

H14 Test for Sediments: presentation of the protocol proposed for ratification by the MEEDDM “Hazardous vs. non-hazardous sediments” Working Group and brief rationale of the choices made

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Preliminary remark

The wish of MEEDDM (French Ministry for Ecology, Energy, Sustainable Development and the Sea) is that the “Sediments” protocol becomes part of the general “waste” context that prevails today (whether in a regulatory manner or not), without conferring on sediments an originality that nearly all waste producers could claim for their own waste.

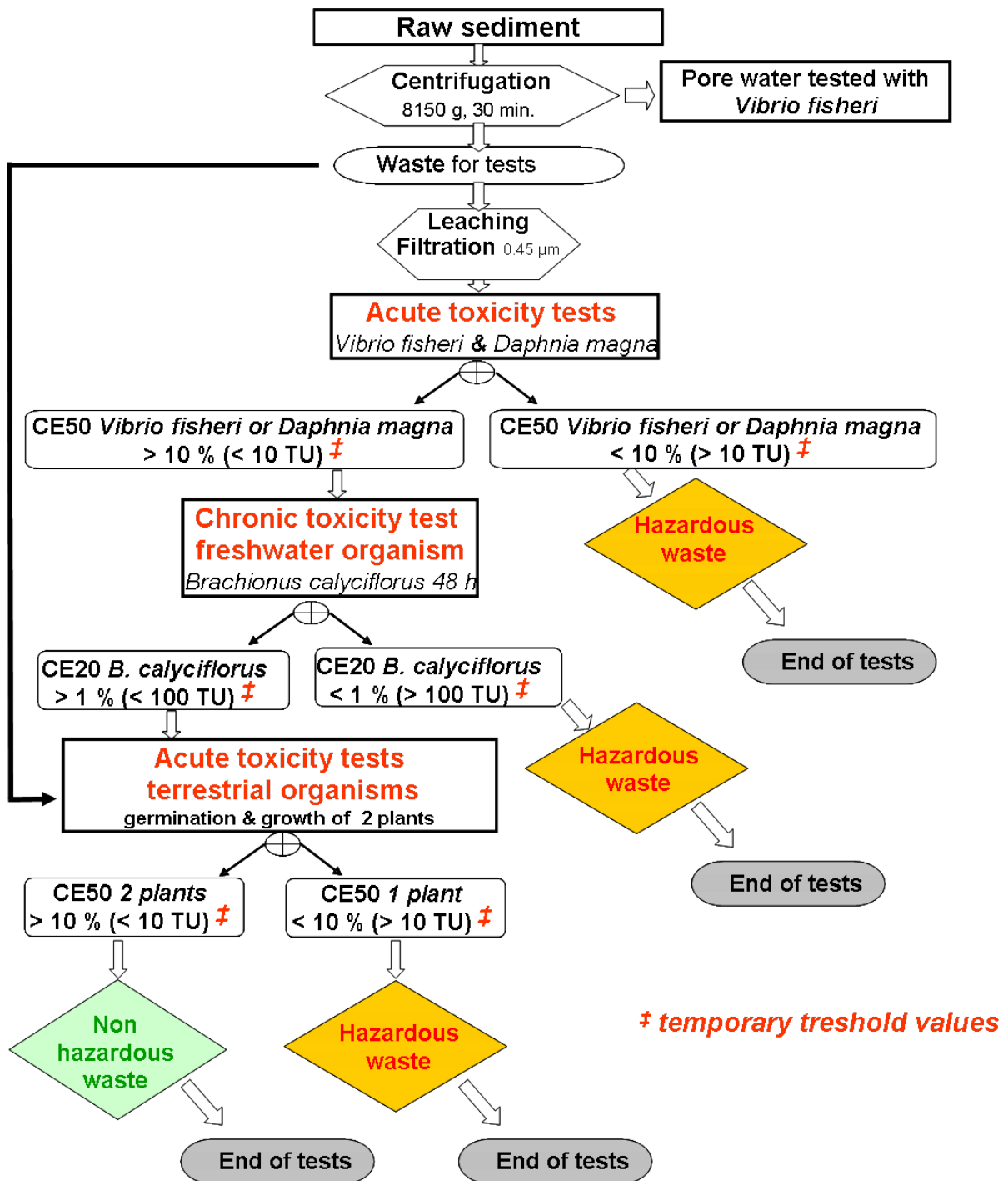
On the basis of this postulate, the proposal is to follow the basic principles of the “Waste” protocol stemming from the work of INERIS in 1998 and optimised in 2000 and 2006, namely:

- To take the toxicity of the eluate (acute toxicity and chronic toxicity) into account.
- To take the toxicity of the crude matrix (acute toxicity) into account.

