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# **Background document for Canadian Federal Environmental Quality Guidelines (FEQGs) for Polybrominated Diphenyl Ethers (PBDEs) in sediment and biota**

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## **Acknowledgement**

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#### OSPAR Convention

The Convention for the Protection of the Marine Environment of the North-East Atlantic (the “OSPAR Convention”) was opened for signature at the Ministerial Meeting of the former Oslo and Paris Commissions in Paris on 22 September 1992. The Convention entered into force on 25 March 1998. The Contracting Parties are Belgium, Denmark, the European Union, Finland, France, Germany, Iceland, Ireland, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

#### Convention OSPAR

La Convention pour la protection du milieu marin de l'Atlantique du Nord-Est, dite Convention OSPAR, a été ouverte à la signature à la réunion ministérielle des anciennes Commissions d'Oslo et de Paris, à Paris le 22 septembre 1992. La Convention est entrée en vigueur le 25 mars 1998. Les Parties contractantes sont l'Allemagne, la Belgique, le Danemark, l'Espagne, la Finlande, la France, l'Irlande, l'Islande, le Luxembourg, la Norvège, les Pays-Bas, le Portugal, le Royaume-Uni de Grande Bretagne et d'Irlande du Nord, la Suède, la Suisse et l'Union européenne.

## Contents

Contents	3
1. Executive Summary	4
2. Récapitulatif	5
3. Introduction	6
4. Synopsis of Background Information	6
4.1 EU Environmental Quality Standards (EQS)	6
4.1.1 EQS protection goal	7
4.1.2 Human Health and Secondary Poisoning Quality Standards (QS) for PBDEs in biota	7
4.1.3 Review of the study used to derive the biota EQS (Branchi et al. (2002))	8
4.1.4 Comparison of the EQS to Environmental PBDE Concentrations	8
4.1.5 Sediment Quality Standard	8
4.2 Canadian Federal Environmental Quality Guidelines (FEQGs)	9
4.2.1 FEQG Protection Goal	9
4.2.2 Proposed FEQG	9
4.2.3 Review of the studies used to derive the FEQGs	11
4.3 Use of FEQGs in MIME PBDE trial assessment	12
4.3.1 Proposed Changes for PBDE MIME assessments	13
5. Conclusions	14
6. Proposals	15
7. References	15

## 1. Executive Summary

Interpretation of monitoring data for hazardous substances in marine sediments and biota requires relevant assessment tools. To this purpose, OSPAR developed Environmental Assessment Criteria (EACs). EACs represent the concentration below which no chronic effects are expected to occur in the marine environment, including the most sensitive species. However, EACs have not been developed for all contaminant/matrix combinations required for OSPAR assessments.

Currently, there are no EACs for the status assessment of polybrominated diphenyl ethers (PBDEs) in sediment and biota. Other assessment criteria available that could be used for PBDE status assessments include the Water Framework Directive (WFD) Environmental Quality Standards (EQS) for biota, and the Canadian Federal Environmental Quality Guidelines (FEQGs) for sediment and biota.

WFD Quality Standards (QS) for human health and secondary poisoning were derived for the sum of 6 PBDE congeners (BDE28, 47, 99, 100, 153, 154) in biota (fish), based on results from ecotoxicological studies on mice. The lower of these QS was assigned as the EQS, which for PBDEs was the human health QS. Application of the EQS must also include a trophic level adjustment to trophic level 5 (for marine food webs); however, in practice this is currently not done. The EQS ( $0.0085 \mu\text{g kg}^{-1}$  wet weight) is very low compared to typically reported environmental concentrations in biota, and most PBDE data for biota will exceed this concentration. The QS derived to protect top consumers from secondary poisoning was  $44 \mu\text{g kg}^{-1}$  wet weight in fish, whole body. A tentative QS for marine sediment ( $310 \mu\text{g kg}^{-1}$  dry weight, normalised to 5% organic carbon) was derived by applying an assessment factor of 50 to the lowest NOEC from long-term studies.

FEQGs are available for individual PBDE congeners in water, sediment and biota and were derived from ecotoxicological testing. The FEQGs for fish were derived using the FEQGs for water (FWQG) and the field-based bioaccumulation factor. The FEQGs for sediment were derived using the FEQGs for fish and the biota-sediment accumulation factor. In biota, FEQGs are higher than the EQS but the FEQG for BDE47 in fish ( $44 \mu\text{g kg}^{-1}$  wet weight) is equivalent to the secondary poisoning QS for the sum of the 6 PBDE congeners.

The FEQGs are currently being trialled for the OSPAR MIME status assessment of PBDEs in sediment and biota. FEQGs rather than the secondary poisoning QS were used, as the FEQGs were available for individual congeners, the QS provides a less stringent assessment of the more toxic homologues, and some data series miss some of the six PBDE congeners that constitute the basis of the QS.

It is proposed that the FEQGs are adopted for OSPAR assessments of PBDE concentrations in biota and sediment.

The FEQGs for sediment are normalised to 1% organic carbon (Environment Canada, 2013). For OSPAR MIME assessments organic contaminant concentrations in sediment are normalised to 2.5% organic carbon, therefore the FEQGs were multiplied by a factor 2.5.

