Tacis

Caspian Environment Programme, Phase 2

Building a Reference Laboratory Structure

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Chapter 1. Executive Summary

1.1. Existing Laboratory Capacity

The laboratory capacity assessment builds upon the contents of a questionnaire, which was completed for each laboratory during a field tour to laboratories in Kazakhstan, Turkmenistan, Russia and Azerbaijan by Latifa Huseynova, and during a field tour to laboratories in I.R Iran by Mohammad Saed Abaee.

Only laboratories identified as major analytical or monitoring laboratories during the first phase of the Tacis project were visited and asked to submit information via the questionnaire. The laboratories are all environmental laboratories, performing analyses of water samples, and in most cases also air, soil or other media.

So far no private or industrial laboratories performing environmental analyses have been investigated to the same level of detail.

1.1.1. Activities of the Existing Laboratories

All laboratories visited perform analyses that may be of interest for the Caspian Environment Programme. They all analyse water of different kinds, some specialising in certain water types. It is apparent that not all laboratories have experience in saline waters. The experience in analysing sediments and biota samples for environmental purposes is very limited, in most cases missing. A very limited number of laboratories have equipment suitable for such analyses.

1.1.2. The Capacities of the Existing Laboratories

Some obvious problems and strengths were revealed at the laboratories during the field tour. Of special importance to the data quality is the almost notorious lack of modern equipment, modern analytical methods and chemical reagents and standards. Limited budgets place a severe restraint on laboratory operations, equipment maintenance and the purchase of high-quality standard materials. The situation in I.R. Iran is in general better on these points. There is also a lack in both in-house and external quality control procedures.

The staff has in general a very good educational background. There is thus an enormous potential to increase the current laboratory capacity if the laboratories can be modernised with appropriate equipment, and the staff receives training on modern analytical methods and modern QA/QC-techniques.

1.1.3. Internal and External QA/QC

The procedures applied for both internal and external QA/QC (Quality Assurance and Quality Control) is not up to modern standards in most laboratories. There appears to be no structure or existing scheme for national or regional inter-comparison exercises within the environmental field. Some laboratories have participated in occasional exercises, but there

are no regular activities. No laboratories appear to be using certified reference materials (CRMs). External references are normally limited to the use of calibrated scales for preparing in-house reference materials, which is not good enough. Internal quality control procedures, such as the use of control samples and control charts, are only in use in a very limited number of the laboratories.

All this highlights how important it is, for improving the data quality in the CEP, to start up reference laboratory activities.

1.1.4. Training of Laboratory Staff

Even though the educational background of the staff is generally very high, they are in many cases not familiar with the state-of-the-art analytical equipment, analytical methods and standardised QA/QC-procedures.

The training needs will be very different for staff from different laboratories, depending on which analyses they will be requested to perform and which equipment they will use in the future. Those laboratories that will mainly perform test-kit analyses will need a certain kind of training, whereas for more advanced equipment and techniques it might prove most useful to send staff away for on-the-job-training at up-to-date laboratories for an extended time period.

1.2. Requirements for Laboratories in the Caspian Region

Today, it is recognised that management of environmental problems requires considerable effort and co-operation, both local and international, and that pollution should be controlled at the source, among other things:

- to prevent the generation of waste that damages the environment,
- to improve the cost effectiveness of environmental management, and
- to identify the specific processes or steps where low-waste technology must be introduced to protect the environment and save natural resources.

An essential component of a well-functioning laboratory system for monitoring sources and large-scale effects is the appointment by the environmental authorities of a Reference Laboratory with sufficient facilities and equipment, a qualified, highly intelligent staff with broad and up-to-date experience in general environmental management, and routine experience in related laboratory activities.

1.2.1. Concept of Traceability

A fundamental principle in practical science is confidence in the ability to measure and compare metrological quantities. Traceability is the process whereby the indication of a measuring instrument can be compared in one or more stages with a national standard for the metrological quantity in question. In each stage, a calibration is performed using a standard with a metrological quantity already determined by calibration with a higher-level standard.

For many parameters related to environmental management, it is difficult to establish an absolute quantity (unit/primary standard) by which all else can be ordered and measured. Instead, one must rely on the precision and "trueness" of analytical methods, supported by the absolute metrological control of measuring instruments wherever possible. The "calibration"/control of analytical capability ensures that the data produced by laboratory units working within a system of laboratories is comparable. By this means a kind of traceability of measurements, analyses, and tests can indeed be achieved.

1.2.2. Need for Establishment of Reference Laboratory Activities

The difficulties in establishing absolute quantities (primary standards) make it essential that the data from various laboratories on the same matrix is scientifically compared, and that such comparisons are periodically conducted, to control that each source of data continues to perform at a sufficient level of quality.

This need for comparable data is the driving force behind the concept of a reference laboratory. The appointment of national or regional reference laboratories within environmental management not only ensures comparable data within a country or region, but also internationally. It is suggested that a reference laboratory can play a role similar to that of a national metrology institute: to ensure comparable and traceable data at the highest level.

Important activities at environmental laboratories to ensure the comparability of laboratory data are:

- The use of certified reference materials (CRM);
- participation in inter-laboratory comparison studies, e.g., proficiency tests;
- documentation and validation of methods; and
- choice of methods, in cases where the obtained analytical results depend upon the technique applied.

1.2.3. The Reference Laboratory

Many European countries have already established or are planning to establish reference laboratories.

To comply with the conditions for reference laboratories currently being applied in Europe, a reference laboratory shall in general conduct its activities with the aim to ensure the comparability of data provided by a community of laboratories, and that the quality of the data is sufficient for environmental purposes. The appointment of a reference laboratory is very much needed to obtain a quality structure within the regional and other environmental laboratories.

To achieve these objectives, a reference laboratory must be capable of providing the following functions:

- evaluation and validation of methods for sampling, sample handling, analysis and data treatment, to provide information to authorities;
- participation in drafting and evaluation of new standards for methods;

- assistance to relevant authorities in preparation of requirements for data quality;
- organisation, co-ordination and conduction of inter-laboratory comparisons;
- service as a link between the laboratory community and laboratories in other countries, including international bodies;
- development of reference materials for use in the laboratories, or provision of information on availability of Certified Reference Materials for environmental laboratories;
- provision of information on all reference laboratory activities to laboratories and institutions functioning within the defined scope of work; and
- training of laboratory personnel in activities to ensure the required quality of results.

The reference laboratory shall conduct the activities outlined above on behalf of the authorities to ensure the quality of sampling techniques, analyses, tests, and measurements.

1.2.4. Laboratories Involved in Environmental Monitoring

It is proposed that the system of laboratories involved in environmental monitoring will consist of some Reference Laboratories (with different specialities) and a number of Regional Laboratories, where the Reference Laboratories will perform reference laboratory activities. One of the Reference laboratories will be responsible for general QA/QC-activities, such as the drafting of QA Manual requirements, auditing of laboratory quality systems and similar.

The establishment of reference laboratory activities will require additional training of staff and expansion of the capacity and the capability of the proposed laboratories, in order to allow them to function as Reference Laboratories.

1.2.5. The Reference Group

The reference laboratory activities will be co-ordinated, planned and evaluated by a group consisting of:

- a representative from each Reference Laboratory
- a representative from a central monitoring laboratory in any country not having a reference laboratory (if any)
- a representative from the CPU

The Reference Group will be collectively responsible for discussions, evaluations and decisions of the reference laboratory activities. They should meet regularly, twice per year is suggested.

1.2.6. Recommendations

It is recommended that:

- One laboratory takes on the role as CEP Reference Laboratory for general quality issues. It is proposed that State Inspectorate in Baku, Azerbaijan, is appointed reference laboratory for general QA/QC
- A number of reference laboratories become responsible for certain groups of parameters (see below).
- One laboratory is designated reference laboratory for sampling and sample handling procedures.
- All laboratories, including the Reference Laboratories, are equipped with a set of basic equipment, to enable them to monitor mainly input and local effects. The Reference Laboratories will, in addition and where needed, be equipped with certain additional equipment in order to be able to perform their tasks as reference laboratories.
- Some laboratories will perform ambient monitoring of the open sea, and will require some additional equipment for this (see “Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs”).
- The Reference Laboratories will, in addition and where needed, be equipped with certain additional equipment in order to be able to perform their tasks as reference laboratories (see “Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs”).
- For more advanced and/or expensive techniques, and where samples can be stabilised and transported, only the reference laboratory, or the reference laboratory and a very limited number of expert laboratories, will perform the analyses.
- Representatives of the Reference Group meet regularly to discuss analytical methods, quality control measures, proficiency testing and data format

Introduction of the internationally accepted requirements, new equipment and new techniques will require upgrading of the competence of the staff at the laboratory. It is, therefore, recommended that training of the staff should be an integrated part of the upgrading activities.

