C146-E375

SHIMADZU
Excellence in Science

GC-MS/MS Handbook for the Analysis of Dioxins in Foods

Procedures for the Analysis of Dioxins in

Extraction of Samples

SpeedExtractor E-914/E-916 (BÜCHI Labortechnik AG)

The SpeedExtractor is your best solution for fast Pressurized Solvent Extraction (PSE). Increase productivity by processing up to 6 samples in parallel. Streamline the workflow of your sample preparation with easy sample loading and ready-to-use extract collection.

Analysis of Samples

GCMS-TQ8050 (SHIMADZU)
(GC-MS/MS Method Package for Dioxins in Foods)

The method package consists of method files registered with the optimal conditions for the analysis of dioxins, as well as a report creation tool that can output the items required by EU regulations. This makes it possible to start an analysis without having to investigate analytical conditions.

Foods Using GC-MS/MS

Purification of Samples

GO-xHT (MIURA CO.,LTD.)

GO-xHT is an easy-to-operate automated system offering high throughput as well as the additional advantage of using less solvents and consumables. It provides labs with high-quality extraction as well as high return on investment thanks to an innovative flow path system.

Extraction of Samples

SpeedExtractor E-914/E-916

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Unrivaled throughput

- 6 times faster than other PSE instruments thanks to parallel extraction concept
- Extract up to 96 samples in an 8 hour shift
- Reduce number of replicates due to identical extraction conditions for up to 6 samples

→ Point 1

Ease of operation

- Unique design of extraction cells allows for ease of sample loading and cell assembly
- High level of automation saves time-consuming preparation and increases safety

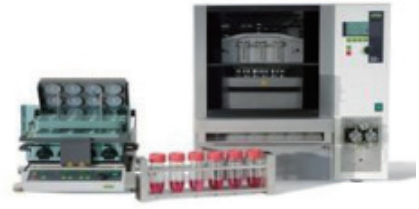
→ Point 2

- The automated sealing eliminates sources of error and guarantees safety

→ Point 3

Low running costs

- Reduced solvent consumption and minimal need for consumables give lower running costs
- Save energy and costs with ECO mode for automated heater control



Point 1 Parallel workflow for high throughput

Significantly increase throughput with the concerted parallel extraction and parallel evaporation. Full compatibility of the SpeedExtractor collection glassware with Syncore™ Analyst and Multivapor™.

Point 2 Extraction made easy

The operation of the SpeedExtractor is easy and intuitive. Apply an established method at the push of a button and extract simultaneously up to 6 samples.

Point 3 Automated sealing

The fail-safe and reliable extraction cells seal automatically using a unique sealing principle and guarantee reproducible results.

Six Steps from the Preparation of Samples, Multi-Sample Extraction, to Multi-Sample Distillation

Step 1 Place the samples in the extraction cell.

Step 2 Attach the extraction cell to the heating block.

Step 3 Close the protective shield and press the button. Six samples (or four samples) are extracted automatically.

Step 4 After the extraction is completed, remove the collection unit.

Step 5 Transfer the samples from the collection vials to the parallel evaporator.

Step 6 Multiple samples are simultaneously distilled by the parallel evaporator.

Purification of Samples

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Heating purification

- The quickness and selectivity of your current DXNs and PCBs analysis can be improved by using this technique. → Point 1

Less than 100 mL of solvent per sample

- It uses only 90 mL of hexane and 2 mL of toluene, without dichloromethane. Thus, this system is both user and eco friendly. → Point 2

No cross contamination in the valve-less system

- There are no solenoid valves on the DXNs and PCBs flow lines. All columns and tubings are disposable. Thus, there is no possibility of cross contamination.

*Please consult the relevant laws and regulations in your country before going for disposal.

