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An empirical approach to the determination of metal regional Sediment Quality Guidelines, in marine waters, within the European Water Framework Directive

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Regional Sediment Quality Guidelines (SQG) for metals from the Basque Country (northern Spain) were determined. These guidelines are proposed to be used for management purposes, within the Water Framework Directive (WFD). SQG were determined on the basis of sediment chemistry, toxicity and benthic community disturbance, from 961 estuarine and coastal samples. The guidelines were calculated using a percentile approach (effect and non-effect data), following normalisation on the fine-sediment content and with non-normalisation. The feasibility of SQG was quantified by the incidence of adverse effects, that is, the ratio of effect/no-effect data, which increased significantly with increasing SQG ranges. The study proposes the following formula to calculate SQG: $SQG_{metal} = SQG^{*}_{metal}\ \mu g \cdot g^{-1}/FC.\ 10^{-2}$ (where FC is fine-sediment content and SQG* is the normalised SQG). The SQG* were 13.5 $\mu g \cdot g^{-1}$ for As, 1 $\mu g \cdot g^{-1}$ for Cd, 39 $\mu g \cdot g^{-1}$ for Cr, 55 $\mu g \cdot g^{-1}$ for Cu, 0.53 $\mu g \cdot g^{-1}$ for Hg, 23 $\mu g \cdot g^{-1}$ for Ni, 78 $\mu g \cdot g^{-1}$ for Pb and 249 $\mu g \cdot g^{-1}$ for Zn. The use of four different organisational biological levels ensures an improvement in the determination of metal regional SQG for the assessment of the chemical and physicochemical status in marine waters, within the WFD.

Keywords: marine sediments; Sediment Quality Guidelines; metals; Basque Country; Water Framework Directive

1. Introduction

The European Water Framework Directive (WFD) [1] aims to achieve 'a good ecological and chemical status', by 2015, within all European water bodies, including estuarine and coastal waters (for details, see refs [2,3]). Most of the methods developed for the implementation of the WFD refer only to assessment of the quality of the water column. Hence, the recent Directive 2008/105/EC [4] establishes environmental quality standards (EQS), for priority substances, in the dissolved fraction for waters. However, according to this Directive, Member States should monitor these priority substances in sediments and should be able to establish EQS at national level (Article 3) [5–10]. In this way, the use of EQS, including ecological targets for waters and sediments (as proposed within the WFD [1] and Directive 2008/105/EC [4]), together with the

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background levels, used as reference conditions for assessing physicochemical status [11–13], are a key objective of the WFD.

There are two levels in the use of chemical indicators (i.e. priority substances) within the WFD [2,11,12,14,15]: (1) classification of the chemical status, which includes only priority substances [9,16]. In this case, priority substance concentrations exceeding the EQS result in a failure to achieve the chemical status; in turn, if concentrations lie below the EQS, the chemical status is achieved [14]; and (2) physicochemical conditions influencing the biological quality, which are related mainly to eutrophication processes, and involve nutrients, oxygen, or turbidity [17,18], but including also chemicals [14]. Hence, the physicochemical status of sediments can be considered as: 'high', when metal concentrations lie below background levels; 'good', when they range between the background level and EQS; and 'moderate', when they exceed the EQS [11,13]. Background values for Basque Country sediments were established by Rodríguez et al. [11], using the maximum likelihood mixture estimation approach; however, EQS were never determined.

The WFD defines an EQS as, 'the concentration of a particular pollutant or group of pollutants in water, sediment or biota that should not be exceeded in order to protect human health and the environment'. Metal concentrations over the EQS can be considered as indicating that the ecosystem will be affected by serious or irreversible harm, or that society has driven the ecosystem to an unstable state [19].

In this sense, Sediment Quality Guidelines (SQG) have been developed elsewhere to deal with many environmental concerns and in response to regulatory programmes [20,21]. SQG are numerical chemical concentrations in sediments intended to be either protective of biological resources, or predictive of adverse effects to those sources, or both [22]. SQG are exactly what they are named – guidelines [23]. They are not definitive indications of whether harm will occur, although typically three levels are set: (1) concentrations below which adverse effects are unlikely; (2) concentrations above which adverse effects are likely; and (3) intermediate concentrations within which adverse effects may or may not occur [23]. In this way, they can be applied, not as a criteria, but as the term suggests, providing guidance values for project managers and decision makers [24], within the context of a regulatory framework (i.e. the WFD) [5]. Hence, SQG would represent the boundary between achieving, or not, the chemical status; or classifying the physicochemical status in the sediments, within the WFD [11].

Deriving SQG for marine sediments is a difficult task [24]. The approaches for the estimation of SQG can be classified into two different groups [21,25,26]. On the one hand, theoretical/mechanistically derived SQG are based upon the equilibrium partitioning approach [27,28], which assumes that chemical activity in the sediment, as indexed by the chemical concentration in the interstitial water, is proportional to the bioavailability of chemicals to sediment-dwelling organisms. According to the equilibrium partitioning approach, the biotic ligand model and the concept of sulfide-bound metals described by the ratio of simultaneously extracted metals and the acid-volatile sulfide concept have been developed to predict the metal concentration in sediment, that is, in equilibrium with the biotic ligand effects concentration [28]. However, mechanistically, approaches cannot be used to describe the partitioning of metal compounds in the various environmental compartments for the reasons described in ECHA [29].

On the other hand, the empirically derived methods are based upon measured chemical concentrations and the corresponding observed biological effects, such as the structural parameters of the benthic community, the laboratory spiked-sediment bioassay approach and field-sediment toxicity [16,30–35].

Moreover, Paya-Pérez et al. [36] propose that if no reliable sediment toxicity data are available, equilibrium partitioning-based approaches can be used to estimate the EQS, but with a high degree of uncertainty. Finally, Paya-Pérez et al. [36] recommend that field data should be considered because the WFD states that the 'EQS derived should be compared and corroborated with any evidence from field or mesocosms studies'. Hence, Paya-Pérez et al. [36] proposes the use of

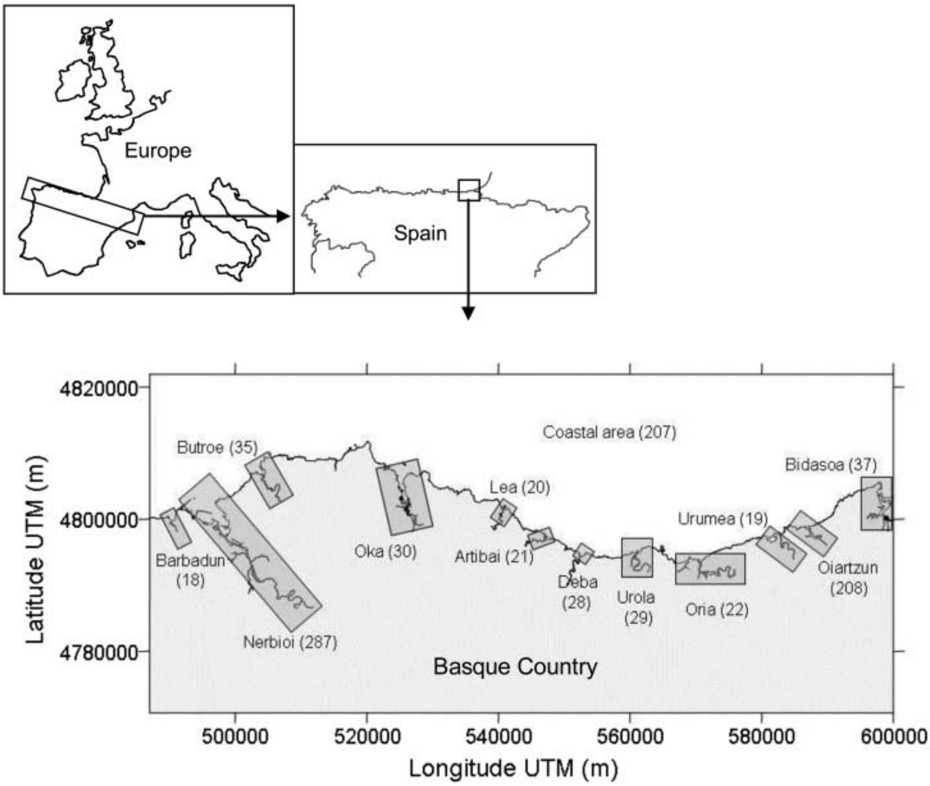

Figure 1. Map of the Basque Country, showing sampling locations.
Note: the number in parentheses indicates the number of samples obtained.

empirical approaches that link biological responses of benthos to chemical contamination in the field. These are based primarily on field data, in which matched sediment chemistry and biological effect data are analysed using various statistical approaches to relate chemical concentrations to the frequency of biological effects [26].

