

# COMMISSION OF THE EUROPEAN COMMUNITIES

COM(80) 831 final  
Brussels, 17 December 1980

Proposal for a  
COUNCIL DIRECTIVE  
on methods for the surveillance and monitoring of the  
environments affected by wastes from the titanium  
dioxide industry

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(Presented by the Commission to the Council)

• COM(80) 831 final

1. Introduction

In implementation of the Action Programme of the European Communities on the Environment of 20 December 1973, the Council adopted on 20 February 1978 (78/176/EEC) the Directive on waste from the titanium dioxide industry, the aim of which is the prevention and progressive reduction, with a view to its elimination, of pollution caused by waste from the titanium dioxide industry.

The Directive stipulates the measures to be taken to achieve this end, in particular the prior authorizations for waste discharge issued by the competent authority of the Member State in whose territory the waste is produced and the drawing up of programmes for the progressive reduction of pollution with a view to its ultimate elimination.

Article 7 of the Directive also stipulates that the Commission will, within one year of notification of the Directive, submit to the Council a proposal on the procedures for the surveillance and monitoring of the environments affected by waste from this industry.

2. Comments on the proposed Directive

2.1. Procedure for drafting the Directive

The Commission has brought together a group of national experts to assist it in drafting the technical part of the Directive. This group, which has met three times, has advised the Commission on the methods of surveillance and monitoring to be used and, in particular, on the list of the relevant parameters to be measured throughout the Community and on the minimum annual frequency of sampling with respect to the different environments receiving waste from the titanium dioxide industry.

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2.2. Aims of the Directive

This Directive will - on the basis of the parameters selected - provide the means of identifying pollution levels in those parts of the environment affected - from a physical, chemical, biological and ecological point of view - by discharges of titanium dioxide waste, of understanding pollution trends and of assessing the progressive reduction of pollution caused by these wastes.

3. Comments on titanium dioxide

3.1. Pigmentary properties

In its crystalline form, titanium dioxide is currently regarded as having the highest refractory index of all the conventional pigments in both its allotropic forms, i.e. anatase and rutile. As a white pigment, it is incomparable and irreplaceable.

3.2. Utilization

On account of its pigmentary properties this product has come into widespread use in many areas, principal among which are paints and varnishes, paper, plastics, rubber, floor coverings, ceramics, and synthetic fibres. The paint and varnish industry accounts for more than half of the total consumption.

3.3. Titanium dioxide manufacturing processes

Manufacturing of titanium dioxide pigments consists in preparing high purity titanium dioxide from an ore in which it is present already but mixed with other substances.

There is no industrial process for manufacturing titanium dioxide by which titanium dioxide can be extracted selectively. The only two existing industrial processes involve digestion of the ore followed by selective operations to extract the titanium compound.

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In a final stage, the titanium dioxide is obtained from the intermediate compound.

These two processes are known as the 'sulphate' and 'chloride' processes respectively.

In the first process, the ore is opened up by sulphuric acid and then the titanium hydroxide is precipitated selectively. Finally, the titanium oxide is obtained by calcination of the hydroxide.

In the chloride process, the ore is chlorinated and the resultant titanium tetrachloride is first separated from the other chlorides mechanically and then distilled. Finally, the titanium oxide is obtained by oxidation of the titanium tetrachloride.

In Europe, the sulphate process is the one most commonly used.

### 3.3.1. Sulphate process

The ore used is either ilmenite or slag.

- Ilmenite is a mineral complex of titanium and iron containing on average 55%  $TiO_2$  and approximately 30% iron;
- slag is obtained from iron ore which contains some 70%  $TiO_2$  10% iron as oxide plus metallic iron.

The main stages involved in the separation of  $TiO_2$  from other compounds are to dissolve nearly all the ore and then to precipitate the titanium selectively as hydroxide. This hydroxide is then calcined to give the titanium oxide.

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The main waste products are:

- (i) residue from the ore digestion process.  
This insoluble residue is composed of unattached ore and insoluble silica;
- (ii) ferrous sulphate in the form of heptahydrate ( $\text{SO}_4\text{Fe}\cdot 7\text{H}_2\text{O}$ ), commonly known as "copperas", resulting from the reduction and crystalization of the solutions formed from the ilmenite digestion stage. This liquor is made up of a complex salt of titanium, ferrous and ferric sulphate;
- (iii) mother liquor produced from the separation of titanium gel after hydrolysis by filtration and/or decantation.  
  
This liquor is made up of a solution of ferrous sulphate in sulphuric acid. It also contains the sulphates of the metals present in trace amounts in the raw material;
- (iv) various gel wash waters and waters from the scrubbing of the calcination gases. They contain dilute sulphuric acid, traces of the salts of iron, titanium and other metals present in the ore.

