

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

The image displays three pieces of analytical equipment used for dioxin and PCB analysis. On the left, a BUCHI SpeedExtractor E-914/E-916 is shown, featuring a white control cabinet with a digital display and a lower unit with a tray of glass flasks. A laptop is connected to the machine, showing a software interface. In the center, a MIURA GO-2EHT/4EHT/6EHT unit is displayed, consisting of a control panel and a series of vertical extraction columns. On the right, a Shimadzu GCMS-TQ NX Series mass spectrometer is shown, featuring a black oven and detector unit with a built-in computer monitor.

BUCHI
SpeedExtractor
E-914/E-916

MIURA GO-2EHT/4EHT/6EHT

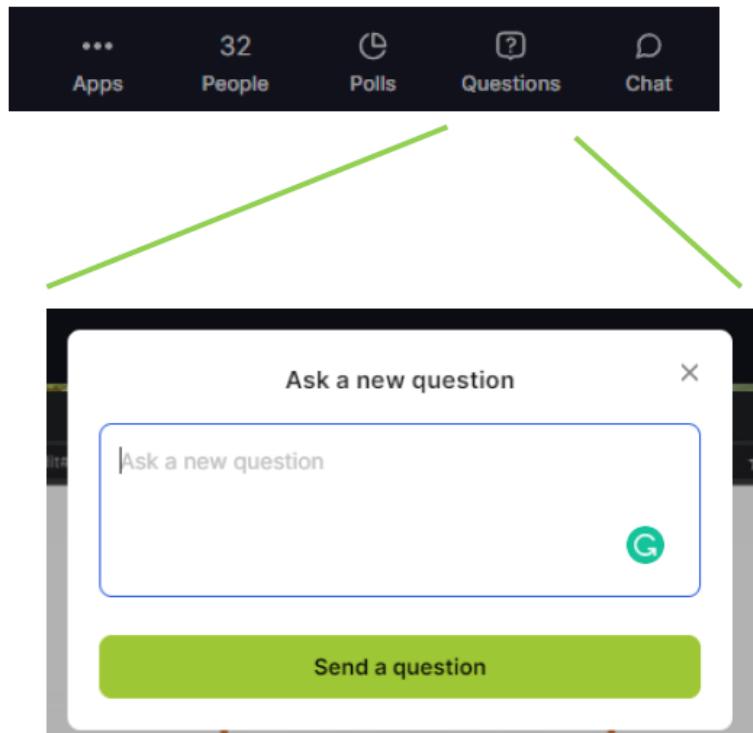
Shimadzu GCMS-TQ NX Series

Pressurized Solvent Extraction
BUCHI SpeedExtractor E-914/E-916

Powerful Clean-Up
MIURA GO-2EHT/4EHT/6EHT

Smart GC-MS/MS Analysis
Shimadzu GCMS-TQ8050 NX

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



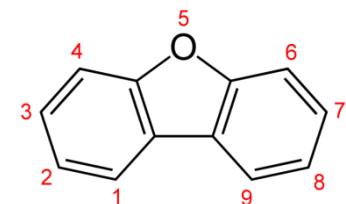
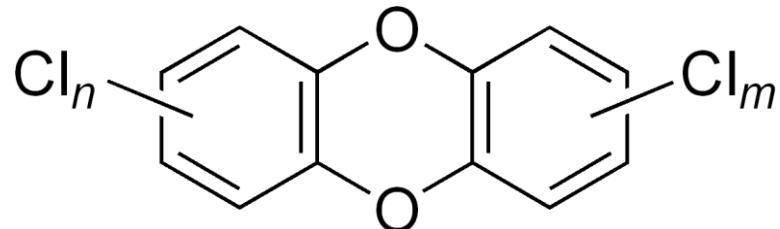
Dioxin determination

