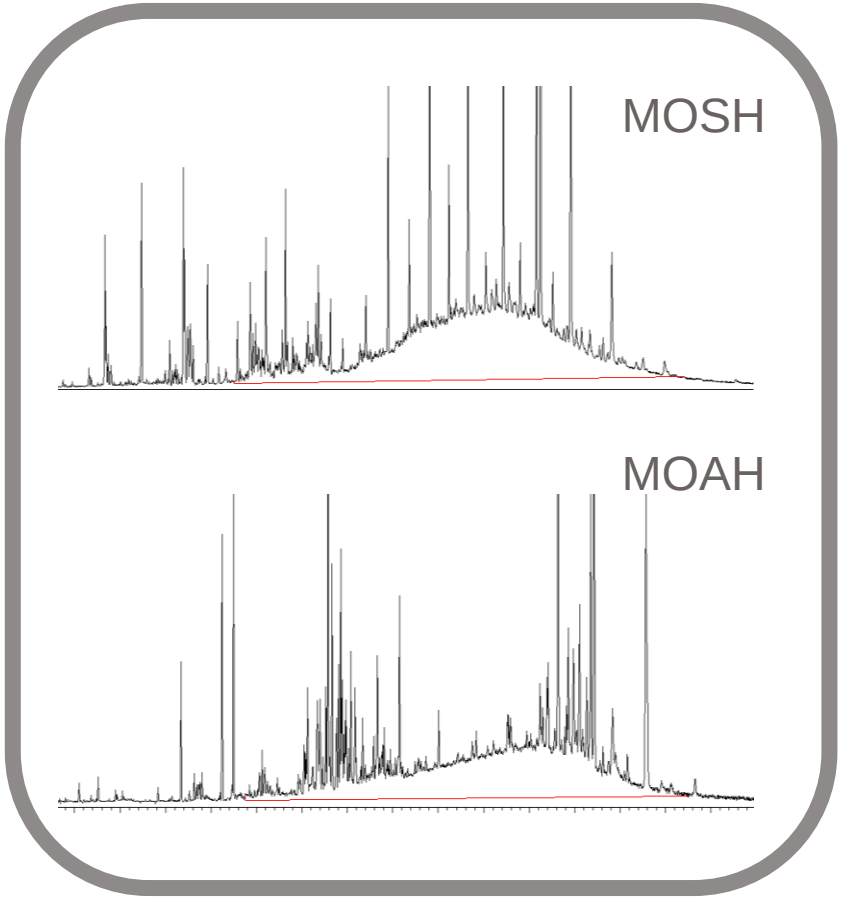


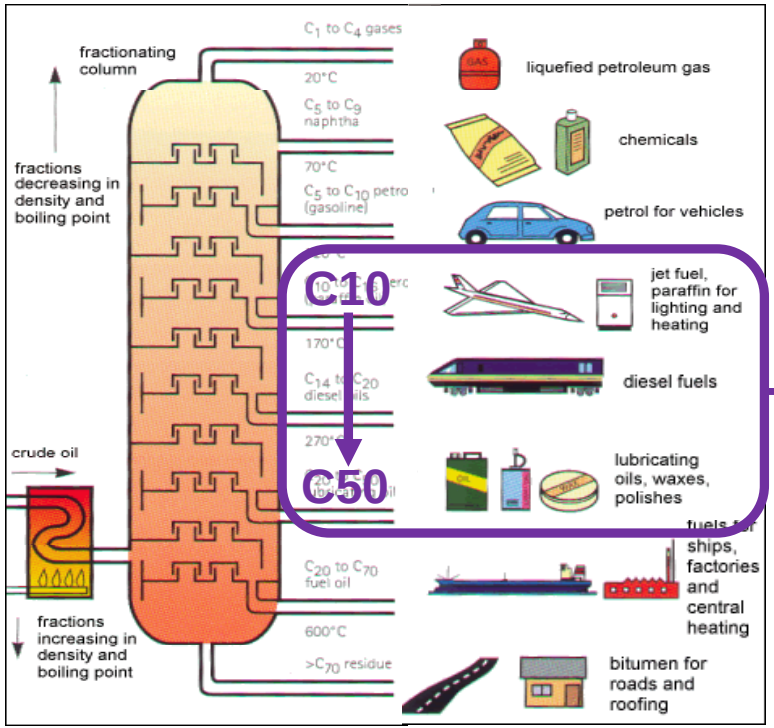
Nestlé Research and Development

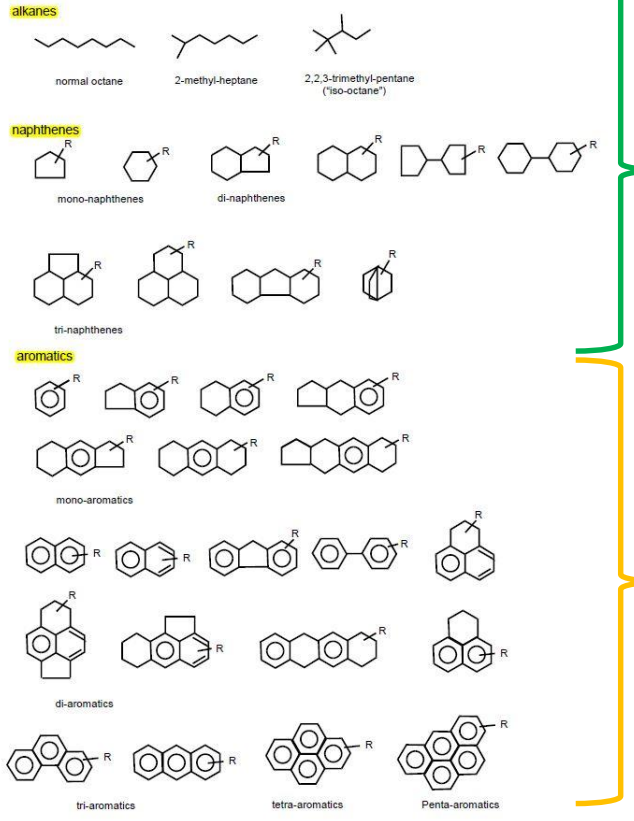
Challenges in the untargeted analysis of mineral oils

Sander koster

Food packaging forum,
