

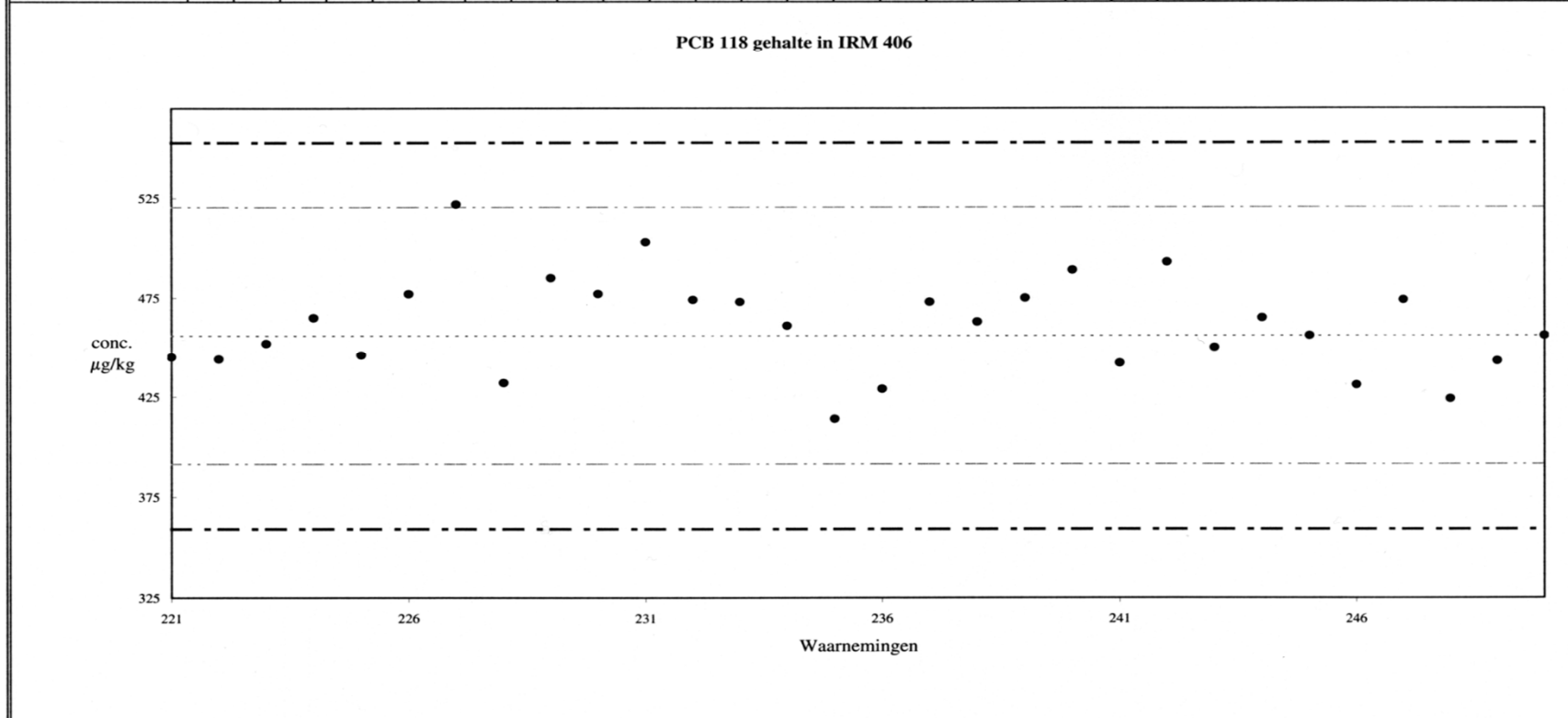
## DISCUSSION ON SECOND WORLDWIDE UNEP INTERLABORATORY STUDY ON POPS

Jacob de Boer, Helena Nilsson, Ike van der Veen, Bert van Bavel, Heidi Fiedler

# Possible mistakes in OCP Analysis

- Separation! - HCHs, chlordanes
- Adsorption - dieldrin, endrin
- Degradation - DDT , dieldrin
- Fractionation: DDE, chlordanes
- Internal standards
- Linearity: Low concentration range ECD is not linear
- Quality standards? Ampouled?

