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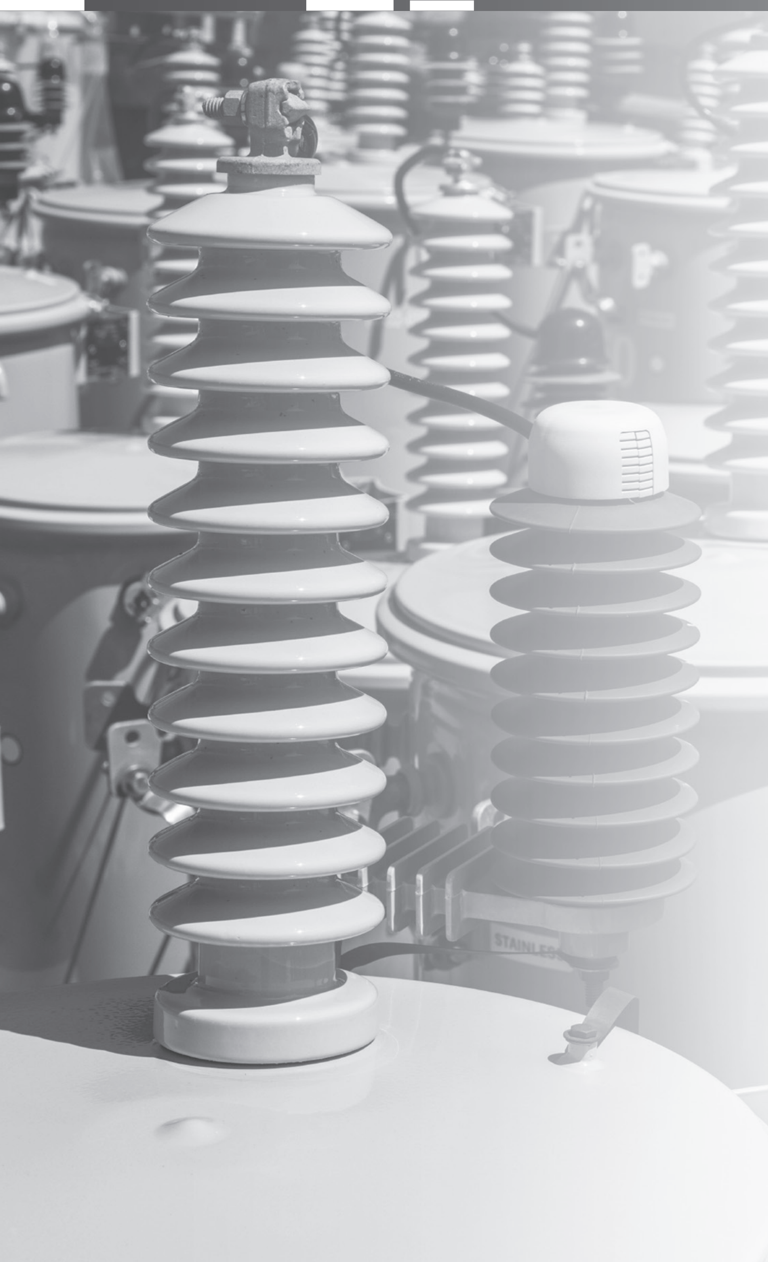

POLYCHLORINATED BIPHENYLS AND POLYBROMINATED BIPHENYLS

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PCB CONCENTRATIONS IN SOIL AND SEDIMENT

This supplementary material to Section 1, Exposure Data, formed part of the original submission and has been peer reviewed.

PCB concentrations in soil and sediment

As polychlorinated biphenyls (PCBs) accumulate in the organic fraction of soil and sediments, the PCB content of the sample will depend on the sample material. For sediments, fine material is generally preferred (OSPAR, 2002). The analysis of soil and sediment samples poses the general question as to whether or not samples should be dried and if so, how. Drying of soil and sediment samples ensures defines water content and facilitates homogenization, thus improving reproducibility in PCB determination; however, it also increases the risk of contamination and losses via volatilization (Smedes & de Boer, 1998; Wilcke *et al.*, 2003). Smedes & de Boer (1998) suggest that freeze-drying is less susceptible to losses than evaporation at elevated temperatures. Water can also be removed by water-absorbing reagents such as sodium sulfate and hydromatrix.