October 4th 2018, Zurich

Composition of Crude oils and Mineral oils

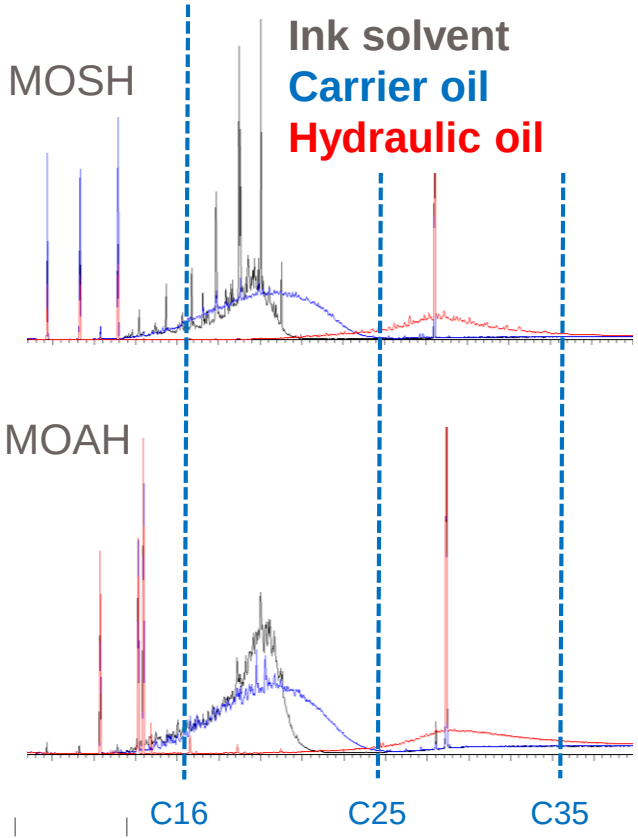




MOSH
(highly branched and alkylated)

MOAH

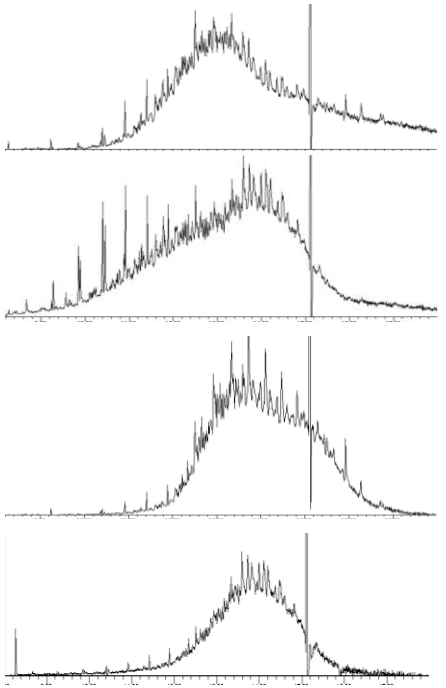
Challenges in the untargeted analysis of mineral oils



