BALTIC SEA ENVIRONMENT PROCEEDINGS

No. 27 C

GUIDELINES FOR THE BALTIC MONITORING PROGRAMME FOR THE THIRD STAGE

PART C. HARMFUL SUBSTANCES IN BIOTA AND SEDIMENTS

BALTIC MARINE ENVIRONMENT PROTECTION COMMISSION
– HELSINKI COMMISSION –

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PREFACE

The Guidelines for the Third Stage of the Baltic Monitoring Programme (BMP) are based on the Guidelines for the Second Stage of the BMP, published by the Commission as Baltic Sea Environment Proceedings No. 12 (BSEP No.12). They have been revised by an expert group nominated by the Commission. The group was chaired by Dr. Gunni Aertebjerg and experts from all the Baltic Sea States participated in the work, with assistance from the International Council for Exploration of the Sea (ICES) and experts of the Baltic Marine Biologists (BMB).

The ninth meeting of the Helsinki Commission (15-19 February 1988) accepted the Guidelines in general as HELCOM Recommendation 9/7. The Commission recommends that the Governments of the Contracting Parties to the Helsinki Convention should apply the Guidelines for the Third Stage of the BMP, i.e. from 1989 to 1993, and also, whenever possible, to follow the Guidelines in the monitoring of the internal waters as well. The data is to be submitted to the data bases of the Commission, as specified in the Guidelines.

The Guidelines for the Third Stage of the BMP are published in the BSEP series as four separate volumes (27 A, 27 B, 27 C, 27 D) and also as one combined volume of loose sheets.

The contents of the Guidelines for the Third Stage of the BMP is as follows:

BSEP 27 A; Part A; Introductory Chapters
27 B; Part B; Physical and Chemical Determinands in Sea Water
27 C; Part C; Harmful Substances in Biota and Sediments
27 D; Part D; Biological Determinands

Volumes B, C and D are intended to be used together with Part A which contains general information on e.g. station networks, sampling requirements and data submission.

Any corrections or proposals for improvements concerning the content of these Guidelines are welcomed, and to be addressed to:

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Possible comments concerning the formats prepared by the ICES should be addressed to the ICES, accordingly, as indicated in the formats.

GUIDELINES FOR THE BALTIC MONITORING PROGRAMME FOR THE THIRD STAGE

C O N T E N T S

of the Baltic Sea Environment Proceedings No. 27 A, B, C, D

C. **HARMFUL SUBSTANCES IN BIOTA AND SEDIMENTS**
(Baltic Sea Environment Proceedings No. 27 C)

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D. **BIOLOGICAL DETERMINANDS**
(Baltic Sea Environment Proceedings No. 27 D)

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C. HARMFUL SUBSTANCES IN BIOTA AND SEDIMENTS

C. I Harmful Substances in Biota

1. Harmful substances in selected species

1.1 Species to be sampled

- Herring (*Clupea harengus*) obligatory
- Cod (*Gadus morhua*) obligatory in areas where normally found
- *Macoma baltica* (only metals) tentative
- *Mytilus edulis* tentative
- *Mesidotea entomon* tentative

Remark:

It is recommended that, in addition to this, the Contracting Parties conduct trend monitoring of contaminants in relevant coastal species. Further details concerning such sampling are described in Chapter C.II.

1.2 Substances to be analyzed

The following harmful substances are selected to be analyzed:

a) Obligatory contaminants

pp'-DDT, pp'-DDE, pp'-DDD, PCBs

Due to the greater accuracy of analyses using capillary column gas chromatography, it is recommended that the determination of individual chlorobiphenyl compounds (CBs) be made obligatory by the end of the Third Stage, with quantification of the following CBs: IUPAC Nos. 28, 52, 101, 118, 138, 153, 180. These are substances for which the levels, despite some decreases, are still of significant magnitude in Baltic biota.

Hexachlorobenzene (HCB), α- and γ-hexachlorocyclohexane (α - and γ- HCH)

These are substances which can be determined with existing analytical competence and for which further information is useful.

Total concentrations of Hg, Cd, Pb

These are metals which, at least in coastal areas and dredge spoil disposal grounds, can be harmful.

b) Tentative contaminants

Chlordanes, dieldrin

These are substances which can be of biological significance and for which more information is needed. Many Contracting Parties already have analytical programmes for these contaminants.

Zn, Cu

These have been tentative elements in the Second Stage of the BMP. However, because they are homeostatic elements in fish, it is doubtful whether the impact of their contaminant burden on the marine environment can be successfully monitored using open sea fish species.

c) Contaminants to be investigated in the future

Polychlorinated camphenes (PCCs, e.g., toxaphene), dibenzodioxins and -furans (PCDDs, PCDFs), PAHs

Analytical methods for these groups of substances (all of them probably of biological significance) are not well developed and much work remains before comparable data will be obtained. It is important that research continues in order to determine their biological impact and to develop efficient analytical methods to be used for serial analyses.

d) Intercalibrations

Laboratories of the Contracting Parties reporting data to the Helsinki Commission are encouraged to participate in any future intercalibration exercises organized by national and international bodies. It is obligatory for reporting laboratories to participate in intercalibrations organized by the Helsinki Commission.

e) Good Laboratory Practice

Laboratories are required to follow a system of Good Laboratory Practice (GLP), as described in, e.g., (15). GLP is a policy for all aspects of the laboratory which influence the quality of the analytical work: for laboratory spaces in which the work is done, for staff, analysts and technicians, for safety and equipment, handling of chemicals, and for recording and reporting the results. This includes the use of standards and certified reference materials, to test methods and ensure that they produce accurate and precise results, and the use of quality control charts. Participation in intercalibration exercises is also part of quality assurance.

1.3 Sampling, sample preparation, analytical standards and reporting procedures

a) Sampling sites

For suitable sampling sites, see the Tables and Figures 1-4 in section A.

The Contracting Parties shall provide results for obligatory species and contaminants from the sampling areas selected as their "responsibility areas". Contracting Parties are encouraged to participate in various optional analytical programmes aimed at improving the monitoring system for the BMP.

In order to save materials from various areas of the Baltic Sea for future analyses of contaminants for which the analytical methods still have to be improved, the Contracting Parties are requested to bank extra material of the species selected as trend monitoring material. If a Contracting Party cannot carry out the annual analysis of obligatory samples, it is recommended to sample and save the material in a national environmental specimen bank. The material shall be saved deep frozen, preferably at -30°C.

b) Test organisms and tissues

Test organisms

Two fish species, cod and herring, have been used as test organisms for the open Baltic Sea since 1979.

Since 1982 when the ICES Cooperative Monitoring Studies Programme began (as a revision of a programme carried out since 1974), there have been differences in the guidelines for monitoring temporal trends in contaminants between the ICES programme for the North Atlantic and the BMP. There has been a wish to harmonize the programmes between ICES and the BMP.

Before considering the details of new BMP trend monitoring guidelines, some principles for trend monitoring programmes have to be defined.

The trend of an environmental contaminant has to be studied for a defined area. The species and the part of its population selected as study material in a trend monitoring programme shall be chosen so that it will represent the defined study area. The selection of a representative material is the most important choice to be made for a monitoring programme. By sampling in

several areas of a marine region, the trend studies in various areas of the region will describe its general contamination.

Although species such as pike (*Esox lucius*) and female juvenile eel (*Anguilla anguilla*) occur locally and may be useful in the study of coastal trends, for pelagic and demersal fish species of the open Baltic Sea there are certain difficulties to fulfill the demand of having a species which throughout its life is stationary.

For various reasons, herring and cod are used as study organisms in the Baltic trend monitoring programme. Some of these reasons include:

a) they can be caught in all parts of the Baltic,
b) they are easy to collect,
c) they are a suitable size for pre-analytical sample treatment,
d) their biology is fairly well known, and
e) they are important commercial species.

However, these two species are migratory and any sampling of them must pay due attention to the period of their life at which they most probably represent the area where they are caught.

Herring

The monitoring programme for the North Atlantic recommends sampling a broad size range of fish, including a broad representation of age classes. However, a recent reassessment indicates that sampling within a constant narrow age band may be an acceptable alternative. It would be unwise to recommend sampling over a broad size range of Baltic herring for the BMP if we accept the principles mentioned at the beginning of this chapter.

Baltic herring spawn along the coasts of the Baltic Sea. Tagging experiments have shown that the herring in the eastern part of the Gulf of Bothnia are stationary (13). In the rest of the Baltic, it is well known that there are different rates of annual growth increments, especially in the earlier age classes of herring. This is interpreted as indicating that herring are a local or regional species at young age classes.

Thus, the young age classes up to the time of sexual maturity are regarded as being more representative of the areas where they are collected than older age classes, for which far distance migration often occurs after the spawning period. Data on organochlorine levels in various catches of Baltic herring have clearly shown that older age classes of herring display a higher variation in organochlorine levels than young age classes, supporting the opinion that young herring are stationary (2).

In order to fulfill the intention of a trend monitoring programme in which different Contracting Parties take responsibility for their regions in the study of trends, young prespawning age classes of herring are recommended to be collected. Because of the need for a certain volume of tissue (especially the liver) for the analytical work, only herring more than 1-2 years old can be used. Thus, herring 2-3 years old are recommended to be sampled.

Cod

In the previous recommendation for sampling cod within the Baltic, it had not been taken into consideration that the northern and eastern areas are only temporarily inhabited by cod, so that it is only in the southern and southwestern areas that cod can be found

on a regular basis. In those areas of the Baltic Sea where cod can be sampled on a regular basis, it is useful to sample cod as a complementary species to herring.

Cod is sampled in the trend monitoring programme in the North Atlantic using a different sampling strategy than that used in the BMP. To obtain more harmonization with the programme in the North Atlantic, it is suggested that the sampling strategy for that area also be used for cod from the Baltic Sea. This is described later.

The large variations in the fat content of cod liver as a result of seasonal variations and due to aging processes in cod imply that any analytical work on cod liver demands special consideration with respect to this fat content variation (11).

Macoma baltica and *Mesidotea entomon*

Macoma and *Mesidotea* (syn. *Saduria entomon*) occur throughout almost the entire Baltic Sea area, inhabiting the coastal zone as well as certain localities of the open sea. *Mesidotea* is the most important benthic species in the open sea areas of the Gulf of Bothnia. There are practical problems in the sampling procedure, making these species not useful for the analysis of organochlorines. For *Macoma*, the variation in growth rate at different sampling sites, the difficulties in determining the age, sex, and the sexual maturity stages of the individuals as well as their infestation by parasites - all phenomena of relevance for the body burden of contaminants - make the species hard to work with. However, for practical reasons, only species with a relatively large individual size can be used. Thus, because *Macoma baltica* and *Mesidotea entomon* are the only large-sized bottom-living species in the Gulf of Bothnia and the Gulf of Finland, they are included as optional species in the BMP.

Mytilus edulis

Although in other sea areas *Mytilus edulis* is considered a coastal species, in the Baltic Sea moving from the Belt Sea to the Baltic Proper, it penetrates into deeper waters and becomes sparse in the "tidal zone". Thus, *Mytilus* is a tentative species in the open sea part of the BMP. *Mytilus* from the open sea should be sampled at the agreed stations (see Section A.2.) at the depth associated with those stations. Data on the sample as indicated in Section C.II.2.c) should be obtained.

Tissue selection

The tissues recommended for analysis of harmful substances are given in Table C.1.

Regarding heavy metal analysis in herring, it is suggested that the liver be used for Cu, Zn, Cd, and Pb analysis. The main reason is the high levels found in liver compared with muscle tissue, but also because the liver reflects the recent exposure history of the fish.

Mercury occurs in similar concentrations in both muscle and liver tissue. However, because variations in feeding habits and in contamination of food may result in short-term fluctuations in mercury levels in the liver, muscle is the preferred tissue for mercury analysis. Furthermore, the correlation between fish muscle mercury levels and the size of the specimen can be used as a means to minimize some of the variance in mercury levels within a sample or between samples.

Because the organochlorines studied here are non-polar substances associated with the fatty tissues of an organism, the fat fraction should be used for their determination.

TABLE C.1. Recommended tissues for analysis of harmful substances

	Hg	Cd	Cu*	Zn*	Pb	PCB	DDTs	HCB	HCH	Chlordane*	Dieldrin*
Herring	M	L	L(M)	L(M)	L	M	M	M	M	M	M
Cod	M	L	L	L	L	L	L	L	L	L	L
Macoma *	ST	ST	ST	ST	ST	W	W	W	W	W	W
Mesidotea *	W	W	W	W	W	ST	ST	ST	ST	ST	ST
Mytilus *	ST	ST	ST	ST	ST						

* Tentative
( ) Optional

M: Muscle
L: Liver
ST: Homogenate of soft tissue
W: Whole animal

For herring, the fat should be extracted from the muscle tissue for DDT and PCB analyses. For cod, however, the fat pool is to a major extent associated with the liver, and thus liver should be used for the analysis of organochlorines.

For *Macoma* and *Mesidotea*, it is not practicable to distinguish between tissues. Thus, homogenates of the entire soft tissues of *Macoma* should be used for heavy metal analyses. For *Mesidotea*, the entire animals should be homogenized prior to analysis.

Sampling procedures

General considerations

Until the effects of physiological variables on contaminant levels are better understood, in monitoring for trend analysis sampling procedures should be carefully observed and should remain the same from year to year. To ensure sample comparability, it is recommended that the samples be collected at the same place and at the same time each year, within the guidelines given below for each species. For best results, it is suggested that the samples be collected by a trained biologist.

The recommended sampling time for each species is given for a specific period of the year, often covering two months for the whole Baltic Sea area. The longer periods given in the sampling recommendations are simply to allow flexibility for the different conditions from south to north in the Baltic Sea area. However, at an individual sampling locality, a narrow sampling period should be observed during the same portion of the appropriate season each year.

Each collection of samples should be reported according to details given in Section C.I.5.

The sampling procedures for each species have been designed to obtain a sample during a relatively stable period for the organisms in terms of migration and physiology. The stability of the habitat during the period preceding sampling is particularly important.

In the Baltic Sea it has been shown that herring have higher organochlorine levels during spring than in early autumn (6, 12). Coastal Baltic fish and freshwater fish from the vicinity of the Baltic show a dramatic increase in organochlorine levels during spring (Edgren et al. 1981, Olsson et al. 1978). For several reasons, spring seems to be an unstable period with regard to physiology (starvation after winter, reproduction, temperature increase etc.) but also the climatic factors might have an influence (ice and snow melting, spring flow).

Because of this, autumn is recommended as the sampling period, a time when many of the above-mentioned processes are avoided. For both coastal fish species and freshwater species, the levels of organochlorine residues have been shown to be stable during the autumn (6, 12).

For herring it is important to stress the presence of both spring and autumn spawning populations. The spring spawning herring shall be used in the BMP and, to obtain such material, reproductive maturity shall be determined. To obtain the stationary prespawning specimens, age shall be determined. To continue the monitoring programme already started, only females shall be used.

For *Macoma*, due to difficulties of identifying age, sex, and reproductive maturity in the large number of organisms to be sampled, a wide cross-section of the population (of adequate specimen size) should be taken and this cross-sectional distribution should remain the same from year to year as far as practicable. The same procedure should be used for *Mesidotea*; however, as reproductive maturity is easy to detect in *Mesidotea*, females carrying eggs should be avoided. *Mesidotea* reproduces at various times throughout the year and thus egg-carrying females will be found during the BMP sampling period.

Only specimens which are not obviously diseased should be used. For *Macoma*, in particular, the degree of infestation by parasites (which differs from year to year and affects the condition of the animals) should be checked in random specimens and recorded. It is suggested that a larger number of specimens than the minimum be collected to allow for possible discards at a later stage.

In collecting herring, a sample should consist of at least 20 females collected at one site. This number is necessary for statistical treatment of the data. See Table C.2. for overall sampling recommendations.

Herring should be caught away from the coast at the sampling areas designated for the Baltic Monitoring Programme. See Maps A.1.-4.

The samples should consist of female spring-spawning fish in their second to third year of life in the southern Baltic (ICES Statistical Areas 23-26) or third year of life in the central and northern Baltic. The specimens should be caught during the months of August and September in the southern and central Baltic, and during September to November in the northern Baltic Sea.

Cod

A sample of fish should consist of at least 25 individuals, and preferably more individuals. The sample should be collected in a length-stratified manner, i.e., the sizes of the fish should span as wide a length range as possible and there should be an equal number of individuals in each length grouping.

The stratification should be based upon an equidistant logged length interval, i.e. the log (upper bound) minus log (lower bound) should be equal for each length interval. The length range of the entire sample should be selected so that the individuals in the lower bound yield sufficient tissue for the chemical analyses, while the upper bound should be selected such that at least five fish can readily be found in the sampled catch. The length range should be divided into five (or more) length intervals of equal size (after log transformation). (See notes on length stratification, below, for an example.) Once the length stratification for a particular species and area has been agreed, this stratification should be strictly adhered to for a number of years. No length interval should be less than 2-3 cm. If the length range is smaller than 2-3 cm, the species is not ideally suited for the proposed analysis.

Each fish should be analysed individually and the following biological variables should always be recorded when sampling for time trend analysis purposes:

- Age
- Total weight
- Total length
- Liver weight when contaminants in liver are determined
  (if another fatty organ is used, the weight should be recorded).
- Sex
- Degree of sexual maturation } where applicable

Notes on length stratification

The main finding from the statistical analyses of data on contaminants in fish tissue is the gain in precision which can be obtained from stratification using biological variables. Although several biological parameters have been shown to be significant as stratification variables in different materials, length appears to be the only parameter which is simple to apply at sea and which shows up as being significant in most cases.

Much discussion has been devoted to whether simple linear or log-linear (multiplicative) models give the better fit. General experience with other fish and other types of data indicate preference for the log-normal model, at least for the present. As the length dependence of the contaminant level is not well understood, sampling should keep the length-contaminant relationship under constant surveillance, i.e., the entire length range should be covered evenly. The length range should be defined from practical considerations, the lower bound ensuring that enough tissue is available for chemical analysis and the upper bound such that at least 5 fish in the largest length interval can readily be found. The length stratification should be determined in such a way that it can be maintained over many years. The length interval should be at least 2-3 cm in size.

It is suggested that the length range be split into 5 length intervals which are of equal size after log transformation. For example, if the length range is 20-70 cm, then the interval boundaries could be (rounded to 0.5 cm) as follows:

cm	No. of fish	Log upper - Log lower
20 - 25.5	5	0.243
25.5 - 33.0	5	0.258
33.0 - 42.5	5	0.253
42.5 - 54.5	5	0.249
54.5 - 70.0	5	0.250
Total	25	

Care should be taken that samples are not unduly clustered within each stratum (length interval). More length intervals could be used and the test of the hypothesized contaminant-length relationship becomes stronger if the lengths are evenly distributed. But the item of major importance is to keep the length stratification identical from one year to the next.

c) Sampling procedures

TABLE C.2. Sampling procedures

Species	Time	Age	Size of organism	Sex	Sample size
Herring	Aug-Sep	1+,2+,3+	N.S.	Female	20 fish
Cod	Aug-Sep	Length	Stratified	Female	25 fish
*Macoma**	Sep	N.S.	> 5 mm	N.S.	40 g
*Mesidotea**	Oct-Nov	N.S.	40-60 mm	N.S.	80 g
*Mytilus**	Oct-Nov	N.S.	N.S.	N.S.	80 g

N.S. = Not Specified
*) = Tentative

d) Specimen data requirements

When the sample has been taken, the appropriate fish specimens should be selected according to the recommendations given for each species in the previous section.

For each fish, the total body weight in grams should be recorded, as well as the total length (length between the nose tip and the tip of the caudal fin) in millimetres.

Each specimen selected should have an intact epidermis and should be placed whole in a polyethylene plastic bag (in no case should PVC plastic be used). The air should be squeezed out and the bag should be sealed and marked with the information specified in Table C.1. For best results, the specimens should be deepfrozen as soon as possible (preferably immediately) and should remain frozen until they are to be prepared for analysis. If any procedure other than fairly immediate deep-freezing is used, this should be noted on the Table C.3.-type data sheet.

The sex should be indicated.

Information on the maturity of the gonads should be recorded.

The age should be determined and should generally be given according to the number of annual rings on the scales or otoliths. For cod, the annual rings on the otoliths should be used along with the length of the fish for the determination of age.

The weight of the liver should be recorded in grammes. Obtaining the correct liver weight can depend on using the appropriate procedure. The complete liver should be removed very carefully during dissection of the partly-thawed specimen. This should be done by a person skilled in the technique to ensure that the full sample is obtained.

Preparation of the sample for heavy metal and organochlorine analyses will be discussed in Section C.I.1.3.g).

e) Macoma baltica and Mytilus edulis
Sampling and specimen data determination

Sampling procedures

The sampling sites for *Macoma baltica* are given in tables and maps in Section A. Samples should be collected in the period of August-September (preferably September).

Although it is difficult to determine the maturity of the gonads, this will remain constant at any individual site when the same month is used for sample collection each year. The sex of the organism is also difficult to distinguish, as this requires microscopic examination, and age is difficult to determine, whether by shell rings or by size-frequency distributions. Both growth rate and maximum size vary greatly in different localities in the Baltic Sea area. Thus, it is not practical to specify overall requirements on these bases for the Monitoring Programme.

Nonetheless, it is important to obtain samples as comparable as possible from year to year. To do this, a representative specimen distribution for the sample should be established for each site and should be utilized each year thereafter. Thus, the first year, organisms should be collected and arrayed to obtain a representative sample of the whole population above the 5 mm length. When possible, the specimens should be arranged in three length classes: small, medium and large. The length of each organism should be measured as the maximum length, regardless of orientation. The number of individuals in each length class should be recorded and this distribution should thereafter be utilized for that site each year that samples are collected. The sample size for *Macoma* is 40 g of soft body tissue. The sample size for *Mytilus* is 80 g of soft body tissue.

Sample data requirements

For bivalves, it is best to carry out the initial post-sampling procedures on board the vessel to avoid a two-step procedure of freezing and re-freezing (which causes variable water losses). Thus, it is recommended that a person skilled in these procedures collect the bivalves and carry out the initial procedures as soon as possible thereafter.

For each sampling site, a Sample Information Sheet (shown in Table C.1.) should be filled out and smaller copies made to label each sample container.

When the organisms have been collected, they should be rinsed externally in clean water from the area of collection to wash away sediments and other foreign matter. They should then be allowed to remain in clean sea water from the area of collection for 12-24 hours to allow them to remove sediments and other foreign matter as pseudofaeces. The specimens should be kept alive at a temperature similar to that observed at the sampling site (preferably in a refrigerator). The storage tank should preferably be of glass.

When this time is over, the total length of each organism should be measured and the information recorded.

