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Automated Sample Preparation and Measurement Workflow for Dioxin Analysis

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Introduction

Dioxins are a class of very toxic compounds found throughout the world in the environment. Equipment sensitivity is of great importance for the analysis of low concentrations of these highly-toxic compounds. Historically, analysis and detection of dioxins was done with magnetic sector-type high-resolution mass spectrometers (HRMS). However, in recent years, the performance of triple quadrupole mass spectrometers (MS/MS) has improved significantly. In addition, the development of the Boosted Efficiency Ion Source (BEIS) offers compound-specific sensitivity up to 4 times greater than previous ion sources and provides accurate quantitation of dioxins at levels comparable to HRMS. Detection limits as low as 20 fg for Tetrachlorodibenzo-p-dioxin (TCDD) were achieved. In this study, we analyzed dioxins in about 250 samples of approximately 40 types of food and animal feed products using a GC-MS/MS with BEIS. We also evaluated the number of analyses possible while maintaining sensitivity at low concentrations in order to verify the durability of the GC-MS/MS instrument.

Analytical Conditions

For the various food samples, pretreatment was performed using an automatic pretreatment unit (extraction: SpeedExtractor (BUCHI); purification: GO-xHT (Miura Co., Ltd.)). Hexane was used as the final solvent for the samples, with a volume of 10 uL. For the STD, a mixture of DF-ST and DF-LCS from Wellington Laboratories was used. The analytical conditions for GC-MS/MS are listed in Table 1.

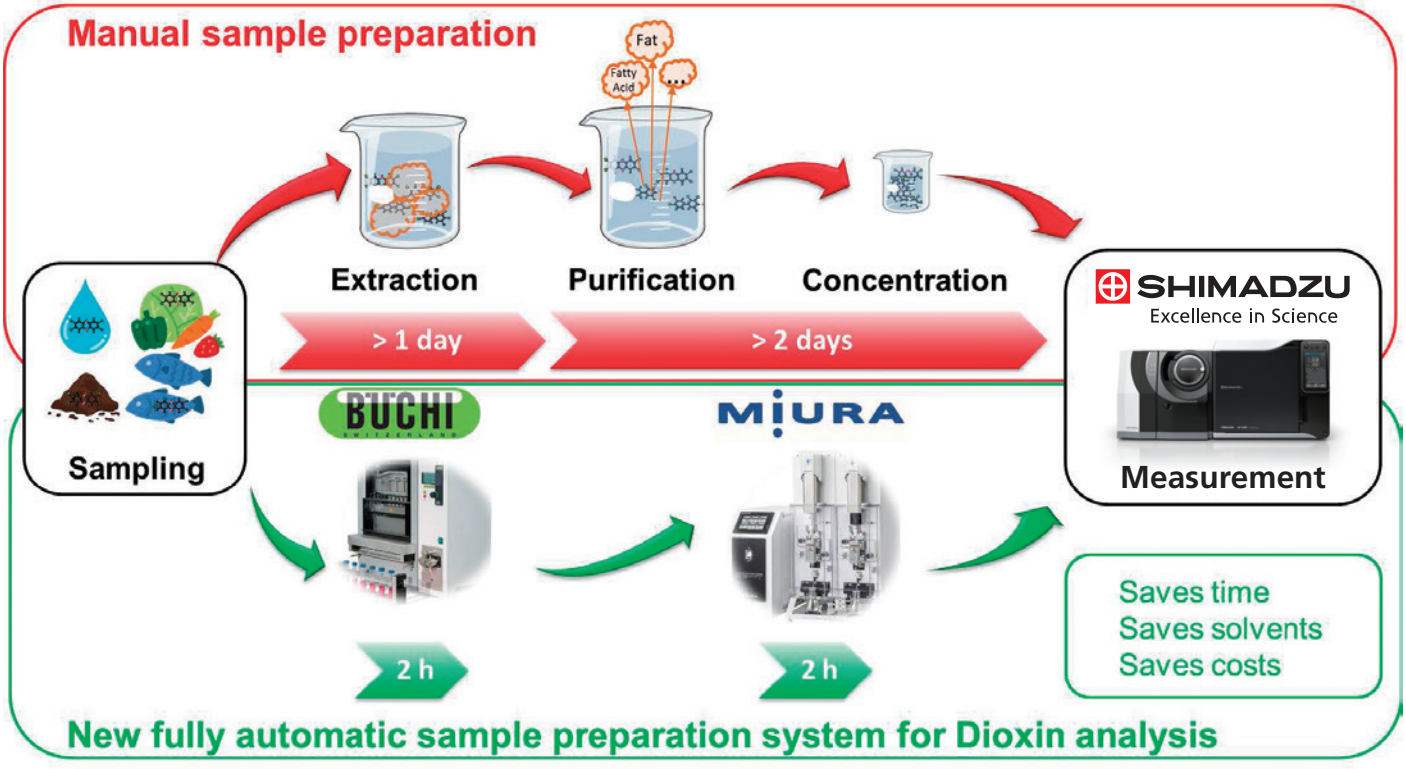
Table 1: Analytical conditions

GC system	**: GC-2030**
Column	: SH-Rxi™-5Sil MS (60 m, 0.25 mm I.D., 0.25 µm),
Injection Mode	: Splitless
Sampling Time	: 1 min
Injection Temp.	: 280 °C
MS system	**: GCMS-TQ8050 NX**
Ion Source Temp.	: 230 °C
Interface Temp.	: 300 °C
Detector Voltage	: 1.8 kV