### 3.3.2. Chloride process

The standard ore used in the chloride process is natural rutile, a scarce ore very rich in  $\text{TiO}_2$  (approximately 96%), or alternatively concentrates generally obtained from ilmenite or synthetic rutile.

The process consists in obtaining titanium tetrachloride from the high-temperature chlorination of a rutile and carbon mixture. After condensation, purification and oxidation of the titanium

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tetrachloride, the titanium dioxide is obtained; the regenerated chlorine is recycled.

This process generates only small amounts of waste but is little used in Europe. But although this process uses concentrates of ilmenite, it does involve an iron removal stage. This pre-treatment leads to the same problems as those associated with waste from the sulphate process.

### 3.4. Composition of the wastes from the titanium dioxide industry

- 3.4.1. In the sulphate process, the main wastes from the industry are: insoluble substances, copperas, mother liquors or strong acids, wash waters or weak acids.

In the chloride process, the main process wastes are the chlorides of manganese, magnesium, calcium, sodium, aluminium, chromium, iron, vanadium,  $ZrO_2$ , titanium dioxide,  $TiCl_4$  and chlorine.

- 3.4.2. The detailed composition of titanium dioxide manufacturing wastes varies according to the ores used, the manufacturing processes and the way in which the solid, liquid and gaseous wastes are treated at each stage of the process.

#### Liquid wastes

Generally, these wastes have the following features:

- high acidity;
- a high iron content, mainly in the form of iron sulphate (0.8 to 14%);
- suspended solids (oxides of Ti, Si, Fe, Al, Ca, Zr);

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- dissolved metals, normally in the following concentrations (in mg/l):

Mn: 400 to 1400; Cr: 4 to 225; Zb: 1 to 125; V: 75 to 110;  
Ni: 4 to 50; Cu: 0.1 to 10; Pb: 3 to 6; As: 0.04 to 4;  
Cd: <0.1; Hg: <0.005.

#### Solid wastes

These are made up of copperas, arising from the processing of raw materials in the sulphate process and by the neutralized residues of ore or slag. Copperas contains some 90% of iron sulphate present as  $\text{FeSO}_4 \cdot 7(\text{H}_2\text{O})$ ; the remaining fraction is made up of titanium dioxide, magnesium sulphate, free sulphuric acid and water of crystallization. The neutralized residues from the sulphate process contain ore or slag which is insoluble at 200°C in 90% sulphuric acid together with filter aids; the waste products from the chloride process comprise unattacked ores, coke and metal hydroxides.

#### Gaseous waste

Principal among these are  $\text{SO}_2$  and  $\text{SO}_3$  which are formed from the residual sulphuric acid (100-120 kg per tonne  $\text{TiO}_2$ ) and the sulphur contained in the fuel used to initiate combustion when the hydrated titanium oxide is calcined. Depending on the conditions in which calcination takes place, the ratio of  $\text{SO}_2:\text{SO}_3$  (calculated as  $\text{H}_2\text{SO}_4$ ) ranges from 1:1.92 to 1:2.80. The amount of gaseous waste produced in the manufacture of one tonne of titanium dioxide ranges from 12 to 22.4 kg  $\text{SO}_2$  and 53.6 to 60.2 kg  $\text{SO}_3$ .

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4. Principal methods of waste disposal used by the titanium dioxide industry

The effects on biotopes due to the various properties of the wastes are closely linked to the nature of the receiving medium. Sea waters are most frequently used for this, but in certain special circumstances the wastes may be discharged in fresh water, stored on land or injected underground into deep strata.

4.1. Discharges at sea

The two principal methods of disposal are:

- (i) by pipeline from the coast
- (ii) discharge at sea from special ships

The first method normally requires prior dilution of the effluent and a discharge point which favours rapid dispersal of the effluent. The acidic solutions are neutralized by dilution and by the buffer effect.

The second method, namely discharge at sea, necessitates the use of specially-equipped tanker ships which enable the acid effluent to be discharged directly into the ship's wake, thus providing rapid dilution and conveyance of the effluent to greater depths. This method is used for soluble wastes (ferrous sulphate and acid solutions).

When titanium dioxide waste is discharged from a ship, the predominant short-term chemical impact is caused by the reduction in pH. According to the guidelines laid down by the Oslo Convention on discharges of titanium dioxide waste, the pH of the receiving waters must reach 6 within a maximum of 5 minutes after discharge.