Laboratory	Responsibilities as Reference Laboratory
State Inspectorate, Baku, Az	Reference Laboratory for general QA/QC, such as: • Support to laboratories in introducing ISO 17025 • Auditing the activities of the other reference laboratories • Auditing the activities of the regional laboratories • Drafting minimum requirements for a QA manual • Drafting a manual for conduction of i/c exercises • Initiating meeting and leading the Reference Group • Training of laboratories in QA/QC
Central DOE, Tehran, Ir	Reference Laboratory for Basic Physical-Chemical Parameters
State Inspectorate, Dagestan, Ru	Reference Laboratory for inorganic trace elements in soil and sediments and biota
Food and Drug, Tehran, Ir	Reference Laboratory for Organic Compounds in biota (and sediments, if included)
Sanitary and Epidemic Station, Atyrau, Kz	Reference Laboratory for Radioisotopes and for Microbiological parameters
Caspian Fisheries, Ru *(unclear)*	Reference Laboratory for Hydrobiological Parameters
State Inspectorate, Baku, Az	Reference Laboratory for Ecotoxicilogical tests (bioassays)
State Committee for Ecology Lab in Baku (Azerekolab), Az	Reference Laboratory for sampling and sample handling procedures

1.3. Training Needs for Implementation of a Reference Laboratory Structure

Even though the importance of the “hardware” (equipment, facilities, etc.) for the data quality cannot be underestimated, it has to be stressed that the “software” (training, QA system, quality requirements, etc.) is of equal, if not higher, importance. It is very easy to consider a laboratory as upgraded when new equipment has been purchased and installed. However, no laboratory should be considered upgraded and running before it has been made absolutely clear that the staff is not only capable of handling the equipment properly, but that the laboratory has a documented system for procedures such as sampling and sample treatment, internal quality control and method validation. If such procedures do not exist, there is a significant risk that the new equipment will not lead to improvement in the data quality at all.

1.3.1. Training in the Handling of Basic Monitoring Equipment

It is proposed that basic monitoring laboratories are equipped with a set of equipment, which should be identical for all these laboratories. The suggested equipment consists of basic measuring tools such as pH-meters, and as far as possible test-kit type equipment for determination of parameters such as nutrients. Such test-kit analyses involve the use of ready-made reagents in disposable test tubes, and simple purpose-made spectrophotometers for reading the results.

The intention is that the equipment provided should also be capable of being handled by staff with little experience in modern analytical methods, thus making it possible to use all laboratories for the basic monitoring. Even though the equipment is meant to be fairly easy to handle, it will be necessary to provide training for some staff from each laboratory, as most of them will be totally unfamiliar with these new instruments. It is proposed that at least two persons from each laboratory (one technician and one chemist or laboratory manager is proposed) receive training by the manufacturer or the agent when the new equipment is installed. Training sessions should if possible be arranged for several laboratories on the same occasion, not only to reduce the costs but also to give the staff a well-needed opportunity to meet and discuss with staff from other monitoring laboratories.

1.3.2. Training in the Handling of Advanced Methods and Equipment

The laboratories that have been proposed as Reference Laboratories have been selected because they already have significant knowledge within their Reference Laboratory field. They have also been selected because they already possess certain pieces of necessary equipment, thereby reducing the needs for both hardware upgrading and specific equipment training. It will nevertheless be necessary to provide training for some of these laboratories when the hardware becomes upgraded.

Apart from analytical equipment, funding will be spent on improving the sampling, sample treatment and sample work-up facilities at the Reference Laboratories. The steps performed prior to the actual chemical analysis of the sample at the laboratory are often neglected parts of the analytical process, and adequate equipment, facilities and training for this should be provided to the Reference Laboratories.

Another crucial but often sadly ignored part of the analysis of biota and sediments is the determination of the so-called co-factors (for example water and lipid content of biota, particle size distribution and organic carbon content of sediments). In order to be able to compare the data for inorganic and organic components between samples with slightly different characteristics, the results are normalised against selected co-factors. The correct determination of co-factors is thus of equal importance to the usefulness of the resulting data as is the measurement of the actual determinant. Experience shows, however, that many laboratories have great difficulties in determining something as seemingly simple as the water content of fish tissue, and that the determination of co-factors in general is given low priority.

We can thus anticipate training needs for the Reference Laboratories, not only in handling new equipment, but for the laboratories handling solid samples, also in sample treatment and in the correct determination of certain co-factors.

1.3.3. Training in Internal Quality Control

Internal quality assurance and quality control are perhaps the most important tools to ensure internal comparability between the produced data from day to day in a laboratory. The fundamental tool for internal quality control is the use of so called control samples, and the corresponding use of control charts for plotting the results. Control samples can be either Certified Reference Materials provided by a competent manufacturer, or in-house material that has been verified against external standards (CRMs).

Before any series of sample measurements is commenced, a control sample is analysed. By the use of simple statistics it is possible to say, objectively, if the determination is under statistical control or not. If it is, the samples can be analysed. If not, the cause for the deviating control sample value has to be established before the series of environmental samples can be analysed. Note that control samples can be used not only to verify the analytical step. By using e.g. CRM sediments or certified biota materials, the whole chain of sample work-up steps can be included in the verification.

During visits to the laboratories it has been noted that a majority of the laboratories use inadequate methods for internal quality control, thus increasing the risk of not discovering temporary or permanent analytical problems. The staff at all involved laboratories, both basic monitoring laboratories and Reference Laboratories, should thus be trained in adequate methods for internal quality control. A short (3-5 days) course is needed. The course should include training on how to prepare samples for internal quality control, on how to apply the samples in practice, as well as on how to interpret and use the results in different types of control charts as a means for internal quality control in a laboratory. An appropriate software package for the laboratories should be selected, to be used for their internal quality control.

1.3.4. Training in Conducting Inter-comparison Exercises

One of the key roles for the proposed Reference Laboratories will be to improve the comparability between data produced by the different laboratories involved in the Caspian Environment Programme through organising and conducting inter-comparison exercises. In an inter-comparison exercise, identical samples are distributed to the participating laboratories, which are asked to analyse the samples for defined parameters and return the answers to the organiser. The concentrations of the samples must be unknown to the participants, but known by the organiser. The organiser writes a report on the outcome of the exercise. In this way, the single laboratory can build up a picture of how well it performs in comparison to other laboratories within the same field, and will also be made aware of their own results that deviate from the mean or assigned value of the distributed samples. The latter information should then be used for improving the analytical performance.

It is vital that the organisers of inter-comparison exercises are capable of:

- producing a homogenous sample batch, with a known uncertainty and within the desired concentration range
- defining the assigned ("true") concentration of the samples, either based on own measurements or on the results reported by the laboratories
- producing and distributing samples that are stable throughout the test period
- receiving data from the participants and handling these data
- defining and documenting a system for maintaining confidentiality
- producing a report which contains the results and some vital statistics
- supporting laboratories in interpreting results and explaining the statistics used

The staff responsible for conducting these exercises should thus receive training on how to perform inter-comparison exercises and on how to evaluate and present the data according to relevant international standards. It is suggested that a few key-persons from each Reference Laboratory participate in a course arranged by the consultants. Some of the Reference Laboratories should be provided with appropriate software for evaluation and presentation of results from inter-comparison exercises according to the statistics defined in the international standard ISO 5275.

1.3.5. Recommendations

It is strongly recommended that the upgrading of laboratory capacity is seen not only as upgrading the equipment and facilities, but that upgrading to a very large extent also is a question of education and training of the involved staff.

It is also recommended to consider the whole analytical chain in the planning of training activities, so that not only the actual analytical part is covered. Of equal importance is training on sampling, sample handling, pre-treatment and the determination of various co-factors used for interpreting the results.

It is furthermore recommended that all laboratories receive training on methods for internal quality control, and that the Reference Laboratories receive training on conducting inter-comparison exercises (external quality control) for the monitoring laboratories.

1.4. Monitoring Parameters and Laboratory Upgrading Costs

1.4.1. Monitoring Medium

Generally, three principal media can be used for the monitoring of an aquatic system: water, particulate matter and living organisms. Each medium has its own set of characteristics for monitoring purposes, such as

- applicability to water bodies,
- specificity to given pollutants,
- possibility of quantification such as flux determination,
- sensitivity to pollution, and

- time-integration of the information received.

Water is by far the most common monitoring medium used to date. The main reason for this is that water samples are comparatively easy and cheap to analyse with standardised methods, and easy to compare to water quality objectives. However, for many substances such as hydrophobic organic micro-pollutants and certain heavy metals, water is not the most appropriate medium to monitor because the water-solubility of these substances is very low and hence they concentrate in particulate matter and biota. Also, the analytical detection limit for some of the substances is very high compared to the water quality requirements for various uses.

So far, relatively limited experience exists in the Caspian Sea of other than water phase monitoring, but it is recommended to introduce monitoring of particulate matter and biota (including ecotoxicological tests, bioassays) on a more regular basis. Some laboratories are monitoring sediments and biota to some degree already, but there is certainly no overall CEP strategy behind these activities.