After draining off the shell liquor, the whole soft body of the organism including the adductor muscle should be carefully removed from the shell and combined with the others to be included in the sample. Care should be taken to avoid excessive tissue damage and thus cause water loss during this procedure. In removing the tissue and placing it in containers, it is very important to utilize the appropriate materials for

cutting tools and storage containers (see Section C.I.1.3.g) for detailed discussion). In particular, for heavy metal analysis, one should avoid direct contact of the tissue to be analyzed with metallic materials; for organochlorine analysis, one should avoid direct contact of the tissue to be analyzed with plastic materials.

For initial preparation of invertebrate samples, the required [1)] amount of soft tissues should be collected in an appropriate container, e.g., a flask with tightly fitting stopper. For heavy metal analysis, 40 g of material are required and a similar amount is needed for organochlorine analysis. For *Macoma*, 40 g only is needed and for *Mytilus*, 80 g is needed, or the amount requested by the analytical laboratory.

The container with the composite sample of organic material should be closed tightly in order to avoid any water loss and stored in a deep-freezer until the respective analytical procedures are carried out.

Prior to analysis, the organic material is homogenized. From the homogenate, subsamples are taken for dry weight, heavy metal and organochlorine determinations.

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1) to be specified by the laboratory in which analysis is made

f) Mesidotea entomon

The above text for *Macoma* and *Mytilus* applies in general also to *Mesidotea*, with the exception that the whole animals of *Mesidotea* are homogenized for analysis and 80 g are required (40 g for trace metal analyses and 40 g for organochlorine determinations).

The sampling sites for *Mesidotea* are indicated in the tables and maps in Section A. The size to sample is 40-60 mm, and specimens less than 30 mm should be avoided as they often show high concentrations of some trace metals. Egg-carrying females should not be sampled.

g) Subsampling and handling

Instrument handling during dissection and subsampling

As mentioned earlier, when directly handling tissues to be used in the analysis of harmful substances, all sources of possible contamination should be avoided. For materials to be used for heavy metal analysis, there should be no direct contact with metallic substances. Similarly, materials to be used in the analysis for organochlorines should not be placed in direct contact with plastics. The next two subsections describe recommended procedures for tools to be used in dissection and sample preparation. As a general rule, the contact time between the sample and the tools should be kept as short as possible. Grinding procedures should be checked very critically to make certain that no contamination can occur.

Subsampling for metal analysis

Cutting tools: Use cut or crushed pieces of glass or quartz knives (the latter are available from e.g., Hans Kürner, D-8200 Rosenheim)

Tools for grinding: Use silica or Teflon mortar with glass pestle for grinding of deep-frozen material. Equipment for homogenizing should be selected carefully according to the above-mentioned criteria.

Tools for holding: Use colourless polyethylene tweezers or haemostats.

After each sample has been prepared, including the samples of different organs from the same individual, the tools should be changed and washed as follows:

1. Wash in acetone or alcohol and distilled water.
2. Wash in HNO_3 (p.a. 1+1) diluted with double distilled water. Tweezers and haemostats in (1+6) acid.
3. Rinse with double distilled water.

The glass plate used during dissection should be cleaned in the same manner. The tools must be kept dust-free between working hours. Plastic tweezers and heamostats are disposable tools and should be replaced after one or two days of work.

Subsampling for analysis of chlorinated hydrocarbons

Cutting tools: Use acid-resistant steel scalpels or cut or crushed pieces of glass.

Tools for grinding: Homogenize using an Ultra Turrax homogenizer or, if the tissue contains mineral particles which will dull the homogenizer, freeze the sample hard in liquid air, then homogenize by mounting it in a Teflon or glass beaker.

Tools for holding: Acid-resistant steel tweezers.

The instruments should be washed as follows:

1. Wash in laboratory detergent and distilled water.
2. Rinse in double distilled water.
3. Rinse with 50:50 acetone (p.a.): 99% ethanol.

Be observant when cleaning the tweezers because the notches on them are difficult to clean. The glass plate used during dissection should be cleaned in the same way. The tools should be kept dust-free.

Subsampling procedures

The dissection room should be kept clean and the air should be freed from particles as much as possible. It is an advantage if the work can be carried out in a hood or under some shelter in order to prevent a direct fall-out of particles onto the sample.

The dissection should be carried out on a clean glass plate using the tools mentioned in the preceding section.

Sample handling

Fish

The dissection of fish is easiest when the material, at least the surface layers of the muscle tissue, is half frozen. For dissection of other organs, the thawing must proceed further, but it is an advantage if, for example, the liver is still frozen. It must be noted that any loss of liquid or fat due to improper cutting or handling of the tissue makes the determinations of dry weight and fat content, and consequently the reported concentrations of harmful substances, less accurate.

The epidermis and subcutaneous tissue should be carefully removed from the fish. Samples should be taken under the red muscle layer. In order to ensure uniformity of samples, the right side dorso-lateral muscle should be taken as the sample. If possible, the entire right dorsal lateral filet should be used as a uniform sample, from which subsamples can be taken after homogenizing for replicate dry weight, heavy metal and organochlorine determinations. If, however, the amount of material so obtained would be too large a sample, a specific portion of the dorsal musculature should be chosen for the sample. It is recommended that the portion of the muscle lying directly under the first dorsal fin be utilized in this case. As both fat and water content vary significantly in the muscle tissue from the anterior to the caudal muscle of the fish, it is important to obtain the same portion of the muscle tissue for each sample. This is necessary in order to ensure comparability.

Based on present knowledge and analytical experience, it is obvious that determinations of lead concentrations especially at the sub-mg/kg level in marine organisms are often significantly influenced by contamination during sampling, sample handling during dissection, and sample pretreatment for analysis. As a consequence, serious doubts are raised about the validity of published measurements of lead in biota of maritime origin as they often seem to be erroneous high.

Chow et al. stated already in 1974 (14) that " ... recent studies of the hazards of lead pollution may be misleading if they are based on analyses of ... animal tissues determined by routine analytical methods carried out without the use of clean-laboratory techniques and without the necessary sensitivity and accuracy ... ". Apart from clean-room procedures as a necessary prerequisite for extreme trace lead analysis the aforementioned authors pointed to further possible sources of systematic errors. The latter might arise from mucosal slime as a potential reservoir of large amounts of lead. They argued from their investigations that mucin secreted by the mucus cells of the epidermis contained a glycoprotein which reacted with water to form mucous slime. It is possible that strong heavy metal complexing sites in epidermal proteins withdraw lead from seawater and incorporate it into the slime. Therefore, additional precautions have to be taken to minimize or avoid transfer of mucosal slime to interior tissues during dissection.

For every tenth fish specimen, a duplicate sample should be obtained to check the whole analytical procedure starting with dissection and subsampling. The duplicate sample should be obtained by taking the corresponding left side dorso-lateral muscle in the same region as is used for the regular right side sample.

After muscle preparation, the liver should be completely and carefully removed while still partly frozen to avoid water and fat loss. Immediately after removing it from the fish, the liver should be returned to the freezer so that it will be completely frozen prior to further handling. This is particularly important for cod liver.

Invertebrates

The procedures for initial handling of *Macoma*, *Mytilus* and *Mesidotea* have been described in Sections C.I.1.3.e) and f). An appropriate sample size should be chosen so that at least duplicate analyses for heavy metals and duplicate dry weight determinations can be carried out.

h) Analytical procedures

General considerations

Each laboratory has developed its own methods of analysis and it is not the intention of this manual to standardize these methods in any way (except dry weight determination). Rather, this section of the manual will mention analytical procedures which are in common use in many Baltic laboratories today. A detailed example of a procedure for analysis of heavy metals is given in Section C.I.2. and a detailed organochlorine determination method is given in (16). An earlier method for the analysis of organochlorines is described in (9). These are intended as examples only.

Fat weight determination

The determination of fat weight is important in the interpretation of the results of organochlorine analyses. Recognizing that each laboratory has its own particular method of measuring fat weight, this manual does not attempt to standardize this determination.

However, a basic factor in the comparability of results of organochlorine analyses from different laboratories involves comparability in the methods of fat extraction. As different fat determination methods may give different results, the results can vary from laboratory to laboratory. A difference in the fat determination will result in a difference on the same magnitude in the organochlorine levels on a fat weight basis. It is an advantage if the residues can be related to a neutral lipid (triglyceride) basis. New techniques, such as dense liquid extraction, by which the fat can be fractionated, are available but not generally used. Thus, it is strongly recommended that the fat weight determination used relate to some recognized procedure, such as the Torrey Method (10), where the extraction is performed using a mixture of chloroform and methanol. This method gives higher values than soxhlet extraction with ether or extraction with ether-hexane mixtures, but provides slightly lower values than the hydrochloric acid/ether extraction method.

When the fat estimation is carried out according to a recognized fat estimation method, it is also rather certain that the chlorinated hydrocarbons will correspondingly be extracted quantitatively.

Dry weight determination

In contrast to the rest of this section, it has been decided to standardize the determination of dry weight. This value should be determined by drying 1-2 g of tissue material in a pre-heated oven at 105°C for 4 hours. If any other method is used it should be related to the above method, so that the results are comparable.

i) Digestion procedures and analytical techniques for the determination of heavy metals

General considerations

There are a number of procedures in common use in Baltic laboratories for digesting samples of biological materials and subsequently analysing them to determine levels of heavy metals. The procedures used by the laboratories taking part in the 1985 ICES baseline study of contaminant levels in Baltic biota are discussed in (3) and detailed descriptions of some of these procedures can be found in the open literature.

The most common general procedure for digestion appears to be wet digestion. This can be done either in an open system or in a closed system (e.g., the bomb technique). Dry ashing can also be used, for instance with oxygen in a closed system (Schöninger system) or with activated oxygen plasma.

Among the common analytical techniques are atomic absorption spectrophotometry, anodic stripping voltammetry and neutron activation analysis.

Standard solutions

It is recommended that laboratories participating in the Baltic Monitoring Programme purchase quantitative standards and prepare their standards according to a common procedure. The ICES intercalibration exercises have shown that when common standards and standard solution preparation procedures are used, the comparability of analytical results among laboratories is enhanced. Thus, the techniques used in intercalibration exercises are recommended here for use in the Baltic Monitoring Programme.

Individual stock solutions of each metal standard (Cu, Zn, Cd, Pb and Hg) should be prepared from stock standards (1 000 ppm) or the equivalent, e.g., Merck AAS standards or British Drug House AA standards. Replicates should be compared.

Mercury

Stock solutions (1 000 ppm) should be prepared in 1N H_2SO_4 or 1N HCl and stored in glass bottles. Fresh stock solutions should be prepared every six months or when the level of solution in the container falls below the halfway mark.

Working solutions should be prepared daily by dilution of the above stock solution using 1N H_2SO_4 together with a sufficient amount of 6% $KMnO_4$ solution to produce a distinct pink colour in the final solution. (Check the mercury content of the potassium permanganate solution as this can contain very high levels of mercury.) In practice, the working solution should be prepared immediately before use and should only have a bench life of about 2 hours.

Other metals

Stock solutions (1 000 ppm) should be made up in 1N acid and can be stored in either glass or plastic bottles. Fresh solutions should be prepared every 6 months or when the level of the solution in the container falls below the halfway mark.

Working solutions should be prepared daily by dilution of the above stock solutions using 1N acid.

j) Extraction and analysis techniques for the determination of organochlorine residues

General considerations

All laboratories working with organochlorine residue analysis of biological material use the same general procedure, namely extraction, clean-up, and gas liquid chromatography (GLC) with electron capture detection (ECD) and quantitation. New techniques to pre-separate different classes of contaminants on adsorption or exclusion chromatography can be used to make the final detection less complicated.

Extraction

To ensure complete extraction of the lipophilic substances DDT, DDD, DDE, PCB, HCB and HCH from biological material, it is necessary to perform a dehydration and disruption of the cells. This can be achieved either by homogenization of the sample with a dehydrating solvent such as 2-propanol or acetone followed by batch extraction, or a biphasic extraction using Na_2SO_4/acetone/hexane (1/1/1; w/v/v) using a ball mill.

Clean-up procedures

In some methods, removal of the fat before GLC analysis is performed in two steps. The first consists of three partitions between hexane and acetonitrile, N,N-dimethylformamide or dimethylsulphoxide. By this method, however, traces of fat are co-extracted and must be removed, e.g., by column chromatography, before GLC analysis. This clean-up procedure is non-destructive but time consuming, and is not necessary if only PCBs, DDT and its metabolites DDD and DDE are to be analyzed. In an alternative method, the fat is easily removed from the extract by means of concentrated sulphuric acid. Several methods described in the literature include an additional separation step in which DDE and PCBs are separated from DDD and DDT by column chromatography before GLC analysis. If this additional column separation step is omitted, confirmation analysis can be performed by treating an aliquot of the extract with potassium hydroxide in ethanol. This treatment will convert DDT to DDE and DDD to DDMU while PCBs and DDE are unaffected. DDE can be converted further to 1,1-dichlorodibenzophenone (DBP) by oxidation of the extract with potassium dichromate in sulphuric acid so that a pure PCB chromatogram can be obtained.

GLC analysis

The final extract can be analyzed with any GLC system having an EC-detector (^{63}Ni). The risk for decomposition of pesticides on the column is a common problem in residue analysis. Therefore, fused silica capillary columns or megabore columns are recommended. A packed column machine can easily be retrofitted with a megabore column, of which a chemically bonded one is best.

It is recommended that extracts be analyzed on columns of different polarities to verify the presence of a particular substance; in other methods, chemical conversion is used, as described above.

Quantitation

DDT and its metabolites are quantitated by comparison of the peak heights or areas in the chromatograms of the sample with the peak heights or areas in the chromatograms of the standard. This procedure normally creates no problem. PCBs can be quantitated in several ways.

In the most common method so far, the PCB standard that has a gas chromatographic pattern most similar to that of the sample is selected, and the heights of the corresponding peaks are compared. Other quantitation procedures are based on the use of a specific GLC-columnn combination with knowledge about the composition and concentrations of the different major components in a technical PCB mixture. The levels of the major components in the sample can be calculated in this method, but it is restricted to the specified GLC conditions mentioned above. Now that individual CBs are available, it is recommended that they be used and the results reported on a compound basis. The latest quantifying techniques make use of chemometrics by which multi-level statistical similarities are used for comparison.

Standard reference materials

Standard reference materials are now available for several types of matrices and their use is encouraged.

Standard solutions

It is recommended that standard solutions be prepared from compounds supplied by, for example, the United States Environmental Protection Agency. The standard prepared from the stock solution should be composed in such a manner that the components included do not interfere with each other on the applied GLC-column, and the resulting chromatogram should give peak heights of the same order.

The preparation of a stock solution (in mg/ml) should be done by weighing approximately 100 mg of the substance with an accuracy of 0.1 mg (equal to 0.1%) in a 100 ml Class A volumetric flask followed by 100 ml (69.2 g) of 2,2,4-trimethylpentane (iso-octane) weighed with an accuracy of 100 mg (0.1%). The stock solution should be checked against previously prepared stock solutions to ascertain that they contain the same concentrations.

After diluting by a factor of one hundred, which is also done by weighing, the final standard solution mixture is made up from the different stock solutions by weighing the proper volumes into a 100 ml volumetric flask adjusted to 100 ml (69.2 g) with iso-octane.

Internal standards

Normally, an internal standard is added directly to the sample. The standards should be chosen so that they follow the compounds through the entire analytical procedure.

k) Reporting of data

For each sample, the concentrations of the measured heavy metals should be reported on a wet weight (fresh tissue) basis and the organochlorines both on a fat weight and a wet weight basis. For *Macoma*, the results should also be reported on a dry weight basis. The reporting format and advice are given in C.I.5 and C.I.6.

TABLE C.3. SAMPLE INFORMATION SHEET

Species:
Analyses to be Conducted:
Person Responsible:
Date of Collection:
Area of Catch:
(including co-ordinates and depth)
Description of Bottom Sediments:
(e.g., rocky, sandy, muddy)
General Environmental Conditions:
Fishing Gear or Sampling Method:
Condition of Organisms:
Method for Determination of Age:
Storage of Sample:
(if other than immediate deep-freezing)

TABLE C.4. SAMPLING PROCEDURES

Species	Location	Depth	Time	Age	Size of organism	Sex	Sample size 1)
Herring	Away from coast	N.S.	Aug-Sep	Central and Northern Baltic 2+, 3+ years Southern Baltic 1+, 2+ years	N.S.	Female	20 fish
Cod	Away from coast	N.S.	Aug-Sep	Length stratified		Female	25 fish
Flounder	Coast, away from rivers	Max. 20 m	Aug-Sep	2+ years	N.S.	Female	20 fish
Pike	Coast, away from rivers	N.S.	Aug-Sep	N.S.	N.S.	Male	20 fish
Eel	Coast, away from rivers	N.S.	Jul-Aug	Yellow stage	40-50 cm	Female	20 fish
Macoma	Open sea and coast	Coast, max. 10 m	Aug-Sep 2)	N.S.	> 5 mm	N.S.	40 g
Mytilus	Open sea and coast	2-5 m	Oct-Nov	N.S.	N.S.	N.S.	80 g
Mesidotea	Open sea	Open sea	Oct-Nov	ca. 2-3 years	40-60 mm	N.S.	80 g
Fucus	Coast	Coast 1-2 m	Oct-Nov	Recent growth	N.S.	N.A.	ca.20 apices

N.S. = not specified
N.A. = not applicable
1) Minimum sample size; it is suggested that more be collected to account for loss
2) Preferably September

2. Analytical procedures for mercury, cadmium and lead in biological material*)

Introduction

The analytical potential of atomic absorption spectrometry (AAS) has gained significantly during the last years with respect to detection ability, precision and accuracy. Improvements in commercial instrumentation have made considerable contributions to the current status of AAS. Therefore, this instrumental technique at present can be regarded as an increasingly valuable routine analytical method frequently used for the performance of trace element analyses in biological and other materials of different origin.

The author wishes to demonstrate by this article his experience in the field of applied atomic absorption spectrometry recommending some methodological approaches for the determination of mercury, cadmium and lead in biological material. Some of the obvious still existing limitations, from the author's perspective, will also be discussed.

Pre-instrumental treatment procedures

Atomspectrometric procedures applied to the determination of trace elements in organic materials have associated calibration problems, as in most cases standard solutions cannot be completely matched to the sample under study.

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Different chemical and physical properties of the sample have a significant influence on the atomization of the element to be determined. Therefore, for the sensitive and reliable determination of trace elements in biological matrices, analytical procedures have been introduced which involve decomposition and further pretreatment of the sample prior to the instrumental measurement.

Among the preferable sample decomposition techniques employed are wet ashing with acids, either in open or closed systems, and low-temperature oxygen plasma-ashing.

In the widely applied wet ashing procedure the sample is treated with acids, mainly nitric, sulphuric and perchloric acids in different ratios (27, 24, 36, 37, 40, 25). Usually large quantities of reagents and large apparatus surfaces are needed for complete destructions, with the consequence that very often serious contamination problems (too high blank values) arise.

Within the concept of providing a blank-optimized sample pretreatment procedure for the ultra-trace element analysis in biological and environmental materials, May et al. (53) developed a $HNO_3/HClO_4$-digestion in specially designed quartz vessels that proved to be very effective with respect to reducing contamination.

A variety of instruments specially designed for acid decomposition in closed PTFE-vessels under pressure and high temperature (pressure decomposition) have been described in the literature (e.g. 17, 18, 35, 42, 68).

The versatile range of systems now available permits the handling of practically any sample quantity required for analysis in the laboratory. For the unexperienced investigator, however, it is important to note that the properties of PTFE can vary considerably. Especially contamination due to desorption of impurities from the inside vessel surface may pose problems. Therefore, it must be strongly recommended that the PTFE-digestion vessel, which is to be used for trace element analysis, has to be checked before use on its qualification for the purpose desired.

In cases where large quantities of reagents and acids and large apparatus surfaces are needed for complete destruction, decomposition with an oxygen plasma excited at high frequency may be given priority over wet decomposition methods. The ashing of organic samples using an oxygen plasma which is excited at high-frequency under low pressure has been known for a long time (26). Based on the work and experience of Carter et al. (20) and Raptis et al. (59), promising powerful plasma decomposition arrangements have recently been introduced by several manufacturers which preclude (with the exception of mercury) the possibility of element losses. Compared to other methods for decomposition, low-temperature O_2-plasma ashing is characterized by low blanks and has almost no consumption of chemicals.

Possibilities for the determination of mercury by cold vapour atomic absorption spectrometry

The most widely used method for the determination of mercury in various materials is the cold vapour atomic absorption technique. In this, Hg is reduced to metallic mercury in acidic solution using a powerful reducing agent, usually stannous chloride. Subsequently the mercury metal is volatilized by a suitable aeration

technique and the mercury vapour carried into an absorption cell where the 253.6 nm wavelength absorbance of mercury atoms is measured.

Organic matrices require a pretreatment according to the afore-mentioned wet decomposition procedures.

The cold vapour technique was first described by Poluektov et al. (57, 58) and later elaborated in more detail by Hatch et al. (34). Since the early 1970s several commercial accessories have been introduced and several modifications of the methodology have been investigated to reach the lowest detection limit and to improve the reliability of mercury determinations (31, 78).

Sodium tetrahydroborate has also been proposed as a reducing agent (47, 60) and found advantageous for several applications because of its higher reducing power and faster reaction. Kaiser et al. (39) observed that several interferences were considerably reduced with this reducing agent, in particular the effect of iodide.

Substantial improvement of the atomspectrometric mercury determination was evident with preconcentration of the analyte element.

Several authors have described the collection of mercury vapour by amalgamation on silver (38, 55, 52) or gold (38, 39, 56, 51, 79). This technique eliminates kinetic interferences due to a different vapourization rate or a different distribution function of the mercury between the liquid and the vapour phases. Further, the sensitivity is increased because the collected mercury can rapidly be released into an absorption cell by heating the absorber. With these methods, detection limits as low as 0.5 ng absolute

(and even lower) are realized but they do depend on blank values and the minimization of any contamination in the system.

Despite the apparent simplicity of the cold vapour atomic absorption technique there, are a number of problems associated with the measurement of mercury, especially at extremely low concentration levels.

The major difficulties arise due to the mobility of this element. Uncontrolled contamination and/or losses of this element through desorption from or adsorption on container surfaces can lead to severe systematic errors.

Several authors reported high rates of mercury adsorption on glass and plastic materials (38, 43). Quartz and glassy carbon seemed to be the best materials, especially after they were pre-conditioned by fuming out with nitric acid (39). Losses can also be found when a desiccant is uncritically used to dry the mercury vapour (Stuttgart, 1978).

A further complication regarding mercury measurements is that mercury in dilute solutions often exhibits unexpected losses on storage.