Background

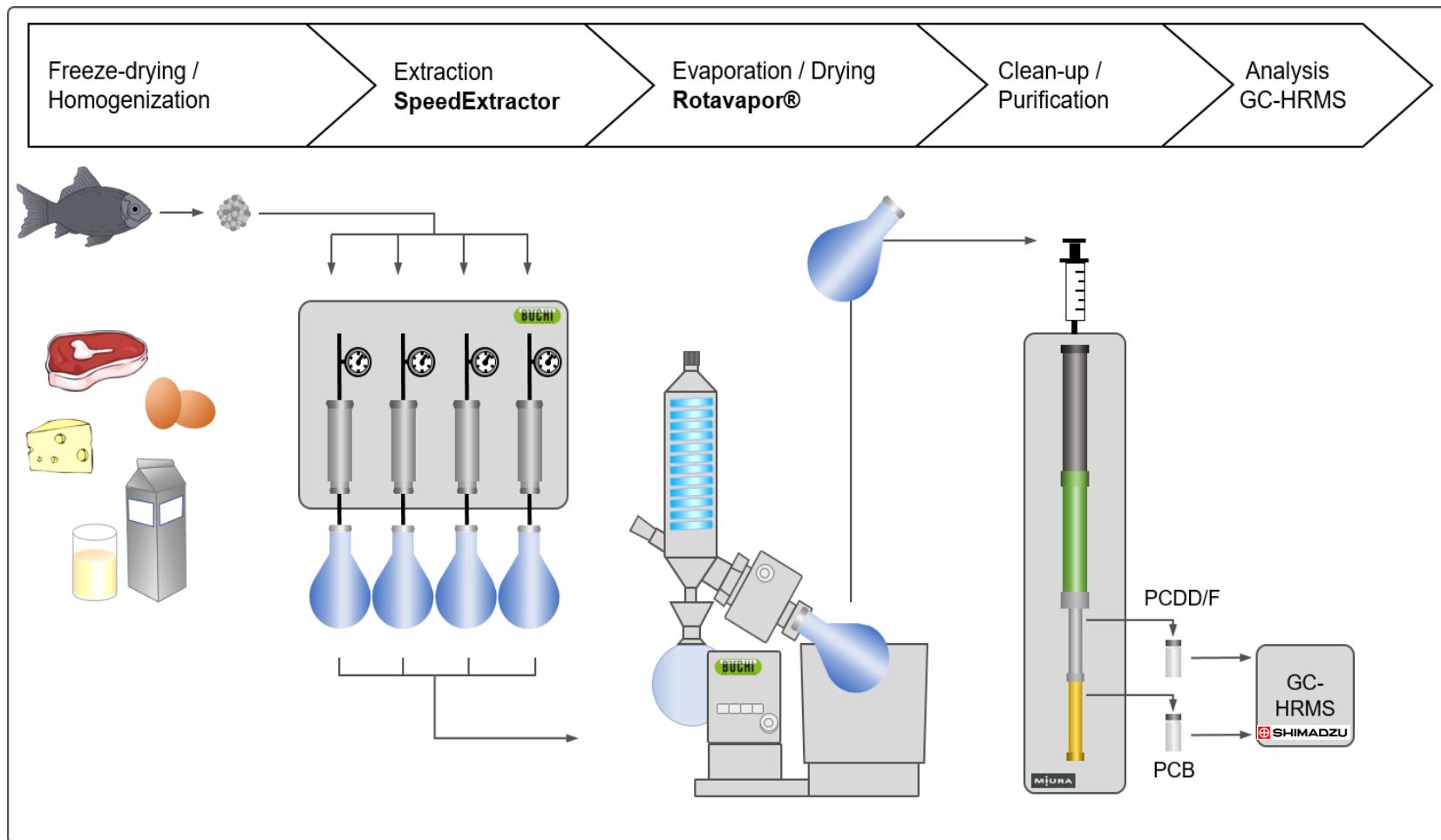
- TCDD was used as herbicide (Agent Orange); other sources: by-product of organochloride production, incineration of Cl-containing substances (e.g. PVC), natural sources (volcanoes), etc.
- ubiquitous in the environment, highly persistent and excellent bioaccumulation
- carcinogen (especially TCDD),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