SQG for metals, calculated by Long et al. [31] and those described by Buchman [32] have been used within the WFD, in assessing chemical and physicochemical status [14]. Nevertheless, according to Chapman [37], the SQG should be used in the region where they were developed, i.e. guidelines derived on site-specific data are able to better predict the toxicity of contaminants for each specific coastal environment [16].

Hence, the aims of this article are to: (1) determine the regional SQG of metals in coastal and estuarine sediments, for the Basque Country, northern Spain (Figure 1), based upon the chemical, toxicity and benthic biotic index values already available in a database; (2) quantify the accuracy of these guidelines to be used in assessing chemical and physicochemical status within the WFD (until EQS could be determined); and (3) compare these SQG with background values calculated for Basque Country and with those SQG values developed using other data and methods, for different areas.

2. Material and methods

2.1. *Study area and sediment sampling*

A total of 961 sediment samples used in this study were obtained from different estuarine (754) and coastal (207) locations along the Basque coast (Figure 1), between 1989 and 2008, including both intertidal and subtidal (0–65 m water depth) samples. Intertidal sediment samples were collected directly by hand, whereas subtidal samples were collected using Van Veen, Day or box-corer grabs. In both cases, the upper 10 cm of sediment was collected. Sediment samples were retained in plastic bottles and stored at 4 °C, until analysis [12]. Sediment was sampled following ISO 5667-15 [38], ICES [39] and OSPAR [40].

2.2. *Data acquisition and analysis*

The sediment chemistry, toxicity and the associated benthic community were analysed. Metal concentrations (As, Cd, Cr, Cu, Ni, Hg, Pb, Zn) were analysed within the fine-sediment content sediment fraction (i.e. $<63\ \mu m$) [11,41–44]. This fraction was obtained by sieving samples previously oven-dried at 60 °C. Analysis of the metal concentrations was carried out using the acid-extractable metal concentration method by means of an acid mixture of HCl/HNO_3 (1:2, v/v). The accuracy of the analytical procedures employed for the analysis of metals in sediment samples was checked using the PACS-2 (NRC, Canada) certified reference material. Recoveries for the certified metals were: 78.3% As, 91.9% Cd, 63.1% Cr, 86.5% Cu, 98.8% Ni, 95.0% Hg, 100.4% Pb, 87.8% Zn (for details of the methodology, see [11,14]. All concentrations are expressed as $\mu g \cdot g^{-1}$ based on dry weight).

SQG values were calculated from metal concentration non-normalised and normalised by fine-sediment content ($<63\ \mu m$). Normalisation on the basis of fine-sediment content was performed by multiplying the metal concentration ($\mu g \cdot g^{-1}$) by the fine-sediment content (%), divided by 100. This normalisation was taken into account previously by Vandecasteele et al. [45], VLAREBO [46] and VROM [47].

The toxicity of the samples was established using three toxicity tests which are frequently performed to evaluate the toxicity of the contaminants present within marine sediments [48–50] and due to their sensitivity to single metals: (1) the Microtox® or bioluminescence inhibition bioassay [51,52]; (2) the 48-h embryo–larval toxicity test of the sea urchin *Paracentrotus lividus* [53]; and (3) the 10-day survival test, with amphipods of the genus *Corophium* [54,55]. All the toxicity tests were performed on the whole sediment (after sieving through 1 mm).

Benthic community disturbance was determined by means of the AZTI's Marine Biotic Index (AMBI) [56], using the software available at http://ambi.azti.es, following the recommendations of Borja and Muxika [57]. The AMBI provides values ranging between 0 and 7, according to a disturbance alteration gradient from no alteration (0) to extreme alteration (7). The benthic macrofaunal responses have been used in developing or testing SQG, in a similar way, in other countries [21,58,59].

The number of determinations for each metal, for toxicity and AMBI is listed in Table 1. For each sample and metal, the data from the four biological effects measured (see above) were considered as different cases. Because of this approach, taking into account that not all the metals

Table 1. Toxicity and AMBI data for each metal generated from a total of 961 of estuarine and coastal surface sediment samples within the Basque Country.[a]

Biological effect	As	Cd	Cr	Cu	Ni	Hg	Pb	Zn
Microtox	106	104	103	128	110	128	124	128
Amphipod mortality	112	104	107	134	116	133	130	134
Embryo success	27	40	33	48	33	47	49	48
AMBI	742	615	750	893	879	861	884	895
Total data	987	863	993	1203	1138	1169	1187	1205

Note: [a] For locations, see Figure 1.

and biological effects were measured in all of the sediment samples, the overall number of sediment samples is not coincident with the total data for each of the metals.

Each of the toxicity and benthic measurements from a sample was ascribed to an effect/no-effect descriptor, following the terminology used by Long et al. [31]. The criteria for the effect entry was assigned when: (1) EC_{50} values for the Microtox® test were $<1000\,mg{\cdot}L^{-1}$ [52]; (2) there was a reduction of at least 20% in amphipod survival, or *P. lividus* embryo success and significant differences (at alpha = 0.05) with regard to the control sediment [60,61]; and (3) the AMBI value was >3.3 (the value above which benthic community is classified, at least, as moderately disturbed, following Borja et al. [56]). For example, this last value has been demonstrated as a useful boundary in detecting impacted and unimpacted areas in aquaculture farms across Europe [62] and mining activity in Greenland [63].

2.3. *Derivation of SQG*

In order to calculate the SQG, two methods have been investigated, as summarised below: (1) the methodology described by Long et al. [31], which determines two values for each metal, the effect range–low (ERL) and the effect range–median (ERM) concentration. The ERM is considered to be associated with likely adverse effects on marine biota. The ERL and ERM were calculated as the 10th and 50th percentile concentration of the effect dataset, respectively; and (2) the methodology by Buchman et al. [32], derived previously by McDonald [30], which establishes two values, the threshold effect level (TEL) and the probable effect level (PEL). The TEL was calculated as the geometric mean of the 15th percentile concentration of the effect dataset, together with the 50th percentile concentration of the no-effect dataset; it represents the concentration below which adverse effects are expected to occur but only rarely. The PEL was calculated as the geometric mean of the 50th percentile concentration of effect dataset and the 85th percentile concentration of the no-effect samples; it represents the concentration above which adverse effects are on a frequent basis expected.

χ^2 tests were performed to evaluate whether the increasing effect percentages with increasing SQG ranges was significant, that is, the ratio of effect/no-effect data differed significantly (at alpha = 0.05) between each range of the SQG. All of these analyses were performed using Statgraphics® Plus 5.0.

3. Results

The SQG values for each metal were derived from the number of data listed in Table 1. The range of concentrations for each metal was: As (1.32–$478\,\mu g{\cdot}g^{-1}$), Cd (0.02–$27\,\mu g{\cdot}g^{-1}$), Cr (0.09–$786\,\mu g{\cdot}g^{-1}$), Cu (1.4–$1132\,\mu g{\cdot}g^{-1}$), Ni (0.21–$290\,\mu g{\cdot}g^{-1}$), Hg (0.009–$61\,\mu g{\cdot}g^{-1}$), Pb (0.05–$2263\,\mu g{\cdot}g^{-1}$) and Zn (15.6–$3708\,\mu g{\cdot}g^{-1}$).

The SQG values, non-normalised by the fine-sediment content, are listed in Table 2. For all of the metals, the TEL and PEL values for the Basque Country were slightly higher than the ERL and ERM values, respectively, especially for As, Cd, Hg and Pb. The SQG values normalised by the fine-sediment content (i.e. SGQ*) (Table 3) are notably lower than the non-normalised values, especially for ERL and TEL, with differences of up to of 25 and 12 times, respectively.

In order to determine the accuracy of the guideline values, the incidence of effects was calculated for the two methodologies (see above). According to Long et al. [31], accuracy was considered as good when: the incidence of the effect was low (<25%), in the minimum effect range; and high (>75%), in the probable effect range. In this sense, the incidence of the effects associated with the ERL* and ERM* values, normalised by the fine-sediment content, were <25% in the

Table 2. Sediment Quality Guidelines for metals ($\mu g \cdot g^{-1}$) calculated on the basis of this study (in bold) (non-normalised by the fine sediment content $< 63\ \mu m$) and previous studies.