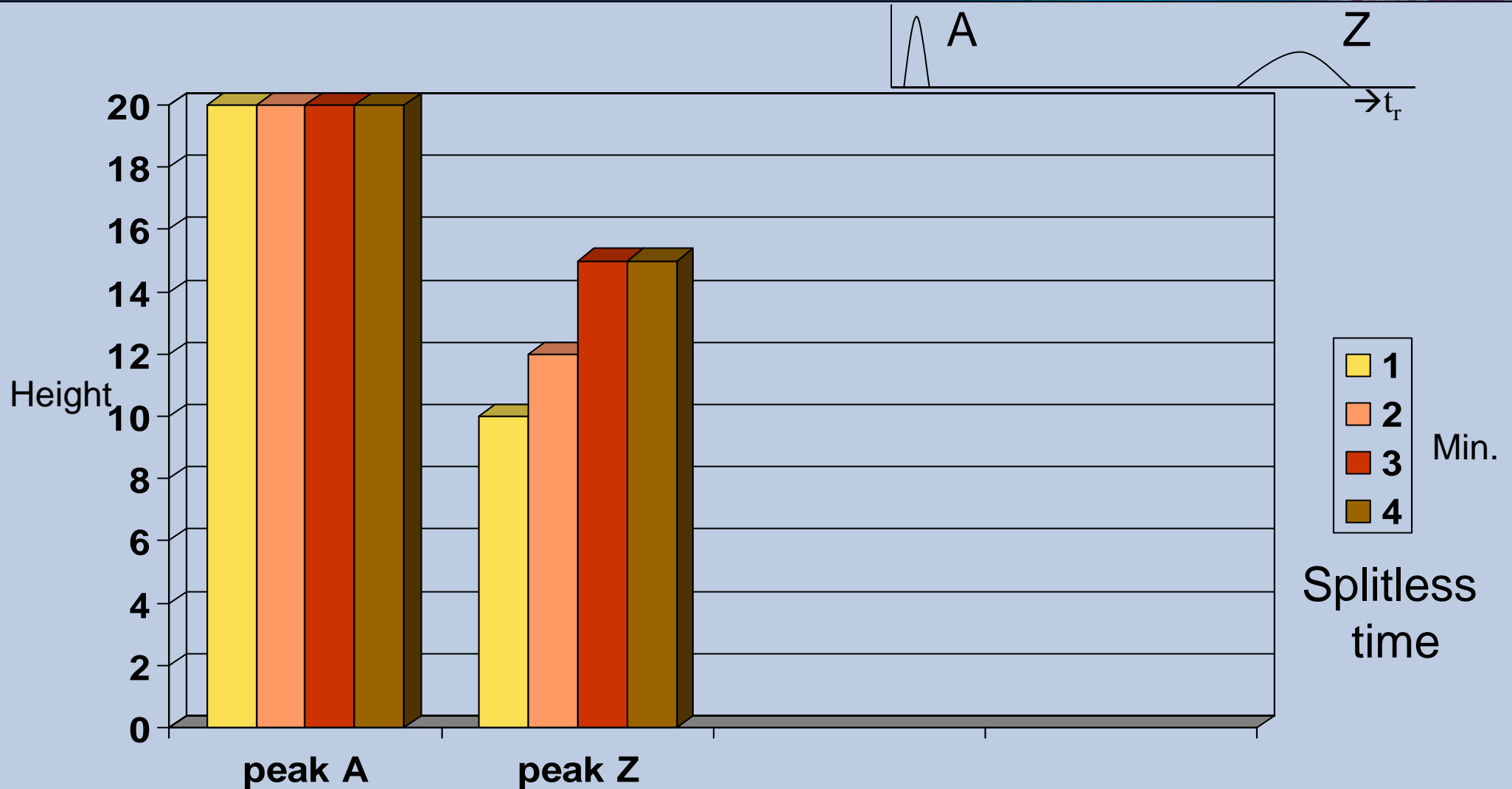
Referentie materiaal :	IRM 406; kabeljauwlever													Periode opbouw kaart												01/1/1991	u/m												17/1/2001											
Referentie waarde :	n.v.t.																									Totaal aantal waarnemingen voor opbouw kaart = 150 waarvan 2 waarnemingen geschrapt.																								
Soort analyse :	PCB analyse volgens ISW A002													<b>Kaart 8</b>																																				
Datum	0	2	2	2	2	0	0	2	2	2	2	0	0	2	0	1	2	1	2	1	0	0	0	2	0	1	0	0	1	1	1	1																		
	7	3	6	6	8	2	9	1	8	2	8	7	4	5	1	2	3	8	7	4	6	7	2	8	1	5	4	1	5	9																				
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	F	M	M	M	M	A	M	J	J	J	J	S	S	O	O	O	N	N	D	F	F	F	J	F	F	F	M	M	A	S																				
	e	a	a	a	a	p	a	u	u	u	u	e	e	c	c	c	o	o	e	e	e	e	a	e	e	e	a	a	u	e																				
	b	r	r	r	r	r	y	n	n	n	n	p	p	t	t	t	v	v	c	b	b	b	b	b	b	b	r	r	g	p																				
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	l	l	l	l	l	l	l	l	l	l	l	l	l	l	l	l	l	l	l	2	2	2	2	2	2	2	2	2	2	2																				