1.4.2. Monitoring of Industrial Water Effluents and River Input

Typical parameters for compliance monitoring of wastewater effluents are: temperature increase, pH, total suspended solids, chlorine, sulphide, ammonia, nitrate, phosphorous, biochemical oxygen demand, chemical oxygen demand, cyanide, oil and grease, phenol, coliform bacteria, and trace elements. Ecotoxicological tests should be applied to get a general picture of the toxicity of the effluent waters (see “monitoring of sediments and biota” below). In addition, certain industrial sites will require the monitoring of specific parameters, depending on their activities.

Many of these parameters can be determined by the use of relatively simple equipment or test kits such as Merck Spectroquant, Dr Lange and Hach equipment. Such test kits consist of prefabricated reagents and measuring instruments. Staff with limited or no expertise in analytical chemistry are able to use such kits after some training.

As mentioned trace organic and inorganic components are included in the list of parameters. Such analyses are to be performed at the respective Reference Laboratory, as the analyses require suitable facilities, considerable analytical expertise and heavy equipment. Furthermore, these parameters are stable (after preservation of the samples), making it possible to collect samples at different locations around the Caspian Sea and send them to the Reference Laboratory for analysis.

1.4.3. Monitoring of Ambient Water

There is a considerable interest in establishing routine ambient monitoring of seawater in the Caspian region. The primary objective of such monitoring is to establish the degree of large-scale pollution and to identify areas where the water quality has decreased due to pollution. Such a monitoring net should also include background monitoring stations, where the natural or normal background values of different parameters can be established.

The parameters to be measured for background monitoring include general physical chemical parameters such as

- indicators for general water quality: temperature, pH,
- gases: dissolved oxygen
- dissolved salts: sodium, potassium, calcium, magnesium, chloride, and sulphate
- nutrients: nitrogen and phosphorus containing compounds, such as ammonia, nitrate, orthophosphate, and organic compounds containing nitrogen and phosphorus.

The parameters to be measured for ambient monitoring (near coastal and open sea monitoring) include general physical chemical parameters such as:

- temperature and salinity
- indicators for general water quality: temperature, pH, suspended solids
- gases: dissolved oxygen and sulphide
- nutrients: nitrogen and phosphorus containing compounds, such as ammonia, nitrate, orthophosphate, and organic compounds containing nitrogen of phosphorus
- COD/BOD (or TOC if concentrations are not high enough)
- oil hydrocarbons
- chlorophyll a

Due to the instability of many of the above-mentioned parameters, it will be necessary to ensure that a few “ambient monitoring” (sea-going) laboratories have the capacity to measure these parameters, as most of them cannot be sampled, preserved and transported with a sufficient degree of reliability.

1.4.4. Monitoring of Sediments and Biota

The objectives of the sediment monitoring would be, as examples:

- To establish a better knowledge of the integrated pollution in selected areas of the Caspian Sea, and
- To be able to assess the retention of pollutants in the sediment, and thereby assess the significance of sediment in pollution transport.

Relevant aspects to cover would be sedimentation rates, particle size distribution, total nitrogen and total phosphorus, organic content and oxygen consumption of the sediment. Also concentrations of trace elements and hydrophobic organic micro-pollutants in sediments would be relevant measurements to take. Sediment monitoring, requiring some specialised analytical equipment and skills, should be performed by very few selected laboratories.

Biological monitoring in the Caspian Sea and its tributaries would have a good potential for detection of presence and assessment of effects of pollution. The general advantage of biological monitoring over water monitoring is that it provides an integrated measure of the pollution load. Two options are considered potentially relevant for the CEP. Both would

require substantial planning and preliminary surveys to establish detailed methodologies, train personnel, determine reference conditions, etc.

- Monitoring of pollution effects on ecosystems by indicator organisms:

By monitoring the absence/presence/abundance of certain indicator species (e.g. benthic macroinvertebrates) a pollution index may be determined for a location. Usually this method is used to assess organic pollution, which would be highly relevant. The usefulness of this approach can only be determined after a preliminary survey to identify appropriate indicator organisms.

The advantage of this kind of monitoring is its simplicity, once a baseline has been established. The disadvantage is the requirement for knowledge of taxonomy and occurrence of native species in the local environment (baseline). This is a clear task for one or two specialised laboratories with the properly trained staff.

- Monitoring by chemical analysis of biotic samples, such as fish, shellfish or plants:

Monitoring of biota would primarily take place in the form of specific surveys at recurrent intervals. The advantages of this kind of monitoring are that it is relevant to human health, and requires less advanced facilities than for determination of the same substances in the water phase, where the concentrations will be much lower. The disadvantage is the higher costs compared with the monitoring of indicator organisms.

The activities are to be seen as a pilot project for introducing sediment and biological monitoring as integral parts of the routine water-quality monitoring programme in the future. The following should be considered:

Planning. It is suggested that the starting point should be the extent of existing experience at the laboratories where sediment monitoring and biological monitoring has already been carried out on a (relatively) regular basis. This means that these laboratories both have certain experience in this field, and also have at least some of the necessary sampling and analytical equipment. The group of Reference Laboratories will be given the task of defining the pilot project. The same group will also decide on the equipment and methodology to apply.

Implementation. The duration of the pilot study will be at least one annual cycle in order to allow for detection of possible seasonal variations. The most significant equipment in need of upgrading for this component is sediment samplers and equipment for sample preparation and work-up. The group of Reference Laboratories will identify the necessary training in sampling, handling, preparation and preservation of samples, which can be given by the consultant in co-operation with experienced local staff.

1.4.5. Equipment and Staff Requirement

The equipment required at each laboratory depends upon the parameters to be measured, the methods to be applied, and the number of samples to be analysed.

The proposed *basic input monitoring*, which all regional laboratories should be able to carry out, requires equipment for measurements of the following parameters:

- Temperature (increase)
- pH
- Total suspended solids
- Oil and grease
- Ammonia, Nitrate, Phosphorous
- Total-N and Total-P
- Biochemical Oxygen Demand
- Chemical Oxygen Demand
- Dissolved Oxygen
- Fluoride, cyanide
- Phenol
- total and faecal coliforms

The two first parameters only require simple measuring instruments. The third and fourth require basic laboratory equipment (filtration equipment, oven and balance). It is proposed that the remaining parameters be analysed by use of test kits (coliforms using the tube method in most cases).

The total cost for a complete set of basic equipment is approximately US$ 80 000. The cost will be reduced for those laboratories where some instruments are already available. A rough estimate of the running costs for a maximum of 500 samples per year amounts to approximately
US$ 10 000.

The reference laboratories and some other specialised laboratories will be required to carry out measurements of other constituents, in addition to the basic set of parameters mentioned above. Some of these laboratories already have the required equipment, but it will be necessary to upgrade the equipment at all of them to a lesser or greater degree.

It is expected that two trained technicians are needed to carry out sampling and analyses for basic parameters. For the specialised parameters, the needs will be individual for each laboratory. Training of staff, both for analytical work and for sampling, is a most important part of the laboratory upgrading.

1.4.6. Choice of Analytical methods

As far as possible all analytical methods used should be international standard methods (ISO or EN standards). The use of harmonised and well-known methods is the best start when trying to reach data comparability. If the existing standard method has to be modified to fit the CEP better, this will be the responsibility of the respective Reference Laboratory.

1.4.7. Recommendations:

It is recommended that establishing or upgrading of environmental laboratory capabilities in the CEP is carried out on the basis of decisions made by the environmental authorities on

- structural and institutional arrangement to be implemented for the laboratories
- laboratories to be established/upgraded

In addition it is recommended that detailed investigations of existing facilities, equipment and possibilities for upgrading of available equipment be carried out.

Furthermore it is recommended that a group is established to plan and implement all necessary activities. It is suggested that representatives from the proposed Reference Laboratories should have a co-ordinating role during the establishment/upgrading of all laboratory capabilities.

Recommended parameters to be monitored, and estimated laboratory upgrading costs, are given below.

