Marine Chemical Monitoring

Policies, Techniques and Metrological Principles

Philippe Quevauciviller
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Philippe Quevauviller
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When I was invited to write this book, my first reaction was to wonder about my legitimacy to embark on this new editorial venture since my active involvement in oceanography seems to me far from current practices. Upon reflection, I however figured out that I have been involved in different facets of analytical developments, some of them related to marine science and policies for about 30 years, and that I could hence give it a try. In the specific field of “metrology” – the science of measurements – we cannot reinvent the wheel and I agreed to contribute a book related to this topic, which would be based on previous editorial projects to which I had contributed as author or editor. Writing or updating the different chapters of this book reminded me about my career path regarding chemical oceanography, which mixes research, analytical practices and policy, and of all the scientists with whom I worked and who contributed to my scientific and personal network. This preface is a tribute to them.

I started my university studies in marine geology in the early 1980s at the University of Bordeaux under the direction of Prof. Michel Vigneault and other scientists who opened my interest in oceanography, in particular Jean-Claude Faugères and Jean-Marie Froidefond. I joined the geochemistry discipline thanks to Francis Grousset at the Institut de Géologie du Bassin d’Aquitaine (IGBA, Geological Institute of Aquitaine Basin) and got my initial experience in environmental chemistry dealing with research into the silica
and dissolved oxygen pathways in a lake environment [QUE 84] under the supervision of Jean-Marie Jouanneau. I was trained by Philippe Pédemay who introduced me to water sampling and titrimetry/colourimetry. This readily taught me all the care that is required to avoid sample contamination and precautions to be taken regarding data accuracy. During this period, I was also involved in monitoring campaigns of physicochemical parameters in estuarine waters in front of the Blayais nuclear plant, navigating from Bordeaux (Port de la Lune) to the Gironde Estuary mouth on the *Ebalia* oceanographic vessel.

My second step into marine science was during my “Portuguese time” in the years 1984–1987 with in the framework of a scientific cooperation between the University of Bordeaux and the Portuguese Environment State Secretary. I inherited a study of a fantastic playground, in the form of an estuary (Sado Estuary) and a coastal environment (Galé Coast) that had to be studied in depth for geomorphology, sedimentology and geochemistry, under the supervision of Jean-Marie Jouanneau, Claude Latouche and Teresa Pera leading to a Doctorate [QUE 87a] in May 1987. I worked closely with outstanding researchers in Lisbon, in particular Alverinho Dias (then at the Portuguese Geological Surveys), Teresa Vinhas (Marine Hydrographical Institute), Carlos Vale (Fisheries Institute) and Leopoldo Cortez (then at the Environment State Secretary), as well as Miguel Aguas with whom I was introduced to modeling of coastal sediment dynamics, which formed the basis of my first paper published in the international literature [QUE 87b].

The “metrological” side of my studies focused on major and trace element determinations in estuarine sediment and biota samples. I discovered the joy of sampling muddy sediments and oysters in the area of Setúbal, carrying samples in the boot of my car on overnight trips to Bordeaux for major and trace element determinations mainly by X-ray fluorescence (XRF). I was also introduced to graphite furnace atomic absorption spectrometry (GFAAS) by Gilbert Lavaux at the IGBA for Cd and Pb measurements in sediments and oyster tissues. The complex pollution pathways of the Sado Estuary engaged me in further research [QUE 89], in particular about mobile forms of
trace metals using sequential extraction approaches (in particular, the
“Tessier scheme”) and speciation because of a collaboration with Prof.
Michel Astruc (University of Pau) and his team. In his laboratory, I
learnt to analyze sediment and biota samples for their butyltin contents
under the supervision of Renault Lavigne, and took this opportunity to
learn anodic stripping voltammetry and cathodic stripping
voltammetry under the supervision of André Castetbon. I did some
further work on sediment analyses, in particular particulate organic
carbon, under the supervision of Henri Etcheber at the Netherlands
Institute for Sea Research (NIOZ). I also met with Wim Salomons to
discuss sequential extraction matters.

