Strategies: Sediment Quality

A Guidance for the Assessment and Evaluation of Sediment Quality

A German Approach Based on Ecotoxicological and Chemical Measurements

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Abstract. The recommendations presented in this paper use an integrated hierarchical approach combining toxicological, chemical and ecological information to assess and evaluate the quality of sediments. For this reason, biological methods, in combination with the tools of chemical analysis, are given priority when examining the quality of the sediment to establish adverse effects. The trigger is the biotest, instead of chemical methods commonly used. The individual methods are presented for a German approach and can be adopted to other countries by applying ISO, OECD methods. Support is provided on how to best integrate data generated using different assessment tools.

Keywords: Bioassays; biological and chemical analysis; ecotoxicology; integrated assessment; sediment assessment; sediment quality; stepwise strategy

Introduction

Until now, risk estimations for sediments have mostly been realised by means of chemical analysis or combinations of chemical/toxicological approaches, like in the sediment triad (Anderson et al. 2001, Chapman 2000). Regulatory authorities rely more often on numerical Sediment Quality Guidelines (SQGs) than the scientific community would do. While numerical SQGs are appropriate to characterise the chemical load of a sediment, two limitations are obvious which predict implications for ecosystems or human health. An increasing number of new substances is released to the environment every year and adds up to an amount impossible to monitor on a routine basis, also if appropriate methods existed for their analyses. In addition, chemical contaminants in sediments rarely affect organisms as single substances, but instead cause adverse effects as diverse mixtures. It is well known that ecosystems and their interactions with stresses are complex. If we reflect this complexity with data from other assessment tools such as toxicity tests, benthic community surveys, and biomarker studies, we need an appropriate method for a reliable evaluation. Burton (2001) proposed a 'weight-of-evidence' approach in which possible conclusions could be derived by interpreting typical response patterns.

To help practitioners like Port authorities, dredgers, competent regulatory authorities in sediment assessment and risk management decisions, a couple of projects were funded in Germany (e.g. Ahlf and Gratzer 1999, Ricking and Pachur 1999, Hollert and Braunbeck 2001). The outcome of these studies were discussed among scientific experts and environmental regulators and it was evident to all that additional work needs to be conducted to determine how to integrate the information derived from multiple chemical and biological lines of evidence. The paper presented here should provide practitioners in sediment assessment/management with technical guidance for how numeric SQGs and other assessment tools such as toxicity tests or benthic community surveys can be integrated into a sediment assessment framework.

Since the basic question is whether biological impairments are caused by contaminated sediments, the biological assessment facilities are the basis for the development of a regulatory scheme. We think that understanding the bioavailability of pollutants is the key issue for assessing sediment quality, which is increasingly being realised as the primary issue for a risk management (Ahlf and Förstner 2001). This scientific point of view is complemented by the use of SQGs in practice.

The recommendations are directed to environmental authorities, which need

- to classify hot spots, rank contaminated sites using chemical and biological methods
- to make decisions for more detailed studies on the site-specific damage of aquatic communities
- to trigger regulatory action and establishing target remediation objectives, e.g. for dredged material.

The following recommendations address the problem of providing an accurate assessment of sediment quality in rivers, lakes and waterways, while considering a cost-effective procedure. The number of modules will depend on the objective of the examination and, in many cases, it will not be necessary to apply the entire range of analytical tools.

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1 Assessment Strategy

This guidance is based on the assumption that the sites at which the sediment quality assessment would be applied have already been identified as contaminated or are suspected to be. Contamination can be detected by a variety of ecotoxicological, biological, or chemical indicators. The process of identifying and evaluating the indication of contamination can be put in concrete forms:

- The water is influenced by discharges.
- No indicators of good water quality are found in the benthic bioecocenosis (e.g. amphipoda, chironomids).
- Organisms, fish in particular, display abnormalities.

When a contaminated site is recognised, the problem owners should define the objectives of an assessment like general quality monitoring, identification of hotspots, or finding remedial options. The approach can be used to set management goals by asking the following questions:

- What are the problems caused by the contaminated sediments?
- To what extent are the sediments contaminated?
- Where are the pollution hotspots?
- Are there other potential sources in the water body posing an ecological problem?
- Who is affected by the problems?
- How big is the problem?
- Which contaminants are toxic?
- What are the various sediment management options?