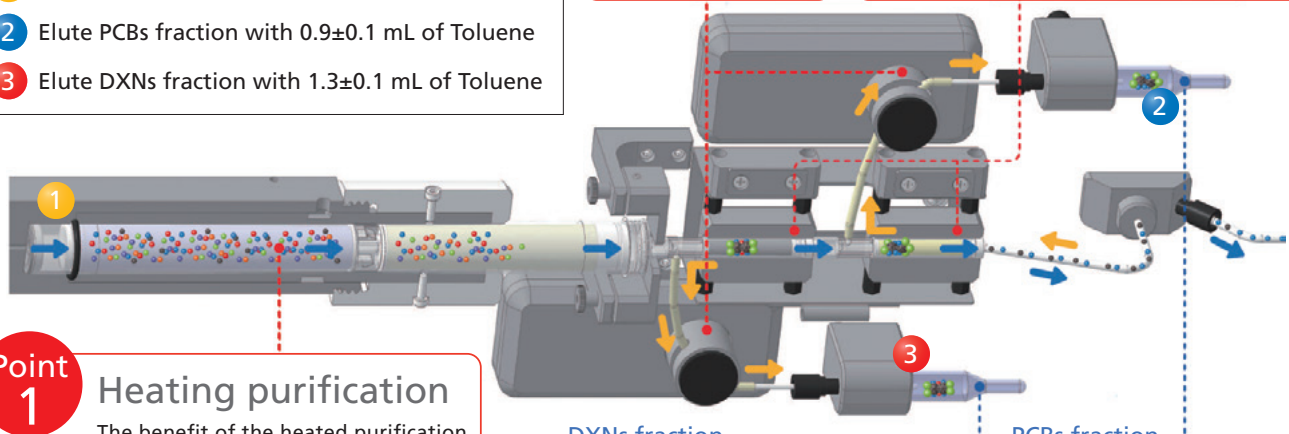
Point 2

Less than 100 mL of solvent

The benefit of a heated concentration column is that the amount of toluene necessary for elution of DXNs and PCBs elution is decreased.

A unique method for solvent flow switching with pinch valves.

1. Clean up with 90 mL of Hexane
2. Elute PCBs fraction with 0.9±0.1 mL of Toluene
3. Elute DXNs fraction with 1.3±0.1 mL of Toluene



Point 1

Heating purification

The benefit of the heated purification column is that DXNs and PCBs can be eluted quickly and a chemical reaction of sample matrices is facilitated with the chemically-modified silica gel.

DXNs fraction

17 isomers of 2,3,7 & 8 substituted Chlorinated dibenzo-p-dioxins, dibenzofurans and 4 isomers of non-ortho PCBs in 1.3 ±0.1 mL of Toluene

PCBs fraction

6 isomers of indicator PCBs, 8 isomers of mono-ortho PCBs in 0.9 ±0.1 mL of Toluene

Four Purification Steps

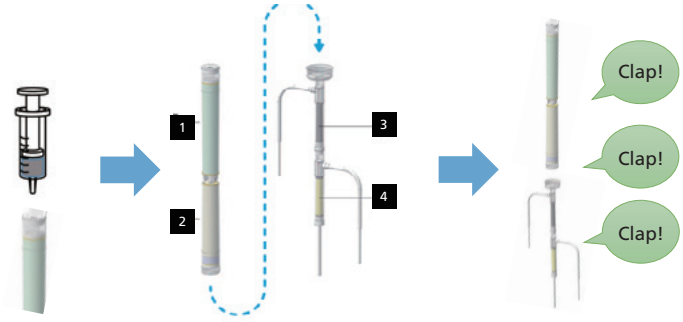
Step 1

Select the column to use.

ø18 column	Filter and Column Size	ø20 column	Filter and Column Size
	$AgNO_3$-Silica gel ø15.6 × 81.3 **1**		$AgNO_3$-Silica gel ø17.6 × 116.9 **1**
	H_2SO_4-Silica gel ø15.6 × 107 **2**		H_2SO_4-Silica gel ø17.6 × 100 **2**
	Carbon ø6 × 38 **3**		Carbon ø6 × 38 **3**
	Alumina ø6 × 38 **4**		Alumina ø6 × 38 **4**

Step 2

Assemble the column and apply the sample to it.



Step 3

Set up the column to the system.