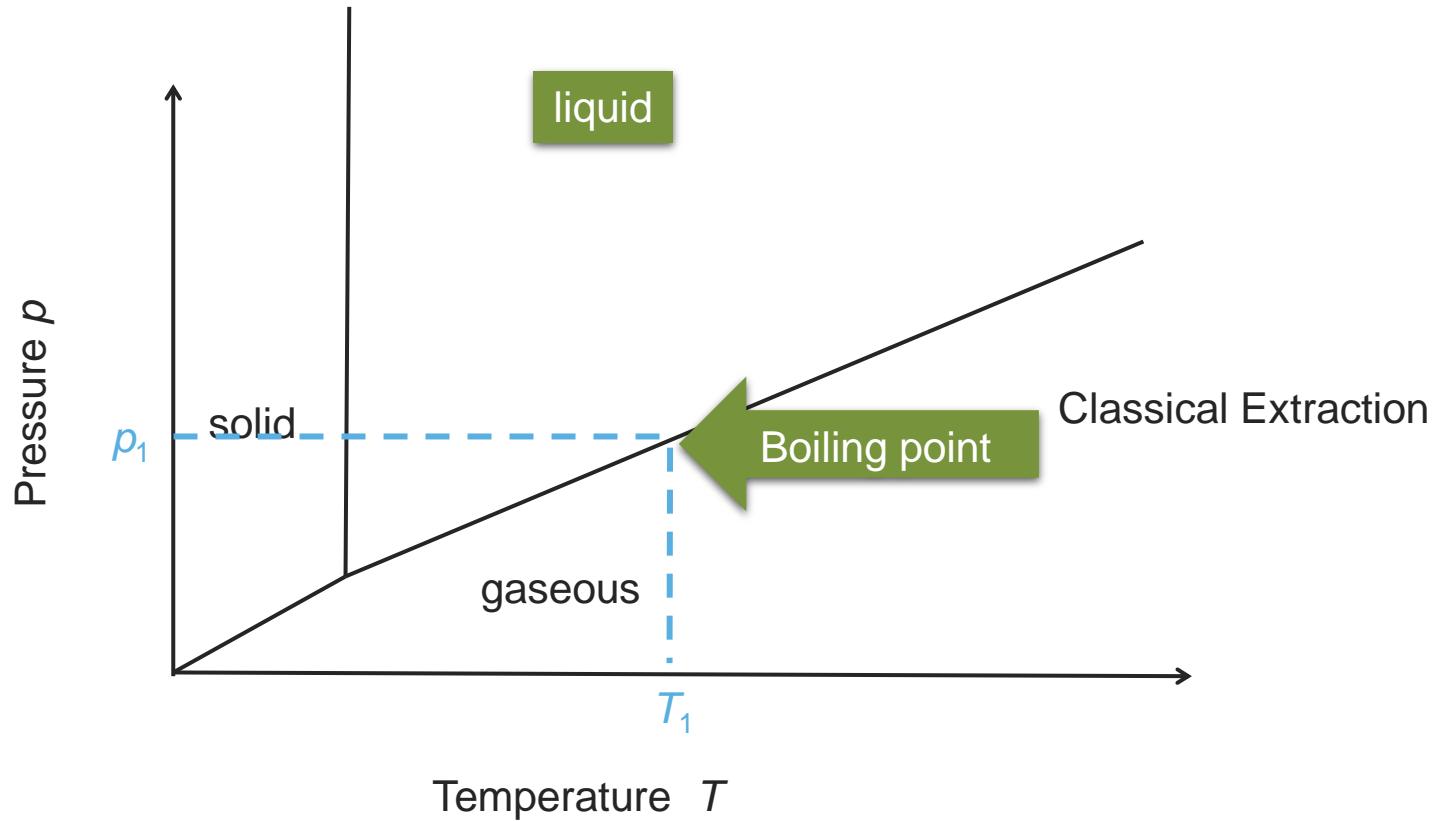
- Higher analyte solubility at high temperatures
- Increased capacity of solvents to solubilize analytes
- Faster diffusion rates
→ improved mass transfer