As a consequence, the resulting diverse objectives need a flexible framework for evaluating sediments. In a conceptual sediment assessment model, chemical, ecotoxicological, and ecological are combined in a hierarchical strategy (**Fig. 1**). In principal, chemical analysis is capable of recording source substances and their metabolites. However, this method provides no data about the effect of the pollutants on organisms, and would not provide any information about synergetic / antagonistic factors. Ecotoxicological testing should be conducted on an appropriate, limited set of species, end points, and exposure routes (Keddy et al. 1995). Bioassays provide data about the effect, without pinpointing the substance and

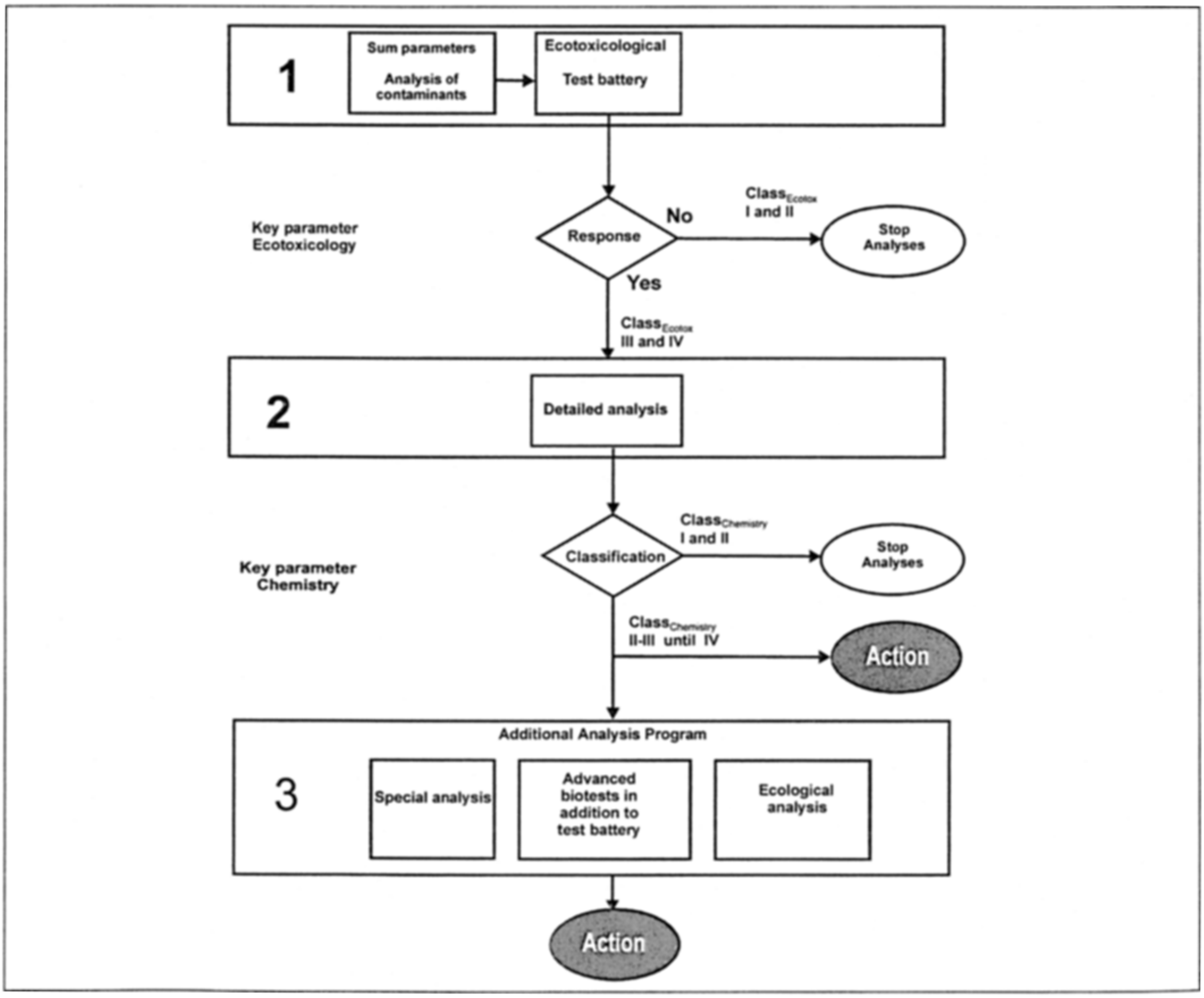

Fig. 1: Hierarchical assessment of contaminated sediments

the potential source. However, if this is carefully done and the results are interpreted as an integrative assessment, there is a clear opportunity to prioritise areas of most concern. Previous studies have shown that a minimum of tests is needed in order to rank sites in a reliable manner (Ahlf et al. 2002). Due to the fact that the effect measured in a biotest does not enable an assessment to be made of the exact damage caused in the ecosystem, the third component in the assessment strategy is an ecological examination of the invertebrate community structure. Structure and/or functional parameters allow the level of damage in a bioecocenosis to be assessed (Chapman 2000). The three phases of the hierarchical procedure will be explained in detail.