For fish, the FEQG will be adjusted to a lipid weight basis (assuming the whole fish used in the toxicity trials had a 5% lipid content) by multiplying the FEQG (on a wet weight basis) by 20. Fish concentrations will then be assessed on a lipid weight basis. For bivalves, the assessment will continue to be done on a wet weight basis (since too few samples have supporting lipid weight measurements), with the FEQGs adjusted to take into account the lower lipid content of shellfish by multiplying the original FEQGs by the ratio  $\text{lipid}_{\text{shellfish}} / 5\%$  (where  $\text{lipid}_{\text{shellfish}}$  is the typical % lipid content of the shellfish species being assessed).

## 2. Récapitulatif

L'interprétation des données de surveillance des substances dangereuses dans les sédiments et le biote marins exige des outils d'évaluation pertinents. À cette fin, OSPAR a développé des critères d'évaluation environnementale (EAC). Les EAC représentent la concentration en dessous de laquelle aucun effet chronique ne devrait se produire dans le milieu marin, y compris chez les espèces les plus sensibles. Toutefois, les EAC n'ont pas été développés pour toutes les combinaisons contaminant/matrice requises pour les évaluations OSPAR.

Actuellement, il n'existe pas d'EAC pour l'évaluation de l'état des polybromodiphényléthers (PBDE) dans les sédiments et le biote. Parmi les autres critères d'évaluation disponibles qui pourraient être utilisés pour l'évaluation de l'état des PBDE, on peut citer les normes de qualité environnementale (NQE) de la directive-cadre sur l'eau (DCE) pour le biote, et les recommandations fédérales canadiennes pour la qualité de l'environnement (FEQG) pour les sédiments et le biote.

Les normes de qualité (NQ) de la DCE pour la santé humaine et l'empoisonnement secondaire ont été établies pour la somme de 6 congénères PBDE (BDE28, 47, 99, 100, 153, 154) dans le biote (poissons), sur la base des résultats d'études écotoxicologiques sur les souris. La plus basse de ces NQ a été attribuée comme norme de qualité environnementale, qui pour les PBDE était la NQ de la santé humaine. L'application des NQE doit également inclure un ajustement du niveau trophique au niveau trophique 5 (pour les réseaux trophiques marins) ; toutefois, dans la pratique, cela ne se fait pas actuellement. La NQE (0,0085 µg kg<sup>-1</sup> de poids humide) est très faible par rapport aux concentrations environnementales généralement signalées dans le biote, et la plupart des données relatives aux PBDE dans le biote dépasseront cette concentration. La NQ dérivée pour protéger les principaux consommateurs contre l'empoisonnement secondaire était de 44 µg kg<sup>-1</sup> de poids humide dans le poisson, corps entier. Une NQ provisoire pour les sédiments marins (310 µg kg<sup>-1</sup> de poids sec, normalisé à 5 % de carbone organique) a été calculée en appliquant un facteur d'évaluation de 50 à la CSEO (concentration sans effet observé) la plus faible tirée d'études à long terme.

Les FEQG sont disponibles pour les congénères individuels des PBDE dans l'eau, les sédiments et le biote et ont été dérivées de tests écotoxicologiques. Les FEQG pour les poissons ont été établies à partir des FEQG pour l'eau (FWQG) et du facteur de bioaccumulation basé sur le terrain. Les FEQG pour les sédiments ont été calculées à partir des FEQG pour les poissons et du facteur d'accumulation biote-sédiments. Dans le biote, les FEQG sont supérieures aux NQE, mais la FEQG pour le BDE47 dans le poisson (44 µg kg<sup>-1</sup> de poids humide) est équivalente à la NQE d'empoisonnement secondaire pour la somme des 6 congénères de PBDE.

Les FEQG sont actuellement testées pour l'évaluation de l'état des PBDE dans les sédiments et le biote, qui est en cours d'élaboration par le groupe MIME d'OSPAR. On a utilisé les FEQG plutôt que la NQ d'empoisonnement secondaire, car les FEQG étaient disponibles pour des congénères individuels, la NQ fournit une évaluation moins rigoureuse des homologues plus toxiques, et certaines séries de données omettent certains des six congénères PBDE qui constituent la base de la NQ.

Il est proposé que les FEQG soient adoptées pour les évaluations OSPAR des teneurs de PBDE dans le biote et les sédiments.

Les FEQG pour les sédiments sont normalisées à 1 % de carbone organique (Environment Canada, 2013). Pour les évaluations OSPAR élaborées par le groupe MIME, les concentrations de contaminants organiques dans les sédiments sont normalisées à 2,5% de carbone organique, les FEQG ont donc été multipliées par un facteur 2,5.

Pour le poisson, la FEQG sera ajustée sur une base de poids de lipides (en supposant que le poisson entier utilisé dans les essais de toxicité avait une teneur en lipides de 5%) en multipliant la FEQG (sur une base de

poids humide) par 20. Les concentrations dans les poissons seront ensuite évaluées sur la base du poids des lipides. Pour les bivalves, l'évaluation continuera à se faire sur la base du poids humide (car trop peu d'échantillons ont des mesures de poids en lipides), les FEQG étant ajustées pour tenir compte de la teneur plus faible en lipides des mollusques et crustacés en multipliant les FEQG initiales par le ratio lipides<sub>Shellfish</sub> / 5% (où lipides<sub>Shellfish</sub> est la teneur typique en lipides (%) des espèces de mollusques et de crustacés évaluées).