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Since the pH of wastes varies according to their source and that different types of ship may be used (differences in size and speed), the following measurements should be taken before the permitted level of discharge is decided:

- (i) the pH at different dilutions of the receiving water should be calculated in such a way as to produce a pH/dilution curve;
- (ii) on the basis of this curve, if a pH of 6 or more must be reached within 5 minutes, the requisite dilution can be calculated;
- (iii) the rate of discharge can be worked out depending on the required dilution on the basis of experimental work carried out on the ship itself (using tracers) or by calculation (using general formulae for the minimum dilution in the ship's wake).

Another possibility is direct measurement of the pH of the waste dissolved in the ship's wake during the discharge operation.

These conditions also presuppose that the dilution engendered by the propeller wake will be adequate to reduce the density difference between the sea water/waste mixture and the receiving water to a sufficiently low level to prevent the formation of dense layers which would impede the subsequent mixing process.

#### 4.2. Discharge into surface waters

This method of disposal is used for acid solutions once they have been neutralized. The neutralization products are disposed of with the ferrous sulphates.

4.3. Storage on land

This type of storage on "tips" is used by small capacity titanium dioxide plants and is most frequently used for the small fraction of insoluble waste materials (i.e. those which cannot be dissolved by sulphuric acid when the ore is opened up).

Furthermore, the acid effluent from this industry can be neutralized by calcium carbonate and slaked lime ( $\text{Ca(OH)}_2$ ) and then stored on land. The same is true for the ferrous sulphates.

4.4. Injection into underground strata

With this disposal method, the previously neutralized acid solutions are pumped into subterranean strata.

4.5. Recycling of the waste products

In some production plants the dilute sulphuric acid solutions are recovered, reconcentrated and used again. In others, varying amounts of copperas (ferrous sulphates) are roasted and converted to sulphuric acid by the addition of sulphur.

Other potential outlets for these products have been contemplated, for example: iron oxide pigments, flux for the cement industry, weed killer and in water treatment for the production of drinking water. But only small quantities of copperas are involved in these applications.

5. Methods of surveillance and monitoring of the environments affected

The methods for the surveillance and monitoring of the environments receiving  $\text{TiO}_2$  wastes discharged at sea or into fresh water, stored or dumped on land or injected into underground strata are based on the provisions of Annex II B of the Directive on waste

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from the titanium dioxide industry, adopted by the Council on 20 February 1978 (78/176/EEC).

5.1. Where the waste is discharged into fresh water or dumped/ immersed at sea, the following three components shall be monitored: the water column, living organisms and the sediment. Periodic checks on the state of the area affected by the discharges will make it possible to monitor changes in the environments affected.

Monitoring of the following parameters shall be compulsory:

(a) In the water column

- pH, the salinity of sea water, dissolved oxygen, turbidity, hydrated iron oxides and hydroxides in suspension, concentration of dissolved salts, toxic metals;

(b) In suspended solids and in sediment

- hydrated iron oxides and hydroxides and toxic metals;

(c) In living organisms (having due regard to the species typical of the discharge zone)

- the levels of iron, titanium, chromium, cadmium, mercury, vanadium, nickel, copper, zinc and lead;

(d) Flora and fauna

Diversity and relative abundance.

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Monitoring of the following parameters shall be optional:

- the level of chromium, vanadium, zinc, lead, nickel, copper, manganese in the water column and sediment and the amounts of suspended solids.

5.2. In the case of storage, tipping or injection, the monitoring shall include:

- tests to ensure that surface waters and ground waters have not been contaminated. These tests relate to a control of acidity, the levels of iron (dissolved and in suspension), calcium, chlorides, titanium and sulphates and to the environment of the storage sites, i.e. the permeability, porosity, structure and stability of the soil, and to a general ecological assessment of the area surrounding the tipping, storage or injection point.

## 6. Sampling

The greatest attention should be paid to the taking of samples which should be regarded as a preliminary phase in the analysis process.

The methods of sampling should be such as to ensure that the conditions in which the measurements are taken can be reproduced and that the methods are representative of the conditions prevailing in the surrounding environment. Article 4(4) of this proposal for a directive lays down the general rules to be observed for sampling.

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7. Minimum annual frequency for sampling and for the analysis of parameters

The frequency of sampling and analysis is the minimum number of sampling operations and analyses to be carried out each year in respect of the various parameters described in the foregoing chapter and depending on the different environments affected by the discharge of waste from the titanium dioxide industry.

The samples should be taken having due regard to local environmental and discharge conditions, i.e. whether the discharges are intermittent or continuous. The exact site of the sample taking and its distance from the discharge point should be specified in each case by the competent authority in each Member State.

Where the results of measurements taken on samples from the discharge zone are consistent or show a significant improvement in environmental quality over the previous year, the frequency of sampling and analysis may, with the prior agreement of the Commission, be reduced by the Member State concerned.