	Authors	Area	Calculation method	Sediment	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn
ERL	[31]	USA	10th percentile EDS	bulk	8.2	1.2	81.0	34	0.2	20.9	46.7	150
ERL	This study	BC	10th percentile EDS	<63 m	**7.5**	**0.2**	**25.6**	**27.0**	**0.1**	**21.0**	**31.0**	**120.5**
TEL	[32]	USA	$\sqrt{\text{15th percentile EDS * 50th percentile NEDS}}$	bulk	7.2	0.68	52.3	18.7	0.13	15.9	30.2	124
TEL	This study	BC	$\sqrt{\text{15th percentile EDS * 50th percentile NEDS}}$	<63 m	**13.0**	**0.4**	**35.1**	**40.4**	**0.3**	**25.8**	**52.9**	**180.4**
NC	[33]	The Netherlans	Effect concentration	bulk	31	1.1	116	36	0.56	35	132	145
ERM	[31]	USA	50th percentile EDS	bulk	70	9.6	370	270	0.7	51.6	218	410
ERM	This study	BC	50th percentile EDS	<63 m	**16.0**	**1.0**	**58.6**	**82.5**	**0.7**	**36.0**	**116.0**	**380.7**
PEL	[32]	USA	$\sqrt{\text{50th percentile EDS * 85th percentile NEDS}}$	bulk	41.6	4.2	160	108	0.7	42.8	112	271
PEL	This study	BC	$\sqrt{\text{50th percentile EDS * 85th percentile NEDS}}$	<63 m	**26.3**	**1.8**	**66.5**	**95.5**	**1.1**	**38.7**	**143.7**	**441.7**
MPC	[33]	The Netherlans	Effect concentration	bulk	190	30	1720	73	26	44	4800	620
PNEC	[13]	Norway	Chronic test	bulk	52	2.6	560	51	0,63	46	83	360
CSI	[58]	USA	Chemical Score Index	bulk	-	0.3	-	100	0.3	-	25	500

Note: BC, Basque Country; CSI, chemical score index; EDS, effect dataset; ERL, effect range–low; ERM, effect range–median; MPC, maximum permissible concentration; NC, negligible concentrations; NEDS, no effect dataset; PEL, probable effect level; PNEC, predicted no effect concentrations; TEL, threshold effect level.

Table 3. Sediment Quality Guidelines* for metals ($\mu g \cdot g^{-1}$) normalised by the fine sediment content ($< 63\ \mu m$), calculated in this study.

	Calculation method	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn
ERL*	percentile 10 EDS	0.42	0.02	1.85	1.45	0.01	0.83	2.03	7.5
ERM*	percentile 50 EDS	8.77	0.52	31.74	43	0.28	21	53	190
TEL*	$\sqrt{\text{percentile 15 EDS} * \text{percentile 50 NEDS}}$	1.25	0.07	5.45	3.4	0.04	2.25	5.42	19
PEL*	$\sqrt{\text{percentile 50 EDS} * \text{percentile 85 NEDS}}$	13.5	1	39	55	0.53	23	78	249

Note: EDS, effect dataset; ERL, effect range–low; ERM, effect range–median; NEDS, no effect dataset; PEL, probable effect level; TEL, threshold effect level.

minimum effect range (<ERL); they were close to 70% in the probable effect range (>ERM), for all of the metals (Figures 2 and 3). Moreover, as shown by the χ^2 tests, the incidence of the effects increased significantly with increasing SQG ranges, in almost all cases, except for Cd. By

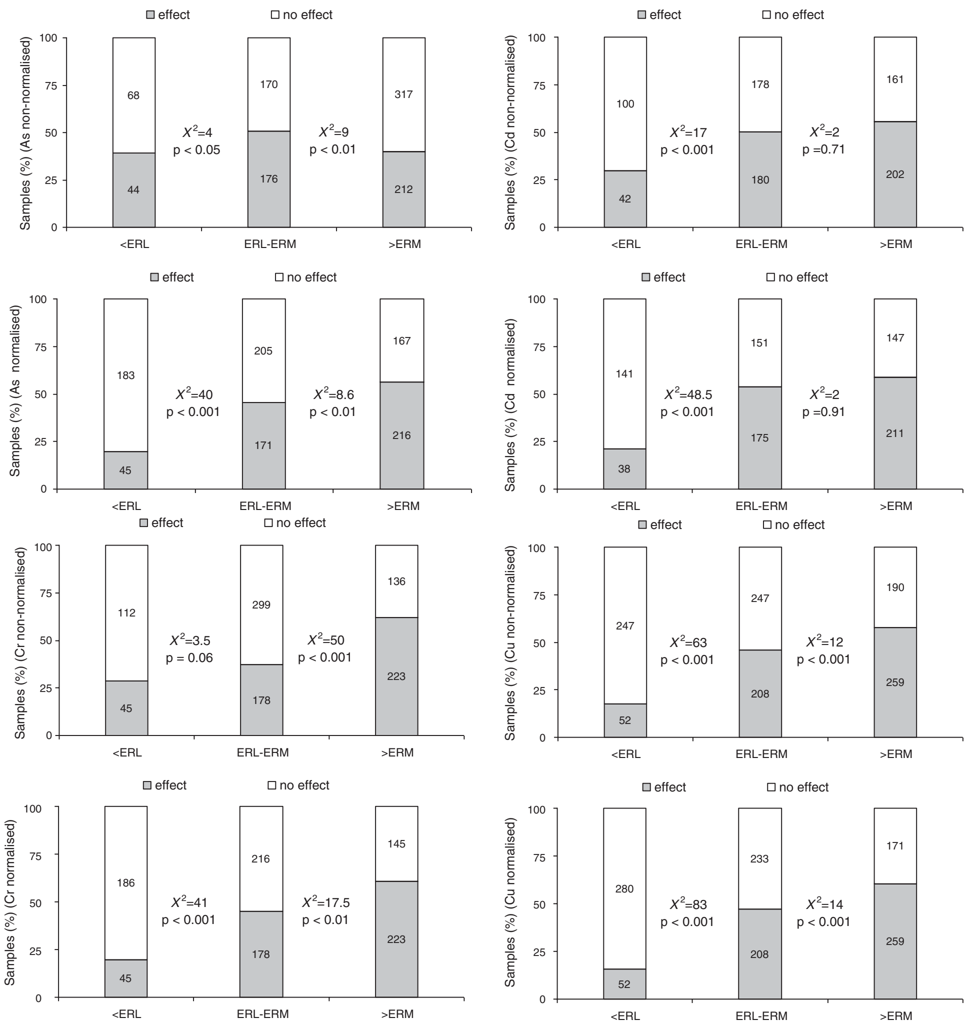

Figure 2. Percentage incidence of effects within concentration ranges (defined by ERL and ERM guidelines for As, Cd, Cr and Cu), normalised and non-normalised by the fine-sediment content. χ^2 values (and associated *p*-values) were calculated to test whether the ratio of no-effect data/effect data (according to the terminology of Long et al. [31]) differed significantly between adjacent ranges. Numbers in bars indicate absolute values.