While most extraction techniques aim to remove as much of the analyte as possible from the soil or sediment matrix for determination of total concentrations, equilibrium sampling techniques (passive sampling) have been applied to analyse the concentration or chemical activity of the freely dissolved, which is considered more relevant in an exposure context (Määnpää *et al.*, 2011).

The Nordic guidelines for chemical analysis of contaminated soil samples describe extraction of PCBs from soil by sonication (Karstensen *et al.*, 1998). Other extraction methods include Soxhlet (Gibson *et al.*, 2005), pressurized liquid extraction (PLE) (Wang *et al.*, 2010) and microwave-assisted extraction (MAE) (Düring & Gäth, 2000), including low pressure MAE (Bruzzoniti *et al.*, 2012). A comparison of Soxhlet, PLE and MAE for the analysis of soil samples showed that MAE and PLE were comparable to Soxhlet and even gave better extraction efficiencies for PCBs with high K_{ow} values (Wang *et al.*, 2010). Dispersive liquid–liquid microextraction, a ternary component solvent system, has also been applied to soil samples (Hu *et al.*, 2009).

According to Webster *et al.* (2013), Soxhlet is the benchmark technique for PCB extraction from sediments. A comparison of different extraction methods for PCBs in sediment concluded lower recoveries for ultrasonic extraction than for PLE and MAE (Zhang *et al.*, 2011b).

Several studies have analysed soil and sediment samples together. EPA method 1668B also describes analytical methods that are suitable for both matrices and suggests a Soxhlet/Dean-Stark extractor for PCB extraction (EPA, 2008a).

Hawthorne *et al.* (2009) used a solvent-free method combining subcritical water extraction with solid-phase microextraction for PCB extraction from soil and sediment. In this method, hot water is used under pressure, so it remains liquid, but polarity and surface tension are reduced, while viscosity is increased (Hawthorne *et al.*, 2009). Avoiding the water phase, headspace solid-phase microextraction has been applied as well, after warming soil and sediment samples (Llompart *et al.*, 1999).

For determinations of total PCB concentrations in soil and sediment, conversion reactions have been pursued, including perchlorination to decachlorinated biphenyl, hydrodechlorination to biphenyl (Wu & Marshall, 2001), and hydrogenolysis to dicyclohexyl (Ehsan *et al.*, 2003). Obviously, information on congener-specific concentrations and PCB patterns are lost with these methods.

Soil and sediment samples usually contain sulfur, which is co-extracted with PCBs and will interfere with subsequent analysis (Smedes & de Boer, 1998). PLE offers the possibility of in-line clean-up in the extraction cell, for example by addition of activated copper (Webster *et al.*, 2013). Copper can also be used in other extraction techniques, for example, mixed with the sample or together with general adsorption chromatographic techniques. Other methods for sulfur removal include saponification, reaction with tetrabutylammonium salts, and gel permeation chromatography (GPC) (see Smedes & de Boer, 1998; and Mechlińska *et al.*, 2012, for reviews).

Apart from sulfur, crude extracts of soil and sediment contain many other natural and anthropogenic compounds that have to be removed before instrumental analysis. In principal, the same techniques as those described for the other matrices are suitable, i.e. alumina (Smedes & de Boer, 1998), silica gel (Jayaraman *et al.*, 2001), Florisil (Castells *et al.*, 2008) and GPC (Wang *et al.*, 2010). According to the EPA (2008a), GPC should be used for all soil and sediment extracts. Data on PCB concentrations in soil and sediment are presented in the Table.