### 3. Introduction

Contaminant monitoring data form the basis of environmental assessments, which aim to characterise the status or quality of the marine environment with regard to chemical pollution. This means that measured concentrations are compared with assessment concentrations describing cut-offs for categories of environmental quality. These assessment concentrations are based on toxicological data and they have significant implications as they are used to classify the status of a marine area. OSPAR Background Assessment Concentrations (BACs) and Environmental Assessment Criteria (EACs) were primarily developed for the assessment of contaminant concentrations. If the mean concentration of a given data set is significantly below the BAC, the concentration is considered “near background” or “close to zero” in case of man-made substances. Compounds with concentrations below the BAC threshold fulfil the ultimate aim of the OSPAR Hazardous Substances Strategy of approaching natural background concentration or zero. OSPAR EACs were developed to assess if concentrations of contaminants were likely to have adverse biological effects on the marine environment. A first set of EACs was proposed by OSPAR in 2004 (OSPAR, 2004), with updates for PCBs and PAHs that became available in 2008 (OSPAR, 2009a; 2009b). The development of EACs used predicted-no-effects-concentration (PNEC) derived from no-observed-effect concentrations (NOEC) or lethal concentrations (LC<sub>10</sub>) and assessment factors, i.e. a safety margin to account for uncertainty related to the transfer of laboratory results to the field (OSPAR, 2012). However, EACs were not agreed for all contaminants/matrix combinations and therefore alternatives or EAC proxies have been used in OSPAR status assessments. For PAHs and metals in sediment, it was decided to use the US-EPA Effects Range system (NOAA, 1999; Buchman 2008). The Effect Range values were set on the basis of ecotoxicological criteria for sediment living organisms (Long *et al.*, 1995), and the Effects Range Low (ERL) set as the lower 10% effect level. As such, it is possible, but unlikely that effects can occur at concentrations lower than the ERL. Further details of assessment criteria used in OSPAR assessments are described in the EAC audit trail (OSPAR, 2019).

OSPAR Environmental Assessment Criteria (EACs) have not been developed for polybrominated diphenyl ethers (PBDEs) in sediment or biota and are unlikely to be developed in the near future. Furthermore, the ERL values used as EAC proxies in OSPAR assessments of heavy metals and PAHs in sediment, are not available for PBDEs. The few alternatives of PBDE assessment criteria (that MIME is aware of) with a broadly similar purpose are the Environmental Quality Standard (EQS) for biota, and the Canadian Federal Environmental Quality Guidelines (FEQGs) for sediment and biota. How these are derived, and the associated issues will be described, and recommendations provided on which are most suitable for OSPAR assessment purposes.

### 4. Synopsis of Background Information

#### 4.1 EU Environmental Quality Standards (EQS)

Environmental Quality Standards (EQS) for biota have been set at an EU level and should protect freshwater and marine ecosystems from possible adverse effects of chemicals as well as human health via drinking water or ingestion of food originating from aquatic environments. At present, EU wide biota EQS values exist for mercury (Hg), hexachlorobenzene (HCB), hexachlorobutadiene (HCBd), PBDEs, fluoranthene, benzo[*a*]pyrene, dicofol, perfluorooctane sulfonic acid (PFOS), dioxins/furans and dioxin-like polychlorinated

biphenyls (PCBs), hexabromocyclododecane (HBCD) and heptachlor/heptachlor epoxide (EU, 2013). The biota EQS values relate to fish, with the exception of PAHs, for which EQS values refer to crustaceans and molluscs. Quality Standards (QS) were also derived for sediment using sediment-dwelling organisms toxicity data, if available.

#### 4.1.1 EQS protection goal

The EQS is the maximum allowable concentration of a contaminant not causing harm and ensuring that aquatic environment and human health are adequately protected. In the process of deriving EQS, Quality Standards (QS) are derived for several different protection goals, i.e. the pelagic and benthic communities in freshwater, brackish and marine ecosystems, the predators of these ecosystems, and human health, using reported studies relevant for each target. Where several assessments were performed for the same compartment (e.g. biota: protection of predators from secondary poisoning and protection of humans from adverse health effects by consuming fisheries products), the QS for the different protection goals are compared and the lowest, most protective value are adopted as the overall EQS for the substance or substance group (EC, 2011a).