Proposed List of Water Quality Variables to be Monitored in the CEP

Type of variable	Type of monitoring					Reference to international standard method
	Back-ground	Input	Ambient	Sediment	Biota	
		Input moni-toring[1]	Near coastal and ambient water (open sea)	Sediment and river-borne particulate material		
General:						
Transparency			X			--
Temperature	X	X	X			--
pH	X	X	X			ISO 10523
Susp. Solids	X	X	X			ISO 11923
DO	X	X	X	X		ISO 5813, 5814
Conductivity	X	X				ISO 7888
Salinity			X			(Grasshoff*)
Total hardness	X					ISO 7980
Chlorophyll-a			X			DIN 38412-16
Nutrients:						
Ammonia	X	X	X			ISO 7150
Nitrate/nitrite	X	X	X			ISO 13395
Total nitrogen		X	X	X		ISO 11905
Phosphorus		X	X			ISO 6878, EN 1189
Total phosphorous		X	X	X		EN 1189
Organic matter:						
BOD_5	X	X	X			EN 1899
COD_{Cr}	X	X	X			ISO 6060
Major ions: (Na, K, Ca, Mg, F, Cl, SO_4)	X					EN/ISO 10304

[1] Monitoring of rivers, sewage, industries

Proposed List of Water Quality Variables to be Monitored in the CEP

Type of variable	Type of monitoring					Reference to international standard method
	Back-ground	Input	Ambient	Sediment	Biota	
Other inorganic: Cyanide		X				ISO 6703
Trace elements: Trace Elements (Notably As, Cd, Cu, Cr, Pb, Hg, Ni, Zn)				X	X	ISO 16590 (Hg) ISO 5666 (Hg) ISO 15586
Organic toxics: Oil hydrocarbons Oil and Grease Phenols		 X X	X X			ISO 10301 -- ISO 6439, 14402
Organic contaminants: Pesticides PCBs, HCBs etc				X X	X X	ISO 6468
Bioassays: Ecotoxicology		X		X		ISO 6341, 12890, 10706, 14669
Microbiological: Faecal coliforms Total coliforms		 X (X)				ISO 4831, 4832

Note 1: In addition to variables listed here, compliance monitoring will have to consider relevant variables associated with special pollution sources .

Note 2: A comprehensive list of priority pollutants is given in the report “Development of Priority List of Chemical Pollutants” (November 2001), and should be consulted when choosing the exact parameters for trace elements (inorganic pollutants) and organic pollutants.

Grasshoff = “Methods of Seawater analysis”, ed. Grasshoff et al., 1999 (Verlag Chemie, Germany)

Estimate of Equipment Cost per Laboratory

Laboratory	Will carry out	Basic equipment	Other laboratory equipment	Total cost for laboratory	Existing equipment assumed to be used
Kazakhstan					
Ministry lab, Atyrau	Basic monitoring	80 000		80 000	
Mangystau Regional lab	Basic monitoring	80 000		80 000	
Atyrau Sanitary and Epidemic Station	Reference Laboratory for Radioisotopes and Microbiology Basic monitoring	80 000	Microscopes, incubators, etc	140 000	Most equipment for microbiology exists but will be modernised
TOO Service Project, Aktau	Basic monitoring	80 000		80 000	
TOO Monitoring, Atyrau Old Airport	Basic monitoring Sampling/preservation for inorganic trace elements Ambient monitoring	80 000	Sample preparation equipment Fluorimeter Spectrophotometer Sampling equipment	160 000	New AAS exists (graphite??) Sampling equipment exists

Laboratory	Will carry out	Basic equipment	Other laboratory equipment	Total cost for laboratory	Existing equipment assumed to be used
Turkmenistan					
Control and Inspection, Turkmenbashi (Kaspekokontrol)	Basic monitoring Ambient monitoring	80 000	Sampling equipment Spectrophotometer Fluorimeter Chemicals, scale	140 000	
Russia					
Inspectorate, Astrakhan	Basic monitoring Background monitoring	80 000	Fluorimeter Spectrophotometer Ion Chromatograph	160 000	
Agrochemical Service Stn, Kalmykia	Basic monitoring	80 000		80 000	

Laboratory	Will carry out	Basic equipment	Other laboratory equipment	Total cost for laboratory	Existing equipment assumed to be used
Inspectorate, Dagestan	Reference Laboratory for Inorganic Trace elements in soil, sedimants, biota and water Basic monitoring Ambient monitoring	80 000	Graphite AAS Flourimeter Spectrophotometer Sample preparation Equipment Sediment sampler	220 000	
Caspian Fisheries Research Institute	Reference Laboratory for Hydrobiology Ambient monitoring		Spectrophotometer	50 000	
Azerbaijan					
State Inspectorate, Baku	Reference Laboratory for General QA/QC Reference Laboratory for Ecotoxicological Tests	80 000	Sample preparation equipment Scale	110 000	AAS
Azerekolab, Ecology, Baku	Reference Laboratory for Sampling Ambient monitoring Background monitoring	80 000	Sampling equipment Fluorimeter Spectrophotometer Ion chromatograph	140 000	Sampling equipment
Sumgayit State Ecology	Basic monitoring	80 000		80 000	
Hydrometeorology State Monitoring	Basic monitoring	80 000		80 000	

I.R. Iran					
Central DOE lab	Reference laboratory for Basic Physical-Chemical parameters Background monitoring Ambient monitoring	80 000	Spectrophotometer Fluorimeter	140 000	CTD and salinometers Sampling equipment
Gorgan DOE lab	Basic monitoring	80 000		80 000	
Sari DOE lab	Basic monitoring	80 000		80 000	
Chaloos DOE lab	Basic monitoring	80 000		80 000	
Anzali DOE lab	Basic monitoring	80 000		80 000	
Research Institute of Petroleum Industry	Basic monitoring	80 000		80 000	
Water and wastewater, Tehran	Basic monitoring	80 000		80 000	AAS
Fisheries Research, Anzali	Basic monitoring	80 000		80 000	
Food and drug, Tehran	Reference laboratory for Organic Compounds in Biota		Sample preparation equipment Standards, chemicals	50 000	GCs, GC-MS
Chemical Engineering Research Centre, Tehran	Should be used for special studies and method development/verification only			no basic costs	
Total cost for CEP				**2 350 000**	

Total cost summary

Investments	Cost, US$	Comments
Equipment for 6 Reference Laboratories	700 000	
Equipment for the 17 other laboratories	1 650 000	
Consultant support for arranging tenders, purchases, starting up Ref Lab activities etc.	210 000	12 m/m estimated
PCs for approx. 15 laboratories	50 000	Some labs lack PCs
Software for Internal QC	252 000	21 laboratories assumed
Software for inter-comparisons	48 000	4 Reference Laboratories
Training	440 000	
Grand total:	**3 350 000**	

Note that cost for local staff is not included

Running costs per year*	Cost, US$	Comments
Basic monitoring, 21 labs	210 000	
Special monitoring, 8 labs	120 000	
Inter-comparison exercises	30 000	
Certified Reference Materials	25 000	
Software upgrading	20 000	
Grand total:	**405 000**	

** "Running costs per year" is an approximation which does not include sampling costs (ship-time, travelling etc) or staff, only chemicals, gases, reagents, consumables etc for the analytical work.*

1.5. Decision Process for Defining Laboratory Capabilities

All laboratories need from time to time to adjust the scope of activities (measurements, tests and analyses) the laboratory offers. New parameters may be introduced and others may be excluded from the laboratory's list of measurements, tests, and analyses. In addition, new analytical techniques may replace older or perhaps more expensive techniques.

Therefore the environmental authorities and the laboratories have to establish tools for how the scope of activities offered to clients can be kept updated and how laboratories can avoid the use of measurements, tests, and analysis of limited value from a regulatory perspective, or which may be unnecessarily expensive.

1.5.1. Performance Characteristics

The important operational characteristics of a method are called *performance characteristics* and they provide the basis for selection of an appropriate methodology. Performance characteristics are not constant for a methodology since analytical values generated tend to be dependent or influenced by the user.