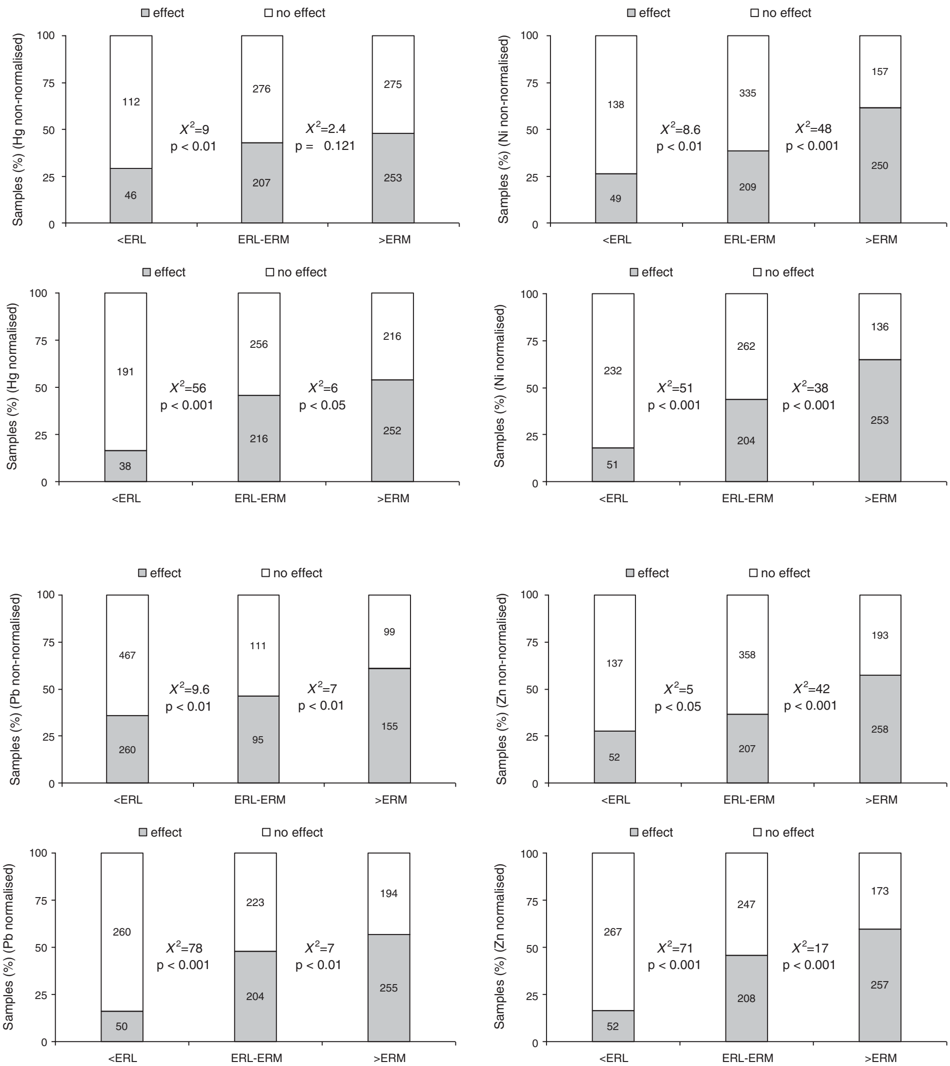

Figure 3. Percentage incidence of effects within concentration ranges (defined by ERL and ERM guidelines for Hg, Ni, Pb and Zn), normalised and non-normalised by the fine-sediment content. χ^2 values (and associated *p*-values) were calculated to test whether the ratio of no-effect data/effect data (according to the terminology of Long et al. [31]) differed significantly between adjacent ranges. Numbers in bars indicate absolute values.

contrast, the incidence of the effects associated with ERL and ERM values non-normalised by the fine-sediment content, were not so consistent; it was >25% in the minimum effect range for all cases, except for Cu. Furthermore, the incidence of the effects did not increase significantly with increasing SQG ranges for Cd, Cr and Hg (Figures 2 and 3).

In the same way, the incidence of the effects associated with the TEL* and PEL* values normalised by the fine-sediment content was <25% in the minimum effect range; it was close to 70% in the probable-effects range, for all of the metals (Figures 4 and 5). Furthermore, as shown by the χ^2 tests, the incidence of the effects increased significantly with increasing SQG ranges for almost all metals, except for As, Cd, Hg and Pb. By contrast, for the non-normalised values, the incidence of the effects was >25% in the minimum effect range for all cases, except for Cu. Furthermore, the incidence of the effects increased significantly with increasing SQG ranges, except for As, Cd and Hg (Figures 4 and 5).

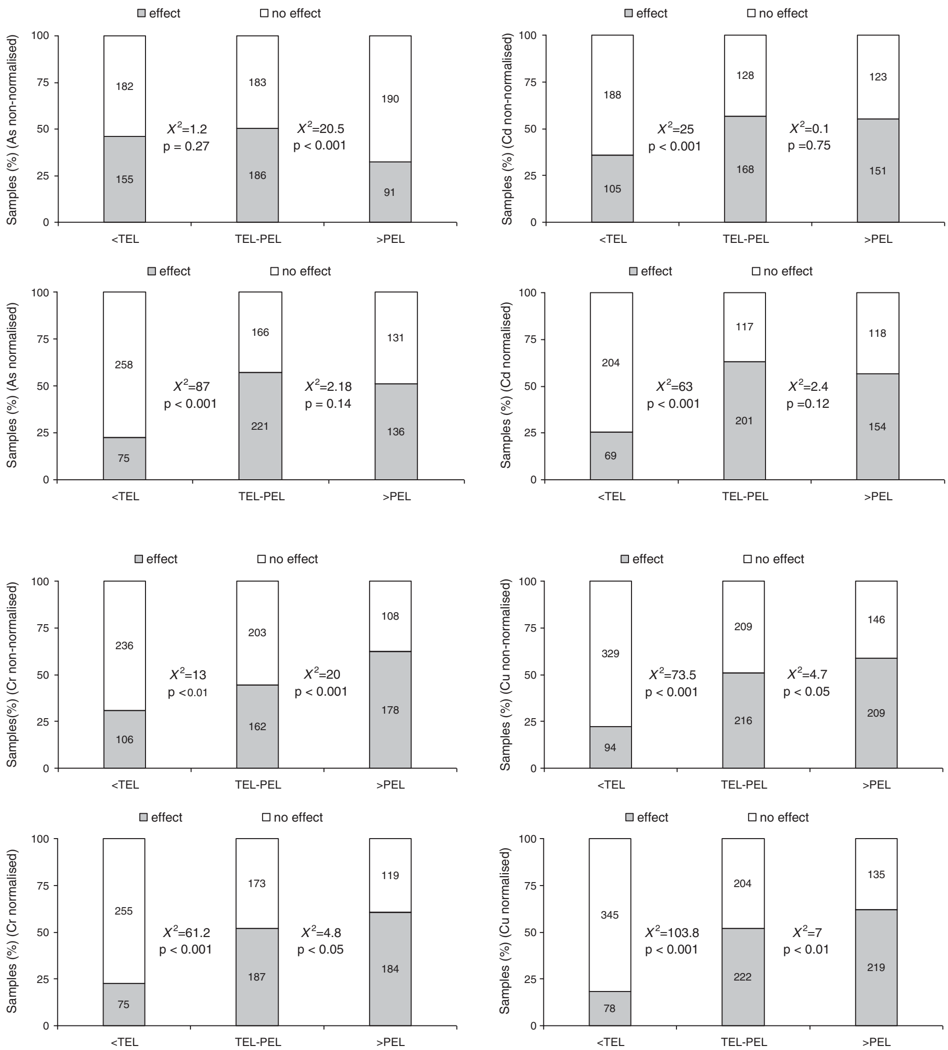

Figure 4. Percentage incidence of effects within concentration ranges (defined by PEL and TEL guidelines for As, Cd, Cr and Cu), normalised and non-normalised by the fine-sediment content. χ^2 values (and associated *p*-values) were calculated to test whether the ratio of no-effect data/effect data (according to the terminology of Long et al. [31]) differed significantly between adjacent ranges. Numbers in bars indicate absolute values.

4. Discussion

4.1. *SQG empirical approaches*

Some authors [20,21] have recognised limitations in the empirical SQG use: (1) the causality is not determined; (2) they may or may not measure the effects of contaminant mixtures; (3) the bioavailability is not resolved; and (4) false positives or Type I errors (i.e. a non-toxic sample classified incorrectly as toxic) and false negatives or Type II errors (i.e. a toxic sample classified incorrectly as non-toxic) would occur. Despite the uncertainties involved in the SQG approach, chemically based empirical numeric SQG derived using different methods and assumptions appear to converge, suggesting important underlying relationships relative to causality [26]. In this sense, some authors [10,64] have recommended the use of empirical SQG due to the