#### 4.1.2 Human Health and Secondary Poisoning Quality Standards (QS) for PBDEs in biota

Commercial PBDE mixtures are classified according to their degree of bromination. The dataset is composed of data on the main components of the pentaBDE and octaBDE mixtures: tetra-, penta-, hexa- and hepta-BDEs. This EQS applies in monitoring terms to the sum of the following six indicator congeners in fish: BDE28 (triBDE), BDE47 (tetraBDE), BDE99 (pentaBDE), BDE100 (pentaBDE), BDE153 (hexaBDE) and BDE154 (hexaBDE). The same mouse study (Branchi *et al.*, 2002) was used to derive the human health and secondary poisoning QSs with different back calculations being used to give the different values. For the human health QS a back calculation from the benchmark dose monitoring (BMDL10) of 9 µg kg<sup>-1</sup> bw gives an internal daily dose of 4.32 10<sup>-3</sup> µg kg<sup>-1</sup> bw d<sup>-1</sup>, when the longest half-life for BDE99 of 1,442 days in humans was considered (9\*ln2/1442). An assessment factor of 30 is applied (4.32 10<sup>-3</sup> /30) to give 0.144 x 10<sup>-3</sup> µg kg<sup>-1</sup> bw d<sup>-1</sup> which corresponds to a QS of to 8.522 10<sup>-3</sup> µg kg<sup>-1</sup> wet weight (ww), assuming a body weight of 60 kg and an uptake of 1 kg per day. Replication of the calculation of the EQS could not be repeated, there will slight differences in the numbers at each step. This may be due to rounding differences at the various stages.

In contrast, such consideration was not applied to the derivation of secondary poisoning QS, mainly applied in monitoring terms to fish, marine mammals or birds. Moreover, there is no clear evidence that the toxicodynamic would be the same than experimental mice. A NOEC of 4 mg kg<sup>-1</sup> food (Branchi *et al.*, 2002 and 2005) was used to derive the secondary poisoning EQS, along with an assessment factor of 90 to give (4/90\*1000) a QS of 44 µg kg<sup>-1</sup> ww.

**Table 1** – Toxicity studies used by European Commission to derive EQSs for PBDE (sum of BDE28, 47, 99, 100, 153 and 154) (in bold: EQS<sub>biota</sub>)

Target	Media	AF	Unit	QS	Endpoint	Species	Exposure period	Test compound	Evaluation criteria	Reference
Human health	N/A	30	µg kg <sup>-1</sup> biota ww	<b>0.0085</b>	120-days LOAEL (n)	Mice (CD-1 Swiss)	From gestational to 21 post-natal	BDE99	Hyperactivity; alterations in anxiety-like behavior	Branchi <i>et al.</i> , 2002 & 2005
Predators (secondary poisoning)	N/A	90	µg kg <sup>-1</sup> biota ww	44	120-day NOEC (n)					
Benthic community	Freshwater/	10	µg.kg <sup>-1</sup> dw 5%OC	1550		<i>L. variegatus</i>	Adult stage	c-pentaBDE	Survival, reproduction	Wildlife, 2000



Target	Media	AF	Unit	QS	Endpoint	Species	Exposure period	Test compound	Evaluation criteria	Reference
	Marine	50	$\mu\text{g kg}^{-1}_{\text{dw } 5\%OC}$	310	28-day NOEC (n)	(oligochaete)				
Pelagic community	Freshwater/	10	$\mu\text{g.l}^{-1}$	0.049	4-day NOEC (n)	<i>Psetta maxima</i> (marine fish)	Larval stage	tetraBDE-47*	Development, mortality	Mhadhbi <i>et al.</i> , 2010
	Marine	100	$\mu\text{g l}^{-1}$	0.0049						

#### 4.1.3 Review of the study used to derive the biota EQS (Branchi *et al.* (2002))

The most stringent QS is the value derived for human health (Table 1) and therefore this was selected as the EQS ( $0.0085 \mu\text{g kg}^{-1}$  wet weight). The main concerns about the study (Branchi *et al.*, 2002) used to derive this value are that the toxic end-point selected does not show monotonic dose-effect relationship, and the assessment factor (30) used. Branchi *et al.*, 2002 administered daily three treatments of BDE99 at concentrations of 0.6, 6 and 30  $\text{mg kg}^{-1}$  ww to female mice by gavage from gestational day (GD) 6 to postnatal day (PND) 21. In this study, prolonged perinatal BDE99 exposure produced a transient hyperactivity, characterised by an inverted dose-response relationship (with main effects at medium dose), that ends around 4 months of age. This suggests that PBDE exerted neurotoxicity on mice. The author hypothesised that transient effects and inverted dose response relationship might be due to 1) by a potential compensation phenomenon in organism under-development or 2) to a prepartum selection, whereby the most vulnerable subjects died during gestation in the high PBDE99 dose groups and the more resistant surviving individuals showed only subtle and transient behavioural alterations during the developmental phase. The study also highlights that this is a preliminary investigation and that more work is needed.

#### 4.1.4 Comparison of the EQS to Environmental PBDE Concentrations

The EQS value of  $0.0085 \mu\text{g kg}^{-1}$  ww is low compared to typically reported environmental concentrations in biota. Furthermore, this concentration is below many monitoring laboratories detection limits. Eljarrat and Barcelo (2018) reviewed 37 articles published between 2012 and 2017, corresponding to 3,510 fish samples. They showed that despite the ban of penta and octaPBDEs in Europe since 2004, PBDE concentrations in river fish samples exceeded the European EQS in all studies, with the exception of 2 studies in Eastern China and Antarctica where PBDE concentrations below  $0.006 \mu\text{g kg}^{-1}$  ww were reported. In Antarctica, PBDE concentrations in fish up to up to  $0.57 \mu\text{g kg}^{-1}$  ww were also reported. A quarter of European river fish samples (25%) exceeded the EQS by up to ten thousand times.