Gegevens over voorgaande periodes	
Gem +3S	--> 552.8
Gem +2S	--> 520.5
Gem	--> 455.9
Gem -2S	--> 391.4
Gem -3S	--> 359.1
N	--> 148
N <small>TOTAAL</small>	--> 150
N <small>GESCHRAPT</small>	--> 2
Stdev	--> 32.3
Gegevens over deze periode	
Gem +3S	--> 534.3
Gem +2S	--> 509.9
Gem	--> 461.1
Gem -2S	--> 412.3
Gem -3S	--> 387.9
N	--> 30
N <small>TOTAAL</small>	--> 250
N <small>GESCHRAPT</small>	--> 7
Stdev	--> 24.4
Gegevens over alle waarnemingen	
Gem +3S	--> 545.3
Gem +2S	--> 513.7
Gem	--> 450.4
Gem -2S	--> 387.2
Gem -3S	--> 355.5
N	--> 243
N <small>TOTAAL</small>	--> 250
N <small>GESCHRAPT</small>	--> 7
Stdev	--> 31.6
Na 30 waarnemingen:	
$S_{(ALLE\ VOORGAANDE\ WAARNEMINGEN)}$	
0.75 < ----- < 1.48	
$S_{(DEZE\ PERIODE)}$	
<b>1.32</b>	
<b>conform</b>	

Waarneming	221	222	223	224	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241	242	243	244	245	246	247	248	249	250	
Gehalte in µg/kg	445	444	452	465	446	477	522	432	485	477	503	474	473	461	414	429	473	463	475	489	442	493	450	465	456	431	474	424	443	456	
opmerking	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s19	s19	s8 (69)	s8 (69)	s19	s19	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s19(76)	s8 (69)	s8 (69)	s19(76)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	s8 (69)	
Afkeuring = X																															

# Discrimination Effects: Optimisation



# Memory Effects

- Contamination in Gass Lines
- Contamination in GC Tubing or Regulators
- Contamination from Septa
- Dirty Liner
- Glass Wool in Liner?
- Too High Concentrations in Previous Injection
- Cross Contamination from Injection Needle

# Injection Parameters

- Volume: 1-2  $\mu\text{l}$ ; smaller volumes save column and avoid front-tailing due to overloading; adjust liner to injection volume (approx. ID 4 mm for 1-2  $\mu\text{l}$ )
- Always inject at same speed (Autosampler!)
- Solvent: Boiling point ca. 100 °C: iso-octane, heptane, nonane (pentane and hexane may evaporate from the autosampler vial)
- Splitless injection: optimise temperature and splitless time (discrimination effect)
- Liner: Straight or tapered: ensure optimum transfer
- Pressure pulse: ensures rapid transfer of analytes to column (may require straight liner)