Toribara et al. (75) found that because of the high oxidation potential of the mercury (II)-mercury (I) system, almost any reducing substance can convert mercury (II) into mercury (I). In studies on the behaviour of dilute solutions of mercury (II), they realized that loss of mercury was due to reduction of some mercury (II) to mercury (I) which then spontaneously disproportionated into mercury (II) and metallic mercury. The latter escaped as metallic vapour from the solution into the gas phase.

The afore-mentioned authors as well as Feldman (23) have well established that oxidizing agents and acids are effective in preserving ionic mercury in solution.

It is recognized that stannous chloride or tetrahydroborate reduce only inorganic mercury species (reactive mercury).

Organic associates, especially organomercury compounds such as methylmercury are not affected (32, and further references cited there). This circumstance can entail a considerable under-estimation of the total mercury present in a sample, if these compounds are not properly destroyed through chemical attack during the decomposition procedure.

Outline of method

The analysis of total mercury described in the following sections involves the following operational steps:

- preparation of the sample to be analyzed in the form of a solution (test solution) through two different decomposition procedures (wet digestion under reflux with acids and pressure decomposition);
- transformation of the element to be determined (analyte) into mono-atomic vapour at ambient temperature through reduction-aeration followed by pre-enrichment on a gold absorber;
- measurement of absorption by the atomic vapour of the analyte at a characteristic spectral line (253.7 nm) after liberation from the gold absorber;
- determination of the concentration of analyte in the sample on which the measurement is carried out by comparison with reference solutions (calibration standards) of known concentrations.

Equipment

a) Instrumental device for wet digestion under reflux with acids

Wet digestion with acids under reflux is carried out in an apparatus the schematic diagram of which is shown in Figure C.1. The apparatus consists of a round bottom flask (A), a condensation reservoir (B) with pressure equalising tube (C), two-way stopcock (D) and dropping tip (E), a high intensity condenser (F), and a safety trap (G). The latter is attached at the outlet of the reflux condenser in order to prevent fumes possibly containing mercury compounds from escaping through the reflux condenser.

b) Instrumental device for acid decomposition in a closed PTFE-vial under pressure and high temperature (pressure decomposition)

Pressure decomposition is carried out in a device developed and described by Kotz et al. (42).

The sample to be decomposed is weighed into a vial of pure, isostatically pressed PTFE with round bottom (capacity 10 ml), and treated with a pure decomposition agent (HNO_3). The PTFE-vial is placed into a pressure jacket made of special stainless steel, which, by means of a safety device, is protected against any inadmissible excess pressure (max. 100 bar). With the aid of special mounting tools the pressure jacket can be defined and closely sealed. Safe and controlled heating of the pressure jacket is accomplished by an aluminium heating block provided with water connection for quick cooling. The heating block, protected by an over-temperature safety device, is connected to a temperature regulator via a thermocouple compensating

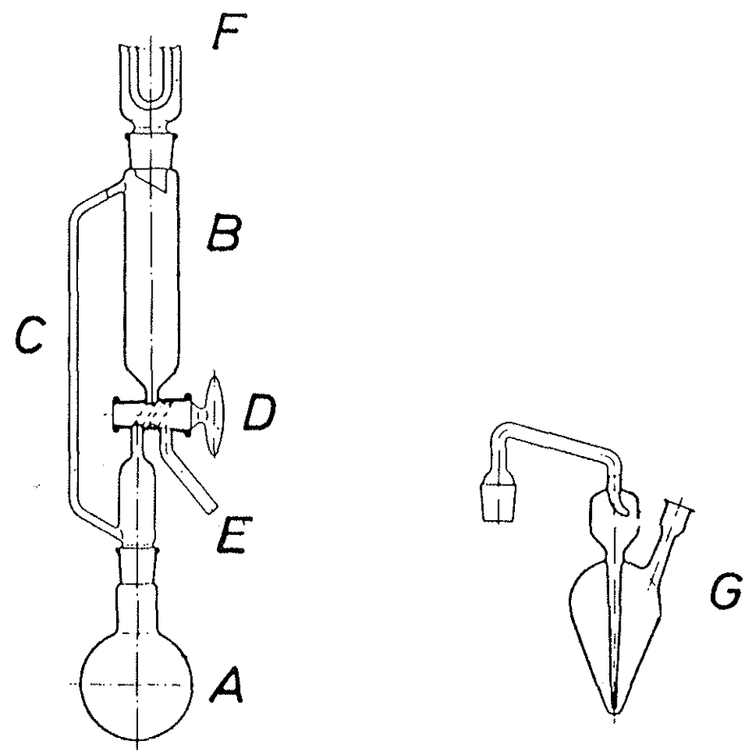

FIGURE C.1. **Schematic diagram of the apparatus for wet digestion under reflux with acids**

A. round bottom flask (100-250 ml),
B. condensation reservoir (capacity ≈ 250 ml, ≈ Ø 45 mm, length ≈ 200 mm),
C. pressure equalising tube,
D. two-way stopcock with standardized PTFE key,
E. dropping tip,
F. high intensity condenser (length ≈ 250 mm),
G. safety trap (capacity ≈ 50 ml).

line and heating line. The temperature is regulated, infinitely variable, from 0° to 250°C.

c) Instrumental device for mercury determination through cold-vapour technique

The device (see Figure C.2.) consists of a gas regulating unit (A) (needle valve, checked by a flow meter), a reaction vessel in the form of a cylindrical round bottom flask, capacity 15 ml (B), fitted to a head for gas washing bottle with sintered disc porosity 1 (C), a Pyrex-glass drying tube (D) containing magnesium perchlorate as a water absorbent.

Two three-way PTFE stopcocks (E, F) are placed between the gas regulating unit, the reaction vessel, and the drying tube to permit the reaction vessel to be by-passed during heating and elution steps.

The mercury collector (G) follows after the water absorbing tube. The collector consists of a silica tube (i. d. 3 mm, length 120 mm) which is packed with a gold coated silica wool plug (50 mg silica coated with 50 mg gold) to form a 50 mm column. The collector tube is closely wound with ca 2 m Nichrome wire (diameter 0.5 mm) connected to a variable transformer (H).

The mercury which is rapidly vaporized and eluted from the column by electrical heating (maximum temperature inside the tube 550°C) is fed directly into the optical cell (I), which is centered in relation to the axis of the light beam (J) and the optical unit (K) (wavelength selector, photoelectric detector and measuring device).

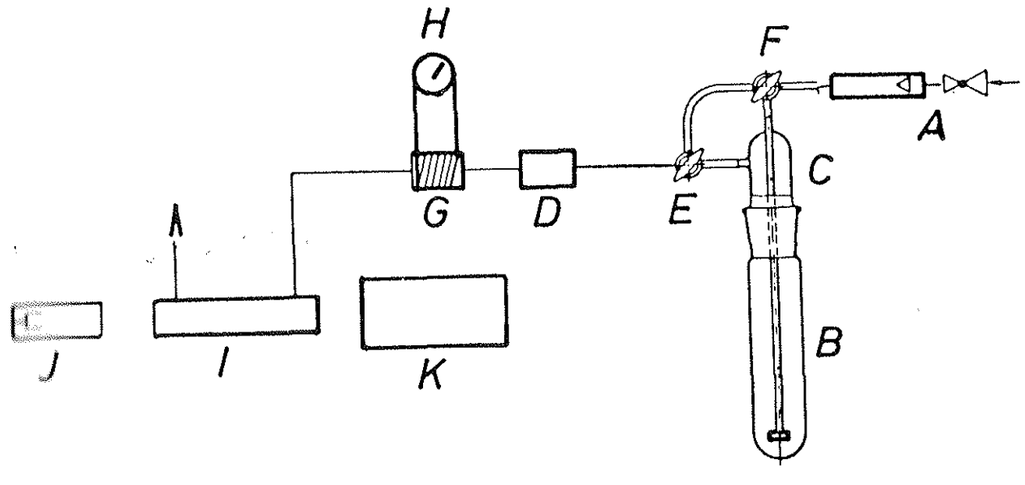

FIGURE C.2. **Schematic diagram of the apparatus for mercury determination by CVAAS**

A. gas regulation unit (needle valve, flow meter),
B. reaction vessel (capacity 15 ml),
C. head for gas washing bottle with sintered disc porosity 1,
D. drying tube (≈ Ø 20 mm, length ≈ 200 mm),
E. and **F.** stopcocks with standardized PTFE keys,
G. mercury collector (Ø 3 mm, length 120 mm) with gold silica wool,
H. transformator,
I. optical cell (≈ Ø 15 mm, length 120 mm),
J. spectral source,
K. optical unit (wavelength selector, photoelectric detector, measuring device).

All the connections in the system are made of PTFE tubing (i. d. 0.5 mm) and PTFE fittings. Nitrogen is used as a purging and carrier gas.

Reagents

Nitric acid, 10 mol/l, high purity grade,
diluted nitric acid, 1 mol/l.
Sulphuric acid 18 mol/l, high purity grade.
Hydrochloric acid, 12 mol/l, high purity grade,
diluted hydrochloric acid 6 mol/l.
Double-distilled water.
Hydroxylamine chloride salt, 69.49 g/mol,
hydroxylamine chloride salt/water 1.4 mol/l.
Magnesium perchlorate GR (for drying).
Stannous chloride hydrate 225.63 g/mol,
stannous chloride hydrate/diluted hydrochloric acid 0.5 mol/l, prepared fresh daily.
The solution is aerated with nitrogen for 30 min. immediately before use to remove traces of mercury.

Procedure

a) Sample digestion under reflux with nitric/sulphuric acid

A sample amount of 10 g (wet weight) at maximum is transferred into the round bottom flask (reaction flask), which is then attached to the condensation reservoir with the stopcock closed.

Ten ml of sulphuric acid and 20 ml of nitric acid are then added into the condensation reservoir and the main water tap is opened to allow the water to flow through the reflux condenser. The safety trap at the outlet of the reflux condenser is filled with 5 ml nitric acid.

When the apparatus has been fitted up fully, the stopcock is opened slowly so that the acid mixture flows into the sample in the reaction flask. The flask is then heated very cautiously under constant observation. When the reaction becomes too violent, the heating is immediately interrupted.

The decomposition process is carried out in three separate phases:

Phase 1) Initial reaction of the organic material with the acid mixture: During this phase the organic material reacts violently with the acid mixture forming nitric oxides and foam. In case the reaction becomes too violent and the foam threatens to reach the reflux condenser through the pressure equalising tube, it is advisable to introduce the acid mixture stepwise into the reaction flask.

Phase 2) Second decomposition step:
After the initial reaction has subsided, the mixture is heated under reflux for at least 60 minutes (up to 3 hours). When the liquid in the reaction flask becomes clear, the sample is ready for the next phase of decomposition.

Phase 3) Complete oxidation of the organic matter:
The stopcock is now closed in order to collect as much acid condensate as possible in the reservoir. Principally, a certain amount of nitric acid should remain in the reaction flask to complete the oxidation of the remaining organic material in the flask. In the event that the liquid in the reaction flask darkens at this stage of the process, the stopcock is opened to introduce a few milliliters of the acid condensate from the reservoir. The stopcock is then immediately closed and the heating procedure is continued in order to allow the rest of the nitric acid in the flask to evaporate into the condensate reservoir. The liquid in the flask should now become colourless, failing which the process of introducing a few more ml of acid condensate is repeated.

When the decomposition process has been completed, the apparatus is allowed to cool down.

The condensate is released through the stopcock into the flask, and the contents of the safety trap attached to the reflux condenser are then poured through the reflux condenser into the reaction flask. The reflux condenser is next removed and the condensation reservoir is rinsed with 10 ml of double-distilled water. The stopcock is kept in the open position to allow the water to flow down into the reaction flask. The apparatus is then fitted up again and the contents of the reaction flask is boiled for another 10 minutes. The solution will now be colourless or slightly yellow. The solution is finally cooled down and transferred into a 100 ml standard flask and filled up to the 100 ml mark with double-distilled water (test solution).

Remark: Samples with a high fat content are not fully oxidized. However, during the analysis of fish flesh, it was established that no residues of Hg were to be found in the fatty phase.

b) Sample decomposition with nitric acid in a closed PTFE-vial under pressure and high temperature (pressure decomposition)

A sample amount of 0.5 g (wet weight) at maximum is transferred into the PTFE digestion vial and the necessary quantity (0.7 ml) of nitric acid is carefully added. The vial is placed into the stainless steel jacket, which is sealed with a screw cap.

The unit is slowly (ramptime 60 min) heated up to about 80°C, controlled by a thermocouple, then heated with rapidly rising temperature up to 140°C (ramptime 45 min). The system is held at this temperature for another 120 min. The screw cap is untightened when the unit has been cooled down to room temperature (Caution! Fumes of nitrogen oxides will be expelled. Therefore, the use of a fume cupboard is absolutely necessary.)

Following this, the PTFE vial is taken out of the jacket with its contents (test solution) available for further investigation.

c) Determination of total mercury by cold vapour atomic absorption spectrometry (CVAAS)

An aliquot of 5 ml of the test solution[1)] obtained according to procedure a) is transferred to the reaction vessel of the unit for the determination of mercury (Fig. C.2.).

In the case of procedure b), the digestion vial is emptied into the reaction vessel of the said unit and rinsed with two portions of 2 ml double-distilled water[2)].

1 ml of hydroxylamine chloride salt solution[3)] is slowly added, followed by 1 ml of stannous chloride solution after 5 min.

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1) The actual amount of test solution required for mercury determination by CVAAS is dependent on the mercury concentration in the organic material under investigation. In order to meet the linear range of the calibration curve, aliquots smaller than 5 ml may be favourable.

2) For the same reason, if necessary, the test solution should be appropriately diluted and only aliquots used for the determination procedure.

3) Nitrogen oxides produced during the sample decomposition procedure form powerful oxidants which inhibit the reduction of Hg^{2+} to elemental mercury. Residues of the said oxidants are removed with hydroxylamine.

The reaction vessel is attached to the system by connecting it carefully with the head for gas washing bottle.

The three-way stopcocks are switched to the purging/collecting position and the flow rate of nitrogen is adjusted to 40 ml/sec.

The mercury is deposited by amalgamation on the gold coated silica wool plug. After purging has been completed (10 min), the stopcocks are switched to the bypass position and the flow rate of nitrogen is increased to 90 ml/sec. The elemental mercury is then vaporized from the Au column by electrical heating of the wire coil using the variable transformer. The absorption at 253.7 nm occurs in the gas cell about 1-2 sec after reaching the mercury elution temperature. The absorption peak is recorded in arbitrary units. When the response has returned to the baseline, the heating is discontinued. After a cooling period of about 5 min, the system is ready for the next cycle.

Calibration

A mercury standard stock solution (0.1 g/l Hg) is prepared by diluting a commercially available atomic spectral standard ("Baker instra-analyzed") with 1M HNO_3. For the working standard (0.1 mg/l Hg) 100 µl of the stock solution are diluted to 100 ml with 1M HNO_3 (prepared fresh daily).

Calibration curves are established by spiking diluted nitric acid (4-5 ml) with 5, 10, 20 and 40 ng Hg^{2+}, respectively. The calibration curve is linear in this range.

Calculation of results

The mercury concentration C (mg/kg) in the organic sample analyzed is calculated by the following equation:

$$C = \frac{m \cdot V_{01} \cdot V_{02} \cdot 10^{-3}}{W} \text{ (mg/kg)}$$

m = amount of mercury (ng) measured in the aliquot of test solution,
V_{01} = volume of test solution (ml) obtained after sample decomposition,
V_{02} = aliquot of V_{01} (ml) used for mercury determination by CVAAS,
W = sample amount (g) used for decomposition.

Precision

The reproducibility of the method was tested by 20 replicate analyses of two different fish flour samples (laboratory internal standards). The results are shown in Table C.5.

TABLE C.5. **Determination of precision: 20 replicate analyses of total mercury in two fish flour samples. All data in µg/kg.**

	Decomposition procedure a)		Decomposition procedure b)	
	mean	s.d.	mean	s.d.
Fish flour 1	21	±2.6	22	±1.7
Fish flour 2	700	±65	685	±59

s.d. = standard deviation

Possibilities for the determination of cadmium and lead by graphite furnace atomic absorption spectrometry

Due to its highly enhanced sensitivity graphite furnace atomic absorption spectrometry is frequently used for cadmium and lead determinations. Most of the commercial furnaces belong to the pulsed-type atomizers, such as the Massmann design (50). Experience has shown that the electrothermal atomization with pulsedtype graphite furnaces in combination with continuum background correction is often subject to serious interferences, both spectral and non-spectral (physiochemical) in origin.

The method of standard additions has been widely used as a way to analyse quantitatively where interferences are present. It is based on the assumption that inherent matrix dependent interferences have an influence on the absorbance both of the added analyte and of the analyte in the original sample to the same extent. The analyst has to consider and to investigate in each individual case whether this pre-supposition is valid.

Slavin et al. (65) stated in a recently published article that a variety of spectral interferences originated from non-uniform absorption by matrix components of the continuum energy used for background correction. Most of these were elemental absorption lines within the bandwidth of the monochromator which resulted in over-correction of the background and therefore low results for the analyte. Similar effects were found in connection with structured molecular absorption, which could, in principal, cause positive or negative errors.

In commercial AAS instruments it is widely recognized that efficient background correction systems, in particular those based on the Zeeman effect, greatly

enhance the possibilities to perform determinations free of spectral interferences. Publications of de Loos-Vollebregt et al. (44, 45, 46) describe in detail the theoretical background and practical application of this method, which is presently well in use.

Several authors, in particular L'vov (48), Sturgeon et al. (71), van den Broek et al. (19), Slavin et al. (62), Hagemann et al. (29), have explained both by theoretical calculations and experimental measurements that many of the observed non-spectral interference problems result in fact from the nonuniform temperature environment provided by conventional pulsed-type furnaces.

The established limitations of such systems have led to the development of modified designs of atomizer furnaces, especially those that operate closer to isothermal atomization conditions.

Of many approaches to achieving isothermal atomization conditions the platform technique has become the most widely applied contribution to graphite furnace atomic absorption spectrometry.

L'vov (48) developed a technique by placing a graphite platform within the pulsed-type furnace which allowed a constant temperature to be achieved for the sample atomization. The temperature of the platform, heated primarily by radiation, lags behind that of the graphite tube wall thus delaying vapourization of the analyte until the tube atmosphere is at a higher and more constant temperature. This leads to a reduction in vapour-phase interferences and produces less effect on the analyte signal from matrix-dependent variations in appearance temperature (62).

Many workers (41, 61, 63, 64, 66, 77) have shown experimentally that physico-chemical interferences are substantially reduced or eliminated when the concept of atomization in a stabilized temperature platform furnace is applied. The basis of this technique lies with vapourization of the sample from a L'vov platform in connection with a procedure which is called "matrix modification". The modifier, a chemical compound such as ammonium hydrogen phosphate or magnesium nitrate which is added to the sample prior to thermal pretreatment and atomization in the furnace, will bind the analyte in a compound, which will delay vapourization of the analyte while permitting a char temperature high enough to remove as much matrix as possible.

In the event that non-spectral interferences observed cannot be overcome by the afore-mentioned approaches, improvements in the analysis may be achieved by adopting pre-instrumental chemical separation procedures. Methods of determination which incorporate chemical separation techniques are much easier to calibrate, as simple standards with a minimum of matrix matching are required to interpolate the analysis of unknown samples from conventional analytical calibration curves. The most frequently applied principle is the conversion of the metal ions into stable, neutral chelate complexes which are transferred to an organic solvent by liquid-liquid extraction. Back-extraction with acid (decomposition of the metal complexes) eventually allows an improvement of the desired selectivity and has the advantage that metals are transferred to a solution in which their concentrations do not change with time. Furthermore, the need to prepare adequate standards by solvent extractions is eliminated. Reference is made in this connection to an earlier method (30), which was recently slightly modified (33). The procedure applied involved sample decomposition in a closed system (pressure

decomposition) followed by solvent extraction with 1,5-diphenyl-thiocarbazone ("dithizone") / toluene, and back-extraction with dilute acid (hydrochloric acid). See also the contribution to ICES Techniques in Marine Environmental Sciences.

As described by Stoeppler et al. (67, 68), other complexing reagents such as dithiocarbamates may be used successfully for solvent extraction. Systems using mixtures of dithiocarbamates (ammonium pyrrolidine-dithiocarbamate and diethyl-ammoniumdiethyl-dithiocarbamate), which were also successfully developed for the determination of trace metals in sea water (21, 22, 49), may also be employed for biological material if appropriately modified.

In view of the relatively low levels of cadmium and lead often encountered in various natural biological materials in the marine environment, improvement of the unfavourable ratio of analyte to matrix by preinstrumental chemical separation and enrichment procedures remains one of the strongest means to increase sensitivity to a realistic degree and to guarantee interference-free analysis.

However, pre-instrumental chemical treatment will be effective only if both contamination and loss of the analyte are properly controlled and minimized. Both contamination and losses represent significant sources of systematic errors (80, 72, 73, 74, 76, 69). Contamination is of particular importance, as the elements of interest usually occur in the environment from a variety of natural and anthropogenic sources.

As the blank value, which originates from impurities introduced by reagents, apparatus, reaction vessels, and laboratory air, primarily affects the reproducibility and actually obtainable limit of detection of an

analysis, special attention must be focused on holding the size and variability of the blank as small as possible (80). For these reasons, precautions have to be maintained throughout the analysis, including the thorough cleaning of labware and purification of reagents prior to use. Sample container and reaction vessels represent potentially one of the largest sources of systematic errors, since impurities leached from the materials for construction may lead to uncontrollable sample contamination. Therefore, much of the analytical reliability in the analysis of extremely low element concentrations will depend on the choice of appropriate container materials and the method of cleaning container surfaces (54).

In general, analyses at low concentrations within predictable error limits are only feasible if certain principles, as postulated by Tölg (74), are followed:

> In the measurement of extreme trace quantities, the size and variability of the blank can be reduced by carrying out the essential operation steps in a reaction room in as rapid a sequence as possible and also with a minimum surface exposure to the reagents.
> All vessels used must as far as possible be of materials (quartz, PTFE, graphite of high purity) that exclude any adsorptive or reactive effects (adsorption losses, contamination by desorption of impurities).
> The optimum conditions for decomposition and further analytical steps are a favourable ratio of the sample amount to surface area and only a small excess of reagents which can easily be purified.
> All possible contamination through the air in the laboratory should be minimized.

It is recognized that in the determination of trace metals such as mercury, cadmium and lead in organic material, conflicting features must be considered in the choice of the optimal analytical procedure. The problems become more obvious as the concentrations of the elements to be determined become lower.