The choice of ecotoxicity tests is inspired in particular by the work of the SEDIMARD-83 project and the conclusions of the scientific expert think-tank that accompanied this project. As regards the MEEDDM “Hazardous vs. non-hazardous sediments” Working Group, numerous exchanges with M. Babut (CEMAGREF), P. Pandard (INERIS) and C. Bazin (POLDEN) largely contributed to the contents of the present note.

The “MEEDDM 2009 H14 Sediments” protocol (figure 1) is designed for application over an 18-month data acquisition phase, followed by a 6-month data processing phase (feedback leading if necessary to modifications of the protocol) and the preparation of statutory instruments.

Figure 1: Protocol proposed for ratification by the MEEDDM “Hazardous vs. non-hazardous sediments” Working Group for the H14 parameter applied to marine and freshwater sediments (October 2, 2009).



1. Factor triggering the implementation of the H14 Sediments protocol

The protocol will be implemented for **all sediments for which the chemical analyses on the < 2 mm fraction exceed one of the S1 level regulatory thresholds of the Order of the 9 August 2006**. The threshold value (0.68 mg/kg) of the parameter “Total PCBs” corresponds to the sum of the concentrations of the 7 PCB indicators: PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153 and PCB 180. The threshold value (22.8 mg/kg) of the parameter “Total PAHs” corresponds to the sum of the concentrations of the 16 PAHs of the US EPA list: naphthalene, acenaphthylene, fluorene, acenaphthene, anthracene, phenanthrene, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno-pyrene, dibenzo(a)anthracene, benzo(ghi)perylene.

The discussions held on the 20 July 2009 between MEEDDM, M. Babut, P. Pandard and C. Mouvet had led to the proposal of the S1 level for continental sediments and the N1 level for marine sediments. Comments made subsequently by other members of the group led to the single referential S1 being chosen. This choice is supported by the following arguments:

- A single referential for all sediments, whether marine or continental.
- For marine sediments, the use of S1 avoids associating an image of potential hazard with sediments that exceed N1, the final destination of which in application of the Order is not necessarily land management, management for which the ecotoxicological protocol has been designed.
- S1 makes reference to values mainly determined on an ecotoxicological basis, whereas N1 and N2 mainly derive from statistical processing of physical and chemical data.

As regards the differences in parameters taken into account in the N1 and S1 threshold values, the analysis carried out by MEEDDM and BRGM is as follows:

- The obligation to carry out PAH measurements for marine sediments is only in anticipation of probable future regulations.
- For continental sediments, the non consideration of tri-butyl tin (TBT) is linked to the current lack of data on the extent of the problem in this medium, the absence of an appropriate “continental” threshold and the wish to optimise the procedure and the total cost of the evaluation of dangerousness.

In our present state of knowledge, the validity of the proposed approach is supported by feedback from VNF (Navigable Waterways of France), which shows that the *Brachionus* ecotoxicity tests on the leachates (L/S = 10) of polluted sediments are systematically negative when the index $Q_{Sm} < 0.5$ ($Q_{Sm} = 0.5$ signifies that the concentration observed for the n parameters taken into account is on average equal to half of the S1 threshold value of each parameter; at the most, the n parameters are the 8 metals/metalloids, total PCBs and total PAHs). VNF (C. Pérard-Albin, personal communication, 3 April 2009) envisages a revision of the threshold for triggering this test, which aims to only carry it out for $Q_{Sm} > 0.5$. The data made available by VNF shows that most of the cases where $Q_{Sm} > 0.5$ correspond to samples in which at least one parameter quite clearly exceeds the threshold value. The application rule proposed herein is thus in agreement with the feedback from VNF.

2. Sampling of sediments intended for the application of the H14 protocol

The number of samples to submit to the H14 protocol varies according to the extent of the dredging operation concerned: the reference values are those of the Circular of the 14 June 2000 for marine sediments and that included in the “*Circulaire technique – Opération de dragage*” (Technical circular – Dredging operation; VNF, p. 12) for continental sediments.

3. How to manage the presence of salt water in marine sediments and deal with both marine and continental sediments in as similar a manner as possible?

Evaluating the ecotoxicity of marine sediments intended for possible land management poses the problem of their natural salinity (which is not not dangerous according to the “Dangerous Substances” Directive), which can bring about a toxicity on germination and plant growth land tests. Furthermore, a chronic test in marine water is lacking whereas the “conventional” waste evaluation requires a chronic test on eluate, which will be salty if it is obtained from crude marine sediment. The natural salinity of the sediment thus has to be reduced before the chronic test on eluate and the acute test on crude waste. This phase of elimination of interstitial water (IW) has an additional interest: it enables exactly the same series of tests to be applied to both marine and continental sediments.