Guidance on the implementation of biota EQS dictates a number of conditions that biota data should comply with in order to meet biota compliance requirements of the WFD (EC, 2009). This includes a stipulation that monitoring data should be converted to a trophic level of four (or five for marine). Most contaminant data for environmental fish monitoring will be for species of a lower trophic level, therefore trophic magnification factors (TMF) will need to be applied to account for the extra magnification step in the marine environment which in most case will give a higher concentration. However, currently databases do not hold information on the trophic level of fish for which contaminant data is submitted.

#### 4.1.5 Sediment Quality Standard

A QS for PBDEs (sum of BDE28, 47, 99, 100, 153 and 154) in sediment was estimated from sediment-dwelling organisms toxicity data. NOEC values of 6.3, 16 and  $3.1 \text{ mg kg}^{-1}$  dry weight were derived from long-term studies (Wildlife, 2000a, b & c). However, the organic carbon content of the sediment used in these tests was

not available, although the organic matter content was reported as <2%. The organic matter content is approximately two times higher than the organic carbon content, and therefore it was assumed that the organic carbon content was <1%. A requirement of the sediment QS is that the concentration should be normalised to a standard organic carbon content of 5%. Therefore, the lowest NOEC was multiplied by 5 to give a value of 15.5 mg kg<sup>-1</sup> dry weight. A tentative QS was derived from this by applying an assessment factor of 50, for marine sediment, to give a QS of 310 µg kg<sup>-1</sup> dry weight.

## 4.2 Canadian Federal Environmental Quality Guidelines (FEQGs)

### 4.2.1 FEQG Protection Goal

Federal Environmental Quality Guidelines (FEQGs) are benchmarks for aquatic ecosystems, which are intended to protect all forms of aquatic life (vertebrates, invertebrates, and plants) from direct adverse effects for indefinite exposure periods *via* the water column (Environment Canada, 2006, 2013). Where concentrations are below the FEQG, adverse effects on aquatic organisms or the wildlife that predate on them are unlikely.

### 4.2.2 Proposed FEQG

The FEQGs for water (FWQGs) were calculated from the lowest toxicity endpoint for each individual congener reported in chronic toxicity tests (critical toxicity values, CTV; Table 2) and then applying an application factor (AF). An AF of 100 was applied to the CTV, 10 to account for the extrapolation from laboratory to field conditions and inter- and intra-species variations in sensitivities, and 10 because PBDEs are persistent and bioaccumulative. Federal Sediment (FSeQGs) and fish tissue (FFTGs) Quality Guidelines were derived for individual congeners from the PBDE-spiked water test using either *Arcatia tonsa* or *Daphnia magna* (FWQGs, Table 2) and the field-based bioaccumulation factor (BAF<sub>field-based</sub>) or biota-sediment accumulation (BSAF) factors (Table 3):

$$\text{FFTG} = \text{FWQG} * \text{BAF}_{\text{field-based}}$$

In the absence of BAFs for hepta-, octa- and deca-BDE, FFTGs could not be derived (Table 3).

Federal Sediment Quality Guidelines (FSeQG) were calculated from FFTGs, converted to dry weight assuming 75% moisture content and divided by appropriate biota-sediment accumulation factor (BSAFs) which was adjusted to 1% organic carbon (Table 3):

$$\text{FSeQG} = \frac{\text{FFTG}/0.25}{\text{BSAF}}$$

Federal Wildlife Dietary Guidelines (FWiDG) are intended to protect mammalian and avian consumers of aquatic biota. These are benchmarks of concentrations of toxic substances in aquatic biota (whole body, ww) which are consumed by terrestrial and semi-aquatic wildlife (Table 4). Federal Wildlife Dietary Guidelines (FWiDG) were derived using the critical toxicity values (CTV) and applying assessment factors such as for Lab to field (10) rodent to wildlife (10) acute to chronic (10) and food intake: body weight ratios (Table 4):

$$\text{FWiDG} = \text{CTV}/\text{AF}$$

**Table 2** – Studies used to derive Federal Water Quality Guidelines proposed (Environment Canada, 2013).

Homologue	Tested chemical	AF <sup>a</sup>	CTV <sup>b</sup> µg L <sup>-1</sup>	FWQG <sup>c</sup> ng L <sup>-1</sup>	Species	Exposure	Endpoint	Evaluation criteria	Reference
triBDE	BDE28	100	4.6	46		From egg to larval stage	5-d EC10	larval development	
tetraBDE	BDE47	100	2.4	24					

pentaBDE	BDE99	100	0.39	<b>4</b>	<i>Acartia tonsa</i>				Wollenberg er <i>et al.</i> , 2005
pentaBDE	BDE100	100	0.023	<b>0.2</b>	(marine copepod)				
hexaBDE	BDE153	100	12	<b>120</b>	<i>Daphnia magna</i>	<24h old larval to adult stage	21-d EC40	lethality, growth, reproduction	Nakari & Huhtala, 2008
heptaBDE	OctaBDE <sup>d</sup>	100	1.7	<b>170</b>	<i>Daphnia magna</i>	<24h old	21-day	survival, growth,	Graves <i>et al.</i> , 1997
octaBDE	OctaBDE <sup>d</sup>	100	≥1.7	<b>170</b>	(freshwater flea)	larval to adult stage	NOEC/LOE C	reproduction	

<sup>a</sup> AF: application factor of 100 (10 to account for the extrapolation from laboratory to field conditions and inter- and intra-species variations in sensitivities, and 10 because PBDEs are persistent and bioaccumulative).