The most common performance characteristics needed to describe a method are

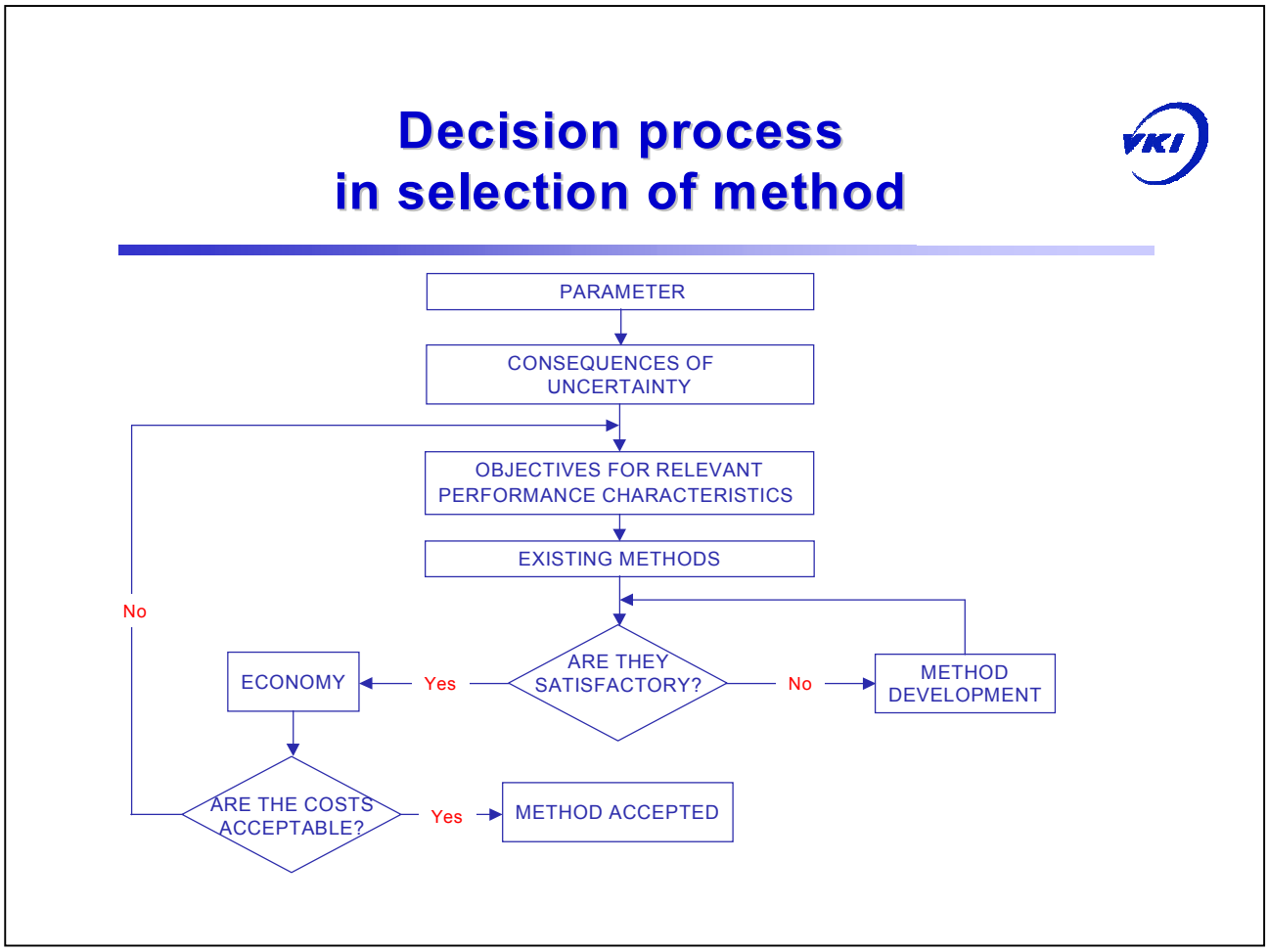
- limit of detection
- accuracy (systematic error)
- precision (random error)

1.5.2. Selection of Method

The choice of method will depend on several factors, the most important being the performance characteristics mentioned above. A stepwise procedure for making the choice is shown schematically in the figure below.

First the parameter to be determined is selected and the possible consequences of uncertainty are evaluated. Based on this, the relevant *performance characteristics and the target values* are selected*: the quality requirements (or quality objectives)* are established for the measurement, testing or the analytical process.

If existing methods are insufficient, a new method must be developed or the strategy for the investigation can be changed, resulting in a change in data quality objectives.



1.5.3. Recommendations

It is recommended that the environmental authorities and laboratories involved in activities related to management of the environment at appropriate intervals evaluate if the applied analytical methods meet quality requirements.

In addition, it is recommended that the environmental authorities and the laboratories at appropriate intervals should evaluate if the applied analytical methods could be replaced by methods that have better performance characteristics and may be also cheaper to perform.

It should be the responsibility of the Reference Laboratory to take the leading role in such activities, which could be initiated by such things as changes in legislation, changes in the water quality or development of new analytical or data evaluation tools. Regardless of such changes, a plan for regular re-evaluation of the performance characteristics must be constructed.

It is further recommended that the laboratories follow the decision process in the selection of methods to be used for parameters that have not previously been measured, tested or analysed.

Proposed Time Schedule for building of Reference Laboratory Structure

Total time = 24 months

Planned Activity	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**
Negotiations with proposed Ref Labs																								
Meeting of Ref Lab group to plan upgrading and ref lab activities																								
Ref Lab group to meet with Hydromet Committee																								
Exact needs of Ref Labs investigated																								
Exact needs of basic labs investigated																								
Call for tenders, evaluation of tenders																								
Equipment purchase and installation																								
Training in basic equipment																								

Planned Activity	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**
Training in advanced methods for Ref Labs																								
Training in Internal QC for all labs																								
Training in conduction of i/c exercises for Ref Labs																								
First i/c exercise on basic water parameters conducted and reported																								
Ref Lab group to discuss the quality needs and the list of monitored parameters and methods																								
Pilot project for sediment and biota monitoring planned and started																								
Pilot project conducted																								
Pilot project evaluated																								
Sediment and biota monitoring implementation																								

Chapter 2. Existing Laboratory Capacity

2.1. Introduction

The document gives a brief description of existing regional-level laboratories in the Caspian Sea littoral states, and an assessment of their current capacity. The capacity assessment builds upon the contents of a questionnaire, which was completed for each laboratory during a field tour to laboratories in Kazakhstan, Turkmenistan, Russia and Azerbaijan by Latifa Huseynova in June and July, 2000, and during a field tour to laboratories in I.R Iran by Mohammad Saed Abaee during the late summer/early autumn 2000. The mission reports and completed questionnaires from the two field tours are given in Annexes A and B, respectively.

Only laboratories identified as major analytical or monitoring laboratories in the first phase of TACIS were asked to submit information via the questionnaire. However, more informal information was nevertheless gathered from other laboratories as well.

The laboratories of the State Inspectorate, Baku, the Azerecolab of the State Committee for Ecology, Baku, and the State Committee for Ecology Laboratory in Sumgayit have also been visited by Mikael Krysell.

2.2. Existing Laboratories

The laboratories visited during the field tour are all environmental laboratories, performing analyses of water samples, and in most cases also air, soil or other media. The laboratories visited do not necessarily contribute data to the CEP today, but are in all cases potential data producers in the future.

So far no private or industrial laboratories performing environmental analyses have been investigated

2.2.1. Kazakhstan

In total, 10 laboratories were visited in Kazakhstan. Of these, four belonged to the Ministry of Natural Resources and Environment, and two to the Health Ministry. Furthermore, two academic institutions and two non-governmental institutions were visited. It should be noted that the Hydrometeorological Institute of Kazakhstan has no laboratory facilities.

There are completed questionnaires from five of these laboratories.

2.2.2. Turkmenistan

In Turkmenistan, the laboratories under the Ministry of Natural Resources and Environment in Turkmenbashi and Ashgabat were visited. The State Committee for Fisheries has no laboratory.

The only completed questionnaire comes from the laboratory in Turkmenbashi.

2.2.3. Russia

Three laboratories were visited in Russia, all being governmental institutions. Two of the laboratories work on general environmental issues, whereas one mainly concentrates on agricultural chemistry.

All three laboratories have completed the questionnaire, though with a varying degree of ambition.

2.2.4. Azerbaijan

Four governmental laboratories were visited in Azerbaijan, three in Baku and one in Sumgayit. Three of these laboratories belong to the State Committee of Ecology, and one was the laboratory of the State Hydrometeorology Committee.

All four laboratories have submitted information to the questionnaire.

2.2.5. I.R. Iran

Ten laboratories have been visited in I.R. Iran. Of these, five where laboratories belonging to the Department of the Environment (DOE) of Iran, three belonging to other governmental/communal services, one was an academic department and one was the Environmental Pollution Department of the Research Centre of the Petroleum Industry.

All laboratories have provided information to the questionnaires, though the four local DOE laboratories have completed a simplified version.

2.3. Comments on the Existing Laboratories

A brief overview of some important aspects of the capacity of the laboratories is given in table 1.“Mission Report Section” refers to the section in either of the summary mission reports, where the laboratory is mentioned. “Waters” as sample type means multiple water matrices (usually fresh surface water, groundwater, drinking water and wastewater).

Overview over Laboratory Capacities

Labs in Kazakstan	Mission report section	Staff – Education	Sample types	Facilities and equipment	QA/QC	External references
Ministry lab, Atyrau	2.1a	19 persons (17 academics + 2 administration)	Waters, soil, air	Poorly equipped. No AAS, GC etc. Some sampling equipment. 2 PCs	Very little info, in-house checks 4/month	Regional i/c exercises Standards from Ekogidrokhimengo Central lab, Almaaty
Mangystau Regional lab	2.1b	6 (5 academics + 1 sampling person)	Waste water, air	Equally poorly equipped. No sampling equipment. 2 PCs	No info	No i/c exercises Standards from GSO for some compounds
Atyrau Sanitary and Epidemic Station	2.2	Lab 17 (7 PhD, 10 tech) + field staff	Waters, soil, air +bacteriology	Slightly better equipped, but no AAS. No info on field equipment or PCs	Carried out according to Sanitary Guideline, state Standard etc	Some i/c between laboratory divisions Standards from Altey Co, Almaaty and St Petersburg