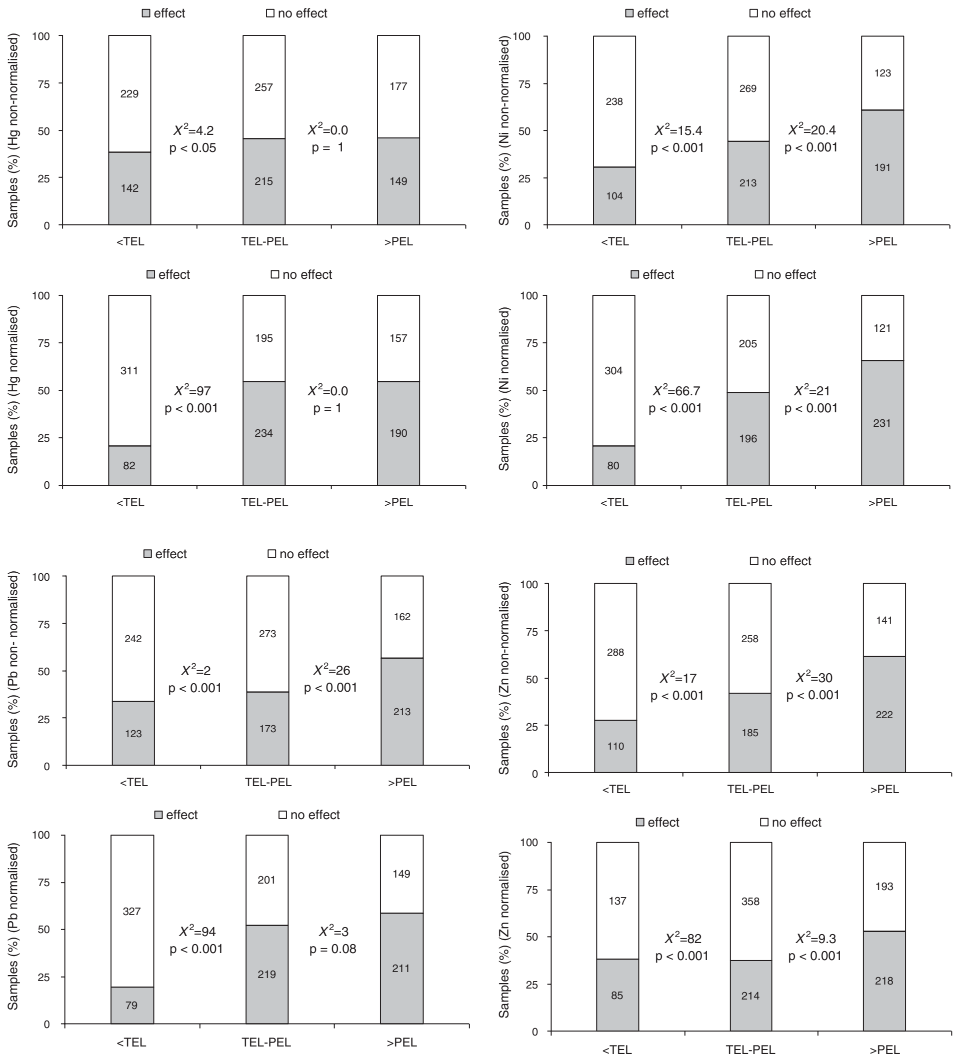

Figure 5. Percentage incidence of effects within concentration ranges (defined by PEL and TEL guidelines for Hg, Ni, Pb and Zn), normalised and non-normalised by the fine-sediment content. χ^2 values (and associated *p*-values) were calculated to test whether the ratio of no-effect data/effect data (according to the terminology of Long et al. [31]) differed significantly between adjacent ranges. Numbers in bars indicate absolute values.

following advantages: (1) they are calculated from a large database of correlative effects by using field-collected sediments (i.e. that available for the Basque Country); (2) they can be applied in a regulatory framework (i.e. the WFD or others), serving as scientific advisory values; (3) they show a wide geographical application (in our case, probably for the same ecoregion, within the Bay of Biscay); and (4) they constitute a fast and reliable way to assess overall environment quality and, if necessary, to set remediation goals, as in this contribution. Moreover, according to Hagopian-Schlekat et al. [65], whole-sediment test systems may provide more environmentally realistic assessment of the effects and biological controls on mixed-metals bioavailability of the meiobenthos.

Hence, two different empirical approaches have been used in the regional SQG calculation for the sediments of the Basque Country. Both of the guideline approaches normalised by the fine-sediment content showed good accuracy due to the significant increasing effect values with increasing SQG ranges for all metals, except for Cd in the ERL*/ERM* values, and for As, Cd, Hg and Pb in the TEL*/PEL* values. In this sense, the accuracy of the guidelines for those metals appeared to be low. Similar inconsistencies have been found in other SQG determinations [31] for Ni. Some of these inconsistences could be related to variables such as pH, hardness, organic matter [66] and to the ratio of the sum of the simultaneously extracted metals (SEM) to acid-volatile sulfide (AVS) concentrations, which greatly affect metal bioavailability and toxicity to aquatic organisms [67]. In Borja et al. [68] the SEM results showed that $> 70\%$ of Ni and 58% of Pb were found as a residual form or in a stable association with the mineral matrix of the sediment; this is indicative of a mineral/detrital origin for this metal [68]. Thus it may be observed despite the high concentration in which these metals were present in the sediments they do not represent any biological risk [55]. In this sense, it is difficult to develop an understanding of the controlling geochemical process which is related to the circulation of metals in the ecosystem as well as their bioavailability and toxicity [55].

Nevertheless, in the ERL/ERM approach, only the effect data matrix is used to estimate ERL and ERM [31]. By contrast, Buchman et al. [32] proposed using not only the effect, but also the non-effect data for the calculation of the SQG. By including the effect and non-effect data in the SQG calculation, the effect of false negatives and positives on the database is minimised, improving the accuracy of the SQG [21].

In the same way, Hübner et al. [10], following an exhaustive evaluation and comparative study of the main international SQG, concluded that the TEL/PEL approach offers a solid and proven scientific basis and extensive comparability, together with a high predictive capacity. By contrast, Fairey et al. [34] have demonstrated that the mean SQG quotient, based upon a combination of the ERM and PEL of different chemicals, offered a high predictive method of acute toxicity to amphipods.

4.2. *SQG normalised versus non-normalised by fine-sediment content*

Based upon the χ^2 test and the incidence of effect results [30,31], those SQG* calculated from normalised metal concentrations produced the best accuracy, particularly in the ERL/ERM approach. This study suggests that a two-tiered normalisation approach in the sediment guidelines calculation, including metal concentration analysis in the fine-sediment content and the adjustment by the fine-sediment content, should be preferred. Vandecasteele et al. [54] agreed that normalisation of heavy metal concentrations towards the fine-sediment content is an appropriate technique for testing the robustness of the data. In the same way, Roach [69] demonstrated that As, Cu, Cr, Ni, Pb and Zn showed strong linear relationships with grain size. This investigation found that the grain-size normalisation approach appeared to be a useful technique for detecting the presence of elevated concentrations of metals, in sediments with a range of grain sizes.

In this sense, by determining the SQG* normalised on the basis of the fine-sediment content, guidelines would be valid for almost all types of sediment within the Basque Country. The following formula is proposed, taking into the account the fine-sediment content:

$$\mathrm{SQG}_{\mathrm{metal}} = \mathrm{SQG}^{*}_{\mathrm{metal}}/\mathrm{FC}\ 10^{-2}$$

where FC (%) is the fine-sediment content and SQG* is the SQG ($\mu\,g \cdot g^{-1}$) normalised by fine-sediment content (see Table 3), in $\mu g \cdot g^{-1}$. These SQG* values were derived on the basis of sediments with 0.1–99.2% fine-sediment content.

Table 4. Background concentrations (BCK) ($\mu g \cdot g^{-1}$) of metals in estuarine and coastal surface sediments (< 63 μm) within the Basque Country, as described by Rodríguez et al. [11]; PEL/BCK ratio and ERM/BCK ratio values.

	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn
BCK	12	0.24	26	33	0.13	29	31	174
PEL/BCK ratio	2	8	3	3	9	1	5	3
ERM/BCK ratio	1	4	2	3	5	1	4	2

Note: ERM, effect range–median; PEL, probable effect level.

4.3. *Comparison with background values and other guidelines*

The two-tiered normalisation by fine-sediment content does not allow comparisons of the guidelines calculated in this study with background values derived by Rodríguez et al. [11] due to differences in the scale (Table 4). In this sense, only comparisons of non-normalised SQG values were performed. As commented in the Introduction, background levels are used in assessing the WFD physicochemical status (representing the boundary between 'high' and 'good'). Hence, it may be expected that background shows values lower than the SQG, with no affection to the biota.

On the one hand, in our investigation, the TEL values for all metals, except for Ni, were higher than the threshold of background values. However, the ERL values were lower than the background for all metals. Clearly, most of these apparent incoherencies arise from differences in the approaches and methodologies used in the background and SQG derivation. At this point, the ERL values could not be considered as a threshold below which sediment toxicity is impossible and above which it is likely [70]. On the other hand, the ERM and PEL values for all metals were higher than the background values.