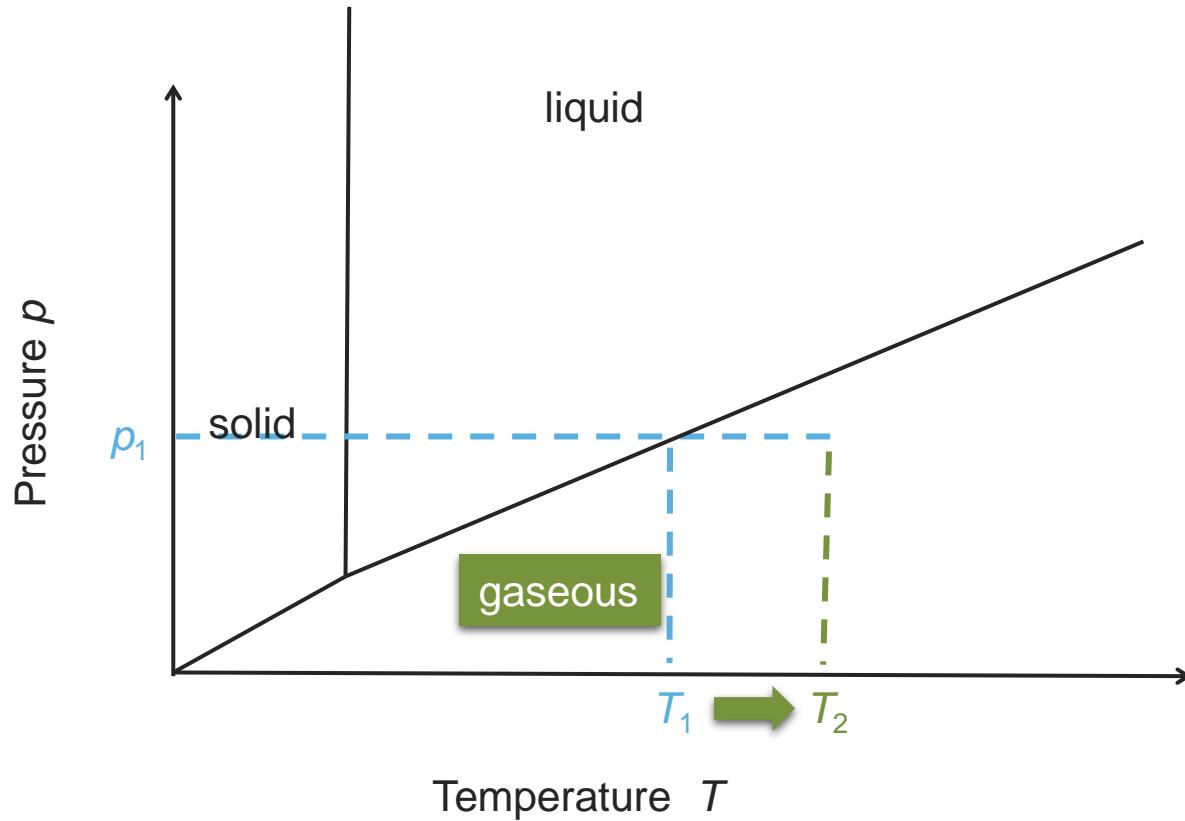
High pressure:

- 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

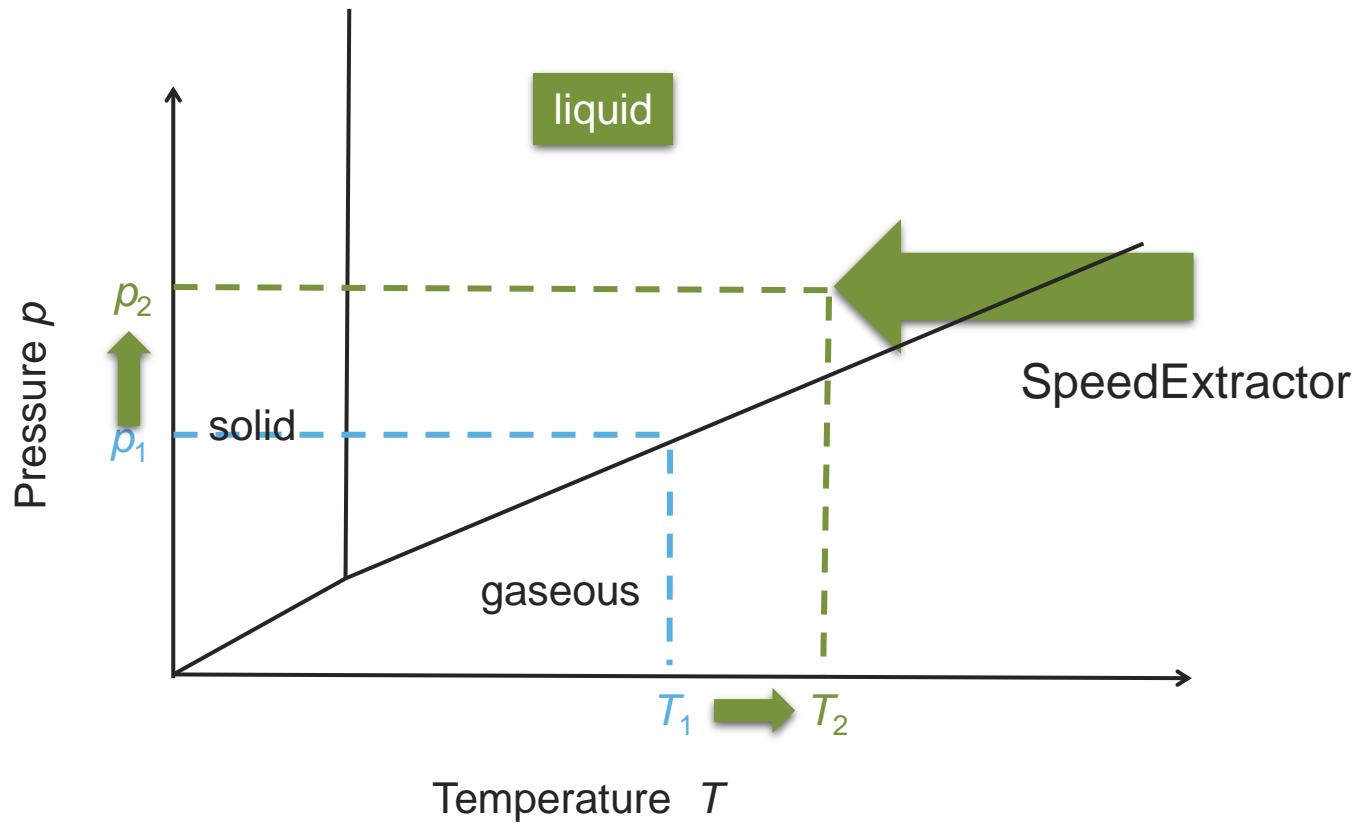
Classical Extraction



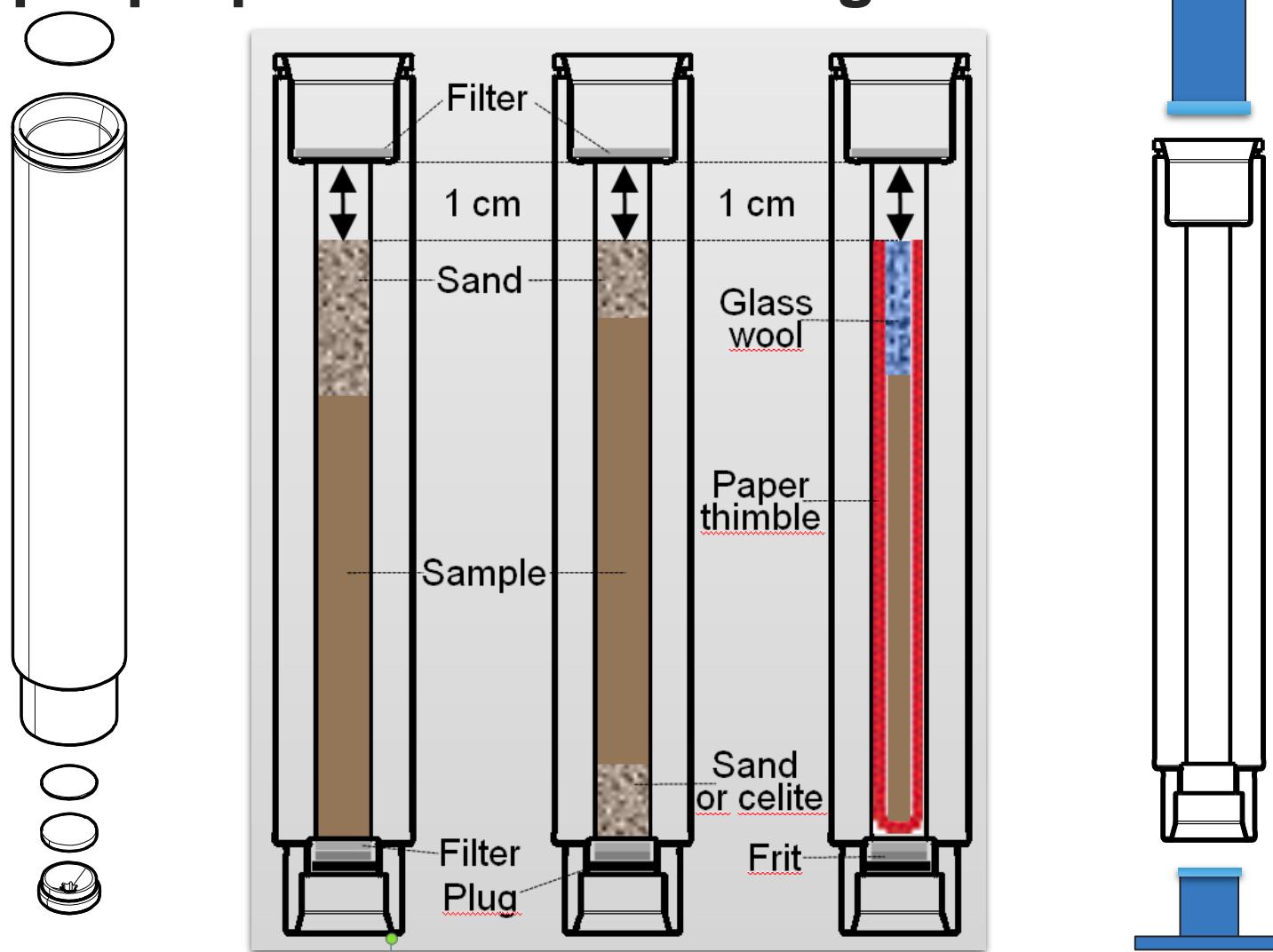
Increase of temperature



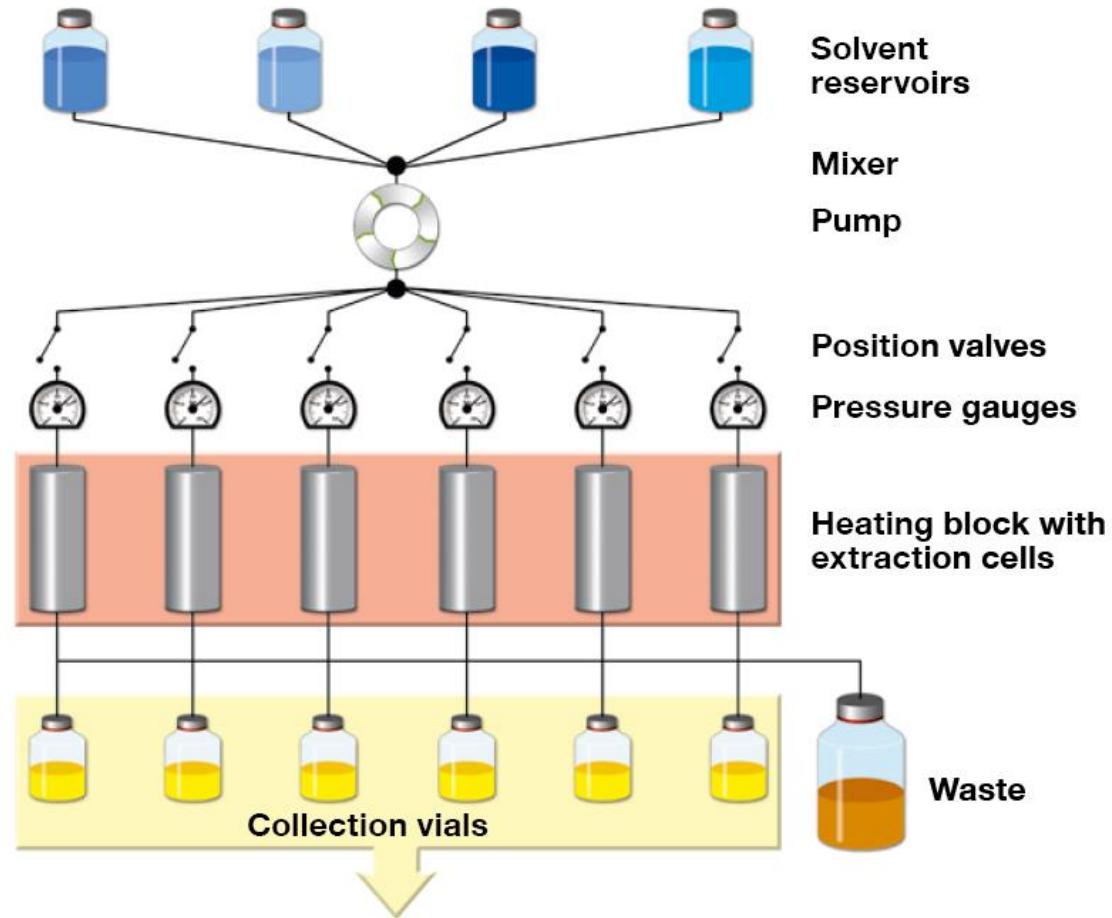
Pressurized Solvent Extraction



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 % 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

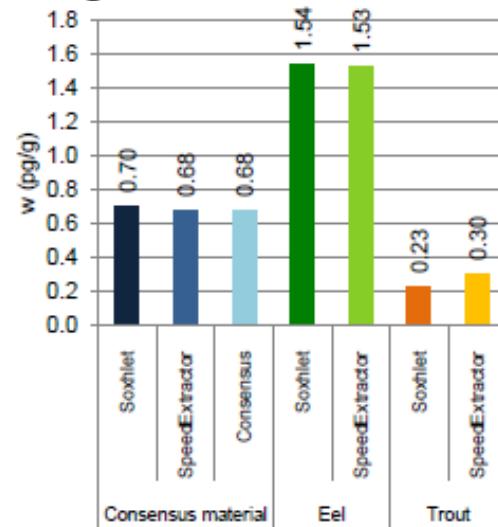


Figure 1: Median of TEQ (WHO 2005) for PCDD/Fs in consensus material. Mean of TEQ In eel and trout. Consensus material: Soxhlet n=1, SpeedExtractor n=1, consensus n=79. Eel and trout: Soxhlet n=1, SpeedExtractor n=3.