Table. PCB concentrations in soils and sediments worldwide

Region, country	Time	Sources	Measurements	PCB concentrations	Reference
Asia					
Lake Baikal, the Russian Federation	May 1992	Four surface soil samples from agricultural fields around Irkutsk	Kanechlors 300, 400, 500, 600 as standards	Range, 1.4–92 ng/g dw	Iwata *et al.* (1995)
Guiyu, Guangdong Province, China	August 2003	Sediments from Lianjiang river close to e-waste site	Total PCbs	Mean, 743 µg/kg dw	Leung *et al.* (2006)
Can Gio, Hue, Hanoi, Viet Nam	2003	15 sediment samples	DL-PCBs as standards	Mean DL-PCBs: Can Gio, 252 ng/kg dw Hue, 265 ng/kg dw Hanoi, 12 720 ng/kg dw	Kishida *et al.* (2010)
Suburban and urban locations, Osaka, Japan	2003	14 sediment samples	DL-PCBs as standards	Mean DL-PCB: Suburban: 2.3 ± 4.7 to 200 ± 210 ng/kg dw Urban: 21 ± 17 to 54 000 ± 78 000 ng/kg dw	Kishida *et al.* (2010)
North-western Arabian Gulf	NR	Sediment core	PCBs 18 17, 31 28, 33, 52, 49, 44, 74, 70, 101, 99, 87 110, 151, 149, 118, 153, 105, 138, 132, 187, 183, 128, 177, 171, 156, 180, 191, 169, 170, 201, 208/195, 194, 205, 206, 209	Range, 875–14 671 pg/g dw for different sediment depths	Gevao *et al.* (2012)
North America					
North America		Soil		Average of 4300 (range, 110–25 000) pg/g dw; however, some sites had concentrations as high as 100 000 pg/g	Li *et al.* (2010)
USA		Soil from 27 rural and/or remote areas		3089 (SE, 1009; SD, 5241) pg/g dw	EPA (2007)
Highly contaminated New Bedford Harbor, Massachusetts, and a control site, USA		Soil concentrations from 34 homes		Geometric mean concentration: New Bedford Harbor: 200 ng/g dw (range, 23–1800); comparison neighbourhood: 60 ng/g dw (range, 15–290)	Vorhees *et al.* (1999)
South and Central America					
Brazil		Bulk deposition from air into soil in urban, suburban, rural, and background sites	28 PCB congeners	Mean and range, 13 100 pg/m² (2400–15 300) in urban, 6000 pg/m² (1430–6800) in suburban, 5000 pg/m² (1400–6100) in rural and 8200 pg/m² (2900–8600) at background sites	Meire *et al.* (2012)

Table 1 (continued)

Region, country	Time	Sources	Measurements	PCB concentrations	Reference
South America		Review of various studies		Average of 1400 (61–9500) pg/g dw	Li *et al.* (2010)
North-eastern Sao Paulo State, Brazil		Soil	Sum of seven congeners	0.02–0.25 ng/g dw Concentrations were higher in forest areas, probably reflecting the influence of the canopy	Rissato *et al.* (2006)
Rio de la Plata estuary, Argentina		Coastal sediments	Sum of 41 congeners	< 0.1 to 100 ng/g, with levels higher near industrialized areas close to Buenos Aires [61 ± 37 mg/g) relative to cleaner northern stations [3.6 ± 2.2 ng/g] The congener patterns indicated transformer oils containing Aroclors 1254 and 1260	Colombo *et al.* (2005)
Santos and Sao Vicente Estuary system, Sao Paulo, Brazil		Sediments	Sum of 30 congeners	Range, 0.03–254 ng/g dw	Bícego *et al.* (2006)
Chile		Sediments from four remote Andean lakes (as an indication of atmospheric transport)	Sum of 43 congeners	Total PCB fluxes in three lakes were similar to those in other remote lakes in the northern and southern hemisphere, but in Lake Venus, on the western side of the Andes, levels were significantly higher (7658 ng/m^2 per year) In all lakes the highest concentrations were in sediments deposited in 1991–1998	Pozo *et al.* (2007)
Africa					
Africa, various locations		Soil		Average, 390 (range, 94–620) pg/g dw. Most sites were < 500 pg/g dw	Li *et al.* (2010)
Metropolitan Durban, KwaZulu-Natal, South Africa		Surface soils	82 congeners	Average (surface soils), 109.64 (SD, 116.07) Average (shallow soils), 19.22 (SD, 33.23)	Batterman *et al.* (2009)
Coastal bays, Egypt,		Estuarine and coastal sediment samples		Range, 39–744 ng/g	Abd-Allah & Abbas (1994)
Alexandria harbour, Egypt		Estuarine and coastal sediment samples		Range, 0.9–1210 ng/g	Barakat *et al.* (2002)
Nile River, near Cairo, Egypt		Sediments	26 congeners	Range, 1461–2244 pg/g	El-Kady *et al.* (2007)
Northern Morocco		Sediments in salt marshes		< 1 ng/g	Piazza *et al.* (2009)

