# Dioxin analysis in food and environmental samples

# Agenda

Maren Sander
Product Manager for Pressurized Solvent Extraction and parallel Evaporation

Sample preparation using **Pressurized Solvent Extraction**

Waldemar Weber
Product Manager for GCMS

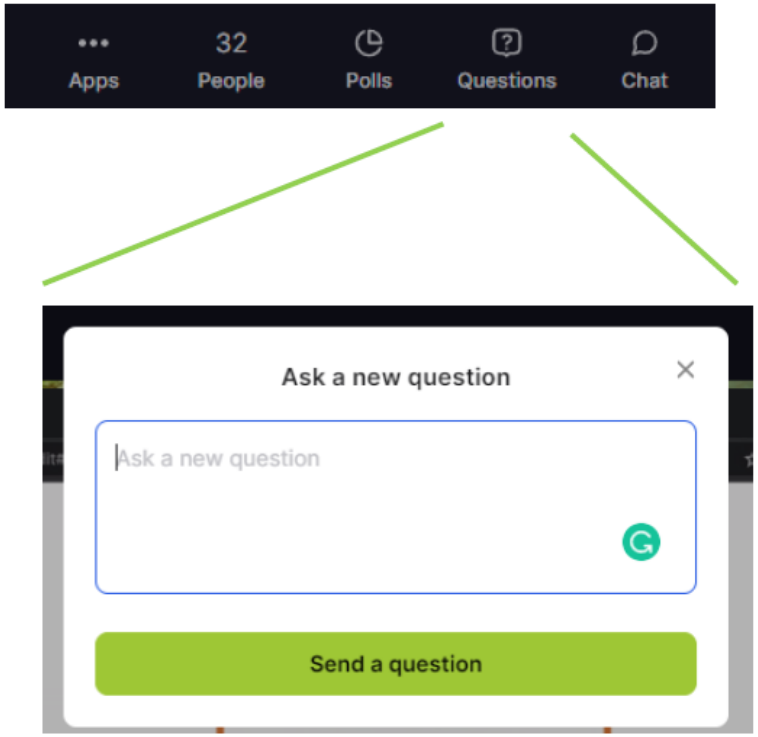
Dioxin analysis using sample purification and **GC-MS/MS**

Dr. Peter A. Behnisch
Director at Biodetection Systems BDS, Netherlands

**Case study** on how to detect dioxins and other POPs in food

# Full solution for Dioxins and PCBs analysis

# Dioxin determination




Use the “Questions” panel in the bottom right hand corner to leave a question during the presentation.

Our panelist will answer at the end of the session or per e-mail.

# Sample preparation for dioxin determination using the SpeedExtractor

The advantage of performing Pressurized Solvent Extraction

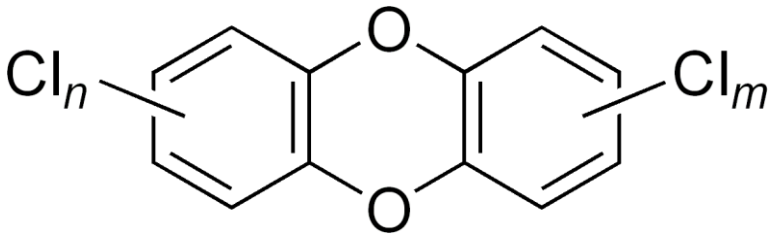
# Dioxin determination

Background

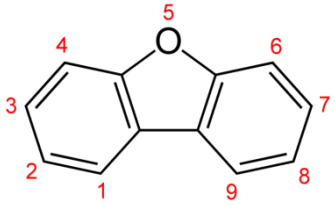
- TCDD was used as herbicide (Agent Orange); other sources: by-product of organochloride production, incineration of Cl-containing substances (e.g. PVC), natural sources (volcanoes), etc.
- ubiquitous in the environment, highly persistent and excellent bioaccumulation
- carcinogen (especially TCDD), increased risk of tumors, etc.