Accordingly, the Member States should communicate to the Commission the results of these measurements and state the frequency they propose to adopt.

8. Preservation and transport of samples

As stated in Article 5, the type of container used for sampling and packaging should not influence the analytical results.

The volume of such sampling bottles should be such as to enable the specified measurements to be carried out in optimum conditions of reproducibility. Since the characteristics of the sample water may alter quite rapidly under the action of the various organisms it contains, such changes as may occur between the moment of sampling and analysis should be kept to a strict minimum by carrying out the analyses as rapidly as possible. .../...

Many investigations have been made with a view to recommending methods by which water samples can be stored without their composition being affected. But it is impossible to lay down any hard and fast rules which would cover every case and every situation and to which there would be no exceptions. The samples needed to measure the parameters should be transported to the laboratory for examination at the earliest opportunity. If immediate analysis is not possible, the samples should be kept under conditions where the containers are protected from any outside contamination and no change in the state of their contents is possible.

9. Methods of measurement

The reference methods of measurement for each parameter selected for the surveillance and monitoring of the environments affected are shown in the Annexes to this proposal.

Laboratories in the Member States using other methods must ensure that the results obtained are equivalent or comparable to those given in the Annexes.

The result obtained from the samples and methods of measurement must be representative of the true average quality of the water analysed. The various operations needed to achieve this result may lead to errors; it is vital that such errors be detected and then eliminated or reduced.

The Annexes to this proposal for a Directive lay down the conditions in which the measurements of certain parameters must be carried out in view of the risk that their value may change during the transport or protracted storage of the water samples in containers. This is the case with temperature and pH.

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10. Action at international and Community level

In the campaign against freshwater and sea water pollution, the Community is a Contracting Party to international conventions aimed at reducing and eventually eliminating this type of pollution. The conventions are:

- The Convention for the Prevention of Marine Pollution from Land-based Sources (Paris Convention) (75/437/EEC);
- Convention for the Protection of the Mediterranean Sea against Pollution (Barcelona Convention);
- Protocol for the Prevention of Pollution of the Mediterranean Sea by Dumping from Ships and Aircraft (77/585/EEC);
- Convention on the Protection of the Rhine against Chemical Pollution (77/585/EEC).

On 17 May 1980, the Community also signed the Protocol on the protection of the Mediterranean Sea against pollution from Land-based sources.

Specific articles of these Conventions either ban the dumping or discharge of certain substances such as acid compounds whose composition and quantity are such that they may adversely affect the quality of sea water or make the dumping or discharge of specific substances, such as titanium, chromium and acid compounds subject to the prior granting of a specific permit or to appropriate programmes and measurements to reduce pollution.

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The Community participates as an observer in the Convention for the Prevention of Marine Pollution by Dumping from Ships and Aircraft, signed in Oslo on 15 February 1972. The Community is closely following the work being done under the auspices of the Standing Advisory Committee to the Scientific Councils of the Oslo Convention and the Joint Working Party on monitoring and continuous surveillance under the Oslo and Paris Conventions; this work concerns the monitoring and continuous surveillance of the sea areas into which titanium dioxide wastes are discharged.

In January 1976, moreover, the Commission put forward a proposal for a Council Directive on the dumping of wastes at sea (OJ C 40, 20 February 1976) which contains a brief review of the legal situation regarding this method of dumping at sea.

Finally, a Directive on pollution caused by certain dangerous substances discharged into the aquatic environment of the Community (76/464/EEC) stipulates that prior authorization must be obtained for any discharge into waters likely to contain one of the substances specified in Annex II, such as titanium, zinc, vanadium, chromium etc.

11. Consultation of the European Parliament and the Economic and Social Committee

Since this proposal for a Directive is based on Article 7 of Directive 78/176/EEC, consultation of the European Parliament and of the Economic and Social Committee is obligatory, as stipulated in paragraph 3 of this Article.



THE COUNCIL OF THE EUROPEAN COMMUNITIES

Having regard to the Treaty establishing the European Economic Community, and in particular Articles 100 and 235 thereof,

Having regard to Council Directive 78/176/EEC of 20 February 1978 on waste from the titanium dioxide industry<sup>1</sup>, and in particular Article 7(3) thereof,

Having regard to the proposal from the Commission,

Having regard to the opinion of the European Parliament,

Having regard to the opinion of the Economic and Social Committee,

Whereas, irrespective of the method and extent of the treatment of wastes from the titanium dioxide industry, the discharge, dumping at sea, storage, tipping or injection into the ground of such wastes must be accompanied, in particular, by measures to monitor the environment affected from a physical, chemical, biological and ecological point of view;