Labs in Kazakstan	Mission report section	Staff – Education	Sample types	Facilities and equipment	QA/QC	External references
TOO Service Project, Aktau	2.4.1	Approx. 7 (1-2 academics)	Groundwater + wastewater, soil, air	Small lab, poorly equipped. Field equipment for hydrographic and metals samples. 2 PCs	QA procedures exist	Appear to participate in i/c exercises No info on standards
TOO Monitoring, Atyrau Old Airport	2.4.2	Approx. 20 (12 academics, mainly chem. engineers)	Waters, sediments, soil, air and biota	Small lab, new bldg constructed. Reasonably equipped, mainly old equipment (new AAS, old chromatographs). Sampling equipment for water, sediments, biota, air. 4 PCs	Some internal i/c exercises Detailed method references QA procedures exist	National i/c by Ekogidrokhimengo, 3-4/yr Standards from Ekogidrokhimengo Central lab, Almaty
Labs in Turkmen.	**Mission report section**	**Staff – Education**	**Sample types**	**Facilities and equipment**	**QA/QC**	**External references**
Control and Inspection, Turkmenbashi	3.1	6-7 persons (4 chem. engineers)	Water and sediments, very basic parameters only	Poorly equipped, no financing for 5 years. Old-fashioned methods (Hg-method for NH_4 and Fe). Primitive sampling equipment for water and sediments.	Some in-house testing through parallel sampling	No i/c exercises No standards bought last 5 years

Labs in Russia	Mission report section	Staff – Education	Sample types	Facilities and equipment	QA/QC	External references
Inspectorate, Astrakhan	4.1	34 (25 academics)	Waters, soil, sediments, air	Reasonably equipped, though mainly old (GCs, AAS, Hg-analyser etc). Some field equipment. 3 PCs	In-house checks once per year QA manual exists	No i/c exercises Standards from ZAO EKROS
Agrochemical Service Stn, Kalmykia	4.2	72 (68 academics: chemists, biologists, agronomers)	Waters, sediments, soil, plants	Reasonably equipped (AAS, but no GC). 1 PC only. Sampling equipment for water and sediments	In-house checks according to standards QA manual exists	Participates in i/c exercises by CINAO, Moscow (annually) Standards from Ekoanalitika and VINIKhSZR (Moscow) and GP VNIIFTRI
Inspectorate, Dagestan	4.3	13 (9 academics)	Waters, soil, air	No info in questionnaire. 1 PC (reasonably equipped acc. to the mission report)	No info	No info

Labs in Azerbaijan	Mission report section	Staff – Education	Sample types	Facilities and equipment	QA/QC	External references
State Inspecto rate, Baku	5.1	11 (7 academics)	Waters, sediments, soil, air, sludge	Relatively well equipped (AAS, GC, LC, Hg-analyser). Equipment rather new in general. Have field equipment for water and sediments. 4 PCs (2 of these for instruments).	In-house tests (replicate samples etc). Use Licensed Methods from State Committee of Ecology	Have participated in international i/c (which?) Standards from EKROS, St. Petersburg, IWAKO, the Netherlands and Azerreakhim Equipment checked by state authorities
Azerekolab Ecology, Baku	5.2	16 (14 academics)	Waters, sediments, soil, biota, air	Relatively well equipped (New AAS, GC, LC). Runs PAH with GC and metals in biota. Sampling equipment for water and sediments. x PCs	EPA and ISO-methods. In-house tests QA manual exists	Have participated in international i/c 1995-1998 (ERTI Laboratory) Standards from IWACO, The Netherlands
Sumgayit State Ecology	5.3	12 (6 academics)	Waters, air	Poorly equipped (No functional AAS or GC). Old equipment. Some field equipment, not much. No PC	Very few QA procedures applied.	No i/c exercises In-house standards only, no material bought

Hydrometeorology State Monitoring	5.4	150+ staff (100academics)	Waters (no wastewater), soil, air	Poorly equipped, old equipment. Limited field equipment. PC??	No info	No info
Labs in I.R. Iran	**Mission report section**	**Staff – Education**	**Sample types**	**Facilities and equipment**	**QA/QC**	**External references**
Central DOE lab	1	20 (15 academics)	Waters, soil, air	Reasonably well equipped (AAS, new GC, old LC). CTD, salinometers and similar also available. 5 PCs	No in-house QC. Standard methods. QA manual, but very few written instructions	Stds from Merck, Fluka, IAEA etc. No national but some international ROPME i/c exercise. No CRMs
Gorgan DOE lab	1	5 (all academics?)	Waters, air	Reasonable equipment (AAS, no GC)	Standard Methods used	Samples have been exchanged
Sari DOE lab	1	3 (2 academics)	Waters	Just very basic equipment (no AAS, GC etc)	Standard Methods used	Samples have been exchanged
Chaloos DOE lab	1	5 (3 academics)	Waters	Basic equipment and an AAS.	Standard Methods. Some QC applied.	None
Anzali DOE	1	13 (several	Waters,	Reasonable. AAS, no GC.	Standard Methods.	Samples have been exchanged

lab		academics)	leachate		Some QC applied	
Labs in I.R. Iran	**Mission report section**	**Staff – Education**	**Sample types**	**Facilities and equipment**	**QA/QC**	**External references**
Research Institute of Petroleum Industry	2	Approx. 20 (15 academics)	Waters, soil, sediments, air, biota	Fairly well equipped (AAS, IR, TOC, no GC??). Have sampling equipment. Several PCs	International Standard methods used. In-house checks performed. QA manual and written instruction	Stds from Merck, Fluka etc Some i/c exercises, not regular (nat and internat) + in-house checks with other department
Water and wastewater Tehran	3	Ca. 25 (20 academics)	Waters, incl, coliforms and microbiology	Well equipped (2*AAS, 2*GC, 2*LC + others). Sampling equipment could be improved. Several PCs	Standard Methods In-house checks. QA Manual, but few written instructions	Stds from Merck, Fluka etc No i/c exercises
Fisheries Research, Anzali	4	Close to 20 persons (17 academics) + supporting adm.	Waters, microbiology	Mainly basic equipment for chemistry (AAS), but some portable equipment available. Sufficient sampling equipment	Standard Methods In-house checks. QA Manual and written instructions	Stds from Merck, Fluka etc Occasional national i/c exercises have been performed.

Labs in I.R. Iran	Mission report section	Staff – Education	Sample types	Facilities and equipment	QA/QC	External references
Food and Drug Control lab	5	More than 100, many academics	Very few water samples, mainly food and drugs	Very well equipped for organic analysis (several GCs and LCs, GC-MS, FTIR). AAS. No sampling equipment or standard water quality parameter analyses. Several PCs	Standard Methods of various kinds. In-house QC. QA Manuals and written protocols	Stds from WHO International i/c exercises (WHO, UNDP), no national.
Chemical Engineering Research Centre	6	15 (14 academics) + support from administration	Waters, sediments, soil, biota, leachate, solid waste (at varying frequencies)	Very well equipped. (AAS, several ICs and GCs, GC-FTIR, GC-MS etc.). No sampling equipment. Several PCs.	Taken from literature, no ordinary standard methods used. Certain in-house procedures exist	Stds from BDH, Merck, Aldrich etc. National i/c exercises

2.3.1. Activities of the Existing Laboratories

It is fair to say that all visited laboratories perform analyses that may be of interest for the Caspian Environment Programme, though it will be important to assess in detail which matrices and parameters the different laboratories have experience in. They all analyse water of different kinds, some specialising in certain water types. It is apparent that not all laboratories have experience in saline waters. The experience in analysing sediments and biota samples for environmental purposes is very limited (missing in most cases), though some experience does exist. A very limited number of laboratories have equipment suitable for such analyses.

2.3.2. The Capacities of the Existing Laboratories

The mission reports (Annexes A and B) summarise the most obvious problems and strengths revealed at the laboratories during the field tour. Of special importance to the data quality is the almost notorious lack of modern equipment, modern analytical methods and chemical reagents and standards. Limited budgets also place a severe restraint on laboratory operations, equipment maintenance and the purchase of high-quality standard materials. The situation in I.R. Iran is in general much better on these points than in the CIS countries, where the situation in Turkmenistan is most serious. There is also a pronounced lack in both in-house and external quality control procedures at most laboratories. All this leads to a low production rate of data, which is also in many cases of unknown quality.

The staff has in general a very good educational background, this is in principle true for all laboratories. There is thus an enormous potential for increasing the current laboratory capacity (both in the sense of data quality and in the number of data produced) if the laboratories can be modernised with relevant equipment, and the staff receive training on modern analytical methods and modern QA/QC-techniques.

2.3.3. Internal and External QA/QC

The procedures applied for both internal and external QA/QC is not up to modern standards in most laboratories. There appears to be no structures or existing schemes for national or regional inter-comparison exercises within the environmental field. Some laboratories claim to have participated in occasional exercises, but there are no regular activities taking place. No laboratories appear to be using certified reference materials (CRMs) to compensate, to some extent, for the lack of inter-comparison exercises. Thus the external references are normally limited to the use of calibrated scales for preparing in-house reference materials, - a procedure which is not good enough (particularly since some laboratories have problems in buying chemicals of the required purity). All this highlights, once more, how important it is for improving the data quality in the CEP to start up reference laboratory activities.