# Composition of Sample Series

VU University Amsterdam



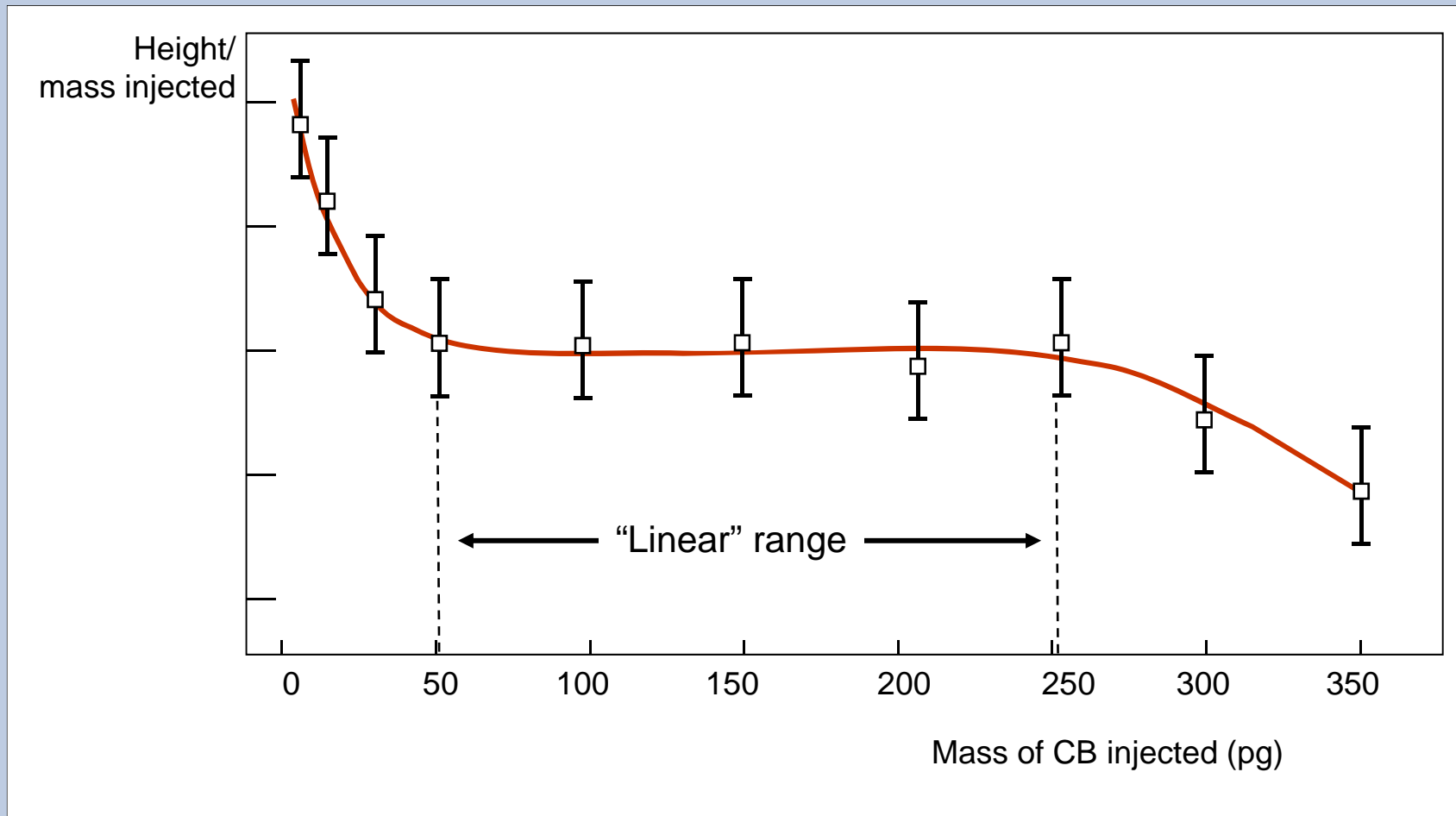
- 1) One Solvent Injection (to 'activate' EC detector)
- 2) Procedure Blank
- 3-22) At random: 5-6 Standards (multi-level) and ca. 12 Samples, and Recovery Standard
- Eventually: One of the Samples in Duplicate
  
- Syringe Standard Added to All (Except Solvent Inj.) before Injection
- Recovery Control Standard: Added to All (Except Solvent Inj.) before Extraction