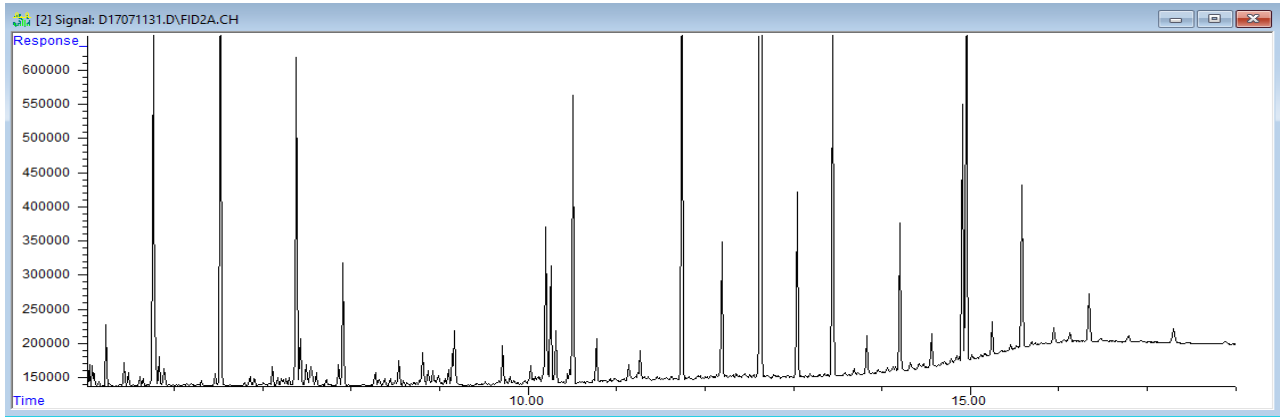
- Mixture of unknowns
- C-range depend on technological function
- No standard available
- Many grades/suppliers.
- Impact geological origin
- Level of hydrogenation
- Allowed vs not allowed.
- Typically FID used, → no confirmation

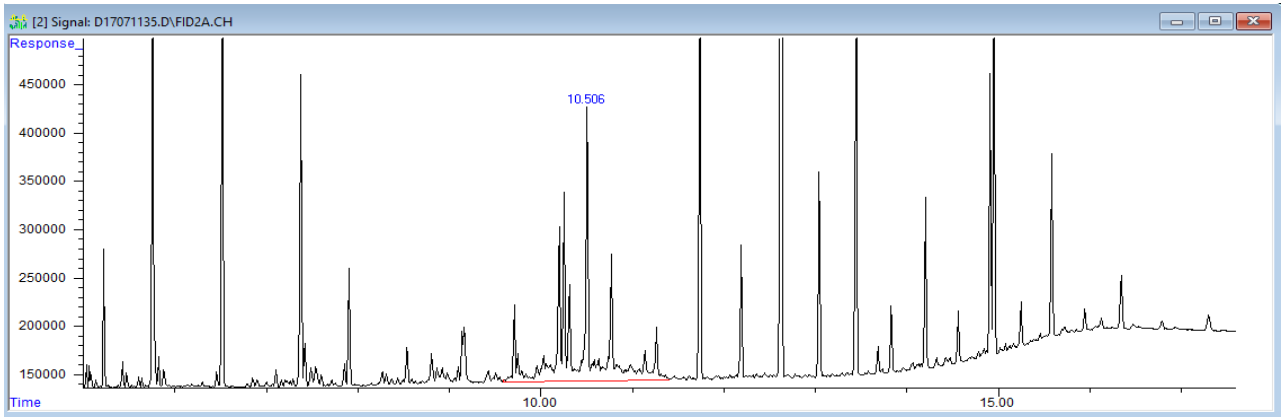
Mineral oils analysis
→ Untargeted
→ Semi-quantitative

4 paraffinic oils (MOSH)