Step 4

Start purification using the method files for DXNs and PCBs. Note that the method files are prepared in advance and do not need to be created.

Analysis of Samples

GCMS-TQ8050 (Method Package for Dioxins in Foods)

The method package consists of method files registered with the optimal conditions for the analysis of dioxins, as well as a report creation tool that can output the items required by EU regulations. This makes it possible to start an analysis without the having to investigate analytical conditions.

■ Method files registered with the optimal conditions for the analysis of dioxins

Optimized analysis conditions (including transition and CE) are pre-registered in the method files. Additionally, the files are registered with retention times and retention indices, and the retention times can be adjusted automatically using the retention time adjustment function (AART: Automatic Adjustment of Retention Time), allowing analysis to start immediately.
Note that a comparison test (an analysis of 44 types and 201 samples of foods and feeds) with magnetic sector GC-MS was implemented using these method files and a Shimadzu TQ, and the performance was confirmed.

Point 1

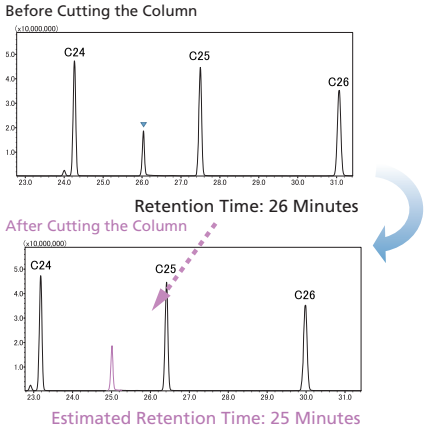
■ Report creation tool, capable of outputting items required by EU regulations

Reports of the analysis of dioxins in foods must contain the results of complicated calculations. A report creation tool is included in this product. It can automatically create reports showing items required by EU regulations. Additionally, it is capable of combining reports on DXNs and PCBs, changing the items displayed, and showing the Limit of Quantification (LOQ) calculation method, providing support to varied customer analyses.

Point 2

Point 1 Automatic adjustment of retention times using the AART function

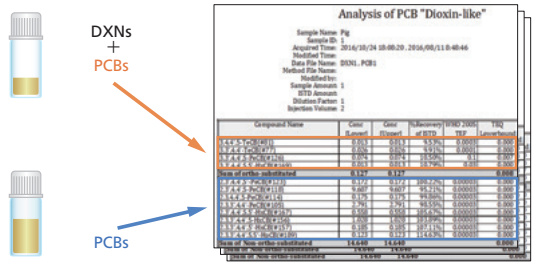
The retention times and retention indices for the target compounds are registered in the method files. The AART function adjusts the retention times for the target compounds using the retention indices for the target compounds and the results of the analysis of an n-alkane standard sample. The retention times and time programs are automatically adjusted even if the retention times for the target compounds change, such as for maintenance of the column tip.



Alkanes with different carbon numbers are investigated at a wide range of retention times, so the retention times of target compounds with a variety of boiling points can be adjusted with high accuracy.

Point 2 Report creation tool, capable of outputting items required by EU regulations

In the analysis of dioxins in foods, DXN analysis samples and PCB analysis samples are prepared from a single sample. However, depending on the pretreatment method, some of the PCBs can become mixed into the DXN sample, so the analysis results for PCBs are sometimes divided into two parts. With this product's report creation tool, even if the analysis results for PCBs are divided into two parts, they can be combined, enabling support for a variety of samples and pretreatment methods.



Reports Can Be Created Combining the Two Analysis Results

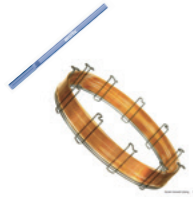
Four Steps from the Preparation for Analysis to Analysis of Samples

Step 1 Preparation for analysis

- Attach a suitable column* for the measurement compounds.
- Attach the insert liner**.
- Start up the instrument's vacuum system.

