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Welcome to the webinar

Sample purification and GC – MS/MS for dioxin analysis

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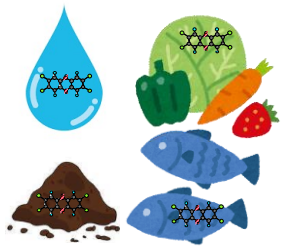
Shimadzu Europa GmbH

Background

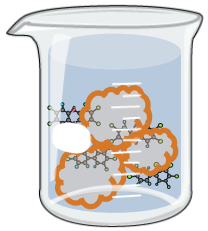
- **Market trend**
  - In the past, Dioxins in food had been analysed by GC HRMS
  - Recently, GC MS/MS was also confirmed as official method
- **Requirement of customer**
  - Start analysis immediately without adjusting the analysis conditions
  - Create reports showing items required by EU regulations
  - Compare the respective quantitative capabilities of GC MS/MS and GC HRMS
  - Fast and simple sample preparation

Manual vs. Automated Sample Prep.

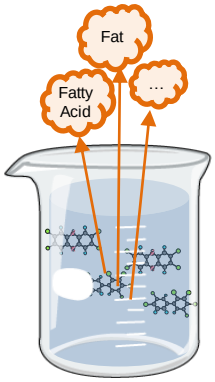
Manual sample preparation



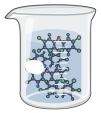
Sampling



Extraction



Purification



Concentration

> 1 days

> 2 days

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Measurement

BUCHI SWITZERLAND

2 h

MIURA

2 h

Saves time
Saves solvents
Saves costs

New fully automatic sample preparation system for Dioxin analysis

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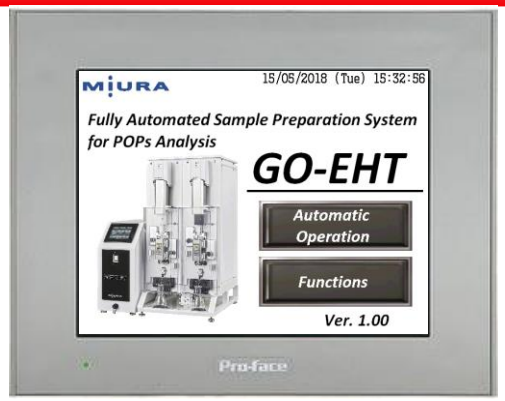
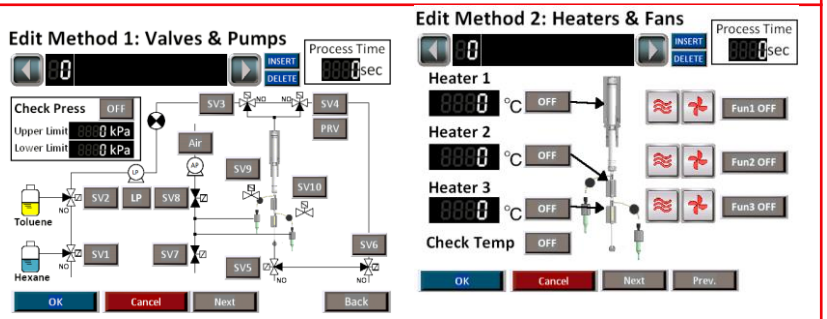
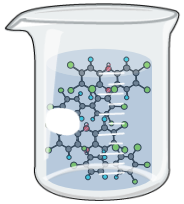
Features of the Miura GO-EHT System

- **High performance clean-up**
  - 4 columns → Better purification
  - Heated purification and elution → Faster elution
- **User-friendly design**
  - Easy to apply sample
  - Easy connections → No washing
  - Disposable parts → No cross-contamination
- **Unique flow switching**

Perfect separation of PCBs and dioxins in one fraction each with excellent quality

New Miura GO-EHT Features

	GO-EHT series
Touch panel	 **7.5 inch**
Power supply	100-240 V, 50/60 Hz
Method programs	 **Flexible**
Basic method time	80 mins (possible to further optimize)
Functions	Dry-air leak check before start
Data log	P, T, Operation/Alarm