The analyst's confidence in any analytical method can be strengthened considerably if appropriate quality assurance is performed. This includes participation in interlaboratory comparisons as well as checking of measurements by independent analytical procedures (e.g., differential pulse anodic stripping voltammetry or isotope dilution spark source mass spectrometry).

Possibilities for the determination of cadmium and lead by graphite furnace atomic absorption spectrometry

A detailed description of a method for the determination of low concentrations of cadmium and lead in biological tissues, prepared by Dr. U. Harms, has been published in the ICES series Techniques in Marine Environmental Sciences as Volume No. 1.

3. Chlorinated hydrocarbons in biological material

A detailed description of an appropriate method for the analysis of organochlorine contaminants in biological tissue, prepared by Dr. L. Reutergårdh, has been published in the ICES series Techniques in Marine Environmental Sciences as Volume No. 7.

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5. Reporting format for contaminants in marine biota

International Council for
the Exploration of the Sea

INTERIM REPORTING FORMAT FOR CONTAMINANTS IN MARINE BIOTA

HELCOM-VERSION

Revised Nov. 1988

REPORTING FORMAT FOR CONTAMINANTS IN MARINE BIOTA

(NOVEMBER 1988)

MAJOR REVISIONS TO THE ICES INTERIM REPORTING FORMAT FOR CONTAMINANTS IN FISH AND SHELLFISH DATED MAY 1986

This HELCOM version of the format for reporting data on contaminants in marine biota is a development of the May 1986 printing of the ICES Interim Reporting Format for Contaminants in Fish and Shellfish.

With the exception of minor editorial changes, this format fully incorporates the Reporting Format for Contaminants in Fish and Shellfish, without any major changes.

The revisions to this format are essentially those to allow the reporting of data on contaminants in other types of marine biota, specifically marine birds (and their eggs) and macrophytes. The details of these extensions to the format are presented in addenda to this document.

International Council for the Exploration of the Sea

Revised
May 1986

INTERIM REPORTING FORMAT FOR CONTAMINANTS IN FISH AND SHELLFISH

1. INTRODUCTION

This Interim Reporting Format for Contaminants in Fish and Shellfish has been developed by a sub-group of the ICES Marine Data Management Working Group for use in the ICES Cooperative Monitoring Studies Programme. It is also intended to be used for reporting data on contaminants in organisms for the Baltic Monitoring Programme of the Helsinki Commission and the Joint Monitoring Programme of the Olso and Paris Commissions.

The format has been devised to provide for the reporting of all data necessary to an evaluation of contaminant concentrations in organisms, with positions provided for comments containing additional information, where necessary. This format is to be considered an interim format and will be revised if and when experience shows this to be required.

Data reported to ICES using this format should be sent to:

Environment Officer
ICES
Palægade 2-4
DK-1261 Copenhagen K
Denmark

2. OVERVIEW OF THE SYSTEM

Three types of forms are included in this format: a Fish/Shellfish Contaminant Master, a Specimen Data Form, and a Tissue Data Form.

The Fish/Shellfish Contaminant Master serves as the master record for the entire series of data obtained on one species from one station or area. This form provides general information on the sample and where it was obtained (see definition of sample in Annex 1).

The Specimen Data Form provides the record for the data on the physical characteristics (length, weight, sex, age, etc.) of the organisms in the sample. If the organisms have been analyzed individually, one Specimen Data Form is filled out for each individual organism. If the entire sample has been analyzed in bulk as an homogenate, then only one Specimen Data Form needs to be filled in for the entire sample. If, however, the sample has been divided into several sub-samples each of which was analyzed as an homogenate, a Specimen Data Form should be filled in for each sub-sample.

The Tissue Data Form is keyed to the Specimen Data Form and is used to record the data on the concentrations of contaminants in each of the tissues analyzed from the organisms. If the organisms have been analyzed individually, one Tissue Data Form is filled in for each tissue analyzed from each organism. For samples analyzed as homogenates, two Tissue Data Forms are filled in for each tissue analysed so that the results of the duplicate analyses of the homogenates can be reported.

Positions have been provided on all forms for comments and additional information. These positions should be used for information which will assist in the interpretation of the data, thus making the data more valuable for future use. One example is the inclusion of information on methods which have been changed from previous years.

It is intended that all types of relevant data be reported on these forms. If codes for any items are missing, they can be developed on request to the ICES Environment Officer at the address given above.

3. DETAILED DESCRIPTION

3.1 General

The following description applies to all three forms. The page number should be inserted in the upper left-hand corner of each page. On the first page of a series, i.e., on the Fish/Shellfish Contaminant Master Form, the total number of pages of forms associated with that Fish/Shellfish Contaminant Master Form should also be inserted.

The upper right-hand corner of the forms should be filled in as follows:

Item	Code Description
1. Laboratory code	Insert a 4-letter mnemonic code for the laboratory reporting the data (see Annex 2).
2. Year	Insert the last two digits of the year in which the samples were taken.
3. Sequence number	On the Fish/Shellfish Contaminant Master Form, insert the number of the Master Form being filled in beginning with 001 for the first Master Form in a year, 002 for the second, etc. All other forms associated with that Master Form will have the same sequence number.

3.2 Fish/Shellfish Contaminant Master Form

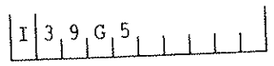
One Fish/Shellfish Contaminant Master is filled in for each species sampled at each station or area on each sampling occasion. The Master Form should be filled in according to the following description.

Item	Code Description
4. Form identifier code	The code M identifies the Fish/Shellfish Contaminant Master Form.
5. Sampling country	Insert the IOC Country Code (see Annex 3)
6. Organization code	Indicate the organization(s) for which data are submitted, as follows: I - International Council for the Exploration of the Sea J - Joint Monitoring Programme (JMP) of the Oslo and Paris Commissions B - Baltic Monitoring Programme (BMP) of the Helsinki Commission More than one designation is allowed.
7. Sampling area indicator	In the first column, indicate the code of the system used to identify the area of sampling as follows: I - ICES Statistical Rectangles B - Baltic Monitoring Programme stations C - Coordinates The remainder of the field should contain the area code, left justified, space filled.

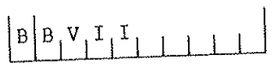
The codes, as presently available, are given in Annex 4, as follows:

Annex 4-1 ICES Statistical Rectangles
Annex 4-2 BMP Stations

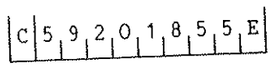
Example 1: A sampling area with the coordinates 55^0 10'N 15^0 30'E would be designated according to the ICES system as:



Example 2: Samples taken at Baltic Monitoring Station BVII would be designated as follows:



Example 3: A sample taken at the coordinates 59^0 20'N 18^0 55'E would be designated according to the coordinates system as:



8. Sampling date	Indicate the year (last two digits only), month, and date of sampling or starting sampling, i.e., catching the fish or shellfish. (The day may be omitted, if desired, and the applicable columns filled with zeroes.)
9. Purpose	Indicate purpose of monitoring in relation to the "New ICES Guidelines for Monitoring Contaminant Concentrations in Fish and Shellfish in the North Atlantic" (May 1982) or other purposes according to the following code: 0 - No specific purpose 1 - Human health 2 - Geographical distribution 3 - Trend determination 4 - Baltic Monitoring Programme
10. Species code	In the first two columns, indicate the species code list which has been used, as follows: 01- ICES 4-digit species code list 02- RUBIN code system 03- US NODC Taxonomic code In the remaining columns,indicate the code for the species sampled, left justified, space filled. NOTE: when reporting to ICES, the RUBIN 8-letter species code is strongly preferred. Codes for common species are contained in Annex 5.
11. Number of individuals used for analysis	Right justified, zero filled. Insert number of individual organisms which have been used for analysis.
12. Number of Specimen Data Forms following this Master	Right justified, zero filled. Insert number of Specimen Data forms following the Master.

13. Comments	Plain language comments can be inserted as needed. Such comments can include detailed coordinates of the sampling location, station name, bottom type, depth to bottom, sampling depth, ship name or code, or any other information of interest when interpreting the data. In addition, any information on the stock of organisms sampled should be given, e.g., stock structure and related biological information.

3.3 Specimen Data Form

If the entire sample was analyzed as an homogenate, only one Specimen Data Form should be filled in. If a sample was divided into several sub-samples each of which was analyzed as an homogenate, a Specimen Data Form should be filled in for each sub-sample. For a sample in which each organism was analyzed individually, a Specimen Data Form should be filled in for each organism. The Specimen Data Form should be filled in according to the following description.

Item	Code Description
1. Laboratory code 2. Year 3. Sequence Number	Insert same information as on Fish/Shellfish Contaminant Master
4. Form identifier code	The code S identifies the Specimen Data Form.
5. Individual or bulked specimens analyzed	Insert the appropriate code, as follows: I - if each individual organism, or parts thereof, has been analyzed separately; P - if the specimens in the sample have been grouped into sub-samples and each sub-sample homogenized and analysed separately (e.g., 25 fish divided into sub-samples of 5 fish each); H - if all the specimens in the sample have been bulked together and homogenized prior to analysis.

6. Specimen or sub-sample number	If reporting results of analyses of individual organisms, these should be ordered according to increasing length, insert 01 for the first (smallest) organism reported, 02 for the second, 03 for the third etc. If several sub-samples were taken and each was analysed as an homogenate, the first sub-sample should be reported as 01, the second as 02, etc. If the entire sample was analyzed as an homogenate, leave these columns blank.
7. Number of individuals in homogenate	If the sample or sub-sample was analyzed as an homogenate, indicate the number of organisms in the homogenate; if individual organisms were analyzed, leave blank.
8. Length	Right justified, zero filled. Insert total length in mm. For individual specimens: Insert value in columns marked "mean". For samples analyzed in bulk: Insert in the appropriate columns the minimum length, maximum length and mean (arithmetic) length of the organisms in the sample (or sub-sample) and the standard deviation of these values.
9. Whole weight	Right justified, zero filled. Insert weight of whole, ungutted fish in grams. For individual fish: Insert value in columns marked "mean". For fish analyzed in bulk: Insert in the appropriate columns the minimum weight, maximum weight and mean weight (arithmetic) of the fish in the sample (or sub-sample) and the standard deviation. For shellfish: Insert the total fresh weight of the soft parts analyzed (tail muscle or whole soft body) in the columns marked "mean".

10. Sex	Insert sex of fish according to the following code: 0 - not determined 1 - male 2 - female 3 - mixed 4 - immature 5 - hermaphrodite
11. Age	Right justified, zero filled. Insert age in years. For individual fish: Insert value in columns marked "mean". For samples analysed in bulk: Insert in the appropriate columns the minimum age, maximum age and mean age (arithmetic) of the fish in the sample (or sub-sample).
12. Shell weight of molluscs	Right justified, zero filled. Insert weight of mollusc shell(s) in grams to one decimal place. This should be the total weight for the shells of all molluscs included on this Specimen Data Form.
13. Number of Tissue Data Forms filled in	Insert the number of Tissue Data Forms which have been completed in association with this particular Specimen Data Form.
14. Comments	Plain language comments can be inserted as needed. Such comments can include the presence of parasites or other disease in the organism, or other information about the organisms sampled.

NOTE: When the muscle tissue of each fish is analysed individually but the livers from several fish are bulked together before analysis, a modification of the reporting procedure should be used. As an example, when there are 25 fish in a sample and the muscle is analysed individually for each fish but the livers from 5 fish at a time are pooled together before analysis, the following reporting procedure should be used: after the Master Form has been filled in, a Specimen Data Form should be filled in for Fish 01, followed by a Tissue Data Form for muscle tissue only, then a Specimen Data Form should be filled in for Fish 02, followed by a Tissue Data Form for muscle only, etc. After the Tissue Data Form for the muscle of Fish 25, a Specimen Data Form should be filled in for the group of Fish 01 to 05 as follows:

5. Individual or bulked specimens analysed - insert a P for partially bulked.

6. Sub-sample number - insert 26.

7. Number of individuals in homogenate - insert 005.

8. Length - insert minimum, maximum and mean length.

9. Weight - insert minimum, maximum and mean weight.

10. Sex - insert if all specimens are same sex.

11. Age - insert minimum, maximum and mean age.

12. Number of tissue data forms filled in - insert appropriate number.

13. Comments - insert comment "bulked livers of Fish 01 to 05".

After this, a Tissue Data Form should be filled in with the appropriate information, using the Sub-Sample Number 26. For the bulked livers of Fish 06 to 10, the same procedure should be followed, assigning this the Sub-Sample Number 27, and so on for the other groups of fish livers.

3.4 Tissue Data Form

If an entire sample was analyzed in bulk as an homogenate, two Tissue Data Forms should be filled in for each tissue analyzed so that the results of the duplicate analyses can be reported. If a sample was subdivided into several sub-samples each of which was analyzed as an homogenate, then two Tissue Data Forms should be filled in for each sub-sample and for each tissue analyzed. For a sample in which each organism was analyzed individually, one Tissue Data Form should be filled in for each tissue analyzed, according to the following description.

Item	Code Description
1. Laboratory code 2. Year 3. Sequence Number	Insert same information as on Fish/Shellfish Contaminant Master
4. Form identifier code	The code T identifies the Tissue Data Form
5. Specimen or sub-sample number	Insert the specimen or sub-sample number given on the corresponding Specimen Data Form.

6. Replicate number	Insert identification number for the replicate in cases where replicate analyses of the tissue or homogenate have been done (eg., 1 for the first replicate and 2 for the second).
7. Tissue analyzed	Insert a code for the tissue analyzed, as follows: 01 - Muscle 02 - Liver 03 - Tail muscle (crustaceans) 04 - Whole soft body (molluscs) 05 - Kidney 06 - Bone 99 - Other (describe in comments)
8. Tissue weight/total organ weight	Right justified, zero filled. Insert in grams to two decimal places. NOTE: This should be the <u>total</u> weight of the tissue or organ for that individual specimen <u>(or a mean weight if several individuals have been bulked)</u>, not the amount taken for analysis. This figure will be used to calculate tissue burdens.
9. Dry weight (%)	Right justified, zero filled. Insert as %, to two decimal places.
10. Fat weight (%)	Right justified, zero filled. Insert as %, to two decimal places. In last column, insert letter code for method used to determine fat weight.
11. No. of contaminants analysed	Right justified, zero filled. Insert total number of contaminants reported for the tissue. If the available number of reporting lines on one form (23) is exceeded, a second form should be used as a continuation sheet, but not be counted as a Tissue Data Form (ie. items 4 to 11 should not be filled out on a continuation sheet).
12. Contaminant code	Left justified, space filled. Insert the code for each contaminant analysed, according to the code list in Annex 6.

13. Basis	Insert a one-letter code for the basis on which the value is being reported, as follows: W - Wet weight (fresh weight) D - Dry weight L - Lipid weight (fat weight)
14. Unit	Insert the unit in which the value is being reported, either: MGKG for mg/kg, or UGKG for µg/kg. as appropriate for the contaminant concerned according to the information given in Annex 6.
15. Qualifier	In the first column, indicate whether the value should be qualified by: > - greater than, or < - less than If not, leave blank. When not detected, use < the detection limit in numerical terms. In the second column, insert a Validation Flag[1]) as follows: Blank - unspecified or quality control check has not been made A - Acceptable: data found acceptable during quality control checks S - Suspect value: data considered suspect (but not replaced) by the data originator on the basis of either quality control checks or recorder/instrument /platform performance.

[1]) From Unesco, 1982. The IOC General Magnetic Tape Format for the International Exchange of Oceanographic Data. Manuals and Guides No. 9, Part 2, p.12.

Q - Questionable Value: data considered suspect (but not replaced) during quality control checks by persons other than those responsible for its original collection, e.g., a data centre.

R - Replaced Value: erroneous or missing data has been replaced by estimated or interpolated value - method by which replacement values have been derived should be described in plain language records.

M - Missing Value: original data erroneous or missing.

16. Value — Insert the concentration of the contaminant, up to three decimal places, using only significant figures.

17. Method — Insert a 3-digit code for the method used to analyze the contaminant. Each laboratory submitting data should maintain detailed records of the methods used to analyze the contaminants and should assign a 3-digit code number for each of these methods. The appropriate code should be inserted here.

18. I/C (Intercalibration) — Insert a 2-digit code for the relevant intercalibration exercise in which the laboratory has most recently participated, according to the code list in Annex 7.

19. Detection limit — Insert the limit of detection of the contaminant according to the method of analysis used.

20. Analytical Laboratory — Insert the code for the laboratory analyzing each contaminant, as appropriate.

21. % Recovery of Standard — Insert in percent (to one decimal place) the ammount of internal standard recovered during analyses for organochlorines.

22. Comments — Plain language comments can be inserted as needed. Such comments can include information relevant to the interpretation of the contaminant values, comments on the methods used, description of an intercalibration exercise, etc.

4. GENERAL REMARKS FOR ALL RECORDS

Except where stated otherwise in the above sections, whenever an element is missing this should be indicated by filling the whole field for that element with blanks.

Any comments, suggestions or questions concerning this format and its use should be forwarded to:

The Environment Officer,
International Council for the Exploration of the Sea,
Palægade 2-4,
DK-1261 Copenhagen K,
Denmark.

Reporting format for contaminants in marine biota

ANNEX 1

GUIDELINES TO BE FOLLOWED FOR MONITORING LEVELS OF HARMFUL SUBSTANCES IN SELECTED SPECIES FOR THE BALTIC MONITORING PROGRAMME

For an indication of the methods to use for the collection, preparation and analysis of samples, see:

THE GUIDELINES FOR THE BALTIC MONITORING PROGRAMME FOR THE THIRD STAGE: Sections C.I. and C.II. 'Levels of Harmful Substances in Selected Species'.

Reporting format for contaminants in marine biota

ANNEX 2

LABORATORY CODES FOR THE REPORTING AND ANALYTICAL LABORATORIES OF THE MONITORING PROGRAMMES - BY COUNTRY

DENMARK	HFLD	Miljøstyrelsens Havforureningslaboratorium Jægersborg Allé 1B DK-2920 Charlottenlund DENMARK
	SCSS	State Chemical Supervision Service Mørkhøj Bygade 26-H DK-2860 Søborg DENMARK
	ICDK	Danish Isotope Center Skelbækgade 2 DK-1717 Copenhagen V DENMARK
FINLAND	IMRF	Institute of Marine Research P.O. Box 33 00931 Helsinki 93 FINLAND
G.D.R	AHZL	Arbeithygienisches Zentrum der chemischen Industrie GERMAN DEMOCRATIC REPUBLIC
	FREI	Forschungsinstitut für NE-Metalle Freiberg 9200 GERMAN DEMOCRATIC REPUBLIC
	BHIR	Bezirkshygiene-Institut Rostock GERMAN DEMOCRATIC REPUBLIC
	IGDR	Institut für Meereskunde Akademie der Wissenschaften de DDR Seestrasse 15 DDR-2530 Rostock-Warnemünde GERMAN DEMOCRATIC REPUBLIC

F.R.G AHHG
FEDERAL REPUBLIC OF GERMANY

BFRG Bundesforschungsanstalt für Fischerei
Laboratory für Radioökologie
der Gewässer
Wüstland 2,
2000 Hamburg 55
FEDERAL REPUBLIC OF GERMANY

BFKG Bundesforschungsanstalt für Fischerei
Institut für Küsten- und
Binnenfischerei
Palmaille 9
2000 Hamburg 50
FEDERAL REPUBLIC OF GERMANY

BFGG BUNDESANSTALT FÜR GEWAESSERKUNDE
Kaiserin-Augusta-Anlagen 15-17
D-5400 Koblenz
FEDERAL REPUBLIC OF GERMANY

DHIG Deutsches Hydrographisches Institut
Bernhard-Nocht-Strasse 78
D-2000 Hamburg 50
FEDERAL REPUBLIC OF GERMANY

NLWG Niedersaechsisches Landesamt
fuer Wasserwirtschaft
An der Scharlake 39
D - 3200 Hildesheim
FEDERAL REPUBLIC OF GERMANY

WGEG Wasserquetestelle Elbe
Focksweg 32 a
D - 2103 Hamburg 95
FEDERAL REPUBLIC OF GERMANY

FITG Fresenius Institut
Chemische und Biologische
Laboratorien GmbH
D-6204 Taunusstein-Neuhof
FEDERAL REPUBLIC OF GERMANY

ISHG Institute Schumacher
Laboratorium für Wasser-, Abwasser-
und Ölanalytik
Dr. Harald Schumacher (Dipl. Chem.)
Sophie-Dethleffs-Str. 4
D-2240 Heide
FEDERAL REPUBLIC OF GERMANY

LWKG Landesamt für Wasserhaushalt und
Küsten
Saarbrückenstraße 38
D-2300 Kiel 1
FEDERAL REPUBLIC OF GERMANY

VUCG Veterinäruntersuchungsamt Cuxhaven
Schleusenstraße
D-2190 Cuxhaven
FEDERAL REPUBLIC OF GERMANY

POLAND IMWP Institute of Meteorology and
Water Management
Wazingtona 42
81-342 Gdynia
POLAND

VHRP Veterinary Hygiene Research Station
Gdansk
POLAND

SFIP Sea Fisheries Institute
Aleja Zjednoczenia 1
81-345 Gdynia
POLAND

IIAP Institute of Ichthyology
Agriculture Academy
Szczecin
POLAND

SWEDEN HRFS Institute of Hydrographic Research
National Board of Fisheries
Box 2566
403 17 Göteborg
SWEDEN

UCKS University College of Kalmar
Inst. Natural Sciences and Technology
Box 905
S-391 29 Kalmar
SWEDEN

MNHS Miljögiftsövervakning PMK
Naturhistoriska riksmuseet
Box 50007
S-104 05 Stockholm
SWEDEN

NSLS National Swedish Environment
Protection Board
Naturvårdsverkets Special Analytical
Laboratory
Box 1302
S-171 25 Solna
SWEDEN

NWLS National Swedish Environment
Protection Board
Naturvårdsverkets Water Quality
Laboratory
S-75008 Uppsala
SWEDEN

LCRS Swedish Environment Protection Board
Laboratory for Coastal Research
S-170 11 Drottningholm
SWEDEN

SERI Swedish Environmental Research
Institute
Sten Sturegatan 42
Box 5207
S-402 24 Gothenburg
SWEDEN

IAMK Institutionen för analytisk och
marin kemi
Chalmers tekniska högskola
S-412 96 Gothenburg
SWEDEN

SLKS AB Svensk Laboratoriet (SWELAB)
Box 903
S-391 29 Kalmar
SWEDEN

USSR DBST Department of the Baltic Sea
Academy of Sciences
Paldiski Street 1
200031 Tallinn
USSR

ASLR Academy of Sciences of Latvian SSR
Institute of Biology
Riga 229021
USSR

ANNEX 3

IOC COUNTRY CODES FOR BMP MEMBER COUNTRIES

Country	Code
Denmark	26
Finland	34
German Democratic Republic	96
Germany, Federal Republic of	06
Poland	67
Sweden	77
Union of Soviet Socialist Republics	90

Reporting format for contaminants in marine biota

ANNEX 4-1

ICES STATISTICAL RECTANGLE CODING SYSTEM

The principle of the statistical rectangle coding system is as follows.
The latitudinal rows, each of which are 30' wide, are numbered (two digits) from 01 at the southern boundary of the ICES statistical area (36°00'N latitude (see ICES No.2 chart)) northwards to 99. The northern limit of the statistical rectangle system is thus 85°30'N latitude.
The longitudinal columns, each of which are 1° wide, are coded according to an alphanumeric system which starts at the western boundary of the ICES statistical area (44°00'W longitude (see ICES No.1 chart)) with A0, continuing A1, A2, A3, to 40°W longitude. East of 40°W the system continues B0, B1, B2..... to B9, C0, C1, C2... C9 etc., using a different letter for each 10° block and covering the entire west-east extent of the ICES statistical area, thus:

A:	44°W - 40°W	G:	10°E - 20°E
B:	40°W - 30°W	H:	20°E - 30°E
C:	30°W - 20°W	J:	30°E - 40°E
D:	20°W - 10°W	K:	40°E - 50°E
E:	10°W - 0°	L:	50°E - 60°E
F:	0° - 10°E	M:	60°E - 68°30'E (M8)

Note that letter I is omitted

When designating a statistical rectangle, the northing co-ordinate is to be stated first. Thus, the rectangle of which the south-west corner is 54°00'N, 3°00'E is designated 37 F3.