Speciation opened a new phase of my oceanographic career thanks
to Olivier Donard, who recruited me to assist in the development of a
hybrid generation GCAAs system for the determination of organotin
compounds. This was done under a contract by the Rijkswatersta
(Dutch Ministry of Water Works) to monitor organotin compounds in
Dutch waters, the results of which would result in a policy leading to
the banning of tributyltin antifouling paints. The policy side was
managed by Remi Laane. With Olivier Donard, we were in charge of
organizing two monitoring campaigns in estuaries, lakes and coastal
waters of the Netherlands, and I took care of hundreds of samples of
sediment, suspended matters, biota and water in partnership with Rob
Ritsema. This experience brought me into marine monitoring work
covering the whole “measurement chain” from sampling to laboratory,
in addition with a practical objective related to policy decision-
making. It also allowed me to gain a second PhD (in environmental
chemistry this time) in 1991 [QUE 91].

This international experience allowed me to join a European
Commission research program, the so-called “Community Bureau of
Reference” (BOR), thanks to meeting Ben Griepink at a conference in
Lisbon (country of Michel Astruc). In this program, I was in charge of
organizing interlaboratory studies and reference material certification
campaigns in the speciation area [QUE 98], which allowed me to
work with outstanding chemists, including Les Ebdon, Peter Craig,
Rita Cornelis, Freddy Adams, Roberto Morabito, and Carmen
Camara, to name a few. One of the BCR projects under my
responsibility, which was started at the request of OSPARCOM and the NSTF, concerned the need to develop a proficiency testing scheme for marine monitoring in order to improve the quality and comparability of measurement data. This formed the basis for the development of the QUASIMEME project [WEC 93] under the chairmanship of David Wells, and many other high-level marine scientists involved in the International Council for the Exploration of the Sea. At this time, I reflected upon the basic metrology principles that are behind environmental monitoring, including marine measurements, and was encouraged to return to research and development, which led me to undertake a HDR diploma in 1999 at the University of Pau [QUE 99a]. The plan to return to a purely scientific career did not concretize, and I decided to shift to policy development instead.

I joined the Environment Directorate-General of the European Commission in 2002. I have been in charge of developing a Directive on Groundwater Protection, a “daughter” directive of the Water Framework Directive (WFD), under the supervision of Patrick Murphy. In doing this, I did not leave the marine sector since the WFD has a coastal component, and I participated in discussions prior to the adoption of the Marine Strategy Directive. Using my quality assurance/quality control (QA/QC) experience, I was able to convince experts within a European group on chemical monitoring activity, which I co-shared with Mario Carere at that time, that a directive dealing with quality principles for chemical monitoring measurements under the WFD would be required. This led to the adoption of the Commission Directive 2009/90/EC, pursuant to Directive 2000/60/EC of the European Parliament and of the Council, laying down technical specifications for chemical analysis and monitoring of water status in 2009 [COM 09].

The sum of experiences gathered over the years allowed me to enter into the editorial world in the early 2000s, e.g. as contributing editor to *Trends in Analytical Chemistry* and as a member of editorial boards on journals, the edition and authoring of several books, and the coordination of a book series on “Water Quality Measurements”, which is composed of 10 books. One of the volumes falls directly
within the scope of the present book [QUE 11b] and much of its substance is hence related to this former publication, with appropriate references.

I was also keen to transmit my combined knowledge of laboratory experience, research and policy developments. Besides my work at the European Commission, I have been invited by André Van der Beken to join his group as associate professor at the Department of Hydrology and Hydrological Engineering at Vrije Universiteit Brussels to teach in an International Masters Programme on “Integrated Water Resource Management”. One of the aspects naturally concerns measurements and metrology principles.

This book consists of five chapters. Chapter 1 provides an insight into historical background, the regulatory framework and science–policy interactions, Chapter 2 deals with monitoring and related QA/QC, Chapter 3 focuses on monitoring types, and Chapter 4 describes general features of analytical methods used in marine monitoring. The book concludes with a discussion about the application of meteorology principles in marine monitoring.