Legal situation

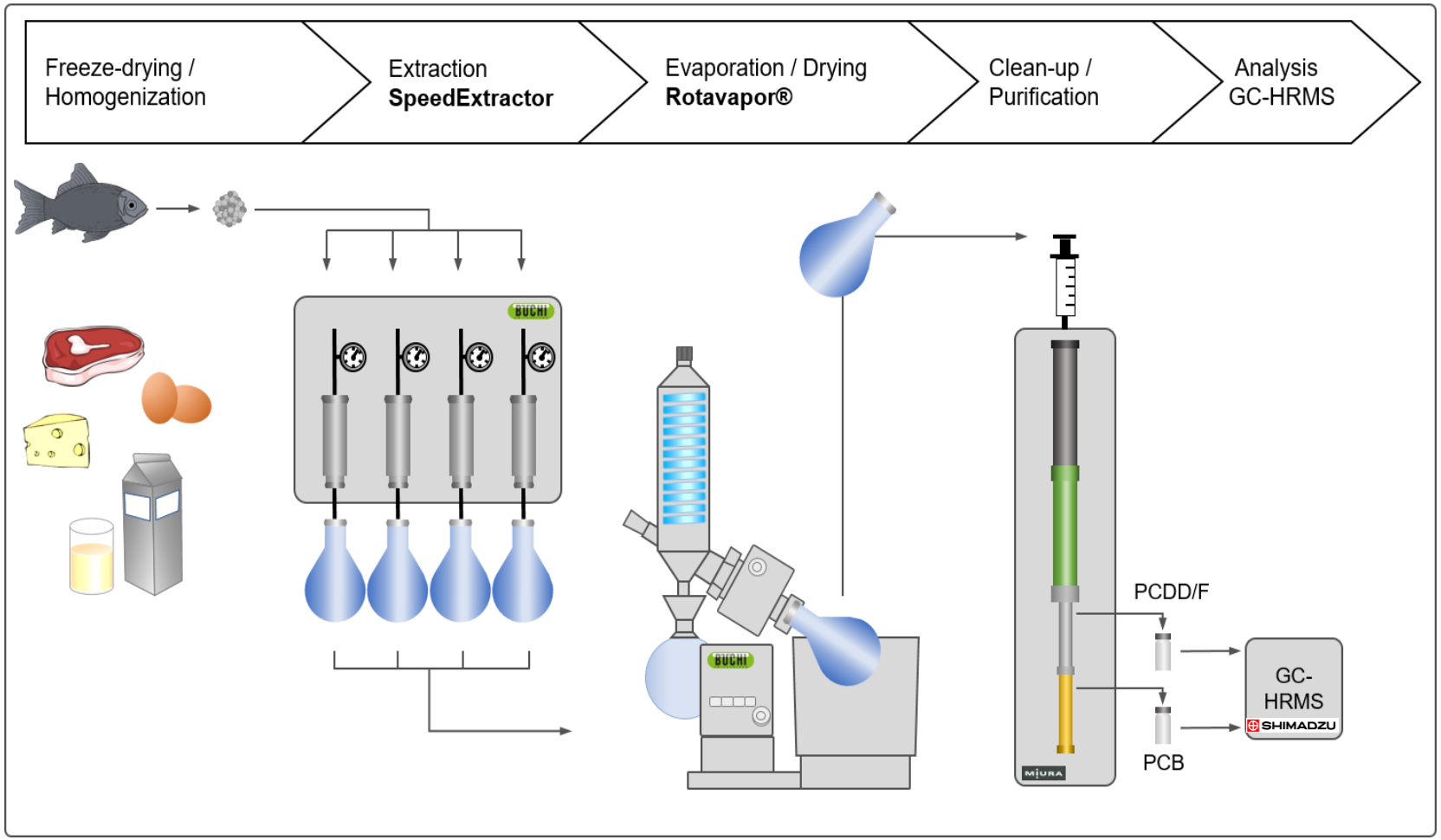
- US EPA regulations on emission and contaminations of air, water, soil, food, feed samples







# Workflow Dioxin determination



# PSE: a general overview

Determination of persistent organic pollutants (POPs) in environmental and food samples, in the chemical, petrochemical and polymer industry, for the determination of pharmaceuticals.