Bjørgesæter and Gray [24] suggested that setting a SQG at four times the background concentrations will provide sufficient protection from metal contamination for the fauna. However, in this study, the ratios between the ERM and the background values (Table 4) ranged from 1 to 3; only for three metals was the ratio > 3, namely Cd and Pb (ratio of 4) and Hg (ratio of 5). The ratios between PEL and the mean background values ranged from 1 to 3, and for three metals the ratio was > 3, namely Cd (ratio of 8), Hg (ratio of 9) and Pb (ratio of 5). In this sense, the folder of four times the background concentrations proposed by Bjørgesæter and Gray [24] would be insufficient to ensure the protection for fauna. From the above discussion, it becomes clear that, in some areas, such as the Basque Country, and depending upon the metal, effects on biota will be identified even at metal concentrations only twice over the background concentrations.

Furthermore, taking into account that the incidence of effect in the ranges above the ERM and PEL was close to 70%, the ERM and PEL values could be considered as a threshold over which effects are frequent. Nevertheless, the observed toxicity could very well be due to co-occurring contaminants, and therefore toxicity identification evaluation (TIE) [71] or spiked-sediment studies [36] can be performed at a regional scale. In other words, ERM and PEL cannot be considered as definitive indicators of toxicity. This conclusion is in agreement with that of Hübner et al. [10].

Moreover, in order to compare the Basque Country SQG values with those developed on the basis of different methodologies and for different areas, eight international SQG values are listed in Table 2. Despite the differences in the analytical and mathematical approaches, the SQG values calculated in this study are within the range of SQG values developed for different areas. In particular, the SQG values developed for Norway [13] (except for As, Cd and Cr) and the USA [72] (except for Pb) are very close to those proposed for the Basque Country.

Furthermore, according to Paya-Pérez et al. [36], results of long-term series from toxicity tests with sediment organisms, combined with assessment factors, are preferred for deriving sediment quality standards due to the generally long-term exposure of benthic organisms to sediment-bound substances. In this sense, comparing the values calculated here with available ecotoxicological data (NOEC/EC_{10}) on spiked-sediment test, the Basque Country SQG values are within the range of those calculated for marine amphipods by Ringenary et al. [73] for Pb, Ciarelli et al. [74] for Cd, Roman et al. [75] for Cu and Hagopian- Schlekat et al. [65] for Ni.

4.4. *Using regional SQG values in the implementation of the WFD*

According to the WFD, 'good' physicochemical status can be considered when pollutant concentrations are between the background level and the EQS [11]. Directive 2008/105/EC [4] establishes the EQS for priority substances (such as Cd, Hg, Ni and Pb) in waters [14,76], but not for sediments. This Directive allows Member States to establish EQS for sediment at a national level and applying those EQS instead of the EQS for water set out in the Directive. Such EQS should be established through a transparent and scientific procedure, to ensure a level of protection to the environment equivalent to the EQS for water, set up at community level, as proposed in this study and in Norway [13]. Nowadays, international SQG for metals, such as those proposed by Long et al. [31] and those described by Buchman et al. [32], are used frequently as the EQS [14]. These SQG values were used by Borja et al. [77] in investigative monitoring for the ecological status assessment within the WFD. In this study, sediment analysis, chemical metal analysis and ecotoxicological approaches were integrated to assess the potential risk associated with a particular pressure (i.e. blast furnace slag disposal). These latter authors have suggested that this integrative approach provided an example of a direct measure of the effects on the biological elements, to be studied under the WFD. Another example of the use of the SQG values within an integrative approach was proposed by Borja et al. [76] within the WFD. Therefore, the determination of EQS values in sediments, for priority substances such as metals, is highly relevant in the implementation of the WFD methodologies [13]; this addresses the physicochemical and ecological status of marine and estuarine water bodies [12].

Moreover, a better understanding of the relationships among SQG, ambient toxicity tests and benthic community metrics is needed. However, this relationship is not simple because metrics generally assess the responses at the community level of biological organisation, whereas sediment guidelines and ambient toxicity tests generally assess or are based on the responses at the organism level [59]. It is important to highlight that the WFD lies with the community structure of different biological quality elements [3]; hence, the protection at the community level should be ensured.

Hence, this study contributes to the assessment of the regional SQG values following the recommendation of Crane [5] in assessing chemical and physicochemical status, to be used within the WFD. Following the WFD, both acute and chronic data should be obtained where possible for the taxa relevant for the water body type concerned. In this study, the use of four different organisational biological levels (bacteria, amphipods, sea urchins and benthic community) ensures an improvement and approximation to EQS level, being the results very close to those obtained by other countries (e.g. Norway) using the WFD recommendations in setting such EQS [13].

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References

[1] Directive 2000/60/EC of the European Parliament and the Council of 23.10.2000, *A framework for community action in the field of water policy*, Official Journal of the European Communities 22.12.2000, pp. 72.

[2] A. Borja, J. Franco, V. Valencia, J. Bald, I. Muxika, M.J. Belzunce, and O. Solaun, *Implementation of the European Water Framework Directive from the Basque Country (northern Spain): A methodological approach*, Mar. Pollut. Bull. 48 (2004), pp. 209–218.

[3] A. Borja, *The European Water Framework Directive: A challenge for nearshore coastal and continental shelf research*, Cont. Shelf Res. 25 (2005), pp. 1768–1783.

[4] Directive 2008/105/EC of the European Parliament and the Council of 16 December 2008 on Environmental Quality Standards in the Field of Water Policy, Amending and Subsequently Repealing Directives 82/7172/EEC, 83/513/EEC, 84/156/EEC, 84/491/EEC and 86/280/EEC and Amending Directive 2000/60/EC.

[5] M. Crane, *Proposed development of sediment quality guidelines under the European Water Framework Directive: A critique*, Toxicol. Lett. 142 (2003), pp. 195–206.

[6] A. Borja, V. Valencia, J. Franco, I. Muxika, J. Bald, M.J. Belzunce, and O. Solaun, *The Water Framework Directive: Water alone, or in association with sediment and biota, in determining quality standards?* Mar. Pollut. Bull. 49 (2004), pp. 8–11.

[7] A. Borja and H. Heinrich, *Implementing the European Water Framework Directive: The debate continues*, Mar. Pollut. Bull. 50 (2005), pp. 486–488.

[8] S. Dueri, J. Castro-Jiménez, and J.M.Z Comenges, *On the use of the partitioning approach to derive Environmental Quality Standards (EQS) for persistent organic pollutants (POPs) in sediments: A review of existing data*, Sci. Total Environ. 403 (2008), pp. 23–33.

[9] C. Maggi, F. Onorati, C.V. Lamberti, and A.M. Cicero, *The hazardous priority substances in Italy: National rules and environmental quality standard in marine environment*, Environ. Impact. Assess. Rev. 28 (2008), pp. 1–6.

[10] R. Hübner, K.B. Astin, and R.J.H. Herbert, *Comparison of sediment quality guidelines (SQGs) for the assessment of metal contamination in marine and estuarine environments*, J. Environ. Monit. 11 (2009), pp. 713–722.

[11] J.G. Rodríguez, I. Tueros, A. Borja, M.J. Belzunce, J. Franco, O. Solaun, V. Valencia, and A. Zuazo, *Maximum likelihood mixture estimation to determine metal background values in estuarine and coastal sediments within the European Water Framework Directive*, Sci. Total. Environ. 370 (2006), pp. 278–293.

[12] I. Tueros, J.G. Rodríguez, A. Borja, O. Solaun, V. Valencia, and E. Millán, *Dissolved metal background levels in marine waters, for the assessment of the physico-chemical status, within the European Water Framework Directive*, Sci. Total. Environ. 407 (2008), pp. 40–52.

[13] T. Bakke, T. Källqvist, A. Ruus, G. Breedveld, and K. Hylland, *Development of sediment quality criteria in Norway*, J. Soil. Sediment 10 (2010), pp. 172–178.

[14] I. Tueros, A. Borja, J. Larreta, J.G. Rodríguez, V. Valencia, and E. Millán, *Integrating long-term water and sediment pollution data, in assessing chemical status within the European Water Framework Directive*, Mar. Pollut. Bull. 58 (2009), pp. 1389–1400.

[15] A. Borja, S.B. Bricker, D.M. Dauer, N.T. Demetriades, J.G. Ferreira, A.T. Forbes, P. Hutchings, X. Jia, R. Kenchington, J.C. Marques, and C. Zhu, *Overview of integrative tools and methods in assessing ecological integrity in estuarine and coastal systems worldwide*, Mar. Pollut. Bull. 56 (2008), pp. 1519–1537.