Table 1 (continued)

Region, country	Time	Sources	Measurements	PCB concentrations	Reference
Port of Tangier, Morocco			86 congeners	164 ng/g	Piazza *et al.* (2009)
Mediterranean coast, Morocco				Range, 0.1–1.8 ng/g	Pavoni *et al.* (2002)
Algiers Bay, Algeria		Sediment		Range, 0.31–323 ng/g dw The profile of the PCBs was very close to that of Aroclor 1260	
Ghar El Melh lagoon, Tunisia			Sum of 20 congeners	Range, 0.454–3.987 ng/g	Ameur *et al.* (2011)
Bizerte lagoon, Tunisia		Sediments from 15 sampling stations	Sum of 20 congeners	Range, 0.89–6.63 ng/g	Derouiche *et al.* 2004
Senegal			Seven indicator PCB congeners	Range, 0.3–19.1 ng/g dw	Bodin *et al.* (2011)
11 African countries (Ghana, Togo, Egypt, Uganda, Kenya, Mauritius, Nigeria, Mali, Zambia, Democratic Republic of Congo, Ethiopia, Senegal, and Barbados)		13 sediment samples	DL-PCBs	Range, 0.006–0.64 pg WHO_{1998}-TEQ/g dw (comparable to low background concentrations in European Union)	ANSES (2011)
Europe					
Slovakia		Soil samples		Range, 1–28 ng/g dw in reference areas Range, 170–8600 ng/g dw from dump areas Range, 43 ng to 53 mg/g dw from hot-spot areas	Kocan *et al.* (1999)
Eastern France		50 wastewater plants		0.05–1.13 mg/kg	ADEME (1998), Preda *et al.* (2010)
Paris region, France		50 wastewater plants		0.5 mg/kg	ADEME (1998), Preda *et al.* (2010)
France		Sludges disposed in agriculture		Mean, 0.19 mg/kg	ADEME (1998), Preda *et al.* (2010)

Table 1 (continued)

Region, country	Time	Sources	Measurements	PCB concentrations	Reference
Saint-Etienne, France		32 soil samples		PCBs 77, 81, 126, 169, 105, 114, 118, 123, 156, 157, 167, 189, 356–5200 ng TEQ/kg PCB-indicators (PCB congeners 28, 52, 101, 118, 138, 153, 180), 3–158 mg/kg	Denys *et al.* (2012)
Krakow, Katowice, and Chorzow, southern Poland	Samples collected in 1994	Surface soil and sediment samples		Soil from Katowice was relatively more polluted than soil from other places	Falandysz *et al.* (2001)
Poland, former Czechoslovakia, the former German Democratic Republic and former Union of Soviet Socialist Republics	1994	Soil samples from former Soviet army bases localized nearby		Elevated concentrations of PCBs, similar to those reported in former military United States bases in Viet Nam	Falandysz *et al.* (1997)
United Kingdom, Norway		46 sites across the United Kingdom; 12 sites in Norway		High relative proportion of the midmolecular weight congeners in the samples from Norway The soils from southern Norway and the United Kingdom contained similar amounts of PCBs per unit area; those from northern Norway contained lesser amounts	Lead *et al.* (1997), Meijer *et al.* (2002)

DL-PCB, dioxin-like polychlorinated biphenyl; dw, dry weight; PCB, polychlorinated biphenyl; NR, not reported; SD, standard deviation; SE, standard error

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