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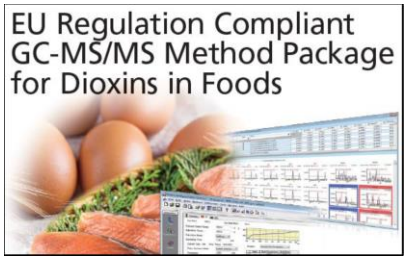
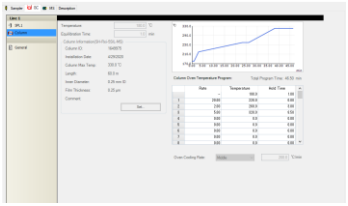
Dioxin Analyzer

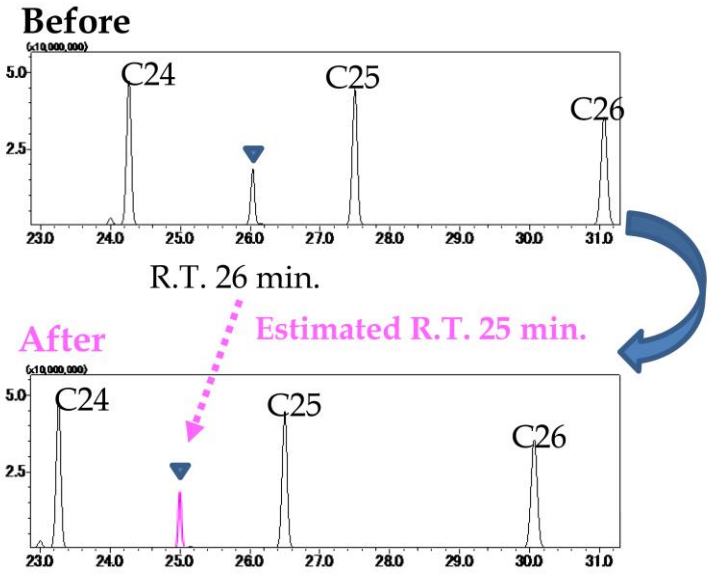


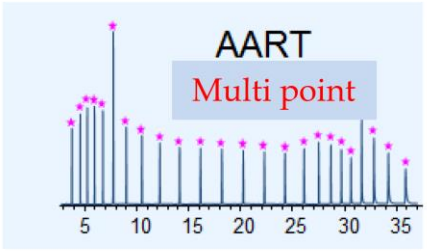
➢ 1. Method Files Registered with the Optimal Conditions

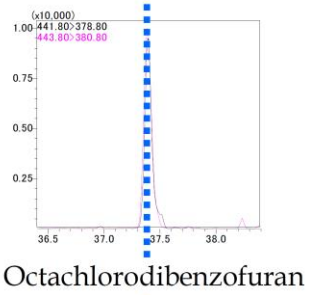
- Method files for DXNs, PCBs, and BFRs



- Automatic adjustment of retention times (AART)







Octachlorodibenzofuran

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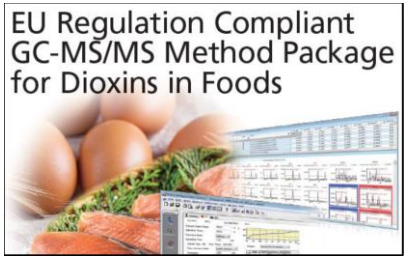
Dioxin Analyzer



- **2. Report creation tool, capable of outputting items required by regulation**

- **3. Method file performance confirmed by the analysis of 44 types/201 samples of foods and feeds**

