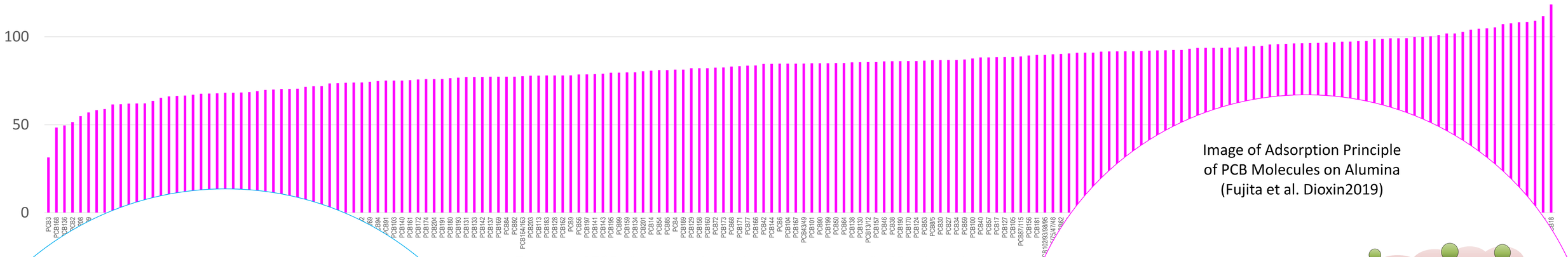
DSP systems
The missing link in your laboratory!

www.dspsystems.eu

A new automated purification method for the analysis of dioxins and all 209 polychlorinatedbiphenyls

Guillaume ten Dam[1], Hiroyuki Fujita[2], Jeroen Markesteijn[3], Chris van Wakeren[1] and Wim Traag[1]
[1] DSP-Systems, Ede The Netherlands | [2] Miura, Matsuyama Japan | [3] NofaLab, Schiedam The Netherlands



Recovery of PCBs from a standard column-set consisting of a 12g silver nitrate silica and 13g acidified silica purification column and a carbon with magnesium oxide and alumina oxide concentration column (Fujita et al. Dioxin2019)

Introduction

In 2015 an automated sample preparation system for the analysis of dioxins and polychlorinatedbiphenyls (PCBs) based on a dichloromethane free principle was introduced in Europe after a successful implementation in Japan the years before. The method has been meant to fulfil to the European requirements and results in two purified fractions containing 1) Dioxins and non-ortho PCBs and 2) mono-ortho PCBs and marker PCBs. In 2019 the method was further developed to fulfil to North American requirements, e.g. the analysis of all 209 PCBs according EPA1668 in addition to the analysis of dioxins (EPA 1613).

A environmental column set for 209 PCB

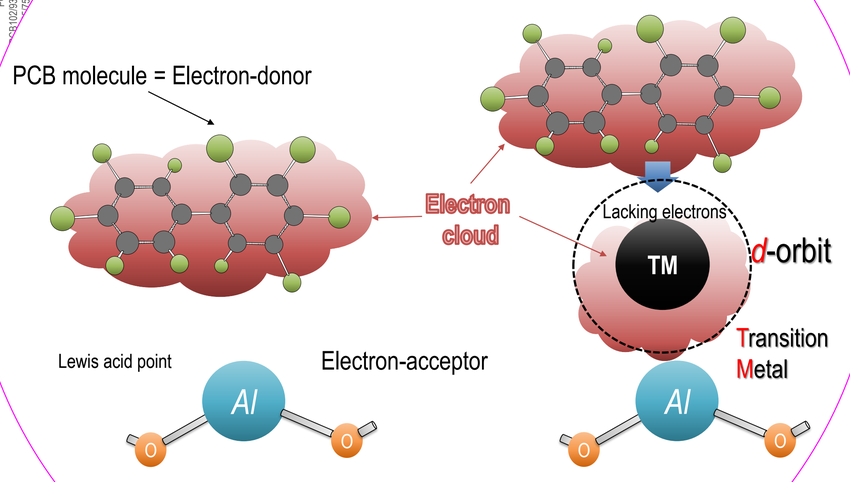
Method development part I

The original method and column setup were evaluated for all 209 PCBs (graph below). Results showed that lower chlorinated congeners were not recovered and most probably were retained on the purification columns while PCB209 ran through the concentration columns and was recovered in the waste.
To achieve suitable recoveries for the lower chlorinated congeners the desorption from the purification columns needed to be enhanced. Experiments were carried out by adding ethyl acetate as modifier and although recoveries improved they remained low for PCB2, 3 and 12. Experiments with environmental column-sets and a larger elution volume showed recoveries up to 45% for PCB3.