<sup>b</sup> CTV: Critical toxicity values calculated from measured (M), nominal (N) or extrapolated (E, Extrapolated value, 55% inhibition at the lowest test concentration (0.2 mg L<sup>-1</sup>) exposure concentrations.

<sup>c</sup> FWQG: Federal Water Quality Guidelines calculated by dividing CTV by an application factor (AF).

<sup>d</sup> Commercial OctaBDE

**Table 3** – Selected bioaccumulation (BAF) and biota-sediment accumulation (BSAF) factors for various PBDEs congeners used to derive fish tissue and sediment quality guidelines (Environment Canada unpublished data). For comparison purposes, BAF reported in the EQS dossier are also indicated in the table.

Homologue	Congener	BAF (Canadian doc.)	BSAF	BAF (EQS dossier)
		L g <sup>-1</sup> ww	µg kg <sup>-1</sup> dw	L kg <sup>-1</sup> ww
triBDE	BDE28	2.5 <sup>a</sup>	10.5	794 (snail)
tetraBDE	BDE47	3.6 <sup>a</sup>	8.9	19.9 x 10 <sup>6</sup> (trout)
pentaBDE	BDE99	0.3 <sup>a</sup>	13.0	5 x 10 <sup>6</sup> (trout)
pentaBDE	BDE100	6.1 <sup>a</sup>	15.8	31.6 x 10 <sup>6</sup> (trout)
hexaBDE	BDE153	3.5 <sup>a</sup>	3.9	
hexaBDE	BDE154			2 x 10 <sup>6</sup> (snail)

dw: dry weight; ww: wet weight;

<sup>a</sup> based on data for mysterysnail (*Cipangopaludina chinensis*), prawn (*Macrobrachium nipponense*), mud carp (*Cirrhinus molitorella*), crucian carp (*Carassius auratus*), northern snakehead (*Ophicephalus argus*) and water snake (*Enhydris chinensis*) (unpublished data).

**Table 4** Derivation of Federal Wildlife Dietary Guidelines (FWIDG) for mammalian and avian species

Homologue	Congener	Species and reference	Endpoint	CTV (µg/kg bw)	AF	Guideline (ng/g wet diet)
tetraBDE	47	Mouse ( <i>Mus musculus</i> ) <sup>a</sup>	Single dose LOAEL	10,500	Lab to field = 10; Rodent to wildlife = 10	44

Homologue	Congener	Species and reference	Endpoint	CTV (µg/kg bw)	AF	Guideline (ng/g wet diet)
pentaBDE	99	Mouse ( <i>Mus musculus</i> )a,b	(Behavioural after 5 months) Single dose LOAEL (Behavioural after 5 months)	800	acute to chronic = 10 FI:BW** = 0.24 (mink) Lab to field = 10 Rodent to wildlife = 10 acute to chronic = 10 FI:BW = 0.24 (mink) 100 (see text)	3
PentaBDE	mixture	Mink ( <i>Mustela vison</i> )c	reduced growth NOAEL 1 µg/g in diet LOAEL 10 µg/g diet	geomean 3.16 µg/g diet		32
PentaBDE	mixture	American Kestrel ( <i>Falco sparverius</i> ) d,e	reproductive and behaviour effects	geomean (see text) 0.13 µg/g diet	Default = 10	13
hexaBDE	153	Mouse – NMRI ( <i>Mus musculus</i> )f	Single dose LOAEL (Behavioural later in adult stage )	900	Lab to field = 10 Rodent to wildlife = 10 acute to chronic = 10 FI:BW = 0.24 (mink)	4
heptaBDE	183	Mouse – NMRI ( <i>Mus musculus</i> )g	Single dose LOAEL (Behavioural after 2 months)	15 200	Lab to field = 10 Rodent to wildlife = 10 acute to chronic = 10 FI:BW = 0.24 (mink)	64
nonaBDE	206	Mouse – NMRI ( <i>Mus musculus</i> )g	Single dose LOAEL (Behavioural after 2 months)	18 500	Lab to field = 10 Rodent to wildlife = 10 acute to chronic = 10 FI:BW = 0.24 (mink)	78
decaBDE	209	Mouse - NMRI ( <i>Mus musculus</i> )h	Single dose LOAEL (Behavioural, Physiological later in adult stage)	2220	Lab to field = 10 Rodent to wildlife = 10 acute to chronic = 10 FI:BW = 0.24 (mink)	9

CTV, critical toxicity values; AF, assessment factors; FI:BW, food intake: body weight ratios