- Animal feed product
- Bétaïne anhydre
- BétaïneHCL 95%
- Bovine fat
- Bovine milk
- Bovine muscle
- Compound fish food
- Dairy product
- Diverse nature
- Eels
- Fish
- Fresh product
- Game liver
- Goat fat
- Goat liver
- Grasses
- Milk
- Molluscs
- Mussels
- Oilcake
- Ovine fat
- Ovine liver
- Oysters
- Pork fat
- Poultry eggs
- Poultry muscle
- Powder
- QC
- Salmon
- Sardine
- Scallops
- Shellfish
- Shrimp
- Thréronine
- Veal fat
- Vitamine K4
- Yellow Pigment
- other

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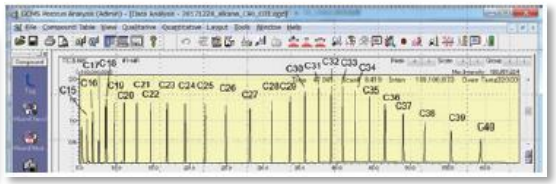
Steps from the Preparation for Analysis

Step 1

Preparation for analysis

Step 2

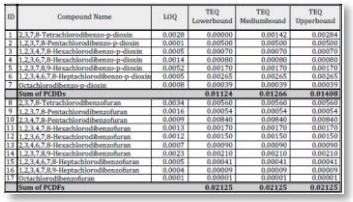


Creation of method files
AART, Calibration curve

Step 3

Analysis of samples

Step 4



ID	Compound Name	LOQ	TEQ Lowerbound	TEQ Mediumbound	TEQ Upperbound
1	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.0028	0.00000	0.00142	0.00284
2	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.0001	0.00500	0.00500	0.00500
3	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.0005	0.00070	0.00070	0.00070
4	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.0014	0.00080	0.00080	0.00080
5	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.0052	0.00170	0.00170	0.00170
6	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	0.0005	0.00265	0.00265	0.00265
7	Octachlorodibenzo-p-dioxin	0.0008	0.00039	0.00039	0.00039
	Sum of PCDDs		**0.01124**	**0.01266**	**0.01408**
8	2,3,7,8-Tetrachlorodibenzofuran	0.0034	0.005601	0.005601	0.005601
9	1,2,3,7,8-Pentachlorodibenzofuran	0.0016	0.00054	0.00054	0.00054
10	2,3,4,7,8-Pentachlorodibenzofuran	0.0009	0.00840	0.00840	0.00840
11	1,2,3,4,7,8-Hexachlorodibenzofuran	0.0013	0.00170	0.00170	0.00170
12	1,2,3,6,7,8-Hexachlorodibenzofuran	0.0012	0.00150	0.00150	0.00150
13	2,3,4,6,7,8-Hexachlorodibenzofuran	0.0007	0.00090	0.00090	0.00090
14	1,2,3,7,8,9-Hexachlorodibenzofuran	0.0023	0.00210	0.00210	0.00210
15	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0005	0.00041	0.00041	0.00041
16	1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.0004	0.00009	0.00009	0.00009
17	Octachlorodibenzofuran	0.0001	0.00001	0.00001	0.00001
	Sum of PCDFs		**0.02125**	**0.02125**	**0.02125**