Whereas, in order to monitor the quality required of the environments affected a regular minimum number of samples should be taken annually so that the parameters specified in the Annexes may be measured; whereas the number of these sampling operations could be reduced in the light of the results obtained; whereas to ensure that the monitoring is effective, samples should also be taken in a zone deemed to be unaffected by the discharges in question;

Whereas in connection with the analyses carried out by the Member States, common reference methods of measurement should be fixed for determining the parametric values which define the physical, chemical, biological and ecological characteristics of the environments affected;

<sup>1</sup> J0 L 54, 25 February 1978

Whereas, for the purposes of surveillance, Member States are at all times free to measure parameters other than those prescribed by this Directive;

Whereas the Member States should communicate to the Commission details of the methods of surveillance and monitoring of the environments affected; whereas it is appropriate that the Commission should draw up a consolidated report to be sent to the Member States;

Whereas certain natural circumstances are outside the control of Member States and that, accordingly, provision must be made for derogation, in certain cases, from this Directive;

Whereas technical and scientific progress may require the rapid adjustment of specific provisions contained in the Annex; whereas to facilitate implementation of the requisite measures, a procedure should be laid down to establish close cooperation between the Member States and the Commission through a Committee on adaptation to scientific and technical progress;

HAS ADOPTED THIS DIRECTIVE

#### Article One

This Directive lays down - pursuant to Article 7(3) of Directive 78/176/EEC - the methods for the surveillance and monitoring of the environments affected from a physical, chemical, biological and ecological point of view, by the discharges of wastes from the titanium dioxide industry.

#### Article 2

For the purpose of this Directive:

- (a) 'environment affected' means surface water, ground water,  
.../...

the sea, the land surface and underground strata and the air into or on which wastes from the titanium dioxide industry are discharged or stored;

- (b) 'sampling point' means the point at which the sample is taken;
- (c) 'reference method of measurement' means a measurement principle or the concise description of a method for determining the parameters specified in the Annexes to this Directive.

### Article 3

1. The relevant parameters for the surveillance and monitoring of the environments referred to in Article 1 are shown in Annexes 1 to 5, subdivided according to the receiving medium.
2. Where a parameter appears in the 'mandatory' column of the Annexes, sampling and analysis must be carried out in respect of the environmental components stated.
3. Where a parameter appears in the 'guide' column of the Annexes, the Member States shall endeavour to perform the sampling and analysis operations for the environmental components shown.

### Article 4

1. The competent authorities in the Member States shall determine the frequency of sampling and analysis for each parameter in respect of a given zone in which discharge, tipping, dumping, storage, deposition or underground injection takes place.
  2. The frequency of sampling and analysis may not be less than the minimum frequencies shown in the Annexes to this Directive. The sampling periods must, as far as possible, be spread over the year in such a way as to obtain a representative picture of the quality of the environments affected.
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3. The competent authority in each Member State shall determine - on a case by case basis - the exact site at which samples are to be taken, the distance of this site from the nearest pollutant discharge point and the depth or height at which the samples must be taken. A general description must be given of the sampling point which will include its permanent features, capable of being codified, and other administrative or geographical information. The description shall be made only once when the typical sampling point is designated.
4. The sampling must be carried out in the zone directly affected by the discharges, special account being taken of local environmental factors and the manner of discharge, i.e. whether intermittent or continuous.

In the case of tidal waters, the samples must be taken at the same depth and under the same conditions, i.e. the same time in relation to high tide, tidal coefficient etc.

5. Where the competent authority finds that the results of measurements on the samples taken in a discharge zone during the previous year are both constant and significant and show that the quality of the environments affected has been detected, the frequency of sampling and analysis may - with the Commission's agreement - be reduced by the Member State concerned;

To this end, the Member States shall communicate to the Commission the results of such measurements and notify it of the frequency they intend to adopt; the Commission may make its agreement subject to certain conditions, in particular the duration of the reduction in frequency. In the likelihood of any deterioration in the quality of the environments affected, the Member States shall take the requisite measures.

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Article 5

1. The reference methods of measurement to be used to determine the relevant parametric values are specified in the Annexes. Laboratories using other methods must ensure that the results obtained are equivalent or comparable.
2. The containers used to carry the samples, the agents or methods used to preserve a part sample with a view to analysis of one or more parameters, the transport and storage of samples and their preparation for analysis must be such that they do not significantly affect the analytical results.

Article 6

In order to assess the state of progress in the surveillance and monitoring of the various environments affected from a physical, chemical, biological and ecological point of view, the competent authorities in the Member States shall ensure that samples are taken and analyses performed in respect of each environmental component specified in the Annexes to this Directive in a zone deemed to be unaffected by the discharges under surveillance.