With this book, I hope to continue to “pass the knowledge” that has been gathered by many individuals in the last two decades, with a special tribute to some of my “masters” and friends without whom I would not have developed such an international and multidisciplinary career, in particular Francis Grousset, Olivier Donard and Ben Griepink.

Philippe QUEVAUVILLER
November 2015
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<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
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<td>AAS</td>
<td>atomic absorption spectrometry</td>
</tr>
<tr>
<td>ACSV</td>
<td>adsorptive cathodic stripping voltammetry</td>
</tr>
<tr>
<td>AE</td>
<td>anion exchange</td>
</tr>
<tr>
<td>AED</td>
<td>atomic emission detector</td>
</tr>
<tr>
<td>AES</td>
<td>atomic emission spectrometry</td>
</tr>
<tr>
<td>AFS</td>
<td>atomic fluorescence spectrometry</td>
</tr>
<tr>
<td>APDC</td>
<td>ammonium pyrrolidine dithiocarbamate</td>
</tr>
<tr>
<td>ASE</td>
<td>accelerated solvent extraction</td>
</tr>
<tr>
<td>ASV</td>
<td>anodic stripping voltammetry</td>
</tr>
<tr>
<td>BBD</td>
<td>butyl butylation derivatization</td>
</tr>
<tr>
<td>BCR</td>
<td>European Community Bureau of Reference</td>
</tr>
<tr>
<td>CCSA</td>
<td>constant current stripping analysis</td>
</tr>
<tr>
<td>CCT</td>
<td>capillary cryogenic trap</td>
</tr>
<tr>
<td>CE</td>
<td>capillary electrophoresis</td>
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</tbody>
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(C)GC  (capillary) gas chromatography
CRM    certified reference material
CSC    cathodic stripping chronopotentiometry
CSV    cathodic stripping voltammetry
CT     cryogenic trap; cryotrapping
CV     cold vapor
CVG    chemical vapor generation
CVT    cold vapor technique
D      distillation
dASCP  derivative anodic stripping chronopotentiometry
DGT    diffusion gradients in thin-film
DPASV  differential pulse anodic stripping voltammetry
DPC    diphenylcarbazide
DPCSV  differential pulse cathodic stripping voltammetry
ECD    electron capture detector
EI     electron impact ionization
ESI    electrospray ionization
Et ethyl
ETAAS  electrothermal atomic absorption spectrometry
Eth ethylation derivatization
EXAFS  extended X-ray absorption fine structure
<table>
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<tr>
<th>Abbreviation</th>
<th>Definition</th>
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<tr>
<td>FAAS</td>
<td>flame atomic absorption spectrometry</td>
</tr>
<tr>
<td>FI</td>
<td>flow injection</td>
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<tr>
<td>FIA</td>
<td>flow injection analysis</td>
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<tr>
<td>FPD</td>
<td>flame photometric detector</td>
</tr>
<tr>
<td>FPLC</td>
<td>fast protein liquid chromatography</td>
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<tr>
<td>GC</td>
<td>gas chromatography</td>
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<tr>
<td>HG</td>
<td>hydride generation</td>
</tr>
<tr>
<td>HPLC</td>
<td>high-performance liquid chromatography</td>
</tr>
<tr>
<td>HR</td>
<td>high resolution</td>
</tr>
<tr>
<td>HS</td>
<td>headspace</td>
</tr>
<tr>
<td>HTFA</td>
<td>trifluoroacetylacetone</td>
</tr>
<tr>
<td>IBMK</td>
<td>isobutyl methyl ketone</td>
</tr>
<tr>
<td>IC</td>
<td>ion chromatography</td>
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<tr>
<td>ICP-AES</td>
<td>inductively-coupled plasma atomic emission spectrometry</td>