Creation of reports

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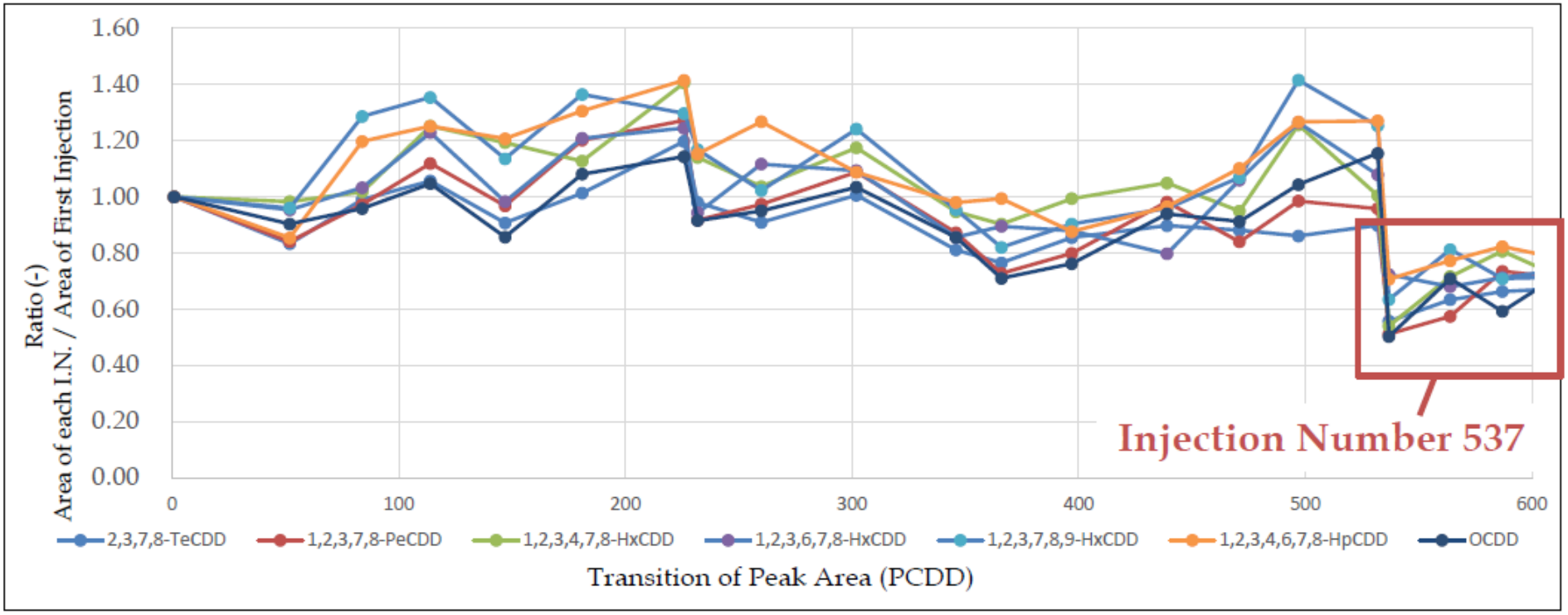
Analysis result

I.D.	Compound Name	Calibration Point Concentration						Average RRF	RRF (level 1)	Dev(%) *Criteria <30%
		Level 1 (pg/uL)	Level 2	Level 3	Level 4	Level 5	Level 6			
1	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.025	0.050	0.100	0.250	0.500	1.000	1.212	1.144	5.60
2	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.025	0.050	0.100	0.250	0.500	1.000	1.089	0.990	9.11
3	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.025	0.050	0.100	0.250	0.500	1.000	1.106	1.157	-4.62
4	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.025	0.050	0.100	0.250	0.500	1.000	1.043	1.043	-0.06
5	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.025	0.050	0.100	0.250	0.500	1.000	1.039	0.936	9.95
6	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	0.025	0.050	0.100	0.250	0.500	1.000	1.033	1.115	-7.86
7	Octachlorodibenzo-p-dioxin	0.050	0.100	0.200	0.500	1.000	2.000	1.180	1.261	-6.84
8	2,3,7,8-Tetrachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.159	1.213	-4.65
9	1,2,3,7,8-Pentachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.047	0.974	6.94
10	2,3,4,7,8-Pentachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.038	0.962	7.35
11	1,2,3,4,7,8-Hexachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.106	1.358	-22.81
12	1,2,3,6,7,8-Hexachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.052	1.134	-7.82
13	2,3,4,6,7,8-Hexachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.000	0.923	7.67
14	1,2,3,7,8,9-Hexachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.021	1.205	-18.09
15	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.097	1.157	-5.46
16	1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.025	0.050	0.100	0.250	0.500	1.000	1.056	1.080	-2.27
17	Octachlorodibenzofuran	0.050	0.100	0.200	0.500	1.000	2.000	0.981	0.975	0.66