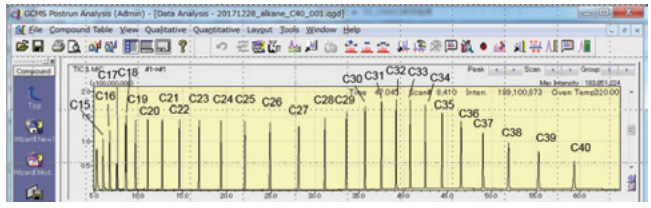
***DXNs analysis column**
SH-Rxi™-5Sil MS (60 m, 0.25 mm I.D., 0.25 µm) P/N: 227-36036-02 (SHIMADZU)
PCBs analysis column
HT8-PCB (60 m, 0.25 mm I.D.) P/N: 054237 (Trajan Scientific)
BFRs analysis column
SH-Rtx™-1614 (15 m, 0.25 mm I.D., 0.1 m), P/N: 227-36265-01 (SHIMADZU)

**Topaz® 3.5 mm I.D. Single Taper Inlet Liner w/ Wool, P/N: 23336 (Restek Corp.)



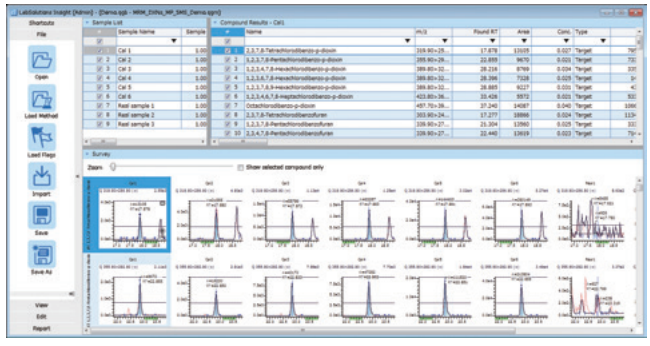
Step 2 Creation of method files

- Analyze the n-alkane standard sample.
- Adjust the retention times of the measurement compounds (AART function).
- Set the parameters for quantitation (including the calibration curve concentrations).



Step 3 Analysis of samples

- Measure and analyze the samples.



Export the results to Excel®.

Step 4 Creation of reports

- Create a report.

Analysis of PCDD/PCDF

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	0.00560	0.00560	0.00560
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**

EU Regulation Compliant GC-MS/MS Method Package for Dioxins in Foods Specifications:

■ Product Contents

Method Files (DXNs analysis method file, PCBs analysis method file, and BFRs analysis method file)
Method File for Adjusting the Retention Times
Report Creation Tool

■ Supported Instruments

GC/MS: GCMS-TQ™8050

■ Operating Environment

Excel: Microsoft® Excel® 2016
Workstation: GCMSsolution™ Ver. 4.45 SP1 or later
LabSolutions™ Insight Ver. 3.2 SP1 or later

■ Recommended Consumables

Insert Liner: Topaz® 3.5 mm ID Single Taper Inlet Liner w/ Wool, P/N 23336 (Restek Corporation)
n-alkanes: C8-C40 Alkane calibration standard (SIGMA-ALDRICH, Cat#:40147-U)
Quantitative Retention Index Standard (C7-C33) (Restek Corporation, 31080)
Column: DXNs Analysis: SH-Rxi™-5Sil MS (60 m, 0.25 mm I.D., 0.25 m), P/N: 227-36036-02 (SHIMADZU)
PCBs Analysis: HT8-PCB (60 m, 0.25 mm I.D.), P/N: 054237 (Trajan Scientific)
BFRs Analysis: SH-Rtx™-1614 (15 m, 0.25 mm I.D., 0.1 m), P/N: 227-36265-01 (SHIMADZU)

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Note: GO-xHT is not available in some countries/regions. Contact your Shimadzu sales representative for more details.
The n-alkanes mixed sample is used in analysis in order to adjust the measurement parameters and retention times in the method files using the AART function. BFR compounds with carbon Nos. C33 and higher are included.
P/N 31080: for customers who only analyze DXNs and PCBs.
P/N 40147-U: for customers who analyze BFRs.

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