#### 4.2.3 Review of the studies used to derive the FEQGs

There are very few ecotoxicological studies available for PBDEs, leading to uncertainty in the toxicity thresholds. The Wollenberger *et al.* (2005) study used to derive the FEQG for BDE47 (together with FEQG for BDE28, BDE99, BDE100) reported on subchronic effects on larval development of a marine copepod *Acartia tonsa* exposed to individual PBDE congeners dissolved in seawater. Larval development is not a typical endpoint, but which could be justified as PBDEs are expected to be endocrine disruptors (Darnerud *et al.*, 2001; Yu *et al.*, 2011). Wollenberger *et al.* (2005) and Nakari & Huhtala (2008) derived threshold values based on extrapolated concentration, as for BDE100, and nominal concentration as for BDE153 and BDE47.

FWQG were derived even if only one ecotoxicity dataset was available, as opposed to EU approach which require a minimum of 3 acute studies at different trophic levels (algae, invertebrates and fish) available.

The application factor used in the Canadian approach only took into account i) the extrapolation from laboratory to field conditions and species sensitivities, and ii) the vPvB (very persistent and very bioaccumulative) characteristics of PBDEs congeners. FWQGs apply to both freshwater and marine environments without applying conversion factors to derive guideline values from freshwater to marine species or/and from marine species to freshwater species.

Justification for developing FEQG for individual congeners is not detailed in the Canadian document. The EU justification for deriving an assessment criterion for the sum of 6 congeners (BDE28, BDE47, BDE99, BDE100, BDE153 and BDE154) rather than individual congeners was (EC, 2011b):

- There is no reliable mixture toxicity predictive model:
- Not enough experimental data on single toxicity of the mixture components to model their mixture effect
- Due to high log  $K_{ow}$  values, there seems to be not even the possibility of using QSAR estimations for setting up a toxicity ranking between the mixture components.

#### 4.3 Use of FEQGs in MIME PBDE trial assessment

The FEQGs currently being trialled in the MIME assessment are shown in Table 5. The FEQGs for biota either relate to fish health (concentrations that should not cause adverse effects on fish) or mammalian wildlife health (concentrations in fish that should not cause adverse effects on mammalian predators). They may not be appropriate for assessing the effect of PBDEs on other groups (such as plants). As such, they might be less protective than an EAC (the concentration that should not cause adverse effects on the most sensitive marine organisms). The FEQGs for sediment relate to the health of sediment-dwelling and pelagic animals and again may not be appropriate for other groups (such as plants).

To apply the FEQGs, several further decisions were required:

- where an FEQG was available for both fish and mammalian wildlife health, the lower, most protective value was chosen
- an FEQG for a homologue group was applied to the individual PBDE congeners within that group.
- biota FEQGs were applied to all fish (typically liver concentrations) and shellfish (soft body concentrations)
- sediment FEQGs were normalised to 2.5% organic carbon (by multiplying by 2.5)
- sediment FEQGs were also applied to non-normalised concentrations (Iberian Sea and Gulf of Cadiz)
- the sediment FEQG for octa BDEs was applied to BD183 (a hepta BDE) since the substance tested in the toxicity tests was a mixture of hepta and octa BDEs

**Table 5** Canadian Federal Environmental Quality Guidelines (FEQGs) for PBDEs currently being trailed in the MIME assessment, alongside the human health and secondary poisoning QS for comparison.

AC	substance	value	tissue (fish)	purpose
biota ( $\mu\text{g kg}^{-1}$ wet weight)				
QS	sum BDE28, 47, 99, 100, 153, 154	0.0085	fish muscle	human health
QS	sum BDE28, 47, 99, 100, 153, 154	44	whole fish	secondary poisoning
FEQG	total tri BDE (includes BDE28)	120	whole fish	fish health
FEQG	total tetra BDE (includes BDE47, 66)	44	whole fish	mammalian wildlife health
FEQG	total penta BDE (includes BDE85, 99, 100)	1	whole fish	fish health
FEQG	total hexa BDE (includes BD153, 154)	4	whole fish	mammalian wildlife health
FEQG	total hepta BDE (includes BD183)	64	whole fish	mammalian wildlife health
FEQG	total octa BDE	63	whole fish	mammalian wildlife health
FEQG	total nona BDE	78	whole fish	mammalian wildlife health
FEQG	total deca BDE (includes BD209)	9	whole fish	mammalian wildlife health
sediment ( $\mu\text{g kg}^{-1}$ dry weight)				
FEQG	total tri BDE (includes BDE28)	44		sediment-dwelling & pelagic health
FEQG	total tetra BDE (includes BDE47, 66)	39		sediment-dwelling & pelagic health
FEQG	total penta BDE (includes BDE85, 99, 100)	0.4		sediment-dwelling & pelagic health
FEQG	total hexa BDE (includes BD153, 154)	440		sediment-dwelling & pelagic health
FEQG	total octa BDE	5600		sediment-dwelling & pelagic health
FEQG	total deca BDE (includes BD209)	19		sediment-dwelling & pelagic health

#### 4.3.1 Proposed Changes for PBDE MIME assessments

The FEQGs for biota are set for whole fish and units are  $\mu\text{g kg}^{-1}$  ww. However, in this form, they fail to account for potential differences in the uptake of PBDEs due to differences in the lipid content of different monitoring species and tissues. The FEQGs were therefore adjusted to a lipid weight basis by assuming the whole fish used in the toxicity trials had a 5% lipid content and multiplying the FEQGs (on a wet weight basis) by 20 (Table 6).