Article 7

Member States may, at any time, measure parameters other than those laid down by this Directive relevant to the surveillance and monitoring of the environments affected.

Article 8

1. The following information shall be given by the Member States in the report they are required to submit to the Commission pursuant to Article 14 of Directive 78/176/EEC:

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(a) details of the methods of surveillance and monitoring carried out by the bodies appointed in accordance with Article 7(2) of Directive 78/176/EEC;

(b) the details referred to in point (a) above shall, in respect of each environment affected by the discharges, include the following information:

- the results of the measurements of the parameters listed in the "mandatory" and "guide" columns provided that the latter parameters are measured;
- the methods of measurement and analysis with their limit of detection, accuracy and precision;
- the descriptions of the sampling point;
- a description of the sampling methods used.

2. In implementation of this Article, the first set of data to be communicated is that gathered during the third year following notification of this Directive.

3. The Commission shall, with the prior agreement of the Member State concerned, publish a summary of the information thus obtained.

4. The Commission will assess the effectiveness of the environmental surveillance and monitoring procedure and will - no later than six years after notification of this Directive - place before the Council, where appropriate, proposals to improve this procedure and, if necessary, to harmonize the methods of measurement including the limit of detection, accuracy and precision and the sampling methods.

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Article 9

Member States may derogate from this Directive in the event of flooding or natural disaster or on account of exceptional weather conditions.

Article 10

The requisite amendments to adapt the methods of analysis and the G & I parameters shown in the Annexes to scientific and technical progress shall be adopted in accordance with the procedure laid down in Article 12.

Article 11

1. A Committee on adaptation to technical progress (hereinafter referred to as the "Committee") consisting of representatives of the Member States and chaired by a Commission representative, is hereby set up.
2. The Committee shall draw up its rules of procedure.

Article 12

1. Where the procedure laid down in this Article is to be followed, matters shall be referred to the Committee by its Chairman, either on his own initiative or at the request of the representative of the Member States.
  2. The Commission representative shall submit to the Committee a draft of the measures to be adopted. The Committee shall deliver its opinion on the draft within a time-limit set by the Chairman in the light of the urgency of the matter. It shall act by a majority of 41 votes, the votes of the Member States being weighted as provided for in Article 148(2) of the Treaty. The Chairman shall not vote.
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3. (a) The Commission shall adopt the proposed measures where they are in accordance with the opinion of the Committee.
- (b) Where the proposed measures are at variance with the opinion of the Committee, or if no opinion is adopted, the Commission shall, without delay, submit a proposal to the Council concerning the measures to be taken. The Council shall act by qualified majority;
- (c) If, within three months of the proposals being submitted to it, the Council has not acted, the proposed measures shall be adopted by the Commission.

Article 13

Directive 78/176/EEC is hereby amended as follows:

- (a) In Article 7(1) the words "and of the environment concerned having regard to its physical, chemical, biological and ecological aspects" shall be deleted.
- (b) Article 8, paragraph 1 point C is amended as follows:  
  
"If the results of the monitoring provided for in Article 1 of Directive            EEC concerning the methods of surveillance and monitoring of environments affected" show a deterioration in the specific environment affected in the area concerned, or
- (c) Annex II(B) is deleted.

Article 14

1. The Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with this



Directive before 1 January 1983. They shall forthwith inform the Commission thereof.

2. The Member States shall communicate to the Commission the texts of the main provisions of national law which they adopt in the field covered by this Directive.

Article 15

This Directive is addressed to the Member States.

ANNEX I

RECEIVING MEDIUM: AIR

COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
Air	SO <sub>2</sub>		12	<ul style="list-style-type: none"> <li>- In the prevailing wind in the zone directly affected by the discharge.</li> <li>- In accordance with those given in Annex II of the Directive of 15 July 1980 on air quality limit values and guide values for sulphur dioxide and suspended particulates. (O.J. L 229 of 30 August 1980, pages 30-48) (80/779/CEE)</li> </ul>

RECEIVING MEDIUM - SEA WATER (estuarine, coastal, open sea)

COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
Water column Non-filtered water or Water filtered through 0.45 μm membrane	Temperature		4	Thermometry
	Salinity		4	Conductimetry
	pH		4	Electrometry. Measurement is to be carried out on the spot at the time of sampling.
	Dissolved O <sub>2</sub>		4	- Winkler method - Electrochemical method
	Turbidity or suspended matter		4	Turbidimetry - Weighing after filtration through 0.45 μm filter and drying at 105°C - Weighing after centrifugation (minimum time 5 minutes, and average acceleration 2,800 and 3,200 g)
	Fe	Cr	4	Atomic absorption spectrophotometry Molecular absorption spectrophotometry
	Ti	Mn, Ni, V, Zn,	4	Atomic absorption spectrophotometry
Suspended solids		Cu, Pb	4	Atomic absorption spectrophotometry polarography
				After appropriate preparation of the sample, wet or dry mineralisation, and purification
	Fe	Cr	4	Atomic absorption spectrophotometry Molecular absorption spectrophotometry