Technology:

- elevated pressures (50 – 150 bar) and temperatures (30 – 200 °C): accelerate the extraction process and reduce solvent consumption

SpeedExtractor:

- automated extraction instrument for simultaneous solid-liquid extraction of up to six samples

# PSE: Advantages

Temperature: → above the boiling point

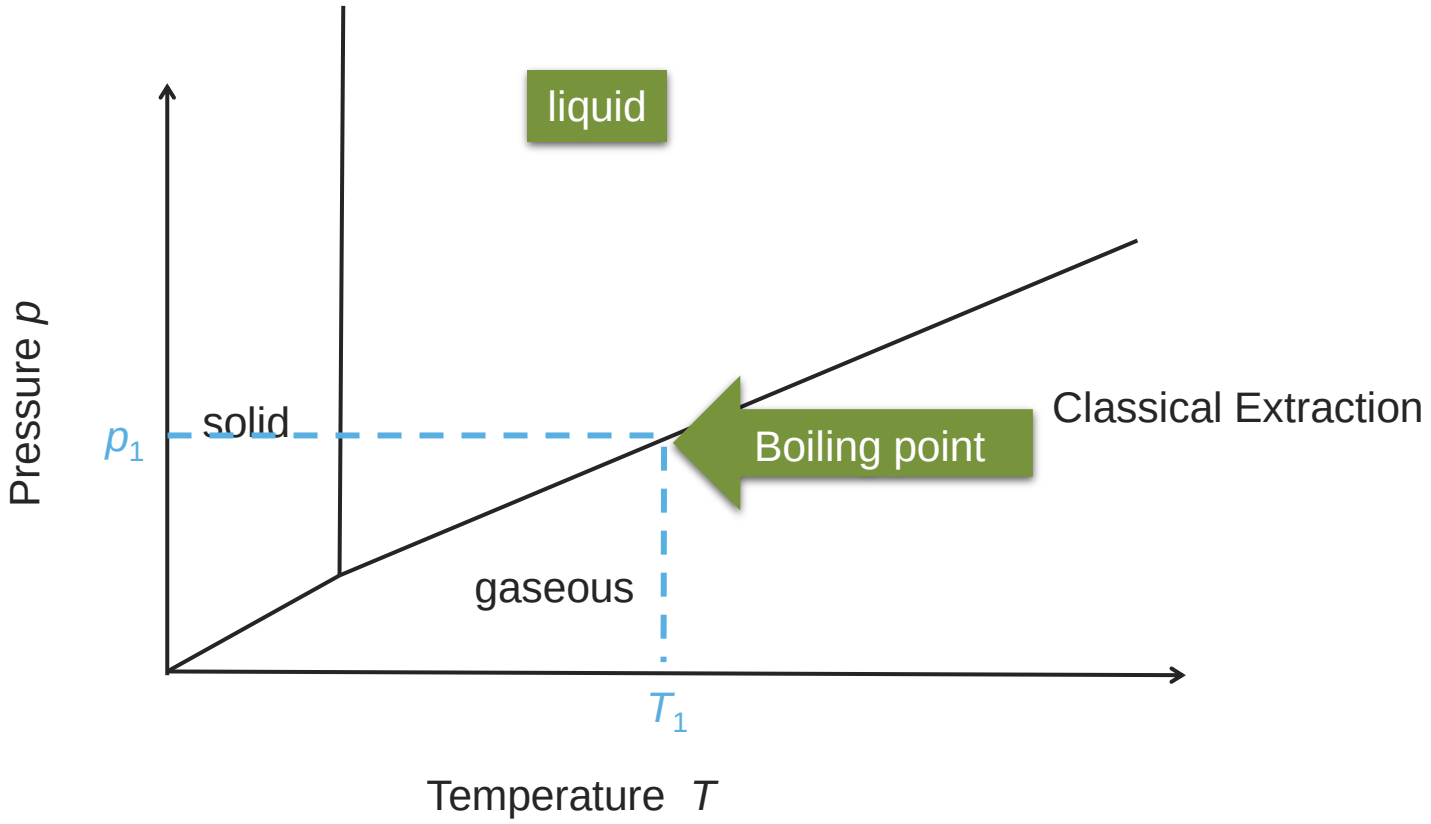
- Higher analyte solubility at high temperatures
- Increased capacity of solvents to solubilize analytes
- Faster diffusion rates