Figure 1: Shimadzu GCMS-TQ8050 NX

Automated vs. Manual Sample Preparation

The sample preparation procedure using the SpeedExtractor and the Miura GOEHT fully-automated system reduces the time for dioxin analysis to just about 2 hours instead of 2 days. Moreover, the system is less labor-intensive, reduces labor costs and enables a higher throughput of samples. Specially prepared columns are used that are already packed and ready for purification. The workflow is shown below:



Extraction using BUCHI's SpeedExtractor

The SpeedExtractor, is an automated instrument used for the parallel extraction of primarily organic compounds from a variety of solid samples. In order to maintain the solvent in a liquid state during the extraction process, the solvent inside the extraction cell is put under pressure. To achieve high recoveries multiple extraction cycles are usually applied. Once the extraction step is finished, the extracts are cooled down in a cooling unit and flushed into collection vials, which can then be easily evaporated. Alternatively, the extract can be collected in round bottom flasks for evaporation using the Rotavapor®. The whole process workflow can be performed in parallel with up to six samples. Extraction cells can accommodate samples sizes from 10 – 120 mL ensuring reliable analysis of high and low polluted samples.

Figure 2: BUCHI SpeedExtractor

Purification using Miura Go-EHT System

GO-EHT 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. Total solvent use is only 110 mL.

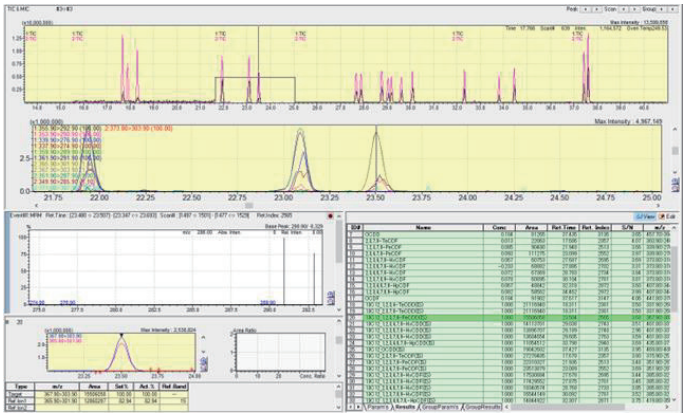
Figure 3: Miura GO-EHT system

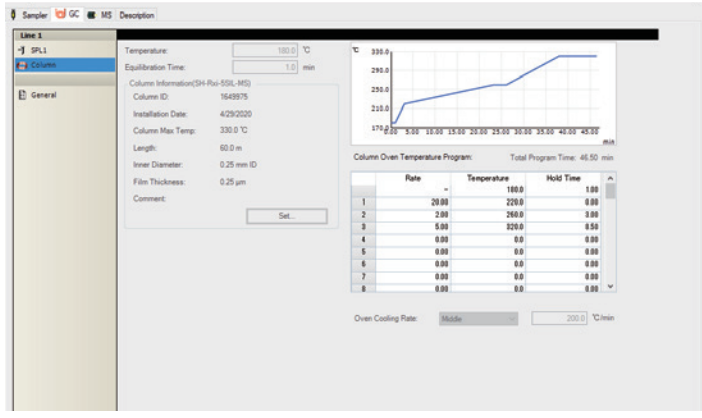
First, the fat is extracted from the food manually or by using an extraction system (e.g. Büchi). The food extract is dissolved in hexane and placed on the Miura column. The columns are inserted into the system and the program is started. The purification, concentration and fractionation of dioxins and PCBs is completely automated. The first two columns 1 and 2 (Figure 3) serve to capture matrix components of the food samples so that only the dioxins and PCBs can proceed. The column treated with silver nitrate removes sulfur-containing components, whereas in the column with sulfuric acid fats are hydrolyzed and basic components are removed.

GC MSMS analysis

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. The workflow is as follows:

- Use the GC-MS/MS method with optimized conditions





- Adjust the retention times automatically by AART function
- Analyse your samples by Shimadzu GCMS-TQ 8050 NX
- Create your reports by a special reporting tool

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Conclusion

The fully automated Shimadzu solution for Dioxin analysis offers many attractive advantages in comparison to the manual workflow. The combination of the Büchi SpeedExtractor with the Miura sample purification system and the Shimadzu GCMS/ MS provides ideal conditions for dioxin and PCB analysis.

References

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[2] Shimadzu Application News No. 01-00171-EN – Nexera Organic Acid Analysis System and its Application.
[3] Shimadzu Technical Report C190-E237A – High-Speed Analysis of Organic Acids Using Shim-pack Fast-OA and pH-Buffered Electrical Conductivity Detection.

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