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COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
	Ti	Mn, Ni, V, Zn	4	Atomic absorption spectrophotometry
		Cu, Pb	4	Atomic absorption spectrophotometry polarography
	Hydrated oxides hydroxides of iron		4	Extraction of the sample under dilute acid conditions (HCl; CH <sub>3</sub> COOH; HNO <sub>3</sub> ; HClO <sub>4</sub> /HNO <sub>3</sub> ), measurement by atomic absorption spectrophotometry. The determination of iron by molecular absorption spectrophotometry can be made without interference if the extraction is made using CH <sub>3</sub> COOH.  The same method of acid extraction must be used for all samples coming from the same site
<u>Sediments</u> In the top layer of sediment (0-1 cm.)	Fe, Ti	Cr, V, Zn, Pb, Ni, Cu, Mn	1	Identical methods to those for measurements in the water column.  After appropriate preparation of the sample (wet or dry mineralisation and purification)  - The quantities of metals may be measured for a specific range of particle sizes  - The quantities of metals are to be expressed in mg/kg of dry matter
	Hydrated oxides hydroxides of iron		1	Identical methods to those for measurements in the water column

COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
<p><u>Living organisms</u></p> <p>Species representative of the site: molluscs, crustaceans and fish and at least two invertebrates including, if possible, one benthic organism (1)</p>	<p>Cr, Cd, V, Hg, Fe, Ti, Zn, Pb, Ni, Cu</p>		1	<p>Atomic absorption spectrophotometry after appropriate preparation of the composite sample of ground flesh (wet of dry mineralisation and purification)</p> <ul style="list-style-type: none"> <li>- For fish, the metals must be measured in muscle. The sample should consist of at least 10 specimens.</li> <li>- The quantities of metals are to be expressed in mg/kg of wet matter</li> </ul> <p>For the molluscs and crustaceans, the metals must be measured in the flesh. The sample should consist of at least 50 specimens.</p> <ul style="list-style-type: none"> <li>- The quantities of metals are to be expressed in mg/kg of wet matter.</li> </ul>
<p>Benthic flora and fauna, fish</p>	<p>Diversity and relative abundance</p>		2	<p>Qualitative and quantitative classification indicating the liste of species, the specimen count per species, the specimen count, density dominance, and in the case of fish, the percentage showing morbid anatomical symptoms.</p>

(1) Species representative of the discharge site: such as mytilus edulis, crangon crangon, flounder, plaice, cod, mackerel, red mullet, herring, sole (or another appropriate benthic species).

ANNEX III

RECEIVING MEDIUM; FRESH WATER

COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
<u>Water column</u> Samples taken at a depth of 0.50 m below the surface at the same time of the year	pH		4	Electrometry. Measurement is to be carried out on the spot at the time of sampling.
	Dissolved O <sub>2</sub>		4	- Winkler method - Electrochemical method
On non-filtered water or water filtered through a 0.45 µm membrane	Turbidity or suspended matter		4	Turbidimetry - Weighing after filtration through 0.45 µm filter and drying at 105°C - Weighing after centrifugation. (minimum time 5 minutes, and average acceleration 2,800 and 3,200 g) and drying at 105°C.
	Dissolved salts		4	Electrometric measurement of conductivity at 20°C
Suspended solids	Fe	Cr	4	Atomic absorption spectrophotometry Molecular absorption spectrophotometry
	Ti	Mn, Ni, V, Zn	4	Atomic absorption spectrophotometry
		Cu, Pb	4	Atomic absorption spectrophotometry; polarography
	Fe	Cr	4	After appropriate preparation of the sample, wet or dry mineralisation, and purification Atomic absorption spectrophotometry Molecular absorption spectrophotometry

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COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
	Ti	Mn, Ni, V, Zn	4	Atomic absorption spectrophotometry
		Cu, Pb	4	Atomic absorption spectrophotometry Polarography
	Hydrated oxides hydroxides of iron		4	Extraction of the sample under dilute acid conditions (HCl; CH <sub>3</sub> COOH; HNO <sub>3</sub> ; HClO <sub>4</sub> /HNO <sub>3</sub> ), measurement by atomic absorption spectrophotometry. The determination of iron by molecular absorption spectrophotometry can be made without interference if the extraction is made using CH <sub>3</sub> COOH.  The same method of acid extraction must be used for all samples coming from the same site.
<u>Sediments</u> On the top layer of sediment (0-1 cm.)	Fe, Ti	Cr, V, Zn, Pb, Ni, Cu, Mn	1	Identical methods to those for measurements in the water column.  After appropriate preparation of the sample (wet or dry mineralisation and purification) - The quantities of metals may be measured for a specific range of particle sizes - The quantities of metals are to be expressed in mg/kg of dry matter
	Hydrated oxides hydroxides of iron		1	Identical methods to those for measurements in the water column