In the MIME assessments, fish PBDE concentrations, which are typically measured in the liver, will be assessed on a lipid weight basis and compared directly against the lipid-adjusted FEQGs.

A more complicated procedure will be required for shellfish (since generally PBDE concentrations in shellfish are reported on a wet weight basis without supporting lipid content measurements). The procedure will replicate that currently used in the MIME assessment for polychlorinated biphenyl concentrations in shellfish, where concentrations are also typically reported on a wet weight basis and the EAC is expressed on a lipid weight basis. Specifically, the assessment will be conducted on a wet weight basis, with the lipid-adjusted FEQGs back-transformed to a wet-weight basis based on the typical lipid content of the shellfish species being assessed (as determined by data in the ICES database). Thus, the monitoring data will be compared to an assessment concentration given by the lipid-adjusted FEQG  $\times$  lipid<sub>shellfish</sub> / 100% (where lipid<sub>shellfish</sub> is the typical % lipid content of the shellfish species being assessed). Note that this is equivalent to comparing the monitoring data to the original FEQG  $\times$  lipid<sub>shellfish</sub> / 5%. Since lipid<sub>shellfish</sub> is typically less than 5%, the assessment concentration used will be lower than the original FEQG. This is consistent with the lower trophic level of shellfish compared to fish. Shellfish are on a lower trophic level than fish, and therefore concentrations will be lower than in fish, since PBDEs can biomagnify.

**Table 6** FEQGs for biota and sediment. The FEQG for fish has been normalised to 5% lipid ( $\times$  20, assuming a 5% lipid content). For sediment the FEQG is normalised to 2.5% organic carbon (FEQG  $\times$  2.5)

	Biota FEQG ( $\mu\text{g kg}^{-1}$ , $\mu\text{g kg}^{-1}$ ww)	Fish normalised to 5% lipid)	Sediment FEQG ( $\mu\text{g kg}^{-1}$ , normalised 2.5% TOC)
BDE28	120	2400	110
BDE47	44	880	97.5
BDE66			97.5
BDE85			1
BDE99	1	20	1
BD100	1	20	1
BD153	4	80	1100
BD154	4	80	1100
BD183			14000
BD209			47.5
			47.6

## 5 Conclusions

For PBDEs in sediment the only assessment criteria currently available are the FEQGs, and therefore for sediment there is currently no other option for status assessments of individual PBDE congeners.

For PBDEs in biota, there are three options: the EQS for human health, the QS for secondary poisoning and the FEQGs. The Environmental Quality Standard (EQS) for PBDEs in biota is the human health EQS. MIME has been tasked to conduct two separate assessment, one with assessment criteria considering human health and one for the environment. The EQS will be included in the assessment for human health. The QS for secondary poisoning is more relevant to use in the environmental assessment than the EQS (human health) and is broadly comparable to the FEQGs (the QS equals the FEQG of BDE47 for mammalian wildlife health). However, the FEQGs are preferred to the QS for three reasons. The QS is for the sum of six PBDE congeners,

so information about individual congener patterns is lost, where the FEQGs relate to specific homologue groups or individual congeners, Second, the QS provides a less stringent assessment of the more toxic homologues; high concentrations of the more toxic penta-PBDEs that might cause environmental harm could go undetected because they are masked by the concentration of BDE47 (which usually dominates the PBDE profile in fish and shellfish). Third, some data series miss some of the six PBDE congeners that are the basis of the QS, so assessment against the QS would not be possible. The FEQGs are based on toxicity tests on a range of organisms but come with many caveats.

## 6 Proposals

MIME propose that the FEQGs be applied as EAC-proxies in the OSPAR assessment of PBDE concentrations in biota (fish and bivalves) and sediment

- For sediment, FEQGs will be normalised to 2.5% organic carbon (by multiplying by 2.5). All data (except for the Iberian Sea and Gulf of Cadiz) will be normalised to 2.5% for comparison.
- For fish the FEQGs will be converted from a ww basis to a lw basis by multiplying by 20, assuming that the fish used to calculate the FEQGs 'typically' had a lipid content of 5%. PBDE fish (muscle and liver) concentrations will be assessed on a lipid weight basis.
- For bivalves, concentrations will be assessed on a ww basis. The FEQGs will be adjusted to take into account the lower lipid content of shellfish by multiplying the original FEQGs by the ratio  $\text{lipid}_{\text{shellfish}} / 5$  (where  $\text{lipid}_{\text{shellfish}}$  is the typical % lipid content of the shellfish species being assessed).

There are clearly inconsistencies between the FEQGs, the CEMP monitoring data, and the underlying principles of an EAC, but these were regarded as small compared to the benefits of having EAC-proxies that could be used to assess status and to monitor progress towards good environmental health.

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