2 Examination Programme Based on ISO/OECD Methods for Parts 1 and 2

The first phase involves characterization both of chemical inventory and toxicological effects. A stepwise interpretation of the results trigger the next phase of the assessment programme (Fig. 1). The modules of each phase contain a set of analytical methods, which are described as follows.

2.1 Ecomorphologic description of the sample site

Prior to the ecotoxicological examinations, an ecomorphological assessment of the sample sites and the catchment area should be performed. The characterization may be presented by means of a mapping of the sediments. Details concerning the area, discharges and outlets, use of the waters, water quality and pollution, as well as the state of the sediments, should be included.

2.2 Sample collection

Sediment samples should be taken to a maximum depth of 50–100 cm.

Phase 1:	composite sample
Rasters:	Raster methods (e.g. centre and satellites on the circumference) Sampling by means of contingency
Phases 2, 3:	Sediment core with different segments

2.3 Processing of the samples and of the test phases

The sediment samples should be stored in the dark at 4°C, according to ISO/DIS 5669-15 Part 15. For the processing of the samples and the test phases in phase 1 and raster samples a homogenisation, and a separation in the fractions <2000 µm and <63 µm should be carried out.

Aqueous elutriates: For the preparation of elutriates the samples have to be rotated with a solid phase/liquid relation of 1:4 (v/v) for 24 h at 4° C

Pore water: Centrifugation should be accomplished immediately after sampling at a speed of at least 2000 g for 30 min in a refrigerated centrifuge maintained at 4°C.

Methanol extract: Extraction with 1 part sediment and 3 parts methanol (v/v) for the determination of the bioavailable fraction (Kwan and Dukta 1990). Use in the bioassays after dilution as aqueous solutions below the toxicity of the solvent.

Acetone extract: For the bioassays of phase 3, apply Soxhlet extraction with acetone and solvent exchange to dimethylsulfoxide. The extracts are used after dilution as aqueous solutions below the solvent-dependent toxicity (Hollert and Braunbeck 1997).

2.4 Modules for the assessment

The flow chart of the assessment strategy (Fig. 1) elucidates the fact that biological effects and bioavailability are the parameters that control the process of the subsequent experimental design (phase 1). Results of chemical-analytical investigations trigger the sequential assessment modules after phase 2.

2.4.1 Toxicity assays

In phase 1, a test battery based on microbial assays is recommended to indicate the ecological hazard potential. A low amount of costs and work, as well as a standardized methodology (OECD, ISO or DIN-guidelines), are important reasons for the chosen combination of the bioassays. The bioassay-set presented was applied in several surveys assessing sediment toxicity of small and large rivers (Ahlf and Gratzer 1999, Hollert and Braunbeck 2001). For a comprehensive assessment of ecotoxicological implications in order to monitor and rank sediment quality of different aquatic systems, the set should be extended. Appropriate methods are recommended in phase 3. As an additional example, if the dredging of contaminated sediments is the objective of the study, an assessment of dioxin-like activities and fish-based assays examining teratogenic and genotoxic effects can be recommended.

Based on the results of the base assessment and sediment chemistry, further examinations should be carried out in phase 3, e.g. biomarkers to assess specific effects on organisms in the field, *in situ*-examinations, bioassays reflecting chronic effects or bioassay-directed fractionations in order to identify hazardous pollutants. These following bioassays are approved.