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COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
<p><u>Living organisms</u></p> <p>Species representative of the substrate</p>	<p>Cr, Cd, V, Hg, Fe, Ti, Zn, Pb, Ni, Cu</p>		1	<p>Atomic absorption spectrophotometry after appropriate preparation of the composite sample of ground flesh.</p> <p>- The quantities of metals are to be expressed in mg/kg of wet matter</p>
<p>Benthic flora and fauna, fish</p>	<p>Diversity and relative abundance</p>		2	<p>Qualitative and quantitative classification indicating the list of species, the specimen count per species, the specimen count, density dominance, and in the case of fish, the percentage showing morbid anatomical symptoms</p>



RECEIVING MEDIUM: STORAGE AND DUMPING ON LAND

COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
Running surface waters	Acidity		2	- Titrimetry
		Cr <sup>(1)</sup>	2	- Atomic absorption spectrophotometry - Molecular absorption spectrophotometry
(a) from the storage area and evacuated to the outside (not recycled)	SO <sub>4</sub>		2	- Gravimetry - Complexometric titration with EDTA - Molecular absorption spectrophotometry
		V <sup>(1)</sup>	2	- Atomic absorption spectrophotometry
(b) around the site in the area affected by the storage and at a point outside this area	Fe <sup>(1)</sup>		2	- Atomic absorption spectrophotometry - Molecular absorption spectrophotometry
		Zn <sup>(1)</sup>	2	Atomic absorption spectrophotometry
Ground waters	Ca		2	- Atomic absorption spectrophotometry - Complexometric titration
		Pb <sup>(1)</sup>	2	- Atomic absorption spectrophotometry - Polarography
Water filtered through a 0.45 μm membrane	Ti		2	- Atomic absorption spectrophotometry
		Ni <sup>(1)</sup>	2	- Atomic absorption spectrophotometry
Around the site including, where necessary, its out-flow points.	Cl		2	- Titrimetry (Mohr method)
		Cu <sup>(1)</sup>	2	- Atomic absorption spectrophotometry - Polarography
		Mn <sup>(1)</sup>	2	- Atomic absorption spectrophotometry

COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
Environment of the storage and dumping site Topography and site management: effect on sub soil and on the ecology of the site	Visual inspection		1	Methods to be chosen by Member States

(1) Also includes a quantitative analysis of the residue after filtration (suspended matter).

RECEIVING MEDIUM: INJECTION

COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
<p><u>Surface waters</u> Water filtered through a 0.45 µm membrane</p> <p>Around the site in the zone affected by the injection</p>	Acidity		1	- Titrimetry
		Cr <sup>(1)</sup>	1	- Atomic absorption spectrometry - Molecular absorption spectrophotometry
	SO <sub>4</sub>		1	- Gravimetric analysis - Complexometric titration with EDTA - Molecular absorption spectrophotometry
		V <sup>(1)</sup>	1	- Atomic absorption spectrophotometry
<p><u>Ground waters</u> Water filtered through a 0.45 µm membrane</p>	Fe <sup>(1)</sup>		1	- Atomic absorption spectrophotometry - Molecular absorption spectrophotometry
		Zn <sup>(1)</sup>	1	- Atomic absorption spectrophotometry
	Ca		1	- Atomic absorption spectrophotometry - Complexometric titration
<p>Under and around the site including outflow points</p>		Pb <sup>(1)</sup>	1	- Atomic absorption spectrometry - Polarography
	Ti		1	- Atomic absorption spectrophotometry
		Ni <sup>(1)</sup>	1	- Atomic absorption spectrophotometry
	Cl		1	- Titrimetry (Mohr method)
		Cu <sup>(1)</sup>	1	- Atomic absorption spectrophotometry - Polarography
	Mn <sup>(1)</sup>	1	- Atomic absorption spectrophotometry	

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COMPONENTS	PARAMETERS		MINIMUM ANNUAL SAMPLING FREQUENCY	REFERENCE METHOD OF ANALYSIS
	IMPERATIVE	GUIDE		
Environment Topography	Ground stability		1	Photographic and topographic survey
	Permeability Porosity		1	Pumping tests Well-logging

(1) Also includes a quantitative analysis of the residue after filtration (suspended matter).