# Detector Maintenance

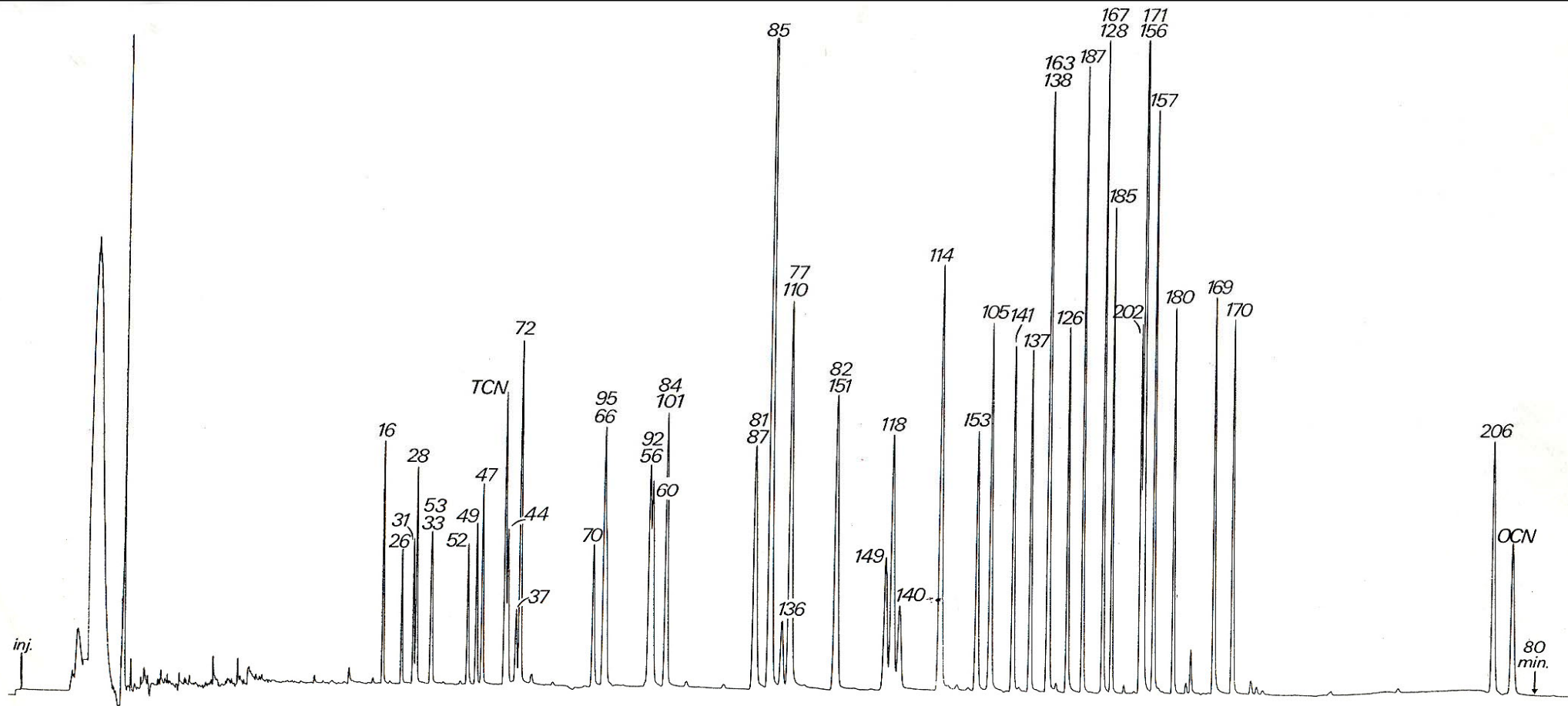
- Negative peaks following analyte peaks → signal of dirty detector, cleaning will be necessary
- Avoid oxygen entering the detector
- Hydrogen as a carrier gas keeps the detector clean
- Optimum temperature approx. 300 °C or higher
- Be very careful with radioactivity: check for leakages annually