</tr>
<tr>
<td>ICP-MS</td>
<td>inductively-coupled plasma mass spectrometry</td>
</tr>
<tr>
<td>ICP-OES</td>
<td>inductively-coupled plasma optical emission spectrometry</td>
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<tr>
<td>ID</td>
<td>isotope dilution</td>
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<tr>
<td>IDMS</td>
<td>isotopic dilution mass spectrometry</td>
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<td>IE</td>
<td>ion exchange</td>
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<tr>
<td>IT</td>
<td>ion trap</td>
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<tr>
<td>Abbreviation</td>
<td>Description</td>
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</tr>
<tr>
<td>LA</td>
<td>laser</td>
</tr>
<tr>
<td>LC</td>
<td>liquid chromatography</td>
</tr>
<tr>
<td>LLE</td>
<td>liquid–liquid extraction</td>
</tr>
<tr>
<td>LOD</td>
<td>limit of detection</td>
</tr>
<tr>
<td>MALDI</td>
<td>matrix-assisted laser desorption and ionization</td>
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<tr>
<td>MCGC</td>
<td>multicapillary gas chromatography</td>
</tr>
<tr>
<td>Me</td>
<td>methyl</td>
</tr>
<tr>
<td>MIBK</td>
<td>methyl isobutyl ketone</td>
</tr>
<tr>
<td>MIP</td>
<td>microwave induced plasma</td>
</tr>
<tr>
<td>MS</td>
<td>mass spectrometry</td>
</tr>
<tr>
<td>MTLPs</td>
<td>metallothionein-like proteins</td>
</tr>
<tr>
<td>MTs</td>
<td>metallothioneins</td>
</tr>
<tr>
<td>MW</td>
<td>microwave</td>
</tr>
<tr>
<td>NaDDC</td>
<td>sodium diethyldithiocarbamate trihydrate</td>
</tr>
<tr>
<td>OCP</td>
<td>organichlonine pesticide</td>
</tr>
<tr>
<td>ORS</td>
<td>octopole reaction system</td>
</tr>
<tr>
<td>OTC</td>
<td>organotin compound</td>
</tr>
<tr>
<td>PFPD</td>
<td>pulsed flame photometric detector</td>
</tr>
<tr>
<td>PLE</td>
<td>pressurized solvent extraction</td>
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<tr>
<td>PLM</td>
<td>permeation liquid membrane</td>
</tr>
<tr>
<td>Pr</td>
<td>propylation derivatization</td>
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<tr>
<td>Abbreviation</td>
<td>Full Form</td>
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<tr>
<td>Preconc</td>
<td>preconcentration</td>
</tr>
<tr>
<td>PSA</td>
<td>potentiometric stripping analysis</td>
</tr>
<tr>
<td>PTFE</td>
<td>polytetrafluoroethylene</td>
</tr>
<tr>
<td>PTI</td>
<td>purge and trap injection</td>
</tr>
<tr>
<td>QF</td>
<td>quartz furnace atomizer</td>
</tr>
<tr>
<td>RP</td>
<td>reversed phase</td>
</tr>
<tr>
<td>SCP</td>
<td>stripping chronopotentiometry</td>
</tr>
<tr>
<td>SE</td>
<td>size exclusion</td>
</tr>
<tr>
<td>SEC</td>
<td>size exclusion chromatography</td>
</tr>
<tr>
<td>SF</td>
<td>sector field</td>
</tr>
<tr>
<td>SIDMS</td>
<td>speciated isotope dilution mass spectrometry</td>
</tr>
<tr>
<td>SPE</td>
<td>solid phase extraction</td>
</tr>
<tr>
<td>SPM</td>
<td>suspended particulate matter</td>
</tr>
<tr>
<td>SPME</td>
<td>solid-phase microextraction</td>
</tr>
<tr>
<td>TFA</td>
<td>trifluoroacetylacetonate</td>
</tr>
<tr>
<td>TMAH</td>
<td>tetramethylammonium hydroxide</td>
</tr>
<tr>
<td>TOF</td>
<td>time-of-life</td>
</tr>
<tr>
<td>USN</td>
<td>ultrasonic nebulizer</td>
</tr>
<tr>
<td>UV</td>
<td>ultraviolet photolysis (treatment)</td>
</tr>
<tr>
<td>UV/vis</td>
<td>ultraviolet/visible</td>
</tr>
</tbody>
</table>
VG vapor generation
WFD Water Framework Directive
XANES sX-ray absorption near-edge spectroscopy
XRF X-ray fluorescence
Abbreviations