Method development part II

The retaining power of the alumina concentration needed to be enhanced to prevent breakthrough of PCB209. To achieve this, the bonding between the packing material and PCBs was re-considered. The bonding arises from the electron donor property of PCBs and electron acceptor properties of the packing material. Hence, it was determined that the electron acceptor capacity of the packing material needed to be increased.
Certain transition metals are very capable electron acceptors as they have empty space in their *d*-orbitals (figure right top). By adding such a transition metal to the packing material it was hypothesised and later confirmed that the recovery of PCB209 would increase (graph above).

Image of Adsorption Principle of PCB Molecules on Alumina (Fujita et al. Dioxin2019)



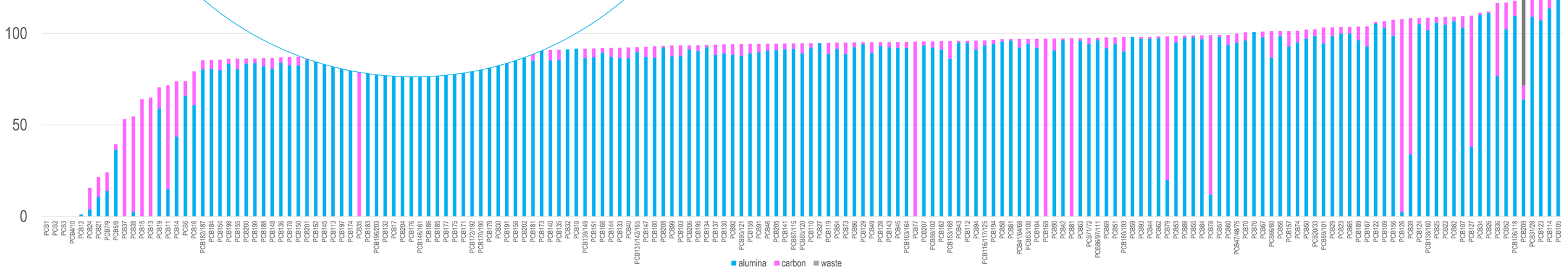
Summary

- A next generation automated sample preparation method for the analysis of dioxins and PCBs from Japan and applied in Europe was evaluated for the North American market.
- The method was adapted by adding a transition metal to the concentration column for stronger retention of PCBs on this column
- The elution from the purification columns was improved by adding ethyl acetate as modifier or by increasing the elution volume.

Method parameters for the standard method and for the method for 209 PCB

Parameter	Hi-Capacity	209-PCB
Size[mm]	20 ϕ	20ϕ
Sample	Up to 5 g fat	Food
		Environmental
Purification (U)	H2SO4-Silica gel	H2SO4-Silica gel
Purification (L)	KMnO4-alumina	KMnO4-alumina
	AgNO3-Silica gel	AgNO3-Silica gel
Concentration(U)	Carbon	N/A
Concentration(L)	Alumina	TM-Alumina
Elution (Hexane)	130mL	130 mL
DXN fraction (Toluene)	1.2 mL	N/A
PCB fraction (Toluene)	1.0 mL	1.2 mL
Run time	120 min	?

Miura GO-eHT



Recovery of PCBs from a standard column-set consisting of a 12g silver nitrate silica and 13g acidified silica purification column and a carbon with magnesium oxide and alumina oxide concentration column

In cooperation with

熱・水・環境のベストパートナー
MIURA

Guillaume ten Dam
Food Valley BTA12
Darwinstraat 7a, 6718 XR Ede
The Netherlands
+31 683627499
Guillaume.ten.dam@dspsystems.eu

In cooperation with

NofaLab Laboratories
NofaGroup Quality in Control