Dioxin determination in ambient air

UniversalExtractor E-800: Dioxin determination in ambient air using Soxhlet extraction

Air quality is an emerging concern for the human population's health, especially in urban areas where anthropogenic activities and pollution sources coexist. Special attention is devoted to the airborne particulate matter (PM), a mixture of microscopic particles and liquid droplets present in the air and once inhaled can get into the lungs and bloodstream depending on their size.

Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) are halogenated aromatic hydrocarbons formed as by-products of thermal processes that may be unintentionally released in the environment [1]. When released, they tend to be persistent in the environment and may undergo long-range transport [2]. They are ubiquitous and unfortunately several reports indicate alarming concentration in the lipid tissues of various organism, especially in the higher level of the food chain because of bioaccumulation. For this reason, they are reported on the Stockholm Convention black list of the persistent organic pollutants (POPs).

The overall toxicity is expressed as TEQ (toxic equivalent quantity), which is a sum parameter calculated from the TEF (toxic equivalent factor) and the corresponding concentration of all congeners present. To establish exposure risks to humans and proper management, the sources and fates of PCDD/Fs is object of detailed study [3].

1. Introduction

The Soxhlet extraction of dioxins from different environmental matrices is described by the regulated methods EPA 1613 [4]. This Short Note describes the extraction of dioxins from particulate matter air samples collected on quartz fiber filters and on polyurethane foam cartridges using the BUCHI UniversalExtractor E-800. The extracts were analyzed by GC-MS.

2. Experimental

Equipment: UniversalExtractor E-800 Pro (chamber heater), Syncore® Analyst with 1 mL appendix vessels, GC-MS.

Samples:
1. Quartz fiber filter (QFF): PM was sampled by using a medium volume sampling device (TCR Tecora; 2.3 m^3/h), daily in two different sites of Northern Italy (registered PM concentration higher than 100 $\mu g/m^3$).
2. ORBO™ 2000, pre-cleaned PUF cartridges. The PUF were used to sample PCDD/Fs from the gaseous phase. In this case, a high-volume sampler (ECHOPUF, TCR Tecora, Italy) was used to collect the gaseous phase. The sampler was operated at 200 L/min, with an average sampling time of 24 h (average sample volume around 288 m^3).

The extractions of the QFF and PUF samples were carried out with the UniversalExtractor E-800. The method parameters are shown in Table 1.

Table 1: Extraction method for UniversalExtractor E-800.

Parameter	Value for QFF	Value for PUF
Extraction method	Soxhlet	Soxhlet warm
Solvent	Dichloromethane	Toluene
Solvent volume	140 mL	150 mL
Extraction time	720 min	800 min
Extraction cycles	80	140
Extraction heating level	8	15
Chamber heating level	-	5
Rinse time	5 min	5 min
Rinse heating level	6	14
Drying time	20 min	20 min
Analyte Protection	on	on
Drying heating level	6	14

After extraction, concentration and clean-up, the dioxins were quantified with GC-MS.

3. Results

The results of the dioxin determinations in air spiked samples are shown in Table 2.

Table 2: Recoveries of dioxin determinations using the UniversalExtractor E-800 and GC/MS.

	QFF Mean [%]	SD	PUF Mean [%]	SD
^{13}C 2,3,7,8 TCDF	**64.8**	14.6	**102.9**	17.2
^{13}C 2,3,7,8 TCDD	**57.2**	4.9	**94.6**	13.9
^{13}C 2,3,4,7,8 PeCDF	**63.9**	10.0	**114.8**	8.0
^{13}C 1,2,3,7,8 PeCDD	**73.7**	3.2	**88.1**	24.9
^{13}C 1,2,3,4,7,8 HxCDF	**70.9**	4.8	**60.0**	6.2
^{13}C 1,2,3,6,7,8 HxCDF	**70.0**	4.5	**60.4**	5.1
^{13}C 2,3,4,6,7,8 HxCDF	**62.2**	10.3	**55.2**	4.8
^{13}C OCDF	**56.4**	7.0	**80.2**	2.7
^{13}C OCDD	**84.4**	7.1	**92.0**	4.5

4. Conclusion

The analysis of PCDDs and PCDFs in air samples were carried out by means of Soxhlet extraction using the UniversalExtractor E-800 and GC/MS. The recovery values of the labelled extraction standard resulted well within the EPA method 1613 indication.

5. Acknowledgement

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6. References

[1] Pitea, D. et al. (2008) Environ. Sci. Technol. 42, 7476–81.
[2] Ssebugere, P., et al., (2014) Sci. Total Environ. 500, 1-10.
[3] Hites, R. A. (2011). Environmental Science and Technology, 45, 16–20
[4] EPA METHOD 1613

For more detailed information refer to Application Note 360/2019