# ECD: Linear Range

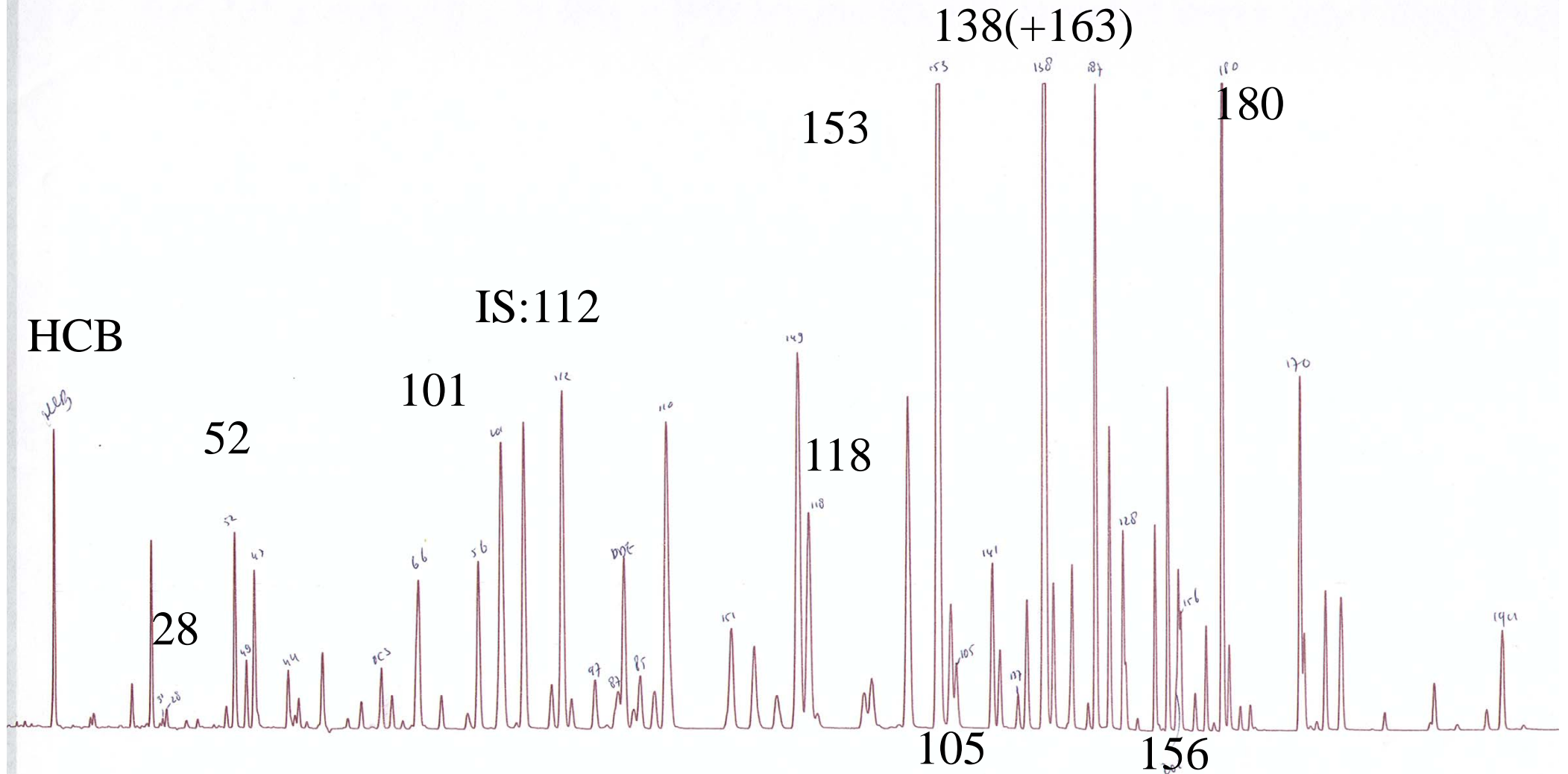


# PCBs on CP Sil 8 Column



18.6 I 12.55

dal Haringvliet oost



PCBs in eel

# Possible mistakes PCB Analysis

- Separation? - Column length
- Standards?
- Internal standards?

# General mistakes

- Calculation errors
- Weight/volume miscalculation
- Fat content
- Multi-level calibration

# Test Materials and Target Compounds

Test material	Compound class
Standard solution	OCP, dl and ndl-PCB, PCDD/Fs, PBDE/PBB, PFAS
Sediment, Netherlands	OCP, dl and ndl-PCB, PCDD/Fs, PBDE/PBB, PFAS
Pike perch fillet, Netherlands	OCP, dl and ndl-PCB, PCDD/Fs, PBDE/PBB, PFAS
Human milk, Sweden	OCP, dl and ndl-PCB, PCDD/Fs, PBDE/PBB, PFAS
Human serum, skiwax technicians, Sweden	PFAS
Air, PUF extract incinerator Sweden	dl and ndl-PCB, PCDD/Fs, PBDE/PBB, PFAS
Water, Amsterdam harbour	PFAS
Transformer oil diluted Aroclor 1254 in toluene	ndl-PCB