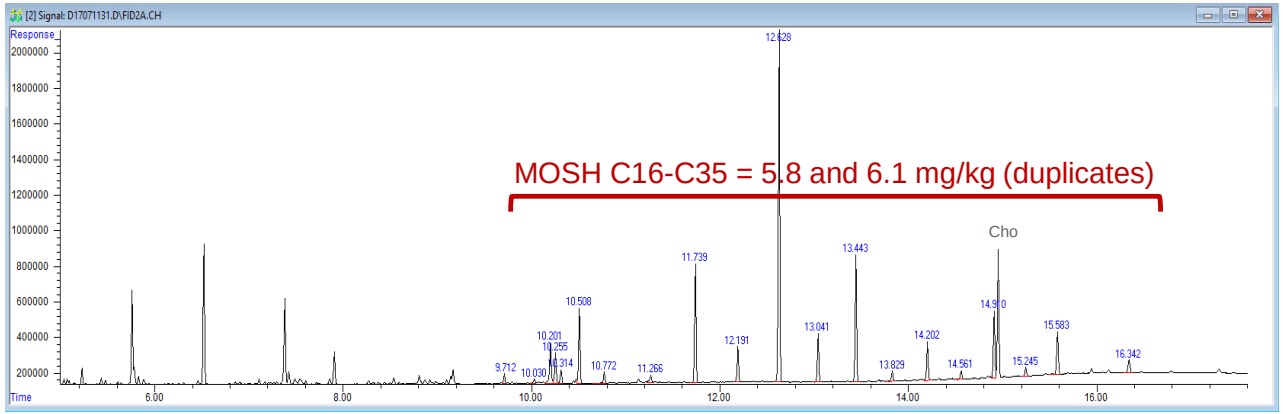
MOSH fraction of a food (I)





- External lab found only MOSH at ~6 mg/kg
- **Where is the expected hump ?**
- Problem of sensitivity ?
- Spiked food with 2.6 mg/kg of mineral oil
- So the method was able to detect 6 mg/kg…

MOSH fraction of a food (II)



Analysis by a third party laboratory using LC-GC-FID-MS:

- Absence of mineral oil confirmed
- Natural hydrocarbons at ~4 mg/kg

- External lab found **only MOSH at ~6 mg/kg**
- Quantification of the natural n-alkanes ?
- Not a problem of detection but of interpretation ?

Later, similar case with a milk powder

External lab: ~2 mg/kg MOSH, no MOAH

NRC: no mineral oils (LoQ ~0.5 mg/kg)

Where do we stand?

Milk powder sent to 9 external labs

Can you perform a mineral oils analysis on this milk powder?

Methodologies used in interlab studies

Laboratory	MS	FID	Saponification	Alox	Epoxidation
1	+	+	+[1)]	-	-
2	+	+	+[2)]	+	+
3	+	+	+[1)]	-	-
4	-	+	-	-	-
5	-	+	-	+	+
6	-	+	-	+	+
7	-	+	-	+	+
8	-	+	-	-	+
9	-	+	+[1)]	-	+
10	-	+	+[3)]	-	+

1) hot, 2) details not given 3) acidic

No alignment

Methodologies used in interlab studies

Laboratory	MS	FID	Saponification	Alox	Epoxidation	Carbon range reported	MOSH (mg/kg)	MOAH (mg/kg)
1	+	+	+[1)]	-	-	C16-C35	3.6*	<0.1
2	+	+	+[2)]	+	+	<C16-C50	4.3	<1
3	+	+	+[1)]	-	-	C16-C35	<0.5	<0.5
4	-	+	-	-	-	C10-C35	<0.03	<0.03
5	-	+	-	+	+	C10-C50	1.1	<0.5
6	-	+	-	+	+	C10-C62	<0.6	<0.15
7	-	+	-	+	+	C10-C62	<0.6	<0.15
8	-	+	-	-	+	<C16->C50	1.4	<0.5
9	-	+	+[1)]	-	+	C10-C35	2.6	0.7
10	-	+	+[3)]	-	+	C10-C50	<1	<1

*measured by FID. Not confirmed by MS.

No alignment

Methodologies used in interlab studies

Laboratory	MS	FID	Saponification	Alox	Epoxidation	Carbon range reported	MOSH (mg/kg)	MOAH (mg/kg)
1	+	+	+[1)]	-	-	C16-C35	3.6*	<0.1
2	+	+	+[2)]	+	+	<C16-C50	4.3	<1
3	+	+	+[1)]	-	-	C16-C35	<0.5	<0.5
4	-	+	-	-	-	C10-C35	<0.03	<0.03
5	-	+	-	+	+	C10-C50	1.1	<0.5
6	-	+	-	+	+	C10-C62	<0.6	<0.15
7	-	+	-	+	+	C10-C62	<0.6	<0.15
8	-	+	-	-	+	<C16->C50	1.4	<0.5
9	-	+	+[1)]	-	+	C10-C35	2.6	0.7
10	-	+	+[3)]	-	+	C10-C50	<1	<1

*measured by FID. Not confirmed by MS.