  → improved mass transfer

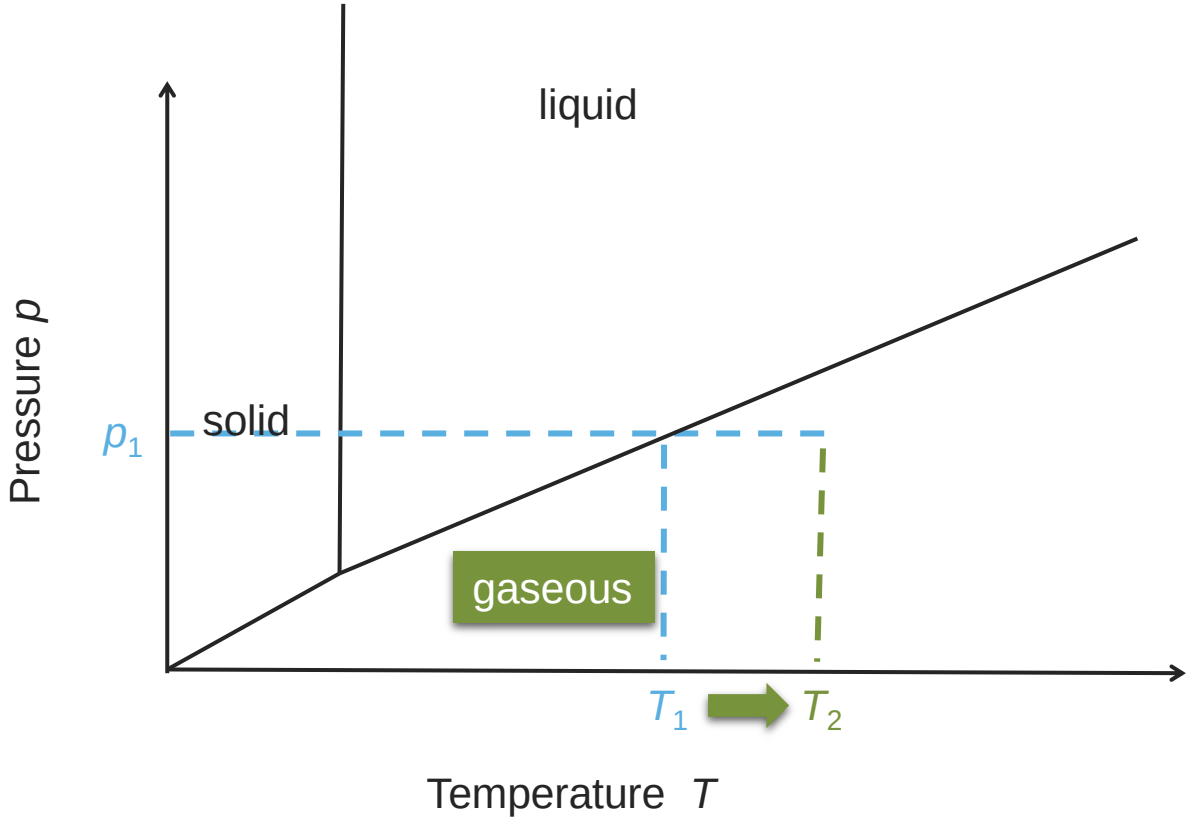
High pressure:

- Better penetration of sample matrix at high temp./press.
- Disruption of strong solute-matrix interactions
- Decreased viscosity of organic solvents
- Extraction from within the sample