The separation of the IW in the ecotoxicity evaluation protocol also corresponds to an operational reality: to reduce the masses transported and enable handling operations such as shovelling, the operators will remove/gather at least part of the IW of the sediment before its transport to the discharge zone or treatment, if any. It will be up to the operators to manage this water according to the appropriate regulations; the potential exotoxicity of this IW will thus be properly taken into account. In order to assess the ecotoxic character of the IW, the *Vibrio fischeri* luminescence inhibition test (also utilisable for fresh water as for salt water) will be applied to it during the validation phase of the MEEDDM 2009 protocol.

4. How to separate the interstitial water from the solid matrix?

In order to generate the material intended for ecotoxicity tests in the laboratory, centrifugation is particularly appropriate since it may be standardised. Work carried out for SEDIMARD-83 showed that an acceleration of 8150 g (attained by quite conventional centrifugation equipment) for 30 minutes makes it possible to recover in a quantitative manner (between 80 and 90 %) the interstitial water from the sediments. The results provided following the application of the protocol should include the dryness of the tested sediment, the procedure used to attain this level of dryness, and, for marine sediments, the conductivity of the eluate (ratio L/S = 10) indicative of any ecotoxic effect on land plants linked to the residual salinity alone.

5. Choice of acute tests on eluates

The two standardised tests for which the most feedback is available are the *Vibrio fischeri* luminescence inhibition test and the inhibition of the mobility of *Daphnia magna* test. The results of SEDIMARD-83 did not show any difference in sensitivity between these two tests; the contamination of the samples studied, although particularly high for certain ports, did not in fact bring about any response from either of the two tests. The number of samples tested remains however extremely limited to draw any conclusions with regard to the choice of one or the other of these tests. Other feedback (mainly regarding effluents) show that these two tests may complement each other in their discriminating character. **During the validation phase of the MEEDDM 2009 protocol, the *Vibrio fischeri* luminescence inhibition test and inhibition of the mobility of *Daphnia magna* test should thus both be applied.**

The desire of the “Hazardous vs. non-hazardous sediments” Working Group is to build up the data base on these acute tests, both in the marine and continental environments; it is for this reason that it has been maintained in the test phase of the national protocol despite the fact it has not appeared to be discriminatory for the several sediments considered in the SEDIMARD-83 study. In particular, it is possible that the typology (different speciation of contaminants, ratios of concentrations between contaminants, the very nature of the

contaminants) or the level of contamination (potentially higher) of continental sediments leads to a certain acute effect.

6. Choice of chronic tests on eluates

Two chronic tests commonly used in the management of waste were compared on harbour sediments in the SEDIMARD-83 project: the *Pseudokirchneriella subcapitata* algae test and the *Brachionus calyciflorus* rotifer test. Among the 5 samples studied, 2 showed a certain ecotoxicity, nevertheless less than 10 toxic units. In one out of two cases, the sensitivity of the rotifer test was greater than that of the algae test. Furthermore, VNF has been using *Brachionus* for several years in the characterisation of fresh water sediments. **Consequently, it is the *Brachionus calyciflorus* rotifer test (NF ISO 20666) that has been retained in the MEEDDM 2009 protocol.** Particular attention must be paid to complying with the temperature conditions under which the test is carried out, feedback other than SEDIMARD having demonstrated the influence of this parameter on the results (the potential effects of substances such as ammonia are not specific to the selected test).

7. Choice of the acute test on “crude” sediment

Land plants and certain representatives of soil fauna are potential targets in the perspective of a land based repository for sediments. These living organisms, dicotyledons and monocotyledons for plants and earthworms for animals, are included in the series of ecotoxicity tests applied to wastes.

In the case of harbour sediments, the SEDIMARD-83 results show that the plant tests *Brassica napus* (rape) and *Avena sativa* (oats) implemented according to the ISO 11269-2 standard are more sensitive than the tests with the earth worm *Eisenia fetida* implemented according to the ISO 11268-1 standard. In so far as the samples of SEDIMARD-83 were tested after elimination of the salty matrix, it could be thought that this difference in sensitivity would also be observed with continental sediments. The lower sensitivity of the earthworm mortality test has also been demonstrated in numerous series of tests on waste. **Consequently, it is the “plant” test (ISO 11269-2) that has been retained for the validation phase of the protocol MEEDDM 2009.** The chironomidae test has not been retained, since it is more appropriate to the evaluation of the ecotoxicity of in-place sediments.

8. Choice of the threshold values for ecotoxicity tests

In the absence of any regulatory data relative to the threshold values of ecotoxicity tests applied to waste, the thresholds proposed in the “MEEDDM 2009 H14 sediments” protocol are those commonly used in the waste sector. There is in fact no reason to take thresholds different to those relating to other types of waste. The fact of adopting these thresholds does not imply that they are considered as particularly appropriate or indisputable. After a validation phase of the protocol, feedback will enable the appropriateness of these threshold values to be assessed. However, if the thresholds have to be re-examined, the revision work will have to examine the hazardous/non-hazardous classification thresholds of wastes in general and not those of sediments alone.