Internal quality control procedures, such as the use of control samples and control charts, are only in use in a very limited number of the laboratories. As this is one of the most

fundamental procedures for verifying the data quality in connection with the analysis, the lack of this practice is worrying.

2.3.4. Training of Laboratory Staff

Even though the educational background of the staff is generally very high, they are in many cases not familiar with the state-of-the-art analytical equipment, analytical methods and standardised QA/QC-procedures. However, as the staff in general has the necessary theoretical background, it should be a relatively simple task to train them in the most recent advances in analytical chemistry.

The training needs will be very different for staff from different laboratories, depending on which analyses they will be requested to perform and which equipment they will use in the future. Those laboratories that will mainly perform test-kit analyses will need a certain kind of training, whereas for more advanced equipment and techniques it might prove more useful to send staff away for on-the-job-training at up-to-date laboratories for an extended period. Some laboratories already maintain equipment and methods of the highest quality and have introduced modern in-house QA procedures, and are thus in less need of such exercises.

Reports that should be read in conjunction with this document:

Annex F: Mission report on Analytical and Monitoring Laboratories and completed questionnaires for 13 laboratories in Kazakhstan, Turkmenistan, Russia and Azerbaijan

Chapter 3. Laboratories' Requirements

3.1. Responsibility of Authorities for Provision of Reliable Data

Decades ago, when the environmental management was at an early stage, most measurements, analyses and tests were performed by individual laboratories, with little contact or co-operation with other laboratories. At that time, environmental problems were mainly perceived as damage to local areas, perhaps caused by specific industrial activities. Later on, it became clear that environmental problems could be transported from one area to another.

Today, it is recognised that management of environmental problems requires considerable effort and co-operation, both local and international, and that pollution should be controlled at the source, among other things:

- to prevent the generation of wastes that damage the environment,
- to improve the cost effectiveness of environmental management, and
- to identify the specific processes or steps where low-waste technology must be introduced to protect the environment and save natural resources.

Furthermore, it is recognised that protection and possible restoration of the ecological balance of natural ecosystems addresses among other things the need for policy and regulatory reforms, the control of pollution from point and non-point sources, and intensive national and international monitoring activities.

In most of the member states of the European Union, it is the responsibility of the environmental authorities to ensure that the environmental perspective is given proper consideration at all levels of the society. Among other things, the authorities should ensure environmental protection and the sustainable use of natural resources as they encourage and promote sustainable development.

In order to carry out these responsibilities, especially in view of the many ways human activity can affect the environment, authorities must be provided with reliable, comparable data from a variety of monitoring and assessment activities.

The provision of reliable laboratory data is a necessary element in describing the environmental state, ensuring that legislation and regulations are adhered to, and demonstrating the expected improvement of the environment. For both national and international environmental management, measurements, analyses, and tests are expected to be of known quality and must be comparable.

The comparison of data, however, has been very difficult, and impossible in some countries, because of the lack of sufficient quality assurance activities as well as limited co-operation between laboratories concerning choice of analytical technique and testing procedure.

An essential component of a well-functioning system is the appointment by the environmental authorities of a Reference Laboratory with sufficient facilities and equipment, a qualified, intelligent staff, broad and up-to-date experience in general environmental management, and routine experience in related laboratory activities. Strengthening this aspect of a laboratory system can greatly facilitate the work of the environmental authorities.

3.2. Samples and Parameters

3.2.1. Monitoring of Water Bodies

The analyses, tests, and measurements to be performed on water bodies at the laboratories should be related to management and control within the environmental sector.

Samples

Analyses, tests, and measurements related to the management and control of water bodies are performed not only on all types of water (ground water, surface water, waste water, leachate, drinking water, etc.), but also on solid sample materials, such as sediment, soil, sludge, waste, deposits, and biological material, where the results of the analyses may contribute to a better description of the environmental condition of water bodies or may give a quantification of the risk for contamination of water bodies.

Parameters

Fields related to the management of the water environment include the following:

- basic physical-chemical parameters;
- inorganic trace elements;
- organic compounds;
- radioisotopes;
- hydrobiological parameters;
- microbiological parameters;
- ecotoxicological tests.

In the following, these fields are subdivided into different groups on the basis of EU Directives and WHO Guidelines related to the protection of the environment.

The groups presented are based both on definitions of the analytical parameters and on the applied analytical techniques. The definitions may create some overlap between the different groups. For example, some ions (e.g., calcium, magnesium, iron, and manganese) are placed both under dissolved salts and under inorganic trace elements, because such compounds can be determined by the use of different techniques.

Another example is phenols, where the analyses can be performed both by spectrophotometry and gas chromatography (GC). Overlap is also found for other organic compounds. Such compounds may be determined by means of GC or high performance liquid chromatography (HPLC), but also by analysis of total organic carbon (TOC), and, if chlorinated, by techniques relevant for organic halogens (-OX.)

It is important that the definitions of the groups are in compliance with the requirements that are formulated by the environmental authorities concerning required techniques and the required quality of the analytical results. For example, the definitions should not include an

analytical technique for a specific parameter if the authorities do not accept the results obtained by the technique.

It is therefore necessary that the definitions and specifications of the different groups are evaluated periodically, e.g., whenever new legislation comes into force. Such an evaluation may reveal a need for updating the definitions of the parameter groups.

Basic Physical-Chemical Parameters

Group 1: Determination of **indicators for general water quality**: pH, alkalinity, conductivity, colour,

turbidity, suspended and dissolved solids, etc.

The measurements are performed using instruments such as pH meters, titration equipment (manual or automatic technique), conductivity meters, balances, ovens, and furnaces.

Group 2: Analyses for **dissolved gases**: oxygen, chlorine (free and bound), and hydrogen sulphide.

Group 3: Analyses for **dissolved salts**: calcium, chloride, fluoride, iron, magnesium, manganese, potassium, sodium, and sulphate.

The analyses are performed using ion chromatography, ion-selective electrodes (fluoride), titration techniques (chloride), spectrophotometry (e.g., iron and manganese), and flame photometry (e.g., sodium, potassium).

Group 4: Analyses for **nutrients**: compounds containing nitrogen and phosphorus, such as ammonia, nitrite, nitrate, orthophosphate, organic compounds containing nitrogen or phosphorus, and silicates.

The measurements are performed using titration techniques, ion chromatography, and spectrophotometry, often combined with automated techniques, such as flow technique (auto analyser or flow injection (FIA)). Sample materials for determination of the nitrogen or phosphorus content of organic compounds must undergo a digestion process before quantification.

Group 5: Analyses for **organic matter**: biochemical oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC), volatile organic carbon (VOC), different fractions of organic halogens (-OX), and chlorophyll.

The analyses are performed using highly standardised methods and include a digestion step and often a subsequent titration step. The analyses of TOC, VOC, and -OX include the use of instruments developed for the determination of these parameters. Spectrophotometry or fluorometry is used for determination of chlorophyll.

Group 6: Determination of different specified fractions of **detergents, cyanide (total and releasable), non-polar extractable substances, extractable substances**, and **phenols**.

The analyses of these specified fractions are performed by highly standardised methods and may include several of the following steps: extraction, evaporation (concentration), and quantification by spectrophotometry (visible or infrared), titration, ion-selective electrodes, or gravimetry.

Group 7: **Determination of taste and odour.**

Determination of taste and odour require most of all specially educated staff and sufficient facilities for performance of the analyses.

Inorganic Trace Elements

Group 8: **Inorganic trace elements** are those elements that can be analysed by means of advanced instruments, such as atomic absorption spectrophotometer (AAS), atomic emission spectrophotometer (e.g., for calcium, magnesium, sodium, and potassium), inductive coupled plasma (ICP) instruments, or other instruments to be used for determination of selected inorganic trace elements, such as hydride-generation instruments or mercury analysers. In most cases, the sample materials must undergo various digestion processes before the trace elements can be estimated.

Analysis for content of trace elements is mostly related to toxic metals, such as arsenic, cadmium, chromium, copper, lead, mercury, nickel and zinc, but several other trace elements are also of interest, e.g., aluminium, barium, beryllium, boron, cobalt, iron, manganese, molybdenum, selenium, silver, tin, and vanadium. Most trace elements are found naturally in the environment. The term "inorganic micropollutants" has typically been related to concentrations above acceptable values. The term **"inorganic trace elements"**, however, is to be preferred as the name for this group of compounds.

Organic Compounds

Group 9: **Organic compounds** in the context of environmental management include compounds, such as methane, phenols, and different classes of hydrocarbons, and organic micropollutants of anthropogenic origin, such as organohalogens.

Analyses of organic compounds are optimally performed by means of gas or liquid chromatography (GC or HPLC) combined with a suitable detector system, such as flame ionisation (FID), electron capture (ECD), or mass spectrometry (MS). In most cases, the sample materials must undergo various pre-treatment processes before quantification of organic compounds can be performed.