**Conventions and legislative units**

- **DIN**: Deutche (German) Industrial Norm
- **EU**: European Union
- **HELCOM**: Helsinki convention for the Baltic Sea
- **MSD**: Marinde Strategy Directive
- **OSPAR**: Oslo-Paris convention for the North Sea
- **SFD**: Shell Fish Directive
- **WFD**: Water Framework Directive

**Atomic absorption techniques**

- **AAS**: Atomic absorption spectrometry
- **CVAAS**: Cold vapor atomic absorption spectrometry
- **HGAAS**: Hydride generation atomic absorption spectrometry
GFAAS  Graphite furnace atomic absorption spectrometry
AFS    Atomic fluorescence spectrometry
STPF   Stabilized temperature platform furnace
HCL    Hollow cathode lamp
EDL    Electronic discharge lamp

**Plasma techniques**

ICP-AES/OES  Inductively coupled plasma-atomic/optical emission spectrometry (synonyms)
ICP-HRMS     Inductively coupled plasma high resolution mass spectrometry
ICP-MS       Inductively coupled plasma mass spectrometry
ICP-QMS      Inductively coupled plasma quadropole mass spectrometry

**X-ray techniques**

ED-SEM       Energy dispersive scanning electron microscopy
EDXRF        Energy dispersive X-ray
XAS          X-ray absorption spectroscopy
XPS          X-ray photon spectroscopy
XRF          X-ray fluorescence
XRD          X-ray diffraction
PXRF         Portable X-ray fluorescence
PIXE         Particle induced X-ray emission
Electrochemical techniques

ASV  Anodic stripping voltametry
CSV  Cathodic stripping voltametry
PSA  Potentiometric stripping analysis

Quality control

IRM  Internal reference material
CRM  Certified reference material
1.1. Introduction

Public awareness about marine pollution and regulatory responses is relatively recent [KRA 11]. The fact that chemical products released into the sea could be hazardous only became “shocking” in the 1950s with the methyl-mercury contamination of tuna and swordfish in Minamata Bay (Japan) in 1956, which resulted in neurological syndromes in the population. Some years later, the wrecking of the Torrey Canyon oil tanker along the Cornish coast (March 1967) and the blowout of an oil platform off the Californian coast in 1969 highlighted the fragility of marine ecosystems in the case of oil spills. The number of dramatic pollution events affecting marine quality would require a full volume but what should be remembered here is that these oil spills resulted in “visible” effects, which led to reactions from the public and changes in opinion that helped create a climate in which legislation was deemed necessary and scientific activities in research and monitoring were encouraged [KRA 11]. Research into the input, transport and fate of pollutants in the marine environment developed in the 1970s, which is reflected by the first issue of the Marine Pollution Bulletin, published in January 1970, which aimed to “spread news of pollution” rather than publishing research results, with the objective of informing policymakers [ANO 70].
In 1977, the International Council of Scientific Union’s (ICSU’s) Scientific Committee on Problems of the Environment (SCOPE) defined “monitoring” as “the collection, for a predetermined purpose, of systematic, inter-comparable measurements or observations in a space–time series, of any environmental variables or attributes which provide a synoptic view or a representative sample of the environment (global, regional, national, or local). Such a sample may be used to assess existing and past states, and to predict likely future trends in environmental features” [HOL 77]. This definition of monitoring, still valid today, turns it into a systematic method of collecting data needed for environmental problem-solving, which is linked to environmental policy [KRA 11].

Monitoring is not only focused on determining concentrations of harmful substances in various compartments of the (marine) environment, e.g. water, sediment and biota. It also includes physical parameters such as salinity, turbidity and pH as well as biological effects [KRA 11]. The basic reasoning behind monitoring has evolved over the years: in the mid-1970s, the focus was on avoiding health hazards, and the knowledge about the fate of pollutants not representing a direct threat to human health was considered to be of lesser importance. Nowadays, monitoring constitutes one branch in a more holistic approach related to marine management and international conventions and regulations (see section 1.3).