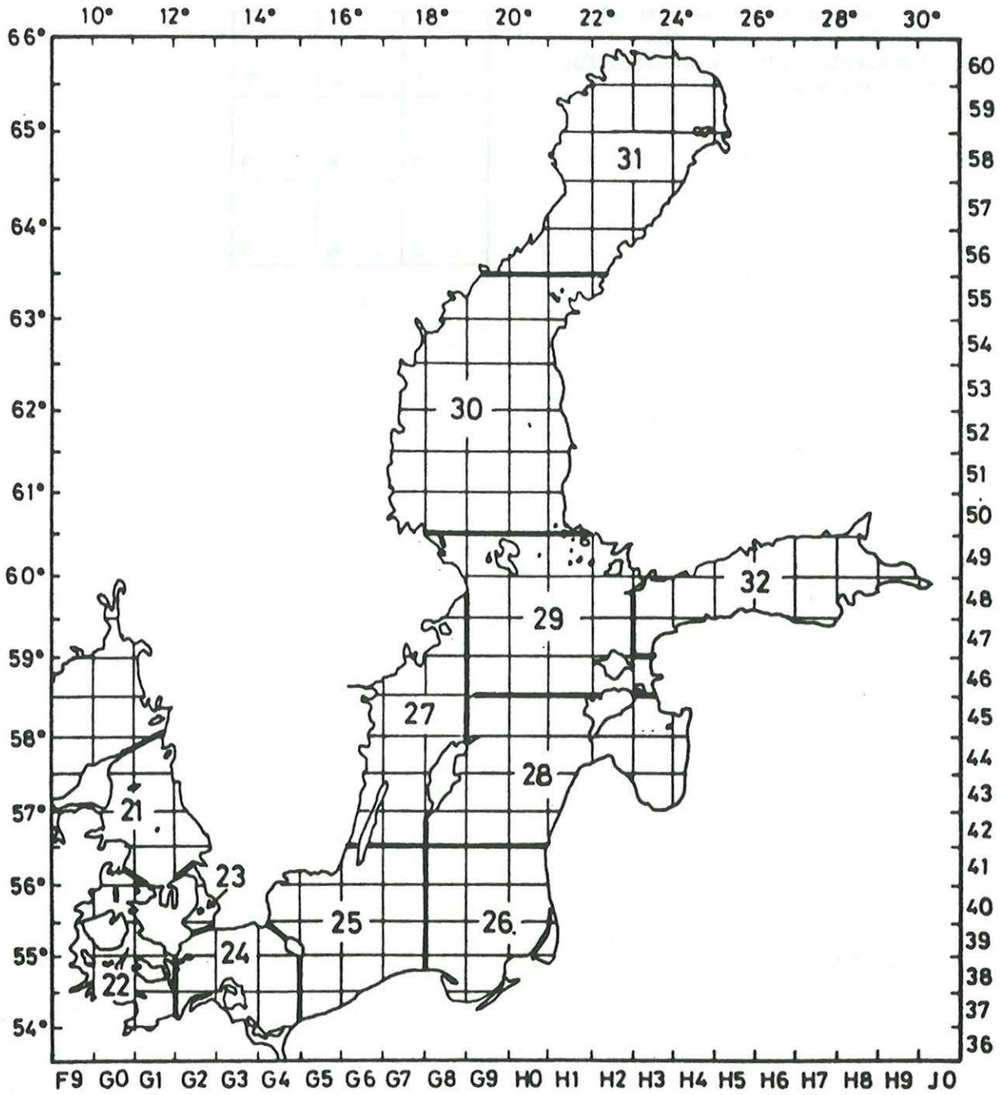
Five charts of the ICES fishing areas with the rectangle network superimposed on them are attached to this document:

1) ICES No.1 - Divisions XIVa, XIVb and Va;
2) ICES No.2 - Divisions of Sub-areas VI and VII, Sub-areas VIII and IX, major parts of Sub-areas X and XII, Sub-divisions Vb_1 and Vb_2;
3) ICES No.3 - Divisions of Sub-area IV and those parts of Sub-areas VI and VII adjacent to UK and Ireland;
4) ICES No.4 - Major parts of Divisions IIa and IIb adjacent to the coast of Norway and of Spitzbergen;
5) ICES 27.3.03.00 (Baltic) - Divisions IIId, IIIb, IIIc and part of Division IIIa brought down to Sub-divisions 21 - 32.

When it is necessary or desirable to specify an area with more precision than is possible with a statistical rectangle designation, a sub-rectangle designation may be given by dividing a statistical rectangle into nine sub-divisions, as follows:

The number of the sub-rectangle should be placed after the alphanumeric code designation for longitude, e.g., 36F84

1	4	7
2	5	8
3	6	9



ICES 27.3.03.00 (Baltic)

Division IIIa includes Sub-division 21 (plus Skagerrak)
Division IIIb is equivalent to Sub-division 23
Division IIIc is equivalent to Sub-division 22
Division IIId is divided into Sub-divisions 24-32

Reporting format for contaminants in marine biota

ANNEX 4-2

SAMPLING STATIONS FOR SHELLFISH IN THE BALTIC MONITORING PROGRAMME OF THE HELSINKI COMMISSION

POSITION		STATION NO./CODE
54°38.0'N	14°17.0'E	P38
59°35.0'N	23°18.0'E	BY25
64°18.3'N	22°21.5'E	BO3
61°55.0'N	19°06.0'E	BVII
59°37.5'N	23°19.7'E	LL11a

If additional stations are sampled, utilize the coordinates of the station.

Reporting format for contaminants in marine biota

ANNEX 5

RUBIN CODES FOR SELECTED SPECIES

RUBIN CODE	LATIN	ENGLISH	FRANCAIS	ICES CODE
ALCA TOR	Alca torda	Razorbill		
ANGU ANG	Anguilla anguilla	European eel	Anguille d'Europe	0202
CANC PAG	Cancer pagurus	Edible crab	Torteau	1101
CLUP HAR	Clupea harengus	Atlantic herring	Hareng	0701
CRAN CRA	Crangon crangon	Common shrimp	Crevette grise	1110
CRAS GIG	Crassostrea gigas	Pacific oyster	Huitres creuse	
ENGR ENC	Engraulis encrasicholus	Anchovy	Anchois	0705
FUCU VES	Fucus vesiculosus	Bladderwrack		
GADU MOR	Gadus morhua	Atlantic cod	Morue fraiche	0402
HIPP HIG	Hippoglossus hippoglossus	Atlantic halibut	Flétan de l'atlantique	0304
HOMA GAM	Homarus gammarus	European lobster	Homard Européen	1106
LIMA LIM	Limanda limanda	Common dab	Limande	0309
MACO BAL	Macoma balthica			1225
MELA AEG	Melanogrammus aeglefinus	Haddock	Eglefin	0407
MERG SER	Mergus serrator	Merganser		
› MERL MNG	Merlangius merlangus	Whiting	Merlan	0415
› MERL MCC	Merluccius merluccius	European hake	Merluche	0403
MYTI EDU	Mytilus edulis	Blue mussel	Moule	1206
MYTI GAL	Mytilus galloprovincialis	Mediterranean mussel	Moule méditerranéenne	
NEPH NOR	Nephrops norvegicus	Norway lobser	Langoustine	1107
OSTR EDU	Ostrea edulis	European flat oyster	Huitre plate	1204
PAND BOR	Pandalus borealis	Prawn	Crevette	1109
PECT MAX	Pecten maximus	Common scallop	Coquilles St. Jacques	1207
PLAT FLE	Platichthys flesus	European flounder	Flet	0311
PLEU PLA	Pleuronectes platessa	European plaice	Plie atlantique	0305
SADU ENT	Saduria entomon syn: Mesidothea entomon			
SARD PIL	Sardina pilchardus	European pilchard (sardine)	Sardine/pilchard	0703
SCOM SCO	Scomber scombrus	Atlantic mackerel	Maquereau	0902
SOLE VUL	Solea vulgaris	Common sole	Sole commune	0312
SPRA SPR	Sprattus sprattus	Sprat	Sprat	0704
STER PRD	Sterna paradisae	Arctic tern		
URIA AAL	Uria aalge	Guillemot		

Reporting format for contaminants in marine biota

ANNEX 6

CODE LIST FOR CONTAMINANTS AND UNITS

CONTAMINANT NAME	CODE	UNIT
Arsenic	AS	MGKG
Cadmium	CD	MGKG
Chromium	CR	MGKG
Copper	CU	MGKG
Mercury	HG	MGKG
Manganese	MN	MGKG
Nickel	NI	MGKG
Lead	PB	MGKG
Zinc	ZN	MGKG
DDE (p,p')	DDEPP	UGKG
DDT (o,p)	DDTOP	UGKG
DDT (p,p')	DDTPP	UGKG
TDE (p,p')	TDEPP	UGKG
DDT(p,p') + DDE(p,p')	DDTEP	UGKG
Σp,p'-DDT	sDDTp	UGKG
ΣDDT	sDDT	UGKG
Dieldrin	DIELD	UGKG
HCH-alpha	HCHA	UGKG
HCH-beta	HCHB	UGKG
HCH-gamma	HCHG	UGKG
Hexachlorobenzene	HCB	UGKG
Polychlorinated terphenyls	PCT	UGKG
Polychlorinated camphenes	PCC	UGKG

... continued

CONTAMINANT NAME	CODE	UNIT
Trans-chlordane	TCDAN	UGKG
Cis-chlordane	CCDAN	UGKG
Trans-nonachlor	TNONC	UGKG
Oxychlordane	OCDAN	UGKG
Heptachlorepoxide	HCEPX	UGKG

Polychlorinated biphenyls:

The contaminant code list associated with polychlorinated biphenyls has been extended to allow the reporting of individual PCB congeners.

The complete code list for PCBs is as follows:

CONTAMINANT NAME		CODE	UNIT
Polychlorinated biphenyls		PCB	MGKG
PCB congeners:			
		CB28	UGKG
(by IUPAC numbers)		CB52	UGKG
		CB101	UGKG
		CB118	UGKG
		CB153	UGKG
		CB138	UGKG
		CB180	UGKG
		CB18	UGKG
		CB31	UGKG
		CB44	UGKG
	CB66/95 =>	CBs01	UGKG
		CB110	UGKG
		CB149	UGKG
		CB187	UGKG
		CB170	UGKG
		CB194	UGKG
		CB206	UGKG
		CB209	UGKG
		CB128	UGKG
		CB137	UGKG
		CB195	UGKG
Defined combinations:		PCB7	MGKG
		PCB7A	MGKG
		PCB6	MGKG
		sCB	MGKG

Method for reporting PCBs:

1) In the case of determining PCB by a technique which does not involve differentiation of individual PCB congeners a single value should be reported using the contaminant code PCB and the unit MGKG.

2) If individual PCB congeners are determined the concentrations for each should be reported separately using the appropriate contaminant (CB) code from the list above (note the unit for reporting individual congeners is UGKG). Codes for other congeners not listed can be generated as required as CB followed by the congener IUPAC number. In addition, a value for the summation of the individual congener concentrations may be reported using one of the 'defined combination' codes above (note the units for reporting summations of congeners is MGKG); these codes refer to specific combinations of PCB congeners as follows:

PCB7 = CB28 + CB52 + CB101 + CB118 + CB138 + CB153 + CB180

PCB7A = [PCB7] + CB44 + CB128 + CB137 + CB194 + CB195

PCB6 = CB28 + CB52 + CB101 + CB138 + CB153 + CB180

sCB = any other combination of PCB congeners (describe in comments).

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ANNEX 7

CODE LIST FOR INTERCALIBRATION EXERCISES

CODE		
1A	1/TM/BT -	ICES First Intercomparison Exercise on Trace Metals in Biological Tissues - 1972
1B	2/TM/BT -	ICES Second Intercomparison Exercise on Trace Metals in Biological Tissues - 1973
1C	3/TM/BT -	ICES Third Intercomparison Exercise on Trace Metals in Biological Tissues - 1975
1D	4/TM/BT -	ICES Fourth Intercomparison Exercise on Trace Metals (Cadmium and Lead only) in Biological Tissues - 1977
1E	5/TM/BT -	ICES Fifth Intercomparison Exercise on Trace Metals in Biological Tissues - 1978
1F	6/TM/BT -	ICES Sixth Intercomparison Exercise on Trace Metals (Cadmium and Lead only) in Biological Tissues - 1979
1G	7/TM/BT -	ICES Seventh Intercomparison Exercise on Trace Metals in Biological Tissues - Part A - 1983
1H	7/TM/BT -	ICES Seventh Intercomparison Exercise on Trace Metals in Biological Tissues - Part B - 1985
1Z		Other Intercomparison/Intercalibration Exercise on Trace Metals in Biological Tissues - Describe in comments

- 0 -

2A	1/OC/BT -	First ICES Intercomparison Exercise on Organochlorines in Biological Tissues (Sample Nos. 2A, 2B) - 1972
2B	2/OC/BT -	Second ICES Intercomparison Exercise on Organochlorines in Biological Tissues (Sample Nos. 3A, 3B) - 1974
2C	3/OC/BT -	Third ICES Intercomparison Exercise on Organochlorines in Biological Tissues (Sample No. 4) - 1978
2D	4/OC/BT -	Fourth ICES Intercomparison Exercise on Organochlorines (mainly PCBs) in Biological Tissues (Sample No. 5) - 1979

2E	5/OC/BT -	Fifth ICES Intercomparison Exercise on Organochlorines (PCBs only) in Biological Tissues - 1982
2F	6/OC/BT -	Sixth ICES Intercomparison Exercise on Organochlorines (PCBs only) in Biological Tissues - 1983
2Z		Other Intercalibration/Intercomparison Exercise in Biological Tissues - Describe in Comments

Page ____ of ____ pages
de

LAB. YR. SEQ.NO.
AN. NO.SEQ.
1. 2. 3.

ICES INTERIM REPORTING FORMAT FOR CONTAMINANTS IN FISH AND SHELLFISH
FORMAT DE RAPPORT PROVISOIRE SUR LES CONTAMINANTS DE POISSONS ET CRUSTACES ET MOLLUSQUES

FISH/SHELLFISH CONTAMINANT MASTER
FORMULAIRE PRINCIPAL SUR LES CONTAMINANTS DE POISSONS/CRUSTACES ET MOLLUSQUES

4. Form identifier code/Code d'identification formulaire [M]
5. Sampling country/Pays effectuant échantillonnage
6. Organization code/Code de l'organisation
7. Sampling area indicator/indicateur zone d'échantillonnage
8. Sampling date/Date d'échantillonnage
   Y Y M M D D
9. Purpose/Objet
10. Species code/code l'espèce
11. Number of individuals used for analysis/Nombre d'individus utilisés pour l'analyse
12. Number of specimen data cards following this master/ Nombre de formulaires de données de specimens accompagnant le présent Formulaire Principal
13. Comments (e.g. detailed coordinates of sampling location, station name, bottom type, depth to bottom, sampling depth, ship name, measurement period, information on stock of organisms, stock structure, etc.)/Commentaires (par ex. coordonnées détaillées de l'emplacement d'énchantillonnage, nom de la station, type de fond marin, profondeur vers le fond, profondeur d'échantillonnage, nom ou code de navire, information sur le stock, structure de stock, période des prises de mesures, etc.)

Date received by ICES
Date reçu par CIEM
Y Y M M D D

(Revised June 1985)

LAB. YR. SEQ.NO.
AN. NO.SEQ.
1. 2. 3.

ICES INTERIM REPORTING FORMAT FOR CONTAMINANTS IN FISH AND SHELLFISH

FORMAT DE RAPPORT PROVISOIRE DU CIEM SUR LES CONTAMINANTS DE POISSONS ET DE CRUSTACES ET MOLLUSQUES

SPECIMEN DATA FORM/FORMULAIRE DE DONNEES DE SPECIMENS

4. Form identifier code/Code d'identification du formulaire [S]
5. Individual or bulked specimens analysed/Spécimens analysés individuellement ou en bloc
6. Specimen or sub-sample number/Numbre de spécimen ou de sous-échantillon
7. Number of individuals in homogenate (if analysed as an homogenate)/ Nombre d'individus dans homogénat (au cas où l'analyse est effectuée en homogénat)
8. Length minimum/longueur minimum
   maximum/ maximum
   mean (or individual specimen value)/ moyenne (ou valeur pour spécimen individuel)
   Standard deviation/Ecart type
   mm
9. Whole weight minimum/poids entier minimum
   maximum/ maximum
   mean (or individual specimen value)/ moyenne (ou valeur pour specimen individual)
   Standard deviation/Ecart type
   g
10. Sex/Sexe
11. Age minimum
    maximum
    mean (or individual specimen value)/ moyenne (ou valeur pour spécimen individuel)
    yr(s)an
12. Shell weight of molluscs/Poids de coquilles des molluscs
    g
13. Number of tissue data forms filled in
    Nombre de formulaires de données de tissus remplis
14. Comments (e.g., disease in organism, etc.)
    Commentaires (par ex. maladie dans l'organisme, etc.)

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Lab Code 1. YR 2. Seq.No. 3.

ICES INTERIM REPORTING FORMAT FOR CONTAMINANTS IN FISH AND SHELLFISH
FORMAT DE RAPPORT PROVISOIRE DU CIEM SUR LES CONTAMINANTS DE POISSONS, CRUSTACES & MOLLUSQUES

TISSUE DATA FORM/FORMULAIRE DE DONNEES DE TISSUS

4. Form identifier code/Code d'identification du formulaire [T]
5. Specimen or sub-sample number/Nombre de spécimen ou sous-échantillon
6. Replicate number/Numéro de réplication d'analyse
7. Tissue analysed/Tissu analysé
8. Total tissue organ weight (g)/Poids total du tissu de l'organe
9. Dry weight (%)/Poids sec (%)
10. Fat weight (%); method code/Poids de graisse (%); code de la méthode
11. Number of contaminants analysed/Nombre de contaminants analysés

12. Contam.Code	13. Basis	14. Unit	15. Qualifier	16. Value	17. Method	18. I/C	19. Det'n Lim.	20. Anal.Lab.	21. %Recovery

22. Comments (methods, etc.)/Commentaires (méthode, etc.)

ADDENDA TO THE ICES REPORTING FORMAT FOR CONTAMINANTS IN MARINE BIOTA

These addenda cover the additional forms, and the descriptions for filling out these forms, for reporting data on contaminants in marine birds (and bird eggs) or seaweeds. They constitute an extension to the format used for reporting data on contaminants in fish and shellfish. When reporting data on contaminants in birds or seaweeds, the Contaminant Master Form and the Tissue Data Form described in the main part of this document can be applied without any alterations, other than appropriate attention to details which may not be covered in this addenda.

However, new alternative Specimen Data forms have been developed, one for birds and one for seaweeds, to accomodate these additional species. These new biota-specific Specimen Data forms (detailed below) occupy the same position in the system for reporting data as the 'Specimen Data Form for fish and shellfish', described in the main part of this document.

Section 3.3 of the main format description describes how the 'Specimen Data Form (for fish and shellfish)' should be filled out when reporting data on contaminants in fish or shellfish. On the basis of these addenda:

- When reporting data on birds, section 3.3 (and the form to which it refers) should be substituted with the revised section 3.3 (b), below, and the 'Bird Specimen Data Form'.
- When reporting data on seaweeds, section 3.3 (and the form to which it refers) should be substituted with the revised section 3.3 (c), below, and the 'Seaweed Specimen Data Form'.

3.3 (b) Specimen Data Form - Birds

The present guidelines for monitoring using marine birds or bird eggs require that samples are analyzed and reported on an individual specimen basis. For such samples, a Bird Specimen Data Form should be filled in for each specimen.

To provide for possible future extensions to the guidelines, the possibility of alternative procedures for sample analysis are accommodated, but should in principle not be required at present. Thus, if the entire sample were to be analyzed as an homogenate, only one Bird Specimen Data Form should be filled in. If a sample were to be divided into several sub-samples each of which was analyzed as an homogenate, a Bird Specimen Data Form should be filled in for each sub-sample.

The Bird Specimen Data Form should be filled in according to the following description:

Item	Code Description
1. Laboratory code 2. Year 3. Sequence Number	Insert same information as on Marine Biota Contaminant Master
4. Form identifier code	The code BS identifies the Bird Specimen Data Form.
5. Individual or bulked specimens analyzed	Insert the appropriate code, as follows: (NB: the code 'I' is appropriate if present guidelines are followed correctly.) I - if each individual organism, or parts thereof, has been analyzed separately; P - if the specimens in the sample have been grouped into sub-samples and each sub-sample homogenized and analysed separately; H - if all the specimens in the sample have been bulked together and homogenized prior to analysis.
6. Specimen or sub-sample number	Insert an identifier number for the specimen concerned: 01 for the first, 02 for the second, etc.
7. Number of individuals in homogenate	If the sample or sub-sample was analyzed as an homogenate, indicate the number of organisms in the homogenate; if individual organisms were analyzed, leave blank.