6.5 ndl-PCBs

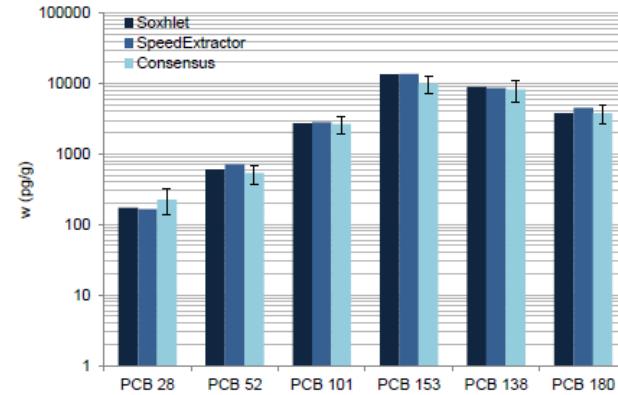


Figure 2: Mean in pg/g fresh weight for ndl-PCBs in consensus material. Results for extraction with Soxhlet and SpeedExtractor and results of consensus. Soxhlet n=1, SpeedExtractor n=1, consensus n=57-60. Error bars: standard deviation of consensus mean.

Application Note



SpeedExtractor E-144
Extraction of PCDD/Fs and PCBs in fish using SpeedExtractor E-144

Determination of dioxins in food stuff

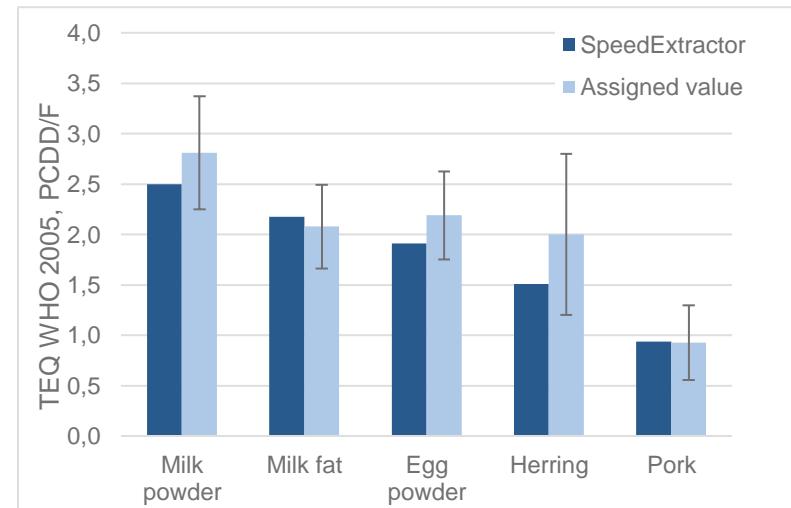
Extraction parameters	
Parameter	Value
Temperature	120 °C
Pressure	100 bar
Solvent	Toluene 70 % Acetone 30 %
Cells	40 mL
Vials	unspecified
Cycles	3
Heat-up	4 / 1 / 1 min
Hold	5 / 5 / 5 min
Discharge	3 / 3 / 3 min
Flush with solvent	0 min
Flush with gas	3 min
Total time	53 min



BUCHI Application Note No. 205-2015
Dioxin determination in foodstuffs
SpeedExtractor® E HT
Determination of Dioxins and Dioxin-like Compounds in Foodstuffs using SpeedExtractor® E HT for the Determination of Dioxins in Dairy, Egg, Fish and Meat, Unived Vaudem, Luc Dassier and Claude Baer, AgroParisTech, Paris, France; Université de Lorraine, Nancy, France; Institut Pasteur, Paris, France.



www.buchi.com Quality in your hands



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



BÜCHI Labortechnik AG