[16] R.B. Choueri, A. Cesar, D.M.S. Abessa, R.D. Torres, I. Riba, C.D.S Pereira, M.R.L. Nascimentog, A.A. Mozetod, and T.A. DelValls, *Development of site specific quality guidelines for North and South Atlantic littoral zones: Comparison against national sediment quality benchmarks*, J. Hazard. Mater. 170 (2009), pp. 320–331.

[17] J. Bald, A. Borja, I. Muxika, J. Franco, and V. Valencia, *Assessing reference conditions and physico-chemical status according to the European Water Framework Directive: A case-study from the Basque Country (Northern Spain)*, Mar. Pollut. Bull. 50 (2005), pp. 1508–1522.

[18] S. Loureiro, A. Newton, and J. Icely, *Boundary conditions for the European Water Framework Directive in the Ria Formosa lagoon, Portugal (physico-chemical and phytoplankton quality elements)*, Estuar. Coast. Shelf Sci. 67 (2006), pp. 382–398.

[19] Anonymous, *Guidance on the application of the ecosystem approach to management of human activities in the European marine environment*, European Marine Strategy – Second Stakeholder Conference, Rotterdam, 10–12 November European Commission, Directorate General, Environment, Directorate D-Water and Environmental Programmes, 2004.

[20] G.A. Burton, *Sediment quality criteria in use around the world*, Limnology 3 (2002), pp. 65–75.

[21] D.J. McCauley, G.M. DeGraeve, and T.K. Linton, *Sediment quality guidelines and assessment: Overview and research needs*, Environ. Sci. 3 (2000), pp. 133–144.

[22] R.J. Wenning and G.C. Ingersoll, *Summary of the Setac Pellston Workshop on the use of sediment quality guidelines and related tools for the assessment of contaminated sediments*, Society of Environmental Toxicology and Chemistry (SETAC), Pensacola, FL, 2002.

[23] M.D. Scrimshaw, T.A. DelValls, J. Blasco, and P.M. Chapman, *Sediment quality guidelines and weight of evidence assessments*, Sustain. Manag. Sediment. Resour. 1 (2007), pp. 295–309.

[24] A. Bjørgesæter and J.S. Gray, *Setting sediment quality guidelines: A simple yet effective method*, Mar. Pollut. Bull. 57 (2008), pp. 221–235.

[25] R. Cormack, *Sediment Quality Guideline Options for 409 the State of Alaska*, Prepared for the Alaska Department of Environmental Conservation Division of Spill Prevention and Response Contaminated Sites Remediation Program, Anchorage, 2001.
[26] G.E. Batley, R.G. Stahl, M.P. Babut, T.L. Bott, J.R. Clark, and L.J. Field, *Scientific underpinnings of sediment quality guidelines*, in *Use of Sediment Quality Guidelines and Related Tools for the Assessment of Contaminated Sediments*, R.J. Wenning, G.E. Bartley, C.G Ingersoll, and D.W. Moore, eds., Society of Environmental Toxicology and Chemistry, Pensacola, FL, 2005, pp. 39–119.
[27] D.M. Di Toro, C.S. Zarba, D.J. Hansen, W.J. Berry, R.C. Swartz, C.E. Cowan, C.E. Pavlou, H.E. Allen, N.A. Thomas, and P.R. Paquin, *Technical basis for establishing sediment quality criteria for nonionic organic chemicals by using equilibrium partitioning*, Environ. Toxicol. Chem. 10 (1991), pp. 1541–1583.
[28] C. Yung-Zeng, Y. Hao, Z. Zhen-Ke, Q. Ming- Zhou, J. Feng, and L. Jun-Jie, *Application of equilibrium partitioning approach to the derivation of sediment quality guidelines for metals in Dianchi Lake*, Pedosphere 17 (2007), pp. 284–329.
[29] ECHA, *The Guidance on Information Requirements and Chemical Safety Assessment*, Guidance for the implementation of REACH, Helsinki, 2008.
[30] D.D. McDonald, C.G. Ingersoll, and T.A. Berger, *Development and evaluation of consensus-based sediment quality guidelines for freshwater ecosystems*, Arch. Environ. Toxicol. 39 (2000), pp. 20–31.
[31] E.R. Long, D.D. MacDonald, S.L. Smith, and F.D. Calder, *Incidence of adverse biological effects within ranges of chemical concentrations in marine and estuarine sediments*, Environ. Manage. 19 (1995), pp. 81–97.
[32] M.F. Buchman, *Screening Quick Reference Tables (SQuiRTs)*, National Oceanic and Atmospheric Administration, Office of Response and Restoration, Seattle Report 08-1, 1999.
[33] T. Crommentuijn, D. Sijm, J. Bruijn, M. Hoop, K. Leeuwen, and E. Plassche, *Maximum permissible and negligible concentrations for metals and metalloids in the Netherlands, taking into account background concentrations*, J. Environ. Manage. 60 (2000), pp. 121–143.
[34] R. Fairey, E.R. Long, C.A. Roberts, B.S. Anderson, B.M. Phillips, J.W. Hunt, H.R. Puckett, and C.J. Wilson, *An evaluation of methods for calculating mean sediment quality guideline quotients as indicators of contamination and acute toxicity to amphipods by chemical mixtures*, Environ. Toxicol. Chem. 20 (2001), pp. 2276–2286.
[35] K.M.Y. Leung, A. Bjorgesaeter, J.S. Gray, W.K. Li, G.C.S. Lui, Y. Wang, and P.K.S. Lam, *Deriving sediment quality guidelines from field-based species sensitivity distributions*, Environ. Sci. Technol. 39 (2005), pp. 5148–5156.
[36] A. Paya-Pérez, P. Whitehouse, H. Wilkinson, J. Rodriguez, and M. David, *Technical guidance for deriving environmental quality standards. Draft revision 5.0. Common Implementation Strategy for Water Framework Directive* (2000/60/EC), 2010.
[37] P.M. Chapman and G.S. Mann, *Sediment quality values (SQVs) and ecological risk assessment (ERA)*, Mar. Pollut. Bull. 38 (1999), pp. 339–344.
[38] ISO 5667-15, *Guidance on preservation and handling of sludge and sediment sample*. Available at www.iso.org.
[39] ICES, *Chemical Measurements in the Baltic Sea: Guidelines on Quality Assurance*, E. Lysiak-Pastuszak and M. Krysell, eds., ICES Techniques in Marine Environmental Sciences, 35 (2004). Available at http://www.ices.dk.
[40] OSPAR, *JAMP Guidelines for Monitoring Contaminants in Sediments*, Agreement 2002-16 (2002). Available at http://www.ospar.org.
[41] U. Förstner and W. Salomons, *Trace metal analysis on polluted sediments*, Environ. Technol. Lett. 1 (1980), pp. 494–505.
[42] S.N. Luoma, *Processes affecting metal concentrations in estuarine and coastal marine sediments*, in *Heavy Metals in the Marine Environment*, R.W. Furness and P.S. Rainbow, eds., CRC Press, Boca Raton, FL, 1990, pp. 51–66.
[43] D.H. Loring and R.T.T. Rantala, *Manual for the geochemical analysis of marine sediments and suspended particulate matter*. Earth-Sci. Rev. 32 (1992), pp. 235–283.
[44] M. Kersten and F. Smedes, *Normalization procedures for sediment contaminants in spatial and temporal trend monitoring*, J. Environ. Monit. 4 (2002), pp. 109–115.
[45] B. Vandecasteele, P. Quataert, B. De Vos, and F.M.G. Tack, *Assessment of the pollution status of alluvial plains: A case study for the dredged sediment-derived soils along the Leie River*, Arch. Environ. Contamin. Toxicol. 47 (2004), pp. 14–22.
[46] VLAREBO, *Decree on Soil Remediation and Soil Protection, ratified by the Flemish Government*. Vlaams reglement betreffende de bodemsanering, 2006.
[47] VROM, *From Funnel to Sieve, Bever Soil Remediation, From Policy to Practice*, Netherlands Ministry of Housing, Spatial Planning and the Environment, 2007. Available at http://international.vrom.nl/Docs/internationaal/VTNZengels.pdf.
[48] ASTM, *Standard Guide for Conducting Static Acute Toxicity Tests with Echinoid Embryos*, American Society for Testing and Materials, E1563-95, Philadelphia, PA, 1995.
[49] USEPA, *Method for Assessing the Chronic Toxicity of Marine and Estuarine Sediment-Associated Contaminants with the Amphipod* Leptocheirus plumulosus, EPA 600/R-01/020, 2001.
[50] OSPAR, *Protocols on Methods for the Testing of Chemicals Used in the Offshore Oil Industry*, OSPAR Commission, 2005.
[51] Y.H. Cheung, A. Neller, K.H. Chu, N.F.Y. Tam, C.K. Wong, Y.S. Wong, and M.H. Wong, *Assessment of sediment toxicity using different trophic organisms*, Arch. Environ. Contam. Toxicol. 32 (1997), pp. 260–267.
[52] M.C. Casado-Martínez, T. Campisi, A. Díaz, R. Lo Re, R. Obispo, J.F. Postma, I. Riba, A.C. Sneekes, J.L. Buceta, and T.A. DelValls, *Interlaboratory assessment of marine bioassays to evaluate the environmental quality of coastal*

sediments in Spain. II. Bioluminescence inhibition test for rapid sediment toxicity assessment, Ciencias Marinas 32 (2006), pp. 1–10.