Historic developments of marine monitoring (with a focus on the North and Baltic Seas) have been described by Kramer [KRA 11] and will not be repeated here. Only key milestones that have led to current regulations will be described. The first landmark in (marine) monitoring is considered to be the United Nations Conference on the Human Environment that was held in Stockholm in June 1972, which resulted in the adoption of a declaration and an action plan. Principle 7 of the declaration stated that “all possible steps shall be taken to prevent pollution of the seas by substances that are liable to create hazards to human health, to harm living resources and marine life, to damage amenities or to interfere with other legitimate uses of the sea”, whereas the action plan recommended that “governments actively support, and contribute to international programs to acquire
knowledge for the assessment of pollutant sources, pathways, exposures and risks” [UNE 72], quoted by [KRA 11]. This conference strengthened the developing environmental (marine) monitoring efforts in various national and international programs, and had some effects on harmonization and structuring marine monitoring plans and activities by active international organizations in the field [KRA 11].

Today, it is well known that marine ecosystems are experiencing unprecedented environmental changes, driven by human activities [ROO 11a]. Issues such as pollution not only from land- and sea-based sources but also from fishing, marine debris, the loss and degradation of valuable habitat and invasions by non-native species are recognized worldwide. However, the initial conventions, and hence research and monitoring, were heavily focused on measurements related to the two major problems at that time: eutrophication and contamination. Furthermore, frequent sampling at sea also became a source of hydrological data in a broader sense, as contaminant data are generally supported by metadata such as salinity, temperature, dissolved oxygen and others. As said above, this has been going on since the late 1960s and early 1970s. Not surprisingly, marine chemical monitoring is presently one of the technically most advanced branches of marine environmental monitoring. Gradually, the focus shifted from pollution to a more holistic approach, often referred to as the ecosystem approach. Chemical monitoring is now only one aspect, albeit an important one, of marine environmental monitoring. Yet, new threats such as climate change and ocean acidification will certainly give a new emphasis on chemical measurements [ROO 11a].

This chapter aims at giving an overview of the existing international organizations and conventions that are central to marine monitoring and research.

1.2. International institutions

Initiatives related to environmental (marine) monitoring were started much before the 1972 Stockholm Conference by a number of international institutions with a certain competition among different
monitoring activities [KRA 11], e.g. a pollution research program developed by the Organisation for Economic Co-operation and Development (OECD) in 1965, marine pollution monitoring activities developed by the International Council for the Exploration of the Sea (ICES) from 1966 onward, etc. In 1969, the UN Food and Agriculture Organization (FAO) convened a “Technical conference on marine pollution and its effects on living resources and fishing”. The same year saw the creation of a joint Group of Experts on the Scientific Aspects of Marine Pollution (GESAMP), cosponsored by the International Maritime Organization (IMO), UNFAO, United Nations Educational, Scientific and Cultural Organization (UNESCO) and World Meteorological Organization. The purpose of this group was to advise the various agencies and UN bodies that were concerned with marine pollution. Later, the World Health Organization, International Atomic Energy Agency and United Nations Environmental Programme (UNEP) joined the initiative [WIN 91]. A strategic vision on scientific aspects of marine environmental protection was developed by this expert group [GES 05].

These developments also resulted in the creation of SCOPE by the ICSU in 1969, which published a report on “Global Environmental Monitoring” [SCO 71] as input for the Stockholm Conference (quoted by [KRA 11]). This report stressed the need for a stronger coordination between a marine pollution monitoring system and Integrated Global Ocean Station System, then under development by the Intergovernmental Oceanographic Commission (IOC) for monitoring the physical conditions of the oceans. Studies based on monitoring of water, superficial sediments and biota for selected critical substances were proposed to be undertaken in pilot areas such as the North Sea, the Baltic Sea, the Mediterranean Sea and the Puget Sound (US). This formed the basis for developing aspects of policy by the IOC and coordinating international science by the Scientific Committee on Oceanic Research (SCOR). Ideas were further developed in the action plan for a Global Environmental Monitoring System [MUN 73] and priority pollutants were defined [AND 88].