Result of the robustness test

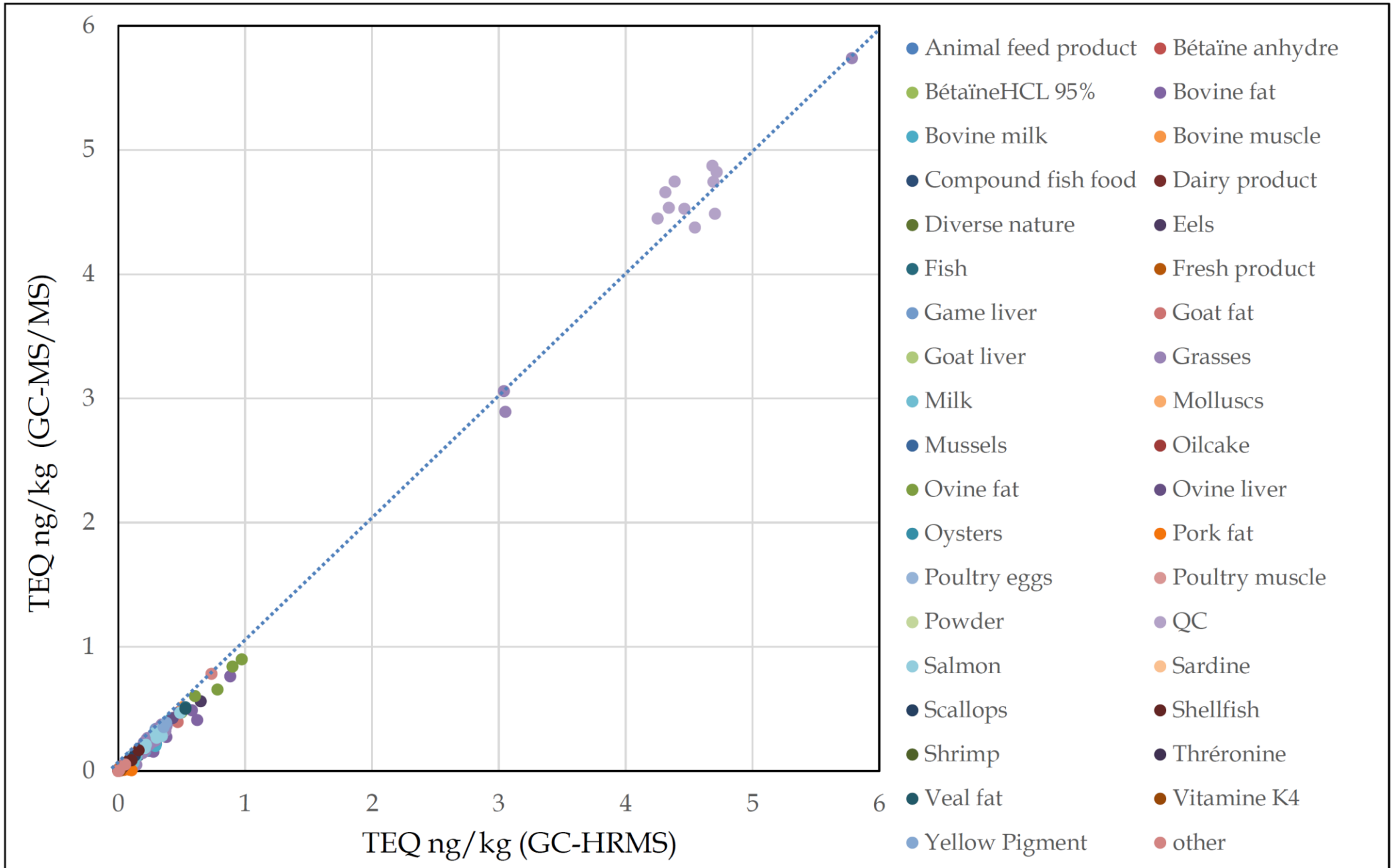
- **Transition of the sensitivity of STD (0.05 pg/uL)**

STD → Test sample



Lifetime was over than 500 injection

Method validation



Dioxins S³ Smart Solution Systems

Extraction

Clean-up

Analysis

Thank You for your Attention