[53] N. Fernández and R. Beiras, *Combined toxicity of dissolved mercury with copper, lead and cadmium on embryogenesis and early larval growth of the* Paracentrotus lividus *sea-urchin*, Ecotoxicology 10 (2001), pp. 263–271.

[54] Environment Canada, *Biological test method*, *Acute test for sediment toxicity using marine or estuarine amphipods*, Report EPS 1/RM/26, 1992.

[55] M.J. Belzunce, O. Solaun, J.A.G. Oreja, E. Millán, and V. Pérez, *Contaminants in Sediments*. *Oceanography and Marine Environment of the Basque Country*, 70, Elsevier Oceanography Series, Amsterdam, 2004, pp. 283–315.

[56] A. Borja, J. Franco, and V. Pérez, *A marine biotic index to establish the ecological quality of soft bottom benthos within European estuarine and coastal environments*, Mar. Pollut. Bull. 40 (2000), pp. 1100–1114.

[57] A. Borja and I. Muxika, *Guidelines for the use of AMBI (AZTI's marine biotic index) in the assessment of the benthic ecological quality*, Mar. Pollut. Bull. 50 (2005), pp. 787–789.

[58] K.J. Ritter, S.M. Bay, R.W. Smith, D.E. Vidal-Dorsch, and L.J. Field, *Development and evaluation of sediment quality guidelines based on benthic macrofauna responses*, 2010. DOI:10.1002/ieam.191.

[59] M.B. Griffith and M. Kravitz, *Relationships among exceedences of sediment guidelines, the results of ambient sediment toxicity tests, and community metrics in estuarine systems*, Estuar. Coast. 31 (2008), pp. 101–114.

[60] T.A. Del Valls, M.C. Casado-Martínez, I. Riba, M.L. Martín-Díaz, J.M. Forja, and E. García-Luque, *Investigación conjunta sobre la viabilidad de utilizar ensayos ecotoxicológicos para la evaluación de la calidad ambiental del material de dragado*, Technical Report for CEDEX Puerto Real, Cádiz, 2003.

[61] M.C. Casado-Martínez, N. Fernández, J. Lloret, A. Marín, C. Martínez-Gómez, I. Riba, and T.A. Del Valls, *Interlaboratory assessment of marine bioassays to evaluate the environmental quality of coastal sediments in Spaìn. III. Bioassay using embryos of the sea urchin* Paracentrotus lividus, Ciencias Marinas 32 (2006), pp. 139–147.

[62] A. Borja, J.G. Rodríguez, K. Black, A. Bodoy, C. Emblow, T.F. Fernandes, J. Forte, I. Karakassis, I. Muxika, T.D. Nickell, N. Papageorgiou, F. Pranovi, K. Sevastou, P. Tomassetti, and D. Angel, *Assessing the suitability of a range of benthic indices in the evaluation of environmental impact of fin and shellfish aquaculture located in sites across Europe*, Aquaculture 293 (2009), pp. 231–240.

[63] A.B. Josefson, J.L.S. Hansen, G.Asmund, and P. Johansen, *Threshold response of benthic macrofauna integrity to metal contamination in West Greenland*, Mar. Pollut. Bull. 56 (2008), pp. 1265–1274.

[64] C.G. Ingersoll, P.S. Haverland, E.L. Brunson, T.S. Canfield, F.S. Dwyer, C.E. Henke, and N.E. Kemble, *Calculation of sediment effect concentrations*, J. Great Lakes Res. 22 (1996), pp. 602–623.

[65] T. Hagopian-Schlekat, G.T. Chandler, and T.J. Shaw, *Acute toxicity of five sediment-associated metals, individually and in a mixture, to the estuarine meiobenthic harpacticoid copepod* Amphiascus tenuiremis, Mar. Environ. Res. 51 (2001), pp. 247–264

[66] G.M. Ogendi, W.G. Brumbaugh, R.E. Hannigan, and J.L. Farris, *Effects of acid-volatile sulfide on metal bioavailability and toxicity to midge (*Chironomus tentans*) larvae in black shale sediments*, Environ. Toxicol. Chem. 26 (2007), pp. 325–334.

[67] J-S. Lee and J-H. Lee, *Influence of acid volatile sulfides and simultaneously extracted metals on the bioavailability and toxicity of a mixture of sediment-associated Cd, Ni, and Zn to polychaetes* Neanthes arenaceodentata, Sci. Total Environ. 338 (2005), pp. 229–241.

[68] A. Borja, J. Franco, V. Valencia, A. Uriarte, and R. Castro, *Red de vigilancia y control de la calidad de las aguas litorales del País Vasco: otoño 1999-verano 2000*. Dpto. de Ordenación del Territorio, Vivienda y Medio Ambiente, Gobierno Vasco. UTE AZTI-LABEIN, unpublished report, 2001.

[69] A.C. Roach, *Assessment of metals in sediments from Lake Macquarie, New South Wales, Australia, using normalisation models and sediment quality guidelines*. Mar. Environ. Res. 59 (2005), pp. 453–472.

[70] T.P. O'Connor, *The sediment quality guideline, ERL, is not a chemical concentration at the threshold of sediment toxicity*, Mar. Pollut. Bull. 49 (2004), pp. 383–385.

[71] A. Macken, M. Giltrap, B. Foley, E. McGovern, B. McHugh, and M. Davoren, *An integrated approach to the toxicity assessment of Irish marine sediments: Application of porewater Toxicity Identification Evaluation (TIE) to Irish marine sediments*, Environ. Int. 35 (2009), pp. 98–106.

[72] K.J. Ritter, S.M. Bay, R.W. Smith, D.E. Vidal-Dorsch, and L.J. Field, *Development and evaluation of sediment quality guidelines based on benthic macrofauna responses*, Integr. Environ. Assess. Manag. DOI: 10.1002/ieam.191.

[73] M.J. Ringenary, A.H. Molof, J.T. Tanacredi, M.P. Schreibman, and K. Kostarelos, *Long-term sediment bioassay of lead toxicity in two generations of the marine amphipod* Elasmopus laevis*, S.I. Smith, 1873*, Environ. Tox. Chem. 26 (2007), pp. 1700–1710.

[74] S. Ciarelli, W.A.P.M.A. Voncka, and N.M. van Straaled, *Reproducibility of spiked-sediment bioassays using the marine benthic amphipod,* Corophium volutator, Mar. Environ. Research 43 (1997), pp. 329–343.

[75] Y.E. Roman, K.A.C. Schamphelaere, L.T.H. Nguyen, and C.R. Janssen, *Chronic toxicity of copper to five benthic invertebrates in laboratory-formulated sediment: Sensitivity comparison and preliminary risk assessment*, Sci. Total Environ. 387 (2007), pp. 128–140.

[76] A. Borja, J. Bald, J. Franco, J. Larreta, J. Muxika, M. Revilla, J.G. Rodríguez, O. Solaun, A. Uriarte, and V. Valencia, *Using multiple ecosystem components in assessing ecological status in Spanish (Basque Country) Atlantic marine waters*, Mar. Pollut. Bull. 59 (2009), pp. 54–64.

[77] A. Borja, I. Tueros, M.J. Belzunce, I. Galparsoro, J.M. Garmendia, M. Revilla, O. Solaun, V. Valencia, *Investigative monitoring within 545 the European Water Framework Directive: A coastal blast furnace slag disposal, as an example*, J. Environ. Monitor. 10 (2008), pp. 453–462.