8. Length of wing	Right justified, zero filled. Insert the length of the wing in mm, as measured from the carpal joint to the tip of the longest feather.
9. Whole weight of bird or egg	Right justified, zero filled. Insert weight of whole bird body or egg, in grams, to one decimal place.
10. Age of bird	Right justified, zero filled. Insert age of bird in days.
11. Thickness of egg shell	Right justified, zero filled. Insert egg shell thickness in mm to three decimal places. Shell thickness should be measured with a micrometer.
12. Maximum length of egg	Right justified, zero filled. Insert the maximum length of the egg in mm, to one decimal place, measured with a slide caliper.
13. Maximum diameter of egg	Right justified, zero filled. Insert the maximum diameter of the egg in mm, to one decimal place, measured with a slide caliper.
14. Number of Tissue Data Forms filled in	Insert the number of Tissue Data Forms which have been completed in association with this particular Bird Specimen Data Form.
15. Comments	Plain language comments can be inserted as needed. Such comments can include the main sources of food for the specimens sampled, detailed information on nest locality or other information about the organisms sampled.

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BIRD SPECIMEN DATA FORM

4. Form identifier code	B,S
5. Individual or bulked specimens analysed	
6. Specimen or sub-sample number	
7. Number of individuals in homogenate (if analysed as homogenate)	
8. Length of wing (carpal joint to tip of longest feather)	mm
9. Weight (whole bird body or egg)	g
10. Age	days
11. Thickness of shell	mm
12. Maximum length of egg	mm
13. Maximum diameter of egg	mm
14. Number of tissue data forms filled in	

15. Comments (e.g., main sources of food, use RUBIN codes where possible; details of sampling locality and nest locality, etc.,)

3.3 (c) Specimen Data Form - Macrophytes

The Macrophyte Specimen Data Form should be filled in according to the following description:

Item	Code Description
1. Laboratory code 2. Year 3. Sequence Number	Insert same information as on Marine Biota Contaminant Master
4. Form identifier code	The code MS identifies the Macrophyte Specimen Data Form.
5. Individual or bulked specimens analyzed	Insert the appropriate code, as follows: I - if the specimens in the sample have been analyzed on an individual plant basis (including situations where the analysis concerned sub-samples which derived from a single individual plant); P - if the specimens in the sample have been grouped into sub-samples derived from different plants and each sub-sample analysed separately; H - if all the specimens in the sample have been bulked together and homogenized prior to analysis, with the sample derived from several distinct plants.
6. Specimen or sub-sample number	Insert the identifier number of the specimen or sub-sample concerned: 01 for the first, 02 for the second, etc.
7. Number of individuals in homogenate	If the sample or sub-sample was analyzed as an homogenate of material derived from several distinct plants, indicate the number of plants concerned; if individual plants (or parts thereof) were analyzed, leave blank.
8. Whole plant or tip	Insert the appropriate code, as follows: P - if the whole plant was analyzed T - if the tips of the plant were removed and analyzed

9. Age of plant	Right justified, zero filled. Insert age of plant in years.
10. Number of Tissue Data Forms filled in	Insert the number of Tissue Data Forms which have been completed in association with this particular Macrophyte Specimen Data Form.
11. Comments	Plain language comments can be inserted as needed. Such comments can include details about the sampling locality or other information relevant to the sample.

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ICES REPORTING FORMAT FOR CONTAMINANTS IN MARINE BIOTA

MACROPHYTE SPECIMEN DATA FORM

4. Form identifier code M, S
5. Individual or bulked specimens analysed
6. Specimen or sub-sample number
7. Number of individuals in homogenate (if analysed as homogenate)
8. Whole plant or tip
9. Age yrs
10. Number of tissue data forms filled in
11. Comments (e.g., details of sampling locality, etc.,)

6. Exchange of data on contaminants in fish and shellfish via magnetic tape

(ICES, rev version May 1986)

Exchange of data on CONTAMINANTS IN FISH AND SHELLFISH on magnetic tape
==========

This document should be read in parallel with "INTERIM REPORTING FORMAT FOR CONTAMINANTS IN FISH AND SHELLFISH", published by ICES (May 1986 revised version). This Format is described in three versions: the full ICES version, the JMP version and the HELCOM version, the latter two are essentially subsets of the first with reference to the particular requirements of the JMP and HELCOM. The magnetic tape format (described below) can be used in conjunction with either of the three versions, where reference is made to specific sections of the full ICES version an equivalent section appears in the JMP and HELCOM subsets.

Magnetic tapes sent to ICES should be:

9 tracks, odd parity
800, 1600 or 6250 bpi (1600 bpi is strongly preferred)
EBCDIC OR ASCII —
no label
fixed blocked records
logical record length 80 characters
blocksize, a suitable multiple of 80

The exchange-format consists of the following record types:

01 - Fish/Shellfish Contaminant Master Record
04 - Specimen Data Record
07 - Tissue Data Record
10 - Contaminant Data Record
13 - Plain Language Record

An entire series of data obtained on one species from one station/area should be organized as follows:

```
01-Master Record
13-Plain Language Records (optional,max 5)
   04-Specimen Record
   13-Plain Language Records (optional,max 5)
      07-Tissue Record
      13-Plain Language Records (optional,max 5)
         10-Contaminant Records (as many as needed)
      07-Tissue Record
      13-Plain Language Records (optional,max 5)
         10-Contaminant Records (as many as needed)
```



```
      .
      .
      .
   04-Specimen Record
   13-Plain Language Records (max 5)
      07-Tissue Record
      13-Plain Language Records (max 5)
         10-Contaminant Records (as many as needed)
      07-Tissue Record
      .
      .
      .
01-Master Record (next series)
```

Record layout for the different record types:

Fish/Shellfish Contaminant Master Record:
****

Field name	Columns	Valid values	Comments
Record id	1-2	01	Mandatory.
Laboratory code	3-6	See Annex 2	Mandatory. All Annexes refer to "Interim Reporting Format...".
Year	7-8	74-present year	Mandatory. Last two digits of the year in which the samples were taken.
Sequence number	9-11	001-999	Mandatory, right justified, zero filled. Consecutive numbers for one year, starting with 001 for the first Master Form in a year, 002 for the second, etc.
Sampling country	12-13	See Annex 3	Mandatory.
Organization code	14-14	0 or 1	Mandatory. If data submitted for ICES insert 1, otherwise 0.
	15-15	0 or 1	Mandatory. If data submitted for JMP insert 1, otherwise 0.
	16-16	0 or 1	Mandatory. If data submitted for BMP insert 1, otherwise 0.
			NOTE: At least one of the columns 14-16 must contain a '1'. Data can be submitted for more than one organization.
Sampling area indicator	17-17	I,J,B, or C	Mandatory.
	18-26	See Annex 4-1 to 4-3	Mandatory, left justified, space filled.

ICES statistical rectangles	27-31	Spaces or see Annex 4-1	Left justified, space filled. ICES will store the sampling area in two systems: in the system reported by the originator, and in the ICES system (ICES will manually convert all sampling areas to the ICES system). It would therefore be a nice help if the participating laboratories would report the sampling area also in the ICES system. If a sample has been collected over more than one statistical rectangle then the most significant rectangle should be inserted. If col 17='I' then simply copy the content of col 18-22 to col 27-31. Col 31 can be used to designate a sub-rectangle within an ICES rectangle, see Annex 4-1.
Sampling date	32-37		Mandatory. In the form YYMMDD, if day not reported then DD=00.
Purpose	38-38	0-4	Mandatory.
Species code	39-60	See Annex 5	The species must be reported in at least one of the coding-systems. If, for instance, only RUBIN-code is used, then insert this code in col 39-46 and fill col 47-56 and 57-60 with spaces.
RUBIN	39-46		
US-NODC	47-56		
ICES	57-60		
Number of individuals used for analysis	61-63	001-999	Mandatory, right justified, zero filled.
Number of specimen data records following this Master record	64-65	01-99	Mandatory, right justified, zero filled.
Filler	66-80	Spaces	For future use.

Specimen Data Record:
****

Field name	Columns	Valid values	Comments
Record id	1-2	04	Mandatory.
Individual or bulked specimens analyzed	3-3	I,P or H	Mandatory.
Specimen or sub-sample number	4-5	00 or 01-99	Mandatory, right justified, zero filled. If the entire sample was analyzed as a homogenate then insert 00.
Number of individuals in homogenate	6-8	000 or 001-999	Mandatory, right justified, zero filled. If individual organisms were analyzed then insert 000.
Length min	9-13		Total length in mm. If individuals analyzed: space filled. If bulk(s) analyzed: space filled if value missing, otherwise right justified, zero filled.
max	14-18		Total length in mm. If individuals analyzed: space filled. If bulk(s) analyzed: space filled if value missing, otherwise right justified, zero filled.
mean or individual	19-23		Total length in mm. Mandatory, right justified, zero filled. (mean=arithmetic mean)
standard deviation	24-28		If individuals analyzed: space filled. If bulk(s) analyzed: space filled if value missing, otherwise right justified, zero filled.

Weight	min	29-33		Whole weight of ungutted fish in grams. If individuals analyzed: space filled. If bulk(s) analyzed: space filled if value missing, otherwise right justified, zero filled.
	max	34-38		Whole weight of ungutted fish in grams. If individuals analyzed: space filled. If bulk(s) analyzed: space filled if value missing, otherwise right justified, zero filled.
	mean or individual	39-43		Whole weight of ungutted fish in grams. Mandatory, right justified, zero filled. (mean=arithmetic mean)
	standard deviation	44-48		If individuals analyzed: space filled. If bulk(s) analyzed: space filled if value missing, otherwise right justified, zero filled.
Sex		49-49	0-5	Mandatory.
Age	min	50-51		In year(s). If individuals analyzed: space filled. If bulk(s) analyzed: space filled otherwise if value missing, right justified, zero filled.
	max	52-53		In year(s). If individuals analyzed: space filled. If bulk(s) analyzed: space filled otherwise if value missing, right justified, zero filled.
	mean or individual	54-55		In year(s). Mandatory, right justified, zero filled. (mean=arithmetic mean)
Shell weight of molluscs		56-60		In grams to 1 decimal place. Space filled if value missing, otherwise right justified, zero filled. NOTE: For numerical fields with implied decimals,"zero filled" means both leading and trailing zeroes. This is also valid for a couple of items in the Tissue Data Record and the Contaminant Data Record
Number of tissue data records belonging to this Specimen record		61-62	01-99	Mandatory, right justified, zero filled.
Filler		63-80	Spaces	For future use.

Tissue Data Record:
****

Field name	Columns	Valid values	Comments
Record id	1-2	07	Mandatory.
Specimen or sub-sample number	3-4	00 or 01-99	Mandatory, right justified, zero filled. Same number as in the corresponding Specimen Data Record.
Replicate number	5-5	0 or 1-6	Mandatory. If no replicates analyzed then insert 0.
Tissue analyzed	6-7	01-06 or 99	Mandatory, right justified, zero filled.
Tissue weight	8-13		In grams to two implied decimals. Space filled if value missing, otherwise right justified, zero filled.
Dry Weight (%)	14-17		In % to two implied decimals. Space filled if value missing, otherwise right justified, zero filled.
Fat Weight (%) and method code	18-21		In % to two implied decimals. Space filled if value missing, otherwise right justified, zero filled.
	22-22		Space filled if method not given and/or value missing in col 18-21, otherwise insert code. Each laboratory gives its own code and it is recommended to describe, or give reference to, method used in Plain Language Records.
Number of contaminants analyzed	23-24	01-99	Mandatory, right justified, zero filled.
Filler	25-80	Spaces	For future use.

Contaminant Data Record:
****

Field name	Columns	Valid values	Comments
Record id	1-2	10	Mandatory.
Contaminant	3-7	See Annex 6	Mandatory, left justified, space filled.
Basis	8-8	W,D or L	Mandatory.
Unit	9-9	M or U (See Annex 6)	Mandatory. M=mg/kg, U=µg/kg.
Qualifier	10-10	space,> or <	See page 13 in "Interim Reporting Format...".
	11-11	space,A,S,Q, R or M	See page 13 in "Interim Reporting Format...".
Value	12-18		In 'unit' to three implied decimals. Space filled if value missing, otherwise right justified, zero filled. NOTE: At first glance it seems to make no sense to allow for 'value missing', since in that case a Contaminant Record would not be filled in, but in col 11 is given option M='Missing value: original data erroneous or missing' and therefore a unique value must be assigned for 'value missing'.
Method	19-21		Mandatory, left justified, space filled. See page 14 in "Interim Reporting Format...".
I/C (Inter-calibration)	22-23	See Annex 7	Mandatory.
Detection limit	24-27		Limit to three implied decimals. Space filled if not given, otherwise right justified, zero filled.
Analytical lab-code	28-31	See Annex 2	NOTE: This item is now Mandatory
% Recovery of Standard	32-35		New Item. % Recovery of internal standard to one implied decimal. Space filled if not appropriate otherwise right justified, zero filled.
Filler	36-80	Spaces	For future use.

Plain Language Record:
****

Field name	Columns	Valid values	Comments
Record id	1-2	13	Mandatory.
Comments	3-62		Left justified, space filled.
Filler	63-80	Spaces	

C. HARMFUL SUBSTANCES IN BIOTA AND SEDIMENTS

C. II Trend Monitoring of Contaminants in the Coastal Zone

1. Choice of monitoring organisms

a) General considerations

In this chapter a general overview is given for using coastal organisms to monitor temporal trends in contaminants such as heavy metals, organochlorines and petroleum hydrocarbons. It must be emphasized, however, that the use of any of the proposed species is optional.

In selecting the monitoring organisms, priority has been given to species with a wide geographical range within the Baltic, availability, adequate size of the organism, and of course their ability to reflect levels of contaminants in the coastal zone. If all the proposed types of organisms are included in a monitoring programme, the results will provide information on the concentrations of contaminants in species at several trophic levels, from algae to predatory birds.

When the monitoring species have been selected, a choice should be made regarding the tissues to be analyzed for each type of contaminant. The general considerations for this choice have been given in Section C.I.1.3.b) of the Guidelines.

In vertebrates, the essential metals copper and, to a lesser extent, zinc are under homeostatic control (1). Because of that, copper in vertebrates will not reflect environmental concentrations adequately. The essential and the non-essential metals except mercury are mainly stored/accumulated in the liver and kidney. These

tissues, and the liver in particular due to its larger size, are therefore the recommended tissue(s) for analysis of heavy metals in vertebrates (see Table C.6.).

Mercury occurs in almost similar concentrations in muscle and liver. However, as muscle tissue generally shows less variation in mercury concentration due to variations in nutritional status, etc. (2), muscle tissue is recommended for mercury analysis.

As heavy metals in macroalgae and bivalves are under poor or no homeostatic control (3), these organisms are highly recommended for metal monitoring purposes.

Although most metals are mainly concentrated in the kidney and hepatopancreas, it is not practicable to distinguish between tissues in bivalves. Thus, homogenates of the entire soft tissues, including the adductor muscle of bivalves, should be used for heavy metal analysis.

In macroalgae, only those parts developed in the sampling year should be used for metal analysis.

In the Baltic Sea, salinity varies from a few parts per thousand in the Bothnian Bay to more than 20 $^{o}/_{oo}$ in the Kattegat. These changes in salinity are accompanied by changes in chemical speciation and bioavailability of heavy metals (4). Thus, comparison of heavy metal concentrations in coastal species from different areas should be done with great care.

The organic xenobiotics to be monitored are non-polar substances mainly associated with lipids. In vertebrates, they should be determined in the lipid fraction of the muscle tissue. In bivalves, analysis should be performed in the lipid fraction of the whole soft body.

TABLE C.6. RECOMMENDED TISSUES FOR ANALYSIS OF HARMFUL SUBSTANCES IN COASTAL AREAS

	Hg Total	Cd	Cu	Zn	Pb	PCBs	DDTs	HCB	HCH	Chlordane	Dieldrin
Bivalves	ST	ST	ST	ST	ST	ST	ST	ST	ST		
Flounder	M	L	L(M)	L(M)	L	L(M)	L(M)	L(M)	L(M)		
Pike	M	L	L+M	L+M	L	M	M	M	M		
Eel	M	L	M	M	L	M	M	M	M		
Birds	M	K	L+M	L+M	L(M)	M	M	M	M		
Fucus	A	A	A	A	A						

ST = Soft Tissue
M = Muscle
L = Liver
K = Kidney
A = Apice
() = Optional

The ability of macroalgae to reflect environmental concentrations of non-polar xenobiotics has not been documented convincingly (3). Thus, macroalgae are not recommended for monitoring organochlorines and petroleum hydrocarbons.

Organic tin

It is recommended that measurements of total organic tin be made in water from harbour areas and in coastal areas with ship and pleasure boat traffic. This proposal is made because it has been shown that the organo-tin compounds used in antifouling paints have serious effects on the larvae and other life stages of molluscs and other marine organisms.

Detailed procedures for the measurement of organic tin and total tin in sea water are described in (15).

b) Macroalgae

Several species of macroalgae have been used successfully worldwide to monitor heavy metals in the coastal zone. The most extensively used species is probably *Fucus vesiculosus*, which has shown good linearity in response to environmental concentrations of heavy metals (3). As *Fucus vesiculosus* is a dominant species in the algal zone in many coastal areas around the Baltic Sea, this species is an obvious candidate for monitoring purposes in the Baltic.

The accumulation of heavy metals in macroalgae is under some influence of their growth rate (3). In spring and summer, growth may be so intense that accumulation of heavy metals cannot "catch up" with the production of metal binding sites in the algae. As the degree of this "growth dilution" varies according to time and site of sampling, care must be taken to sample algae outside their growth season.

c) Bivalves

The blue mussel *Mytilus edulis* is by far the most commonly used organism to monitor contaminants in the coastal zone. Its extensive use in both national and international monitoring programmes is due to a number of qualities that almost makes this species the monitoring organism par excellence (3).

Mytilus edulis can be found in the coastal zone in nearly all temperate waters. It is widely distributed in the Baltic Sea area except in the northern part of the Bothnian Bay and the inner parts of the Gulf of Finland (5).

Going from the Belt Sea to the Baltic Proper, *Mytilus edulis* penetrates into deeper waters and becomes sparse in the "tidal zone". Their growth rate also decreases and they attain a smaller size. As size, age and growth rate are closely linked to vertical distribution in the Baltic, mussels for time trend monitoring should be sampled at approximately the same depth each year.

In *Mytilus edulis*, the reproductive cycle and nutritional status cause rather large variations in the different body constituents during the year (6) and, as the various contaminants differ in their affinity to lipids, proteins and carbohydrates, mussels must be sampled in periods showing only minor changes in body constituents. Otherwise, high or low concentrations of contaminants in mussels may result, even though the environmental concentrations remain the same.

d) Fish

To gain additional information about contaminant loads in the coastal zone, monitoring in predatory fish might be useful. Fish are not, as macroalgae and bivalves, strictly stationary. Thus, they are not equally suited for monitoring very localized differences in contaminant concentrations (7). However, if they are caught in their nonmigratory periods, they may provide valuable information about contaminant loads in restricted areas.

In the Guidelines for the Second Stage it was recommended to use young "yellow" eel (*Anguilla anguilla*, pike (*Esox lucius*) and flounder (*Platichthys flesus*). Of these, only eel and pike are stationary species. Flounder, used in many countries in their coastal monitoring programmes, make annual migrations and are thus less suitable for trend monitoring studies in the coastal zone.

Eel can be found in all areas of the Baltic Sea, while the geographical range of pike is more restricted. Flounder cannot be caught in the Gulf of Bothnia (8), and belongs more to the open part of the sea in the eastern and northern part of the Baltic.

Since the coastal programme is optional, and especially applies to the regional interests of the individual Contracting Party, it is recommended that Contracting Parties make a choice of species for trend study purposes from among the three mentioned above, considering the comparability, stationariness and availability of the species along their coasts.

As both organochlorine and heavy metal concentrations in fish show annual variations not related to variations in contaminant load (9, 3, 10), sampling

should be carried out in periods during which concentrations show only minor variations. For the three proposed species, sampling should be carried out in the autumn.

e) Birds

Birds can provide valuable information to a monitoring programme because they indicate the concentrations of contaminants at a higher trophic level than fish; they also integrate the contaminant levels of the individual fish or shellfish they consume. Additionally, the data obtained may be more easily correlated with population figures than can be done with fish or bivalves because bird populations and reproduction are more easily determined.

In the choice of bird species for test organisms, diet is an important factor. The bird species chosen must feed primarily on marine organisms of a particular type and not on miscellaneous materials, such as human garbage.

The experiences from the last 20 years show that fish-consuming birds can be useful also in a Baltic Monitoring Programme, as part of a temporal trend monitoring system (11). Comparing available data on organochlorine levels in eggs of fish-consuming birds and in herring both used as test organisms in a Baltic trend study has disclosed a much clearer trend for the bird egg series than for the fish series (11). The bird species used was guillemot (*Uria aalge*) nesting in a colony in the central part of the Baltic. Eggs of another bird species of the family Alcidae, the razorbill (*Alca torda*), also nesting in colonies along the Baltic coast, have shown much the same homogeneity with respect to both organochlorine levels and mercury (12). Since the two bird species are stationary within

the Baltic Sea area all the year and thus no far distance migration occurs, their eggs can be used for chemical analysis in trend studies.

These two species have also been monitored biologically with respect to both population status, reproductive rate, and egg shell thickness (12, 13). These parameters are easy to follow compared to reproduction and population parameters for fish and bivalves.

The alcids do not nest in all areas of the Baltic. As an alternative, two other fish-consuming bird species can be recommended, the arctic tern (*Sterna paradisae*) and the merganser (*Mergus serrator*). Since these two species migrate during winter time to areas outside the Baltic, fledglings are recommended as material for analysis. They shall be sampled just before they are nearly full grown and are just about to learn to fly. At this stage they will represent the fish fauna of the area where they have been hatched.

2. Sampling procedures

a) General considerations

For a discussion of the general considerations regarding sampling procedures and the information which should accompany the sample, see Section C.I.1.3.b) of the Guidelines.

The purpose of the sampling procedures outlined below is to ensure that samples are as homogeneous as possible. By following the procedures, more rigorous comparisons between yearly samples or between samples from different areas can be made.

For all the proposed species, sampling should be done once a year.

b) Macroalgae

In addition to load, growth rate is the most important factor influencing heavy metal concentrations in *Fucus vesiculosus*. To minimize bias due to variations in growth rate, the algae should be sampled in October/November at 1-2 m depth. Apices (i.e., tips of the algae, representing recent growth) of similar length, free of epiphytic organisms, from several plants (ca. 20) should be sampled. Fertile portions of the plants should not be collected.

Sample data parameters and reporting system

At each sampling occasion, a sample information sheet (Table C.3. in Section C.I.) should be filled out.