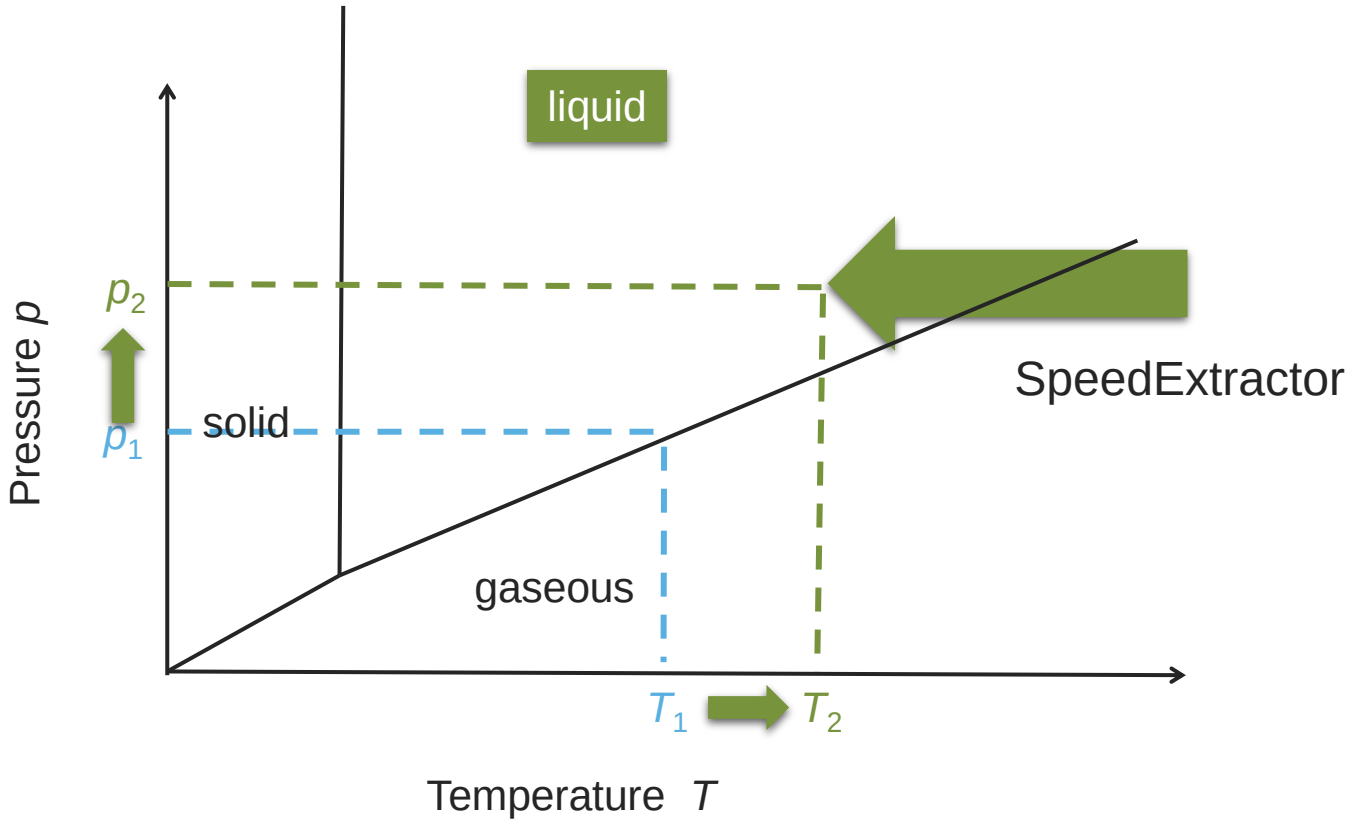
# Classical Extraction



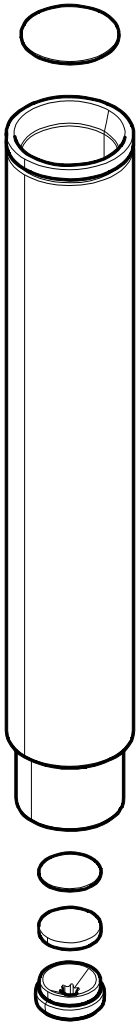
# Increase of temperature

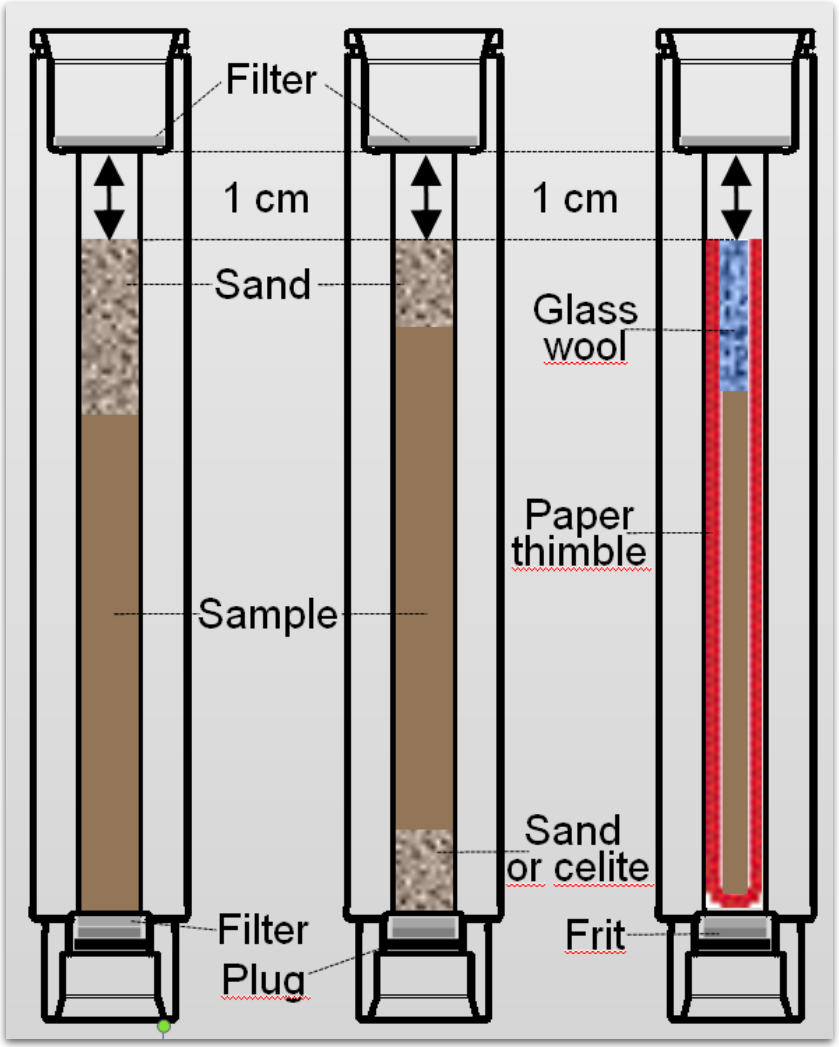


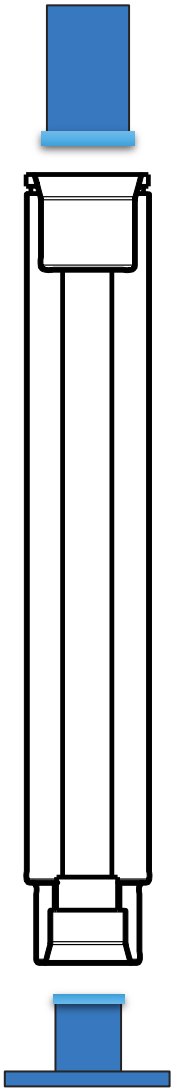
# Pressurized Solvent Extraction



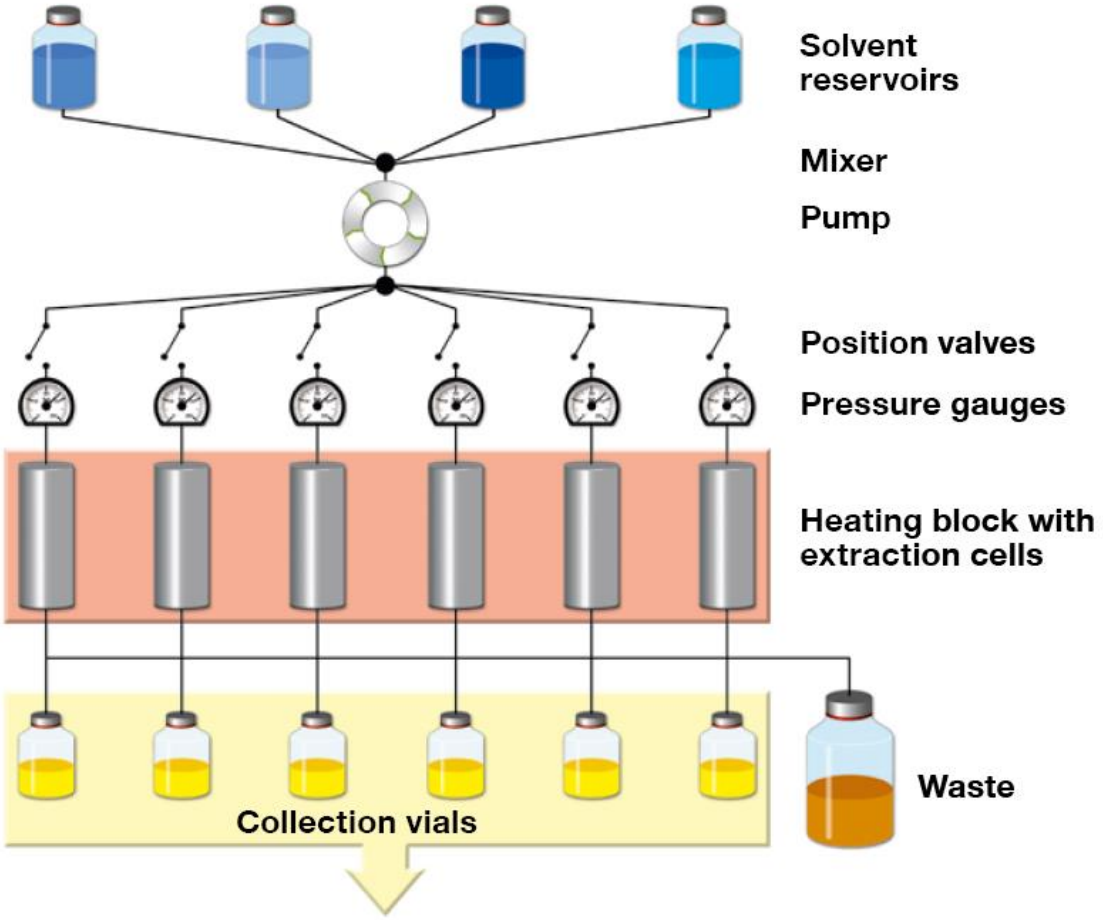
# Sample preparation and sealing





# Parallel approach – 6 samples in 