Ecotoxicological test battery (phase 1):

- Algal growth inhibition test (elutriate) – DIN 38412 L33
- Bioluminescence bioassay (elutriate) – DIN 38412 L34
- Bacterial contact assay (sediment) – DIN 38412 L48

Advanced biotests in addition to test battery (phase 3):

- Nematoda bioassay (sediment), supplementary to phase 1 as a minimum set for monitoring sediment quality – (Traunspurger et al. 1997, Ahlf et al. 2002)
- Chironomus bioassay (sediment) – (ASTM 1994)
- Lemna bioassay (elutriate) – (US-EPA 1996)
- Fish egg assay (Teratogenicity; sediment) – (Ensenbach 1998)
- Mutagenicity (Ames test, extract or elutriate) – (Maron and Ames 1983, OECD 1997)

- Genotoxicity (comet assay, extract or elutriate) – (Berbner et al. 1999)
- Cytotoxicity (RTG-2 cells, extract or elutriate) – (Hollert et al. 2000)
- Endocrine potential (dot blot assay, extract) – (Islinger et al. 1999)
- Bioassay-directed fractionation (extract) – (Brack et al. 1999, Hollert et al. 2000)
- Coupling of photolytic modification of sediment-associated contaminants – (Boese et al. 2000)

2.4.2 Chemical analysis

The investigation is performed with standard analysis methods according to the investigation programme of ATV/ HABAK and ARGE ELBE.

Analysis of contaminants: sum parameters (level 1)

- pH-value – DIN38414 S5, EN12176
- Total organic carbon (TOC) – DIN38409 H3
- Dry matter – DIN38414 T6
- Particle size – DIN19683-2
- Oxidation-reduction potential – Electrode
- Total phosphorus – DIN38414 S12
- Total nitrogen – Kjeldahl
- Mineral oil carbon hydrates (MCH) – DIN38409 H53

Analysis of contaminants: detailed analysis (level 2)

- Heavy metal such as Pb, Cr, Cd, Cu, Zn, Ni, Hg and additional As – DIN38406 S7 extracted with aqua regia
- PAH-EPA – DIN38414 F18
- PCB_6 – DIN38414 S20
- HCH, HCB – DIN38407 F2
- DDD, DDE – DIN38407 F2

New approaches for heavy metals could easily be included in the phase 2 analysis, as they are practicable to more laboratories.

Analysis of contaminants: special analysis (level 3)

- C/N – (Meyers and Ishiwatari 1992)
- H_2S – (Ahlf and Gratzer 1999)
- TBT, TPT incl. metabolites – DIN-preversion
- o-phosphate release – (Hupfer and Steinberg 1997)
- AVS-SEM – (Howard and Evans 1993)
- Sequential extraction of heavy metals – (van Ryssen et al. 1999)
- Acid neutralisation capacity – (Förstner 1989)
- Xenoestrogenic substances – (Safe and Gaido 1998)
- PCDD/F – E DIN 38414-24: 04.98

2.4.3 Ecological analysis

Ecological analysis is the next step to complement ecotoxicological and chemical examination. Macroinvertebrate field surveys and laboratory bioassays could yield different types of information on ecotoxicological effects, and both are therefore recommended in sediment risk assessment procedures (Peeters et al. 2001). The advantage of this approach is that no ecologically relevant stressor is excluded, simultaneously this is a disadvantage for an interpretation of the results considering various natural habitat factors. According to Chapman (1996), significant changes in bioecocenosis structures may be interpreted as a weight-of-evidence for pollution-induced degradation of examined sites. As a regular tool, however, the technique requires much specialized knowledge and is too time-consuming.

3 Evaluation of Quality Criteria for Contaminated Sediments

Level 1

The evaluation of toxicological data includes singular results as well as an integration of the whole test battery. To value the results of the individual biotests (level 1), we suggest the following classification (concerning the effects of the original sample):

Alga growth inhibition test	$G_A \geq 4$
Bioluminescence bioassay	$G_L \geq 8$
Bacterial contact assay	$\geq$50% inhibition of the undiluted sample

High toxicity of elutriates in singular samples can be proved by monitoring the dilution grade, the G-grade. It is the smallest grade of dilution factor G causing an effect below 20%.

An integrated assessment that is based on 4 bioassays, including the nematoda test from phase 3, was performed by assessing response patterns. 5 scaled toxicity classes of possible biotest response patterns were identified on the basis of 3 large-scaled sampling surveys in Germany. This classification was transformed into an expert system, using fuzzy logic to account for biotest immanent variability. Actually, this approach has been applied to develop a non-site-specific classification system for freshwater sediments (Heise et al. 2000). These classes were described as follows:

Class 1:	No single biotest is above medium toxicity and even the average inhibition is low
Class 2:	Higher extractable toxicity with no or low inhibition in any of the other biotests
Class 3:	High particle associated toxicity with low or medium toxicity in the elutriate tests.
Class 4:	High total inhibition, high elutriate toxicity, one test shows stimulation
Class 5:	High total inhibition with high toxicity in elutriate testing (hot spots).