The apices should be cleaned for epiphytic organisms using a polyethylene or glass spatula and rinsed well in sea water from the collection site. After draining as well as possible, the pooled sample is placed in a polyethylene bag which should be sealed and labelled. The sample is kept deep frozen until analysis.

c) Bivalves

Mytilus edulis should be collected at appropriate coastal sampling sites at a depth of 2-5 m in October/November. In the northern Baltic Proper and the Gulf of Bothnia, samples may be taken down to a depth of 15 m.

As both growth rate (size/age relationship) and dominant size within populations vary greatly among locations in the Baltic, it is not practical to specify overall requirements on these bases.

Nonetheless, it is important to obtain samples as comparable as possible. This can partly be achieved by choosing similar sized individuals (within 5 mm). As a check of similarity between samples, the Condition Factor could be calculated in a subsample of 10 specimens. The Condition Factor of each *Mytilus edulis* in a subsample is calculated as

$$\frac{W}{L^3}$$

where W (in mg) is the soft body weight and L (in cm) is the shell length.

The Condition Factor is largely independent of size and usually attains values between 3-5 in autumn.

Each sample to be analyzed should contain 25-50 individuals (at least 50 g soft body weight).

Sample data parameters and reporting system

See Sections C.I.1.3.e) and C.I.1.5 or C.I.1.6 of the Guidelines for recommendations on this subject.

If sampling is carried out in non-turbid waters, the cleansing period in sea water can be omitted.

d) Fish

The sampling procedures for each species have been designed to obtain a sample during a relatively stable period for the organism in terms of migration. More detailed discussion of the factors considered can be found in Section C.I.1.3.b) of the Guidelines.

Flounder should be sampled close to the shore to ensure that they will be representative of the area of catch. Because the fish start to migrate to deeper waters in

late fall, samples must be collected before that time. Samples of flounder should consist of females in their third year of life caught in September using a gill net. The maximum recommended sampling depth is 20 m.

Male pike should be sampled near the coastline in August/September. No other requirements have been specified because no correlations for size or age versus levels of organochlorines have been found. Mercury levels correlate only with size but not with age (14). This correlation can be calculated and a mean value and the variance for a standardized weight can be estimated for comparison between years. Thus, it does not seem important to select only fish of the same age class or year class for monitoring mercury or organochlorines.

Mercury levels have been shown to differ between sexes in pike of similar size (14), and thus only one sex should be used. The males have been chosen because a higher proportion of males is generally caught using gill nets.

Female yellow eel should be sampled in August (July) near the coastline. As age is difficult to determine, no standard age requirement can be made, except that the eels are in the yellow stage. The total length of the fish should be 40-50 cm.

To avoid the direct influence of river inputs of contaminants and to get more regionally representative data, the fish should be caught at some distance from river mouths.

For all the three species, a sample should consist of at least 20 specimens from the same site. The same number of specimens should be collected each year.

Specimen data requirements and reporting system

The overall recommendations on this subject can be found in Sections C.I.1.3.d) and C.I.1.5 or C.I.1.6 of the Guidelines. All of the information requested in Section C.I.1.3.d) should be obtained for samples of the three species discussed here, with the following exceptions. Information on the maturity of gonads should be recorded for flounder, but not for pike and eel. Age need not be determined for eel and pike; for flounder, however, the age should be determined and recorded according to the number of otolith annual rings.

e) Birds

Birds should be sampled from only one area or population for each bird species. A total sample should include ten birds per species, each bird collected from a different nest or duckling brood.

Fledgling birds should be collected just before they learn to fly. Because of the rapid growth rate during this period of the bird's life, it is important that the birds collected are at the same age in days for each year of sampling.

When collecting the birds, the main types of food for that species and area should be identified, as within a species the birds may have different feeding habits in the different parts of the Baltic.

Ten undeveloped eggs of the alcids shall be collected in the beginning of May. The female normally produces only one egg. If the egg is collected in the beginning of the nesting season, the egg will be replaced by the female with a new one.

The eggshell shall be saved after drying at room temperature. Measurements of eggshell thickness shall be carried out in order to monitor the quality of the eggshell.

Young merganser leave the nest just after hatching and follow the mother in the gathering of food. When the ducklings are essentially fully grown in August, they will begin to fly. They should be collected just before that, with only one bird taken per brood.

Arctic terns remain in their nesting colonies until the time comes for them to learn to fly. Thus, they should be collected from their colonies (one per nest) just before they are ready to leave them.

Productivity (number of eggs and production of fledglings) and population size (number of birds) can be followed by field work in the investigated area during the reproductive season.

A sample information sheet should be filled out with information on sampling area, main sources of food and other relevant information (similar to Table C.3. in Section C.I.).

Specimen data requirements and reporting system

After collection, the bird should be grasped just under the base of the wing and, using the forefinger and the thumb, it should be squeezed until the heart is stopped. For each bird, the total body weight in grams should be recorded and the age should be estimated in number of days. The size should be estimated by measuring the wing length from the carpal joint to the tip of the longest feather. This measurement should be made on a flattened wing. Thereafter, each whole bird should be placed in a polyethylene bag. The air should

be squeezed out and the bag sealed and labelled. The birds should be deep frozen as soon as possible thereafter and should remain frozen until they are prepared for analysis.

Each bird should be assigned a specimen identification number and the relevant information concerning it should be recorded on a Bird Specimen Data Form.

Upon dissection, the liver and kidneys should be completely and carefully removed and weighed.

3. Subsampling and handling

a) Tool handling during dissection and subsampling

The tools and procedures described in Section C.I.1.3.g) of the Guidelines should be carefully observed to avoid contamination of the samples during preparation for analysis.

b) Subsampling procedures

Macroalgae

Prior to analysis, the algae sample is homogenized. Subsamples are taken for dry weight and heavy metal determinations.

Bivalves

The procedures for initial handling of a bivalve mollusc have been described in Section C.I.1.3.e) of the Guidelines. An appropriate sample size should be chosen so that at least duplicate analyses for heavy metals, organochlorines and duplicate dry weight and lipid (in case of organochlorines) determinations can be carried out.

Fish

A description of the preparation of fish tissue samples is contained in Section C.I.1.3.g) of the Guidelines.

Birds

Working with a partly thawed specimen, the pectoral muscle of the fledgling birds should be sampled from under the subcutaneous fat layer. The right pectoral muscle should be used. For every fifth bird, a duplicate sample should be taken using the left pectoral muscle.

The liver should be removed carefully and completely for use in copper, zinc and lead analyses. As the kidneys of a bird are easy to obtain, they should be carefully removed for cadmium analysis.

Bird eggs shall be blown after drilling a hole at the equator of the egg. Only undeveloped eggs can be blown. The soft egg material shall then be homogenized.

4. References in chapter C II (Trend Monitoring of Contaminants in the Coastal Zone)

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14. Olsson, M. 1975. Mercury levels as a function of size and age in Northern Pike, one and five years after the mercury ban in Sweden. AMBIO 5: 73-76.

15. Jensen, A. & Cheng, J. 1989. Determination of organic and total tin in sewater by extraction and graphite furnace atomic absorption spectroscopy. ICES Techniques in Marine Environmental Sciences No. 8. (In prep.)

C. HARMFUL SUBSTANCES IN BIOTA AND SEDIMENTS

C. III Harmful Substances in Sediments

1. Contaminants in sediments

A critical assessment of data on contaminants in sediments from the Baltic Sea is presently being conducted by ICES for the Helsinki Commission. Although this work has not yet been completed, the preliminary conclusion is that monitoring of contaminant concentrations in sediments in the open areas of the Baltic Sea need only be conducted at a frequency of once every five, or possibly even ten, years depending on the sedimentation rate and other characteristics of the specific areas to be monitored. However, for certain substances, particularly phosphorus, there may be a requirement for more frequent monitoring, at least in certain areas. Detailed proposals for monitoring contaminants in sediments will be prepared by ICES after the critical assessment of sediment data in the Baltic Sea, and other relevant information, have been completed and evaluated.

2. Reporting format for contaminants in sediments

International Council for
the Exploration of the Sea

INTERIM REPORTING FORMAT FOR CONTAMINANTS IN SEDIMENTS

June 1987

International Council for the Exploration of the Sea

June 1987

INTERIM REPORTING FORMAT FOR CONTAMINANTS IN SEDIMENTS

(VERSION 1, DRAFT 2)

1. INTRODUCTION

This Interim Reporting Format for Contaminants in Sediments has been drafted on the basis of the present requirements of monitoring programmes for contaminants, primarily trace metals, in sediments.

2. OVERVIEW OF THE SYSTEM

Four types of forms have been included in the system: a Sediment Sampling Methods Form, a Sediment Analytical Methods Form, a Sediment Contaminant Master Form, and a Sediment Contaminant Data Form.

The Sediment Sampling Methods Form records information on the type of sediment sampler used, the method of sample storage or preservation, the method of grain size analysis, and the method of structural analysis of a sediment core. The number of Sediment Sampling Methods Forms to be filled in will depend on the number of combinations of methods used.

The Sediment Analytical Methods Form records information on the method of dissolution or extraction used on a sediment sample and the method of analysis for a particular contaminant by the analytical laboratory involved for the year concerned. A series of these forms should be filled in and should precede the first Master form; they should describe the methods associated with any contaminant subsequently reported. One form will need to be filled out for each contaminant according to the method of extraction or dissolution of the sediment used. Thus, the number of forms will depend on the number of contaminant-extraction combinations used in the data series being reported. Each contaminant analysis reported later on the Sediment Contaminant Data Forms is unambiguously associated with a specific Sediment Analytical Methods Form by means of a Method Identifier code.

The Sediment Contaminant Master Form serves as the master record for the series of data obtained at one sampling station on one sampling occasion. This form provides general information on when and where the sample was obtained.

The Sediment Contaminant Data Form provides the record for the data on the sediment samples obtained. The initial parameter to be recorded is the sediment grain size range which has been analyzed, after which is recorded the depth of the core slice analyzed. Thereafter, the concentrations of all parameters determined can be recorded, linked with the Sediment Analytical Methods Form by the Analytical Method Identifier Code. This form is very flexible, providing the opportunity to record data on any contaminant or parameter measured in a grab sample or at all depths in a sediment core.

Positions have been provided on all forms for comments and additional information. These positions should be used for information which will assist in the interpretation of the data, thus making the data more valuable for future use. One example is the inclusion of information on methods which have been changed from previous years.

It is intended that all types of relevant data be reported on these forms. If codes for any items are missing, they can be developed on request to the ICES Environment Officer.

3. DETAILED DESCRIPTION

3.1 General

The following description applies to all forms. The page number should be inserted in the upper left-hand corner of each page. On the first page of the series, i.e., on the first Sediment Sampling Methods Form, the total number of pages of forms associated with the series should be included.

3.2 Sediment Sampling Methods Form

A Sediment Sampling Methods Form is filled in for each combination of the type of sediment sampler used, method of sample storage used, method of grain size analysis used and method of structural analysis of the core used. The Sampling Method Identifier Code from the appropriate Sediment Sampling Methods Form is inserted on the Sediment Contaminant Master Form as a reference for the sampling methods used for that sample.

The Sediment Sampling Methods Form should be filled in according to the following description.

Item	Code Description
1. Form identifier code	The code "S" identifies the Sediment Sampling Methods Form.
2. Country	Insert the IOC Country code (see Annex 2), the same information appears on the Sediment Master Form.
3. Year	Insert the last two digits of the sampling year.
4. Reporting Laboratory	Insert the four-letter mnemonic code (see Annex 1) for the laboratory reporting the data, the same information appears on the Sediment Master Form.
5. Analytical Laboratory	Insert the four-letter mnemonic code (see Annex 1) for the analytical laboratory concerned.

6. Sampling method identifier	Insert a method identifier code for the combination of methods described on this form. It is suggested that a code "1" is given for the first Sampling Methods form for each type of sampler, then "2", "3", etc., for additional forms, if more than one combination of type of sampler with other methods given on this form is used. This item links the method information which follows with the sample taken in the Sediment Contaminant Master, and the code should be inserted in item 14 of this Master Form.
7. Type of sampler	Insert code according to the code list given in Annex 6.
8. Method of storage/ sample preservation	Insert code for method of sample storage or sample preservation. Each laboratory should maintain detailed records of the methods used and should assign a code number for each of these methods.
9. Method of grain size analysis	Insert code for method of grain size analysis. Each laboratory should maintain detailed records of the methods used and should assign a code number for each of these methods.
10. Method of structural analysis	Insert code for method of structural analysis of cores. Each laboratory should maintain detailed records of the methods used and should assign a code number for each of these methods.
11. Comments	Plain language comments can be inserted as needed.

3.3 Sediment Analytical Methods Form

The Sediment Analytical Methods Form should be used to record all details of the methods used in the extraction or dissolution of the sediment and the analysis of a particular contaminant. This removes the need to duplicate this information repeatedly on other forms where data for the same contaminant/extraction method are included. Thus, a series of these forms is filled in, preceding the first Sediment Contaminant Master, to cover all combinations of methods used by a particular laboratory in that year. The methods forms can therefore be referred to repeatedly from the Sediment Contaminant Data Forms.

Often it will only be necessary to fill in one Sediment Analytical Methods Form for each contaminant reported. If more than one method description applies to any one contaminant, then additional methods forms should be filled in for that contaminant, each with a different Analytical Methods Identifier (see description of Method Identifiers on page 6). If analysis of a contaminant is carried out by more than one analytical laboratory, the appropriate number of methods forms should be filled in for each laboratory.

It should be noted that the sampling and analytical methods are described on these two forms by means of codes which should be linked to a full description of the details, including types of equipment, that is retained in the laboratory. If the laboratory is participating in the Joint Monitoring Programme, these details should also be sent to the Secretariat of the Oslo and Paris Commissions.

The Sediment Analytical Methods Form should be filled in as follows.

Item	Code Description
1. Form identifier code	The code "A" identifies the Sediment Analytical Methods Form.
2. Country	Insert the IOC Country code (see Annex 2), the same information appears on the Sediment Master Form.
3. Year	Insert the last two digits of the sampling year.
4. Reporting Laboratory	Insert the four-letter mnemonic code (see Annex 1) for the laboratory reporting the data, the same information appears on the Sediment Master Form.
5. Analytical Laboratory	Insert the four-letter mnemonic code (see Annex 1) for the analytical laboratory concerned.
6. Parameter/Contaminant code	Insert the code for the parameter/contaminant for which the methods data apply, according to the codes given in Annex 4.
7. Analytical method identifier	Insert a method identifier code for the combination of methods described on this form. It is suggested that a code "1" is given for the first Methods Form for each contaminant, then "2", "3" etc. for additional forms, if more than one combination of methods has been used for that contaminant. This item links the

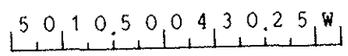
	method information which follows with any analysis (of the contaminant identified in item 6 by the laboratory identified in item 5) for which this Identifier code is reported on a Sediment Contaminant Data Form (item 7).
8. Method of extraction/ condition when extracted	Insert code for the method of sediment extraction, using the appropriate code for the general method (see Annex 5) in the first three columns and a code for the individual laboratory version of this method, assigned by the data originator, in the fourth column. Each laboratory should maintain detailed records of the methods used and should assign a code number for each of these methods. An additional column for reporting the condition of the sample when extracted should be filled in according to the codes in Annex 5.
9. Method of analysis of parameter/ contaminant	Insert a 3-digit code for the method used to analyse the contaminant. Each laboratory submitting data should maintain detailed records of the methods used to analyse the contaminants and should assign a 3-digit code number for each of these methods. The appropriate code should be inserted here.
10. Limit of detection of parameter/ contaminant	Right justified, zero filled. Insert the detection limit for the method of analysis used to determine the contaminant reported in the units appropriate to that contaminant (see Annex 4). The limit of detection is defined here as that concentration of analyte which yields an analytical response equal to three times the standard deviation of the complete procedural blank.
11. I/C (intercalibration)	Insert a 2-digit code for the relevant intercalibration exercise in which the laboratory has most recently participated, according to the list in Annex 3.
12. Comments	Plain language comments can be inserted as needed. Such comments can include information of relevance to the interpretation of the methods used, description of an intercalibration exercise, etc.

3.4 Sediment Contaminant Master Form

One Sediment Contaminant Master is filled in for each sampling station or area on each sampling occasion. The Master Form should be filled in according to the following description.

Item	Code Description
PART I	
1. Form identifier code	The code "M" identifies the Sediment Contaminant Master Form.
2. Sampling country	Insert the IOC Country Code (see Annex 2).
3. Sequence number or core number	Insert the number of the Master Form being filled in beginning with 0001 for the first Master form in a year, 0002 for the second, etc.
4. Sampling date	Indicate the year (last two digits only), month, and date of sampling. (The day may be omitted, if desired, and the applicable columns filled with zeroes.)
5. Sampling time	Insert the time at which sampling commenced (optional parameter to permit correspondence with the Hydrographic Data Format).
6. Sampling area co-ordinates	Insert coordinates of the sampling area, as follows: Note that decimal fractions of minutes are recorded and not seconds. (1) A sample taken at the coordinates 59^0 20.15'N 18^0 55.3'E would be designated according to the coordinates system as: 5 9 2 0.1 5 1 8 5 5.3 0 E (2) A sample taken at the coordinates 50^0 10.5'N 4^0 30.25'W

would be designated according to the coordinates system as:



7. Depth of water	Insert the depth of water at the sampling station in meters. If the sampling takes place in an estuary, the water depth should be taken from a sea level map.
8. Total length of core	If a sediment core has been taken, insert the total depth of the core in centimeters.
9. Estimated sedimentation rate	If desired, record the estimated sedimentation rate at the area of sampling, in mm/yr.
PART II	
10. Reporting Laboratory	Insert the four-letter mnemonic code for the laboratory reporting the data (see Annex 1).
11. Organization code	Indicate the organization(s) for which data are submitted, as follows: I - International Council for the Exploration of the Sea J - Joint Monitoring Programme of the Oslo and Paris Commissions B - Baltic Monitoring Programme of the Helsinki Commission
12. Sampling area designation (eg. JMP Area (sub-area))	If the organization code J has been included in item 11 (above) then insert, left-justified, the code number of the JMP area sampled. If appropriate, insert a sub-area or station number, leaving one blank after the area number.
13. Purpose of monitoring	Indicate purpose of monitoring according to the following code: 0 - No specific purpose 2 - Geographical distribution 3 - Temporal trend determination
14. Number of Sediment Contaminant Data Forms following this Master	Right justified, zero filled. Insert number of Contaminant Data Forms following this Master.

15. Sampling method identifier code	Insert appropriate sampling method identifier code.
16. Comments	Insert plain language comments, as needed.

3.5 Sediment Contaminant Data Form

The Sediment Contaminant Data Form records data on the various 'parameters' which are associated with each sample. In this connection, parameters include measurements such as grain size distribution and chemical analyses of sediment constituents as well as information on the portions of the sediment for which the subsequent data are being reported, e.g., grain size fraction analyzed and depth of core slice. A list of the parameters, with associated codes and units, is given in Annex 4. A Sediment Analytical Methods Form should be filled in for all parameters involving chemical or radiochemical measurements.

Each line on the Contaminant Data Form (items 4-8) records data for one 'parameter'. One form can include data for all parameters associated with a sample taken at a particular station. Grain size fraction and, for sediment cores upper and lower core slice depth, are 'key' parameters; all parameters which follow a particular grain size and core depth record are assumed to be parameters associated with analyses of the portion of the sample of that grain size range (and at that core depth, if applicable). Thus, the first record line on a Sediment Contaminant Data Form should contain data on the maximum grain size of this fraction, and the second line should contain data on the percentage of the total sediment this fraction comprises. If a grab sample is analyzed, no data need be given on the depth of the sediment sample. If a core is analyzed, the next two lines of data should give the upper and lower depth of the core slice analyzed. Thereafter, all parameters associated with that portion of the sample are reported on the following lines. A new core depth record and/or a new maximum of grain size indicates that subsequent parameter records are associated with analyses of this next portion of sample.

The Sediment Contaminant Data Form should be filled in according to the following description:

Item	Code Description
1. Year	Insert the last two digits of the sampling year, the same as on the Sediment Contaminant Master Form.
2. Sequence number (core number)	Insert the Sequence number (or core number), the same information as on the Sediment Contaminant Master.

3. Form identifier code

The code "C" identifies the Sediment Contaminant Data Form.

4. Parameter/contaminant code

Insert the code for the parameter or contaminant according to the codes given in Annex 4.

5. Analytical Method Identifier code

If relevant, insert the appropriate Analytical Method Identifier code corresponding to that inserted on the associated Sediment Analytical Methods Form for the methods of extraction and analysis for the contaminant or geochemical parameter reported on this line.

6. Qualifier

In the first column, indicate whether the value reported under item 7 should be qualified by:

> - greater than, or
< - less than.

If not, leave blank. When not detected, use < the detection limit in numerical terms.

In the second column, insert a Validation Flag as follows:

Blank - unspecified or quality control check has not been made

A - Acceptable: data found acceptable during quality control checks.

S - Suspect Value: data considered suspect (but not replaced) by the data originator on the basis of either quality control checks or recorder/ instrument/platform performance.

Q - Questionable Value: data considered suspect (but not replaced) during quality control checks by persons other than those responsible for its original collection, e.g., a data centre.

R - Replaced Value: erroneous or missing data has been replaced by estimated or interpolated value - method by which replacement values have been derived should be described in plain language records.

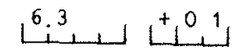
M - Missing Value: original data erroneous or missing.

7. Parameter value or Contaminant concentration

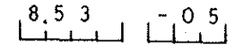
Decimal point justified, blank filled. Insert the parameter value or the concentration of the contaminant as obtained from the analysis of the sediment, in the appropriate units (see Annex 4), using scientific notation. Note - Do not use normalized values. The value should be inserted in the four columns on the left, with the sign and power of ten on the three columns on the right.

Examples:

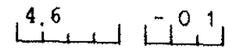
A grain size of 63 µm should be written:



A lead concentration of 85.3 mg/kg should be written:



A moisture content of 0.46 (=46%) should be written:



9. Analytical Laboratory

Insert the four-letter mnemonic code (see Annex 1) for the analytical laboratory. This is a mandatory item and should correspond to the code reported on the Sampling and Analytical Methods Form which contains details of the methods applied.

10. Comments

Plain language comments can be inserted as needed. Such comments can include information relevant to the interpretation of the contaminant values or sample data.

All questions or inquiries concerning this format and its use should be directed to the ICES Environmental Officer, I C E S, Palægade 2, DK-1261 Copenhagen K, Denmark.