20 min




# Parallel approach – Parallel evaporation

# Determination of dioxins in fish

| Extraction parameters | |
|---|---|
| Parameter | Value |
| Temperature | 100 °C |
| Pressure | 100 bar |
| Solvent | Dichloromethane 50 %<br>n-Hexane 50 % |
| Cells | 80 mL |
| Vials | 240 mL |
| Cycles | 3 |
| Heat-up | 5 / 1 / 1 min |
| Hold | 10 / 10 / 10 min |
| Discharge | 4 / 4 / 4 min |
| Flush with solvent | 2 min |
| Flush with gas | 10 min |
| Total time | 1 h 25 min |

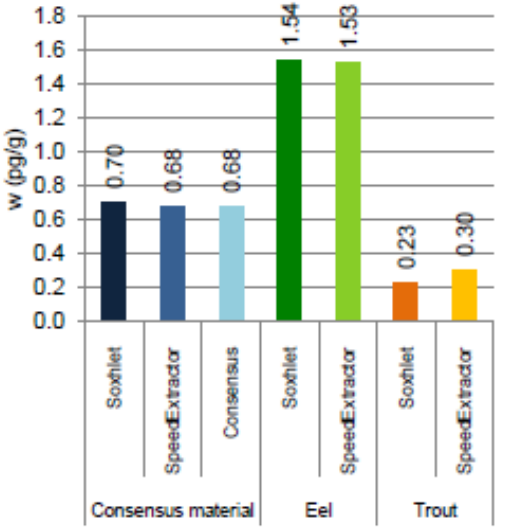



Figure 1: Median of TEQ (WHO 2005) for PCDD/Fs in consensus material. Mean of TEQ in eel and trout. Consensus material: Soxhlet n=1, SpeedExtractor n=1, consensus n=79. Eel and trout: Soxhlet n=1, SpeedExtractor n=3.