Level 2

To classify the data of the chemical analyses, we applied the scheme of the Hamburg bureau of Strom and Hafenbau (Maaß et al. 1997), modified to non-Elbe specific levels of contamination, as shown in **Table 1**. As temporary valuation standards for sediment contamination, we use data from samples of river mud, and the ATV-standards concerning sewage and garbage (ATV 1997). It must be stated that this temporary valuation is based on the limits for drinking water and living conditions of aquatic creatures only. No long-time effects, or even the fact that the NOEC-data of many organisms are below targets, are considered properly.

Class II concentration targets for substances in the sediment are meant to be points of reference rather than limits (Ahlf

Table 1: ATV classification scheme (ARGE-Elbe) with additional compounds according to Maaß et al. (1997) adapted to non Elbe River contaminated samples (DDT, HCB; HCH, Ricking and Pachur 1999). Trace metals are given in the fraction <20 µm

Chemical	**I**	**I-II**	**II (QG)**	**II-III**	**III**	**III-IV**	**IV**	**V**	**VI**
TM [µg/g]									
Hg	0.2 – 0.4	<0.5	<0.8	<5	<10	<25	>25		
Cd	0.2 – 0.4	<0.5	<1.2	<5	<10	<25	>25		
Pb	25 – 30	<50	<100	<150	<250	<500	>500		
Cu	20 – 30	<40	<60	<150	<250	<500	>500		
Zn	90 – 110	<150	<200	<500	<1000	<2000	>2000		
Cr	60 – 80	<90	<100	<150	<250	<500	>500		
Ni	10 – 30	<40	<50	<150	<250	<500	>500		
As	3 – 5	<10	<20	<40	<70	<100	>100		
Organics									
DDT, DDD, DDE, HCH, PCB, each congener [ng/g]	n.m.	<2	<5	<10	<20	<50	>50		
HCB [ng/g]	n.m.	<2	<5	<10	<20	<50	>50		
EPA-PAH [µg/g]	< 1		1 – 4		4 – 10		10 – 20	20 – 35	>35
MCH (DIN H 18-IR) [µg/g]	n.m.		50 – 250		250 – 500		500 – 1000	1000 – 2500	
TeBT, TBT, each [ng/g]	n.m.	<10	10 – 25	25 – 75	75 – 150	150 – 250	>250		

QG: quality goal; **MCH:** mineral oil alkanes

and Gratzer 1999). What options have to be taken when concentrations are too high is down to the particular problem, but further analysis has to be done.

The analysis programme stops

- After level 1: when the test battery is showing no or low effects. (< Class$_{ecotoxicology}$...II)
- After level 2: when the target is fulfilled (< Class$_{chemistry}$...II). If the target is fulfilled although level one proved to be positive (2 tests over 80%), further examinations are required. If the target is not fulfilled, and the contaminating substance is identified, special actions have to be taken. Those options include remediation, excavation, sub-aquatic deposition with capping.

The results of step three are very complex and can only be valuated in relation to the results of steps one and two. Since its methods are meant to specialise or broaden these results, step three is about examining special sections of the whole, according to previous results and objectives. For that reason, ecological sediment examination is a part of the third phase and an option to get more information (Ingersoll et al. 1997). The ecological analysis includes the analysis of the biocenosis structure. In our experience, it seems to be suitable for a comprehensive stream assessment to use a combination of saprobic and ecotoxicological index (Hollert et al. 2002a). Despite these assessments with individual methods, there continues to be uncertainties on how to best integrate data generated using different assessment tools such as toxicity tests, benthic community evaluations, bioassay directed analyses, and sediment chemistry for accurate assessment of sediment quality and the risks associated with various sediment management options (Ingersoll et al. 1997). To overcome a part of the problem, fuzzy logic-based ranking could be used to integrate all kind of results. An integration procedure was successfully performed combining 23 bioassay results, 26 chemical parameters, and one ecological index for classifying sediment quality (Ahlf et al. 2002, Hollert et al 2002b).

4 Outlook

With the recommendations above, integrated evaluation can be realized through sediment contamination analysis, and the various results will add up to criteria of sediment quality. The modular approach allows one to adopt the scheme to other countries with different standard methods of sediment analysis. The framework has to be put into practice by Port authorities, dredgers, competent authorities' methods and will then help practitioners in sediment assessment formulate risk management decisions.

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