Reporting format for contaminants in sediments

ANNEX 1

LABORATORY CODES FOR THE REPORTING AND ANALYTICAL LABORATORIES OF THE MONITORING PROGRAMMES - BY COUNTRY

DENMARK	HFLD	Miljøstyrelsens Havforureningslaboratorium Jægersborg Allé 1B DK-2920 Charlottenlund DENMARK
	SCSS	State Chemical Supervision Service Mørkhøj Bygade 26-H DK-2860 Søborg DENMARK
	ICDK	Danish Isotope Center Skelbækgade 2 DK-1717 Copenhagen V DENMARK
FINLAND	IMRF	Institute of Marine Research P.O. Box 33 00931 Helsinki 93 FINLAND
G.D.R	AHZL	Arbeithygienisches Zentrum der chemischen Industrie GERMAN DEMOCRATIC REPUBLIC
	FREI	Forschungsinstitut für NE-Metalle Freiberg 9200 GERMAN DEMOCRATIC REPUBLIC
	BHIR	Bezirkshygiene-Institut Rostock GERMAN DEMOCRATIC REPUBLIC
	IGDR	Institut für Meereskunde Akademie der Wissenschaften de DDR Seestrasse 15 DDR-2530 Rostock-Warnemünde GERMAN DEMOCRATIC REPUBLIC

F.R.G	AHHG	FEDERAL REPUBLIC OF GERMANY
	BFRG	Bundesforschungsanstalt für Fischerei Laboratory für Radioökologie der Gewässer Wüstland 2, 2000 Hamburg 55 FEDERAL REPUBLIC OF GERMANY
	BFKG	Bundesforschungsanstalt für Fischerei Institut für Küsten- und Binnenfischerei Palmaille 9 2000 Hamburg 50 FEDERAL REPUBLIC OF GERMANY
	BFGG	BUNDESANSTALT FÜR GEWAESSERKUNDE Kaiserin-Augusta-Anlagen 15-17 D-5400 Koblenz FEDERAL REPUBLIC OF GERMANY
	DHIG	Deutsches Hydrographisches Institut Bernhard-Nocht-Strasse 78 D-2000 Hamburg 50 FEDERAL REPUBLIC OF GERMANY
	NLWG	Niedersaechsisches Landesamt fuer Wasserwirtschaft An der Scharlake 39 D - 3200 Hildesheim FEDERAL REPUBLIC OF GERMANY
	WGEG	Wasserguetestelle Elbe Focksweg 32 a D - 2103 Hamburg 95 FEDERAL REPUBLIC OF GERMANY
	FITG	Fresenius Institut Chemische und Biologische Laboratorien GmbH D-6204 Taunusstein-Neuhof FEDERAL REPUBLIC OF GERMANY
	ISHG	Institute Schumacher Laboratorium für Wasser-, Abwasser- und Ölanalytik Dr. Harald Schumacher (Dipl. Chem.) Sophie-Dethleffs-Str. 4 D-2240 Heide FEDERAL REPUBLIC OF GERMANY

LWKG Landesamt für Wasserhaushalt und Küsten
Saarbrückenstraße 38
D-2300 Kiel 1
FEDERAL REPUBLIC OF GERMANY

VUCG Veterinäruntersuchungsamt Cuxhaven
Schleusenstraße
D-2190 Cuxhaven
FEDERAL REPUBLIC OF GERMANY

POLAND

IMWP Institute of Meteorology and Water Management
Wazingtona 42
81-342 Gdynia
POLAND

VHRP Veterinary Hygiene Research Station
Gdansk
POLAND

SFIP Sea Fisheries Institute
Aleja Zjednoczenia 1
81-345 Gdynia
POLAND

IIAP Institute of Ichthyology
Agriculture Academy
Szczecin
POLAND

SWEDEN

HRFS Institute of Hydrographic Research
National Board of Fisheries
Box 2566
403 17 Göteborg
SWEDEN

UCKS University College of Kalmar
Inst. Natural Sciences and Technology
Box 905
S-391 29 Kalmar
SWEDEN

MNHS Miljögiftsövervakning PMK
Naturhistoriska riksmuseet
Box 50007
S-104 05 Stockholm
SWEDEN

NSLS National Swedish Environment Protection Board
Naturvårdsverkets Special Analytical Laboratory
Box 1302
S-171 25 Solna
SWEDEN

NWLS National Swedish Environment Protection Board
Naturvårdsverkets Water Quality Laboratory
S-75008 Uppsala
SWEDEN

LCRS Swedish Environment Protection Board
Laboratory for Coastal Research
S-170 11 Drottningholm
SWEDEN

SERI Swedish Environmental Research Institute
Sten Sturegatan 42
Box 5207
S-402 24 Gothenburg
SWEDEN

IAMK Institutionen för analytisk och marin kemi
Chalmers tekniska högskola
S-412 96 Gothenburg
SWEDEN

SLKS AB Svensk Laboratoriet (SWELAB)
Box 903
S-391 29 Kalmar
SWEDEN

USSR

DBST Department of the Baltic Sea
Academy of Sciences
Paldiski Street 1
200031 Tallinn
USSR

ASLR Academy of Sciences of Latvian SSR
Institute of Biology
Riga 229021
USSR

Reporting format for contaminants in sediments

ANNEX 2

IOC COUNTRY CODES FOR BMP MEMBER COUNTRIES

COUNTRY	CODE
Denmark	26
Finland	34
German Democratic Republic	96
Germany, Federal Republic of	06
Poland	67
Sweden	77
Union of Soviet Socialist Republics	90

Reporting format for contaminants in sediments

ANNEX 3

CODE LIST FOR INTERCALIBRATION EXERCISES ON CONTAMINANTS IN SEDIMENTS

CODE	INTERCALIBRATION EXERCISE
7A	First JMG intercalibration exercise on trace metals in marine sediments - 1980
7B	Second JMG intercalibration exercise on trace metals in marine sediments - 1983
7C	Baltic Sediment Intercalibration Exercise - Part A - 1983
7D	Baltic Sediment Intercalibration Exercise - Part B - 1984
7E	ICES First Intercalibration Exercise on Trace Metals in Marine Sediments (1/TM/MS) - 1984
7Z	Other Intercomparison/Intercalibration Exercise on Trace Metals in Sediments - Describe in comments.

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8A	JMG Intercalibration on Analyses of PCBs in Sediments - 1987
8Z	Other Intercomparison/Intercalibration Exercise on Organochlorines in Sediments - Describe in comments.

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Reporting format for contaminants in sediments

ANNEX 4

CODE LIST FOR PARAMETERS/CONTAMINANTS

PARAMETER/CONTAMINANT NAME	CODE	UNITS	METHODS FORM REQUIRED	MANADATORY FIELD
Grain size, maximum	GSMAX	µm	no	yes
Amount in grain size fraction	GSAMT	none	no	yes
Moisture content	MOCON	none	no	yes
Core slice depth from surface, upper	SDEPU	cm	no	yes
lower	SDEPL	cm	no	yes
Replicate number	REPLN		no	no
Aluminium	AL	g/g	yes	no
Arsenic	AS	g/g	yes	no
Cadmium	CD	g/g	yes	JMP
Chromium	CR	g/g	yes	no
Cobalt	CO	g/g	yes	no
Copper	CU	g/g	yes	JMP
Iron	FE	g/g	yes	no
Lithium	LI	g/g	yes	no
Mercury	HG	g/g	yes	JMP
Manganese	MN	g/g	yes	no
Nickel	NI	g/g	yes	no
Lead	PB	g/g	yes	JMP
Scandium	SC	g/g	yes	no
Titanium	TI	g/g	yes	no
Vanadium	VA	g/g	yes	no
Zinc	ZN	g/g	yes	JMP
Lindane (γ-HCH)	HCHG	g/g	yes	(JMP)
Polychlorinated biphenyls	PCB	g/g	yes	(JMP)
Chlorobiphenyl congeners (CBs) (by IUPAC numbers)	CB28	g/g	yes	(JMP)
	CB52	g/g	yes	(JMP)
	CB101	g/g	yes	(JMP)
	CB118	g/g	yes	(JMP)
	CB138	g/g	yes	(JMP)
	CB153	g/g	yes	(JMP)
	CB180	g/g	yes	(JMP)
Sum of these CBs	PCB7	g/g	yes	(JMP)
Hexachlorobenzene	HCB	g/g	yes	no

.... continued ....

ANNEX 4 - continued

Redox potential	REDOX	mV	no	no
Total Nitrogen	NTOT	g/g	yes	no
Total Phosphorus	PTOT	g/g	yes	no
Calcium carbonate	CACO3	g/g	yes	no
Inorganic carbon	CINOR	g/g	yes	no
Organic carbon	CORG	g/g	yes	no
Loss on ignition	LOIGN	g/g	yes	no
Cesium-137	CS137	mBq/g	yes	no
Lead-210 (unsupported)	PB210	mBq/g	yes	no
Plutonium-239	PU239	mBq/g	yes	no
Plutonium-240	PU240	mBq/g	yes	no
Radium-226	RA226	mBq/g	yes	no
Thorium-234 (unsupported)	TH234	mBq/g	yes	no

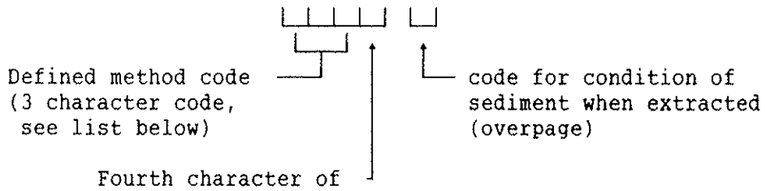
Reporting format for contaminants in sediments

ANNEX 5

CODE LIST FOR METHODS OF SEDIMENT EXTRACTION

The following codes should be used in reporting the 'Method of extraction / condition when extracted' item on the Sediment Analytical Methods form, (item 8).

The field for this item has the following format:



SEDIMENT EXTRACTION METHOD CODE	DESCRIPTION
a) inorganic contaminants	
HCL	Extraction with dilute HCl.
HAC	Extraction with acetic acid.
HNO	Extraction with 1:1 HNO_3.
AQR	Extraction with "aqua regia" (HNO_3/HCl = 1:3).
SAD	Extraction with mixture of strong mineral acids without HF (e.g. $HClO_4$ and/or H_2SO_4 in addition to HNO_3).
HFO	"Total" digestion with mineral acids including HF, in open vessels, evaporation of excess HF before analysis.
HFC	as HFO above, but with digestion performed in closed vessels (pressurized decomposition).
HFB	as HFC above, but with complexation of excess HF with H_3BO_3.
ALK	Alkaline fusion digestion.
SCE	Selective chemical extraction of metal species in particulate phases (e.g. by hydroxylamine, oxalate, H_2O_2, dithionite, ammonium acetate), define procedure used in comments.

... continued

SEDIMENT EXTRACTION METHOD CODE	DESCRIPTION
b) organic contaminants	
EXP	Extraction of (organic) contaminants by shaking with polar solvents.
EXN	Extraction of (organic) contaminants by shaking with non-polar solvents.
EXC	Extraction of (organic) contaminants by continuous treatment in a Soxhlet or similar apparatus.
EXH	Separation of (organic) contaminants from sediment slurries using water steam distillation.
EXO	Other principles of extraction/separation of (organic) contaminants from sediment samples, define procedure used in comments.

CODE FOR CONDITION OF SEDIMENT WHEN EXTRACTED	DESCRIPTION
1	Oven dried.
2	Freeze dried.
3	Fresh material.

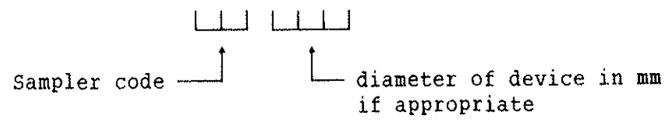
Reporting format for contaminants in sediments

ANNEX 6

CODE LIST FOR TYPES OF SEDIMENT SAMPLER

The following codes should be used in reporting the 'Type of sampler' item on the Sediment Sampling Methods form, (item 7).

The field for this item has the following format:



SAMPLER CODE	DESCRIPTION
GS	Grab sampler.
BC	Box corer.
GC	Gravity corer.
VC	Vibro-corer.
DC	Diver operated corer.
DD	Drilling device.
OS	Other sampling device: define in comments.

INTERIM REPORTING FORMAT FOR CONTAMINANTS IN SEDIMENTS
VERSION 1 (DRAFT 2)

SEDIMENT SAMPLING METHODS FORM

1. Form identifier code [S]
2. Country [ ]
3. Year [ ]
4. Reporting Laboratory [ ]
5. Analytical Laboratory [ ]
6. Sampling method identifier [ ]
7. Type of sampler / diameter (mm) [ ] [ ]
8. Method of storage/sample preservation [ ]
9. Method of grain size analysis [ ]
10. Method of structural analysis [ ]
11. Comments _____

Page __ of __ pages

INTERIM REPORTING FORMAT FOR CONTAMINANTS IN SEDIMENTS
VERSION 1 (DRAFT 2)

SEDIMENT ANALYTICAL METHODS FORM

1. Form identifier code [A]
2. Country
3. Year
4. Reporting Laboratory
5. Analytical Laboratory
6. Parameter/Contaminant code
7. Analytical method identifier
8. Method of extraction / condition when extracted
9. Method of analysis of parameter/contaminant
10. Limit of detection of parameter/contaminant
11. Intercalibration exercise
12. Comments

INTERIM REPORTING FORMAT FOR CONTAMINANTS IN SEDIMENTS
VERSION 1 (DRAFT 2)

SEDIMENT CONTAMINANT MASTER

PART I

1. Form identifier code [M]
2. Country
3. Sequence number (core number)
4. Sampling date Y Y M M D D
5. Sampling time
6. Sampling area coordinates o ' o ' E/W
7. Depth of water (m)
8. Total length of core (cm)
9. Estimated sedimentation rate (mm yr^{-1})

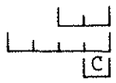
PART II

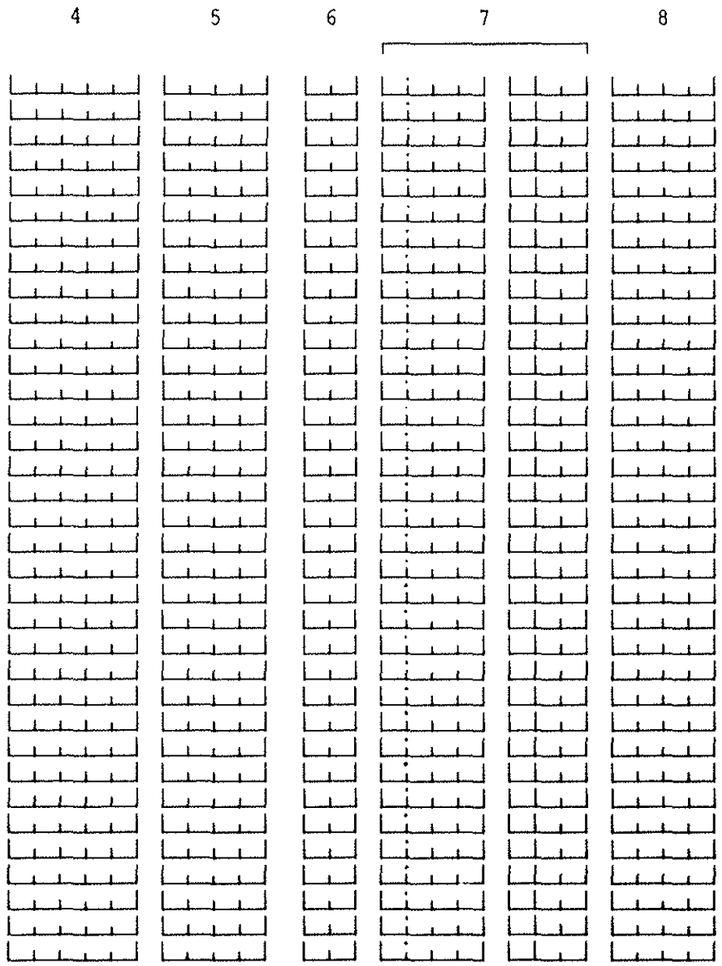
10. Reporting Laboratory
11. Organization
12. Sampling area designation (eg. JMP area)
13. Purpose of monitoring
14. Sampling method identifier code
15. Number of sample data forms following this master
16. Comments

INTERIM REPORTING FORMAT FOR CONTAMINANTS IN SEDIMENTS
VERSION 1 (DRAFT 2)

SEDIMENT CONTAMINANT DATA FORM

1. Year
2. Sequence/Core number
3. Form identifier code





4. Parameter/contaminant code
5. Analytical method identifier code
6. Qualifier
7. Parameter/contaminant value (using scientific notation)
8. Analytical Laboratory
9. Comments _____

_____

_____

BALTIC SEA ENVIRONMENT PROCEEDINGS

No. 1 JOINT ACTIVITIES OF THE BALTIC SEA STATES WITHIN THE FRAMEWORK OF THE CONVENTION ON THE PROTECTION OF THE MARINE ENVIRONMENT OF THE BALTIC SEA AREA 1974-1978 (1979)*

No. 2 REPORT OF THE INTERIM COMMISSION (IC) TO THE BALTIC MARINE ENVIRONMENT PROTECTION COMMISSION (1981)

No. 3 ACTIVITIES OF THE COMMISSION 1980
- Report on the activities of the Baltic Marine Environment Protection Commission during 1980
- HELCOM Recommendations passed during 1980
(1981)

No. 4 BALTIC MARINE ENVIRONMENT BIBLIOGRAPHY 1970-1979 (1981)

No. 5A ASSESSMENT OF THE EFFECTS OF POLLUTION ON THE NATURAL RESOURCES OF THE BALTIC SEA, 1980
PART A-1: OVERALL CONCLUSIONS
(1981)*

No. 5B ASSESSMENT OF THE EFFECTS OF POLLUTION ON THE NATURAL RESOURCES OF THE BALTIC SEA, 1980
PART A-1: OVERALL CONCLUSIONS
PART A-2: SUMMARY OF RESULTS
PART B: SCIENTIFIC MATERIAL
(1981)

No. 6 WORKSHOP ON THE ANALYSIS OF HYDROCARBONS IN SEAWATER
Institut für Meereskunde an der Universität Kiel, Department of Marine Chemistry, March 23 - April 3, 1981
(1982)

No. 7 ACTIVITIES OF THE COMMISSION 1981
- Report of the activities of the Baltic Marine Environment Protection Commission during 1981 including the Third Meeting of the Commission held in Helsinki 16-19 February 1982
- HELCOM Recommendations passed during 1981 and 1982
(1982)

No. 8 ACTIVITIES OF THE COMMISSION 1982
- Report of the activities of the Baltic Marine Environment Protection Commission during 1982 including the Fourth Meeting of the Commission held in Helsinki 1-3 February 1983
- HELCOM Recommendations passed during 1982 and 1983
(1983)

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* out of print

No. 9 SECOND BIOLOGICAL INTERCALIBRATION WORKSHOP
Marine Pollution Laboratory and Marine Division of the National Agency of Environmental Protection, Denmark, August 17-20, 1982, Rønne, Denmark
(1983)

No. 10 TEN YEARS AFTER THE SIGNING OF THE HELSINKI CONVENTION
National Statements by the Contracting Parties on the Achievements in Implementing the Goals of the Convention on the Protection of the Marine Environment of the Baltic Sea Area
(1984)

No. 11 STUDIES ON SHIP CASUALTIES IN THE BALTIC SEA 1979-1981
Helsinki University of Technology, Ship Hydrodynamics Laboratory, Otaniemi, Finland
P. Tuovinen, V. Kostilainen and A. Hämäläinen
(1984)

No. 12 GUIDELINES FOR THE BALTIC MONITORING PROGRAMME FOR THE SECOND STAGE
(1984)

No. 13 ACTIVITIES OF THE COMMISSION 1983
- Report of the activities of the Baltic Marine Environment Protection Commission during 1983 including the Fifth Meeting of the Commission held in Helsinki 13-16 March 1984
- HELCOM Recommendations passed during 1983 and 1984
(1984)

No. 14 SEMINAR ON REVIEW OF PROGRESS MADE IN WATER PROTECTION MEASURES
17-21 October 1983, Espoo, Finland
(1985)

No. 15 ACTIVITIES OF THE COMMISSION 1984
- Report on the activities of the Baltic Marine Environment Protection Commission during 1984 including the Sixth Meeting of the Commission held in Helsinki 12-15 March 1985
- HELCOM Recommendations passed during 1984 and 1985
(1985)

No. 16 WATER BALANCE OF THE BALTIC SEA
A Regional Cooperation Project of the Baltic Sea States; International Summary Report
(1986)

No. 17A FIRST PERIODIC ASSESSMENT OF THE STATE OF THE MARINE ENVIRONMENT OF THE BALTIC SEA AREA, 1980-1985; GENERAL CONCLUSIONS
(1986)

No. 17B FIRST PERIODIC ASSESSMENT OF THE STATE OF THE MARINE ENVIRONMENT OF THE BALTIC SEA AREA, 1980-1985; BACKGROUND DOCUMENT
(1987)

No. 18 ACTIVITIES OF THE COMMISSION 1985
- Report on the activities of the Baltic Marine Environment Protection Commission during 1985 including the Seventh Meeting of the Commission held in Helsinki 11-14 February 1986
- HELCOM Recommendations passed during 1986
(1986)*

No. 19 BALTIC SEA MONITORING SYMPOSIUM
Tallinn, USSR, 10-15 March 1986
(1986)

No. 20 FIRST BALTIC SEA POLLUTION LOAD COMPILATION
(1987)*

No. 21 SEMINAR ON REGULATIONS CONTAINED IN ANNEX II OF MARPOL 73/78 AND REGULATION 5 OF ANNEX IV OF THE HELSINKI CONVENTION
National Swedish Administration of Shipping and Navigation; 17-18 November 1986, Norrköping, Sweden
(1987)

No. 22 SEMINAR ON OIL POLLUTION QUESTIONS
19-20 November 1986, Norrköping, Sweden
(1987)

No. 23 ACTIVITIES OF THE COMMISSION 1986
- Report on the activities of the Baltic Marine Environment Protection Commission during 1986 including the Eighth Meeting of the Commission held in Helsinki 24-27 February 1987
- HELCOM Recommendations passed during 1987
(1987)*

No. 24 PROGRESS REPORTS ON CADMIUM, MERCURY, COPPER AND ZINC
(1987)

No. 25 SEMINAR ON WASTEWATER TREATMENT IN URBAN AREAS
7-9 September 1986, Visby, Sweden
(1987)

No. 26 ACTIVITIES OF THE COMMISSION 1987
- Report on the activities of the Baltic Marine Environment Protection Commission during 1987 including the Ninth Meeting of the Commission held in Helsinki 15-19 February 1988
- HELCOM Recommendations passed during 1988
(1988)

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