**6.5 ndl-PCBs**

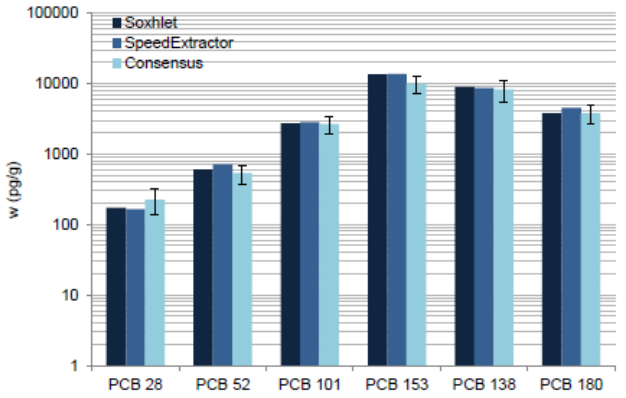
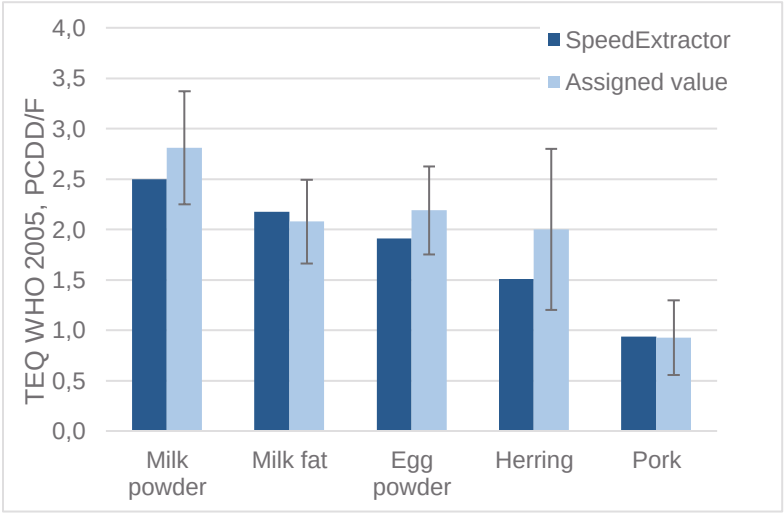



Figure 7: Mean in pg/g fresh weight for ndl-PCBs in consensus material. Results for extraction with Soxhlet and SpeedExtractor and results of consensus. Soxhlet: n=1, SpeedExtractor n=1, consensus n=57-60. Error bars: standard deviation of consensus mean.

# Determination of dioxins in food stuff

| Extraction parameters | |
|---|---|
| Parameter | Value |
| Temperature | 120 °C |
| Pressure | 100 bar |
| Solvent | Toluene 70 %<br>Acetone 30 % |
| Cells | 40 mL |
| Vials | unspecified |
| Cycles | 3 |
| Heat-up | 4 / 1 / 1 min |
| Hold | 5 / 5 / 5 min |
| Discharge | 3 / 3 / 3 min |
| Flush with solvent | 0 min |
| Flush with gas | 3 min |
| Total time | 53 min |





# Compliance with regulations

- USEPA Method 3545A (OCP, OPP, BNA, TPH, PCDD, herbicides and semi-volatiles)
- Accepted under Contract Laboratory Program (CLP) SOW OLM04.2
- USEPA Method 6860/6850: perchlorate from solid waste; PSE for extraction and clean-up
- Chinese Method GB/T 19649-2005 for 405 pesticides in grains and grain products
- German Method L00.00-34 (extended and revised version of DFG Method S 19) for pesticides in foodstuffs
- ASTM Standard Practice D-7210 for additives in polymers and D-7567 for gel content of polyolefins


# Quality in your hands