-fluorenes
4. dibenzothiophene
5. me-dibenzothiophenes
6. C2- & C3-dibenzothiophenes
GC analysis of the different fractions

5. Tri-aromatics (MOAH-3)
Comprehensive two-dimensional gas chromatography (GCxGC)

**GC-GC** (Or 2D-GC)

- Typical heart-cut technique, target compounds
- Characterization of the whole sample: many, many heart-cuts

Comprehensive 2D-GC ➔ GCxGC
Two- dimensional gas chromatography

Heart-cut 2D-chromatography (2D-GC or GC-GC)

Chromatogram from first dim. column

Chrom. 2nd dim. column
Comprehensive two-dimensional gas chromatography

Comprehensive 2D-chromatography (GCXGC)
The use of sample dimensionality

1D by size

1D by colour

1D by shape

2D by size and colour

2D by size and shape
Schematic diagram of a GCxGC system

1. injector, 2. detector, 3. 1st column, 4. 2nd column, 5. column connection, 6. modulator
1. injector, 2. detector, 3. 1st column, 4. 2nd column, 5. column connection, 6. CO\textsubscript{2} nozzles, 7. valves
Cryogenic focusing

1. Carrier gas flows through the stationary phase in the 1st dim. column. The coolant is introduced at this point to cool the stationary phase.

2. As the carrier gas continues through the 2nd dim. column, the coolant is applied to further cool the stationary phase, enhancing the focusing of the sample.

3. The cooled stationary phase facilitates the separation of compounds, allowing for more effective analysis by GC (Gas Chromatography).
One of the two cryo jets
Orthogonality in GCxGC

polarity

volatility
1D-separation

2D-separation

2D-representation
3D-Representation of a GCxGC separation (detail)
Advantages of GCxGC

1. Increase in separation power
2. Sensitivity enhancement
3. Structured chromatograms
Enhanced sensitivity

Non-modulated

Detector response

Retention time (minutes)

Modulated

Detector response

Retention time (minutes)
Signal enhancement in GC×GC

Separation of an urban air sample

Courtesy Allistair Lewis
Ordening of chemical classes on two independent columns

Retention index 1st column

- ▲ alkanes
- ◇ cyclo alkanes
- △ alkenes
- ○ aromatics

Filled: normal side chain
Separation of a kerosene
“Ordered” or “structured” chromatograms

(GC×GC separation of a non-aromatic solvent)
Examples of the separation of MOSH and MOAH fractions

On non-polar stationary phases MOAH are coeluted with MOSH, but carbon numbers do not correspond:
Methyl anthracene (C\textsubscript{15}) at n-C\textsubscript{21}, Chrysene (C\textsubscript{18}) at n-C\textsubscript{27}, Pyrene (C\textsubscript{16}) at n-C\textsubscript{24}

(These separations have been performed in the Kantonales Labor, Zurich, Switzerland)
MOSH in wheat

(This separation has been performed in the Kantonales Labor, Zurich, Switzerland)