No alignment | No alignment | No consistency

External labs requested to return samples

Results external labs

Laboratory	MOSH (mg/kg)	MOAH (mg/kg)
1	3.6	<0.1
3	<0.5	<0.5
4	<0.03	<0.03
5	1.1	<0.5
9	2.6	0.7

Results re-analysis by NRC (LC-GC-FID/MS)

LOQ ~0.5 mg/kg.

Recovery OK

→ Mineral oils not observed in any sample.

?

Conclusion

-Mineral oils analysis: not an easy task!

→ Alignment needed.

-Mineral oils findings in milk powder up to ~6 mg/kg food are unreliable.

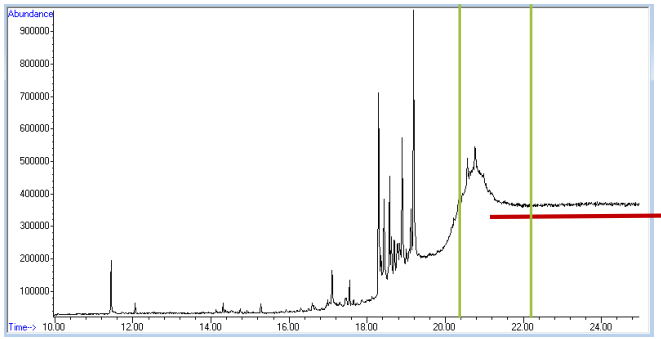
-Mineral oils in vegetable oils (EN 16995): LoQ 10 mg/kg

→ What to do with MOH values below 10 mg/kg?

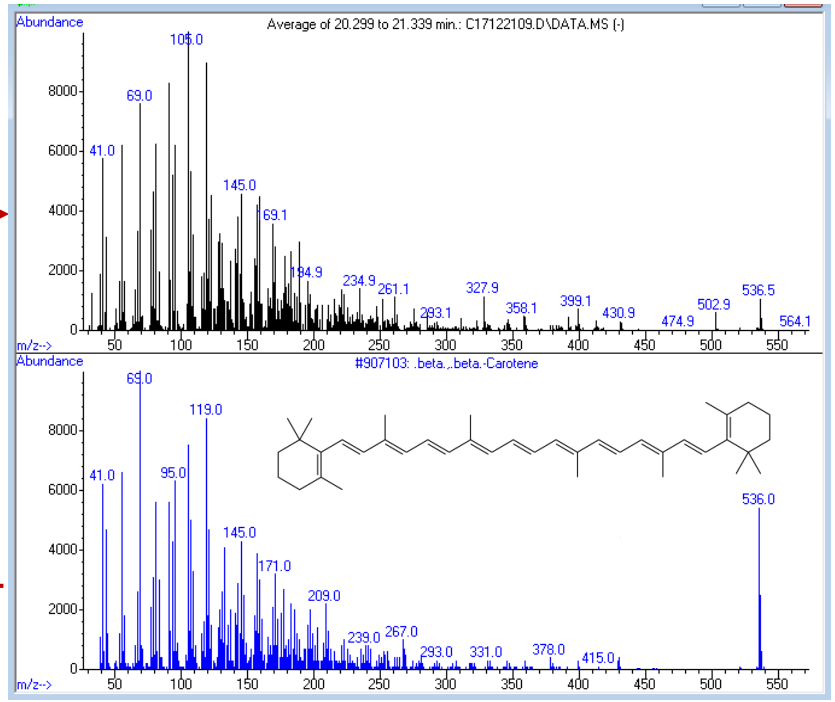
→ More ‘real’ life surveys and p-tests needed!

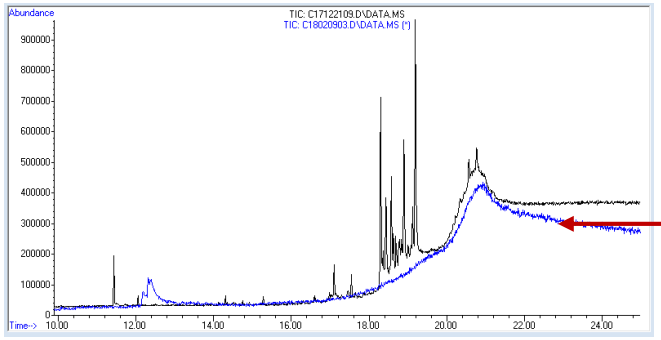
MOAH profile of an milk powder: power of MS

MS confirmation a need!
(though epoxidation would have removed the carotenoids)



MS spectrum





Injection of standard

carotenoids! (see also Grob et al.)

Acknowledgments