Radioisotopes

Group 10: The **radioisotope** group includes radioactive compounds, determined using equipment measuring alpha, beta, and gamma activity or these activities determined individually. Radioactive compounds are typically isotopes of cesium, hydrogen, radium, radon, tritium, strontium, uranium, and yttrium.

Hydrobiological Parameters

Group 11: **Hydrobiological parameters** include qualitative and quantitative characterisation of living organisms, such as algae and bottom organisms.

The analyses require the use of microscopes, etc.

Microbiological Parameters

Group 12: **Microbiological parameters** include investigations of different types of microorganisms, such as coliform bacteria (total and faecal) or other indicator organisms for water hygiene.

The analyses are performed using cultivation techniques involving use of incubators.

Ecotoxicological Tests

Group 13: **Tests for toxicity** for different test organisms (e.g., microorganisms, algae, daphnia, fish) under specified conditions and within a fixed time period.

Toxicity tests are performed using equipment for test systems, microscopes, thermostats, oxygen meters, and other measuring equipment.

Group 14: **Fate studies** estimating the fate (e.g., degradability, bioaccumulation, evaporation, absorption, or distribution) of substances under specified conditions.

The studies include physical-chemical characterisation and degradation studies (most of all, biodegradation studies) under specified conditions.

3.3. Requirements for Laboratories

A useful perspective for environmental laboratories includes the concept of traceability with regard both to equipment related to national standards and to environmental management, the establishment of reference laboratory activities, the parallel structures within each laboratory, and the implications of accreditation and approval.

3.3.1. Concept of Traceability

A fundamental principle in practical science is confidence in the ability to measure and compare metrological quantities. Traceability is the process whereby the indication of a measuring instrument (or a measure) can be compared in one or more stages with a national

standard for the metrological quantity in question. In each stage, a calibration is performed using a standard with a metrological quantity already determined by calibration with a higher level standard (the calibration hierarchy).

Exact definitions of calibration and traceability are given in the publication "International Vocabulary of Basic and General Terms in Metrology", International Bureau of Weights and Measures (BIPM), 1993.

At the international level, decisions concerning the international measurement system (SI) and the realisation of **primary standards** (standards that are designated or widely acknowledged as having the highest metrological qualities and whose value is accepted without reference to other standards of the same quantity) are made by the BIPM, which also organises interlaboratory comparison studies on the highest level.

From the primary standard, the **national standard** is established. In the most cases, a national metrology institute (the highest authority in metrology in almost all countries) maintains a national standard, the source of traceability for the physical quantity within the country.

The requirement of traceability is also a fundament of accreditation/approval procedures. The European Co-operation for Accreditation of Laboratories (EAL) (Publication reference EAL-G12, "Traceability of Measuring and Test Equipment to National Standards, November 1997") gives guidance on how to comply with traceability requirements in relevant standards, such as those ISO 17025 (corresponding to EN 45001). At appropriate intervals, an accredited laboratory shall compare its **reference standard** with the national standard.

According to the EAL document, traceability is characterised by a number of essential elements:

- An unbroken chain of comparisons, going back to the national or international standard;
- Measurement uncertainty: to be calculated for each step according to agreed methods and stated so that an overall uncertainty for the whole chain may be calculated;
- Documentation: each step in the chain must be performed according to documented and generally acknowledged procedures; and the results must also be documented;
- Competence: the laboratories of bodies performing one or more steps in the chain must supply evidence for their technical competence, e.g., by maintaining accreditation;
- Reference to an SI unit: the chain of comparisons must end at a primary standard;
- Re-calibrations: calibrations must be repeated at appropriate intervals; the length of these intervals will depend on a number of variables, e.g., uncertainty required, frequency of use, way of use, stability of the equipment.

At individual laboratories, in-house systems should whenever possible ensure that all measuring and test equipment used in a laboratory is calibrated regularly against its own reference standard**,** which is calibrated against a reference standard at an accredited laboratory.

For many parameters related to environmental management, it is difficult to establish an absolute quantity (unit/primary standard) by which all else can be ordered and measured. Instead, one must rely on the precision and "trueness" of analytical methods, supported by the absolute metrological control of measuring instruments wherever possible. The "calibration"/control of analytical capability ensures that the data produced by laboratory units working within a system of laboratories is comparable. By this means, within the science of environmental management and control, a kind of traceability of measurements, analyses, and tests can indeed be achieved.

3.3.2. Need for Establishment of Reference Laboratory Activities

The difficulties in establishing absolute quantities (primary standards) make it essential that the data from various laboratories on the same matrix is scientifically compared, and that such comparisons are periodically conducted, to control that each source of data continues to perform at a sufficient level of quality.

This need for comparable data is the driving force behind the concept of a reference laboratory. The appointment of national or regional reference laboratories within environmental management not only ensures comparable data within a country or region, but also internationally. It is emphasised that many environmental management activities involve laboratories from several countries.

It is suggested that a reference laboratory can play a role similar to that of a national metrology institute: to ensure comparable and traceable data at the highest level (i.e., smallest uncertainty (highest precision) and/or lowest detection limit). Authorities appointing environmental reference laboratories should therefore take into consideration the criteria used for appointment of metrology institutes.

Important activities at environmental laboratories to ensure the comparability of laboratory data are:

- The use of certified reference materials ("reference materials accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence", BIPM 1993);
- holding of or participation in interlaboratory comparison studies, e.g., proficiency tests;
- documentation and validation of methods; and
- choice of methods, in cases where the obtained analytical results depend upon the technique applied.

3.3.3. Parallel Structures For Laboratories

The structure of laboratories involved in environmental management can be formulated in two ways:

- the physical structure, which describes the laboratory with regard to the various aspects of sampling, analysis, testing, and other measurements and defines both capability and capacity, and
- the quality structure, which describes the status of the laboratory with regard to the requirements necessary to ensure quality data and the responsibility of the laboratory in this respect.

In a publicly funded system of laboratories, a formal assessment (delineation) of the physical laboratory structure would help ensure that all required laboratory activities can be performed, avoid duplication of work and work capacity, and allow resources to be allocated where they are of most benefit. The physical structure can also be evaluated on the basis of its capability to deliver the amount and kinds of data required by the system user, in this case, the environmental authorities.

Requirements concerning the quality structure of laboratories, on the other hand, apply to all laboratories (both public and private) that provide any kind of data for environmental management. A minimum requirement for a quality structure at any testing laboratory is given in the standard ISO 17025 “General requirements for the competence of calibration and testing laboratories”. The ISO Standard includes the requirements for a quality system, to be documented in a quality manual, but the standards do not include requirements concerning the needed quality level of the data. Such quality requirements (performance criteria, e.g., choice of methods, required precision, required detection limit) must be clearly defined by the users of the laboratory data, e.g., the environmental authorities or other organisations or clients. A quality system can then ensure that these requirements are met by the laboratory.

3.3.4. Requirements in ISO Standard 17025

The ISO Standard 17025 “General requirements for the competence of calibration and testing laboratories” has recently been adopted.

The International Standard specifies the general requirements for the competent authority to carry out measurements, tests and analysis including sampling. The ISO Standard is applicable to all laboratories regardless of the number of personnel or the extent of the scope of measurements, tests, and analyses.

The ISO Standard covers both management requirements and technical requirements. The management requirements define requirements for organisation of the laboratory, for a documented quality system, and document control and other activities related to quality management of laboratory activities. Fulfilment of all requirements for the management of a laboratory in the ISO Standard allows the laboratories to act as an independent third party body.

The technical requirements define requirements related to the competence of personnel, the accommodation and environmental conditions, and methods to be used for measurements, tests, and analyses. In addition the technical requirements include use of reference materials and participation in interlaboratory comparisons to assure and document the quality of obtained results.

Additional information can be found in the ISO Standard.

3.3.5. Definition of Scope of Laboratory Activities

The European Co-operation for Accreditation of Laboratories (EAL) (Publication reference EAL-P10, "The Scope of Accreditation and Consideration of Methods and Criteria for the Assessment of the Scope in Testing, February 1997") gives guidance on how to define the scope of accreditation. However the guidance can also be used to define the scope of laboratory activities.

Scope parameters are:

- Testing field (e.g. chemical, biological);
- The material considered (e.g. water, sediment, air);
- The measurement, test, or analysis.

In addition the measurement, test or analysis should be identified in terms of:

- Quantity (property to be estimated);
- Range of measurements (detection limit);
- Uncertainty (precision).

Descriptions of fields, testing materials and measurement, test and analyses relevant for laboratories involved in environmental management are found in section 2 of this document.

3.4. The Reference Laboratory

Many European countries have already established or are planning to establish reference laboratories.

Denmark established the first reference laboratory in 1976. In 2000 the Danish National Environmental Protection Agency operate several reference laboratories. In the Nordic countries, several laboratories conduct reference laboratory activities.

Some former Eastern European countries e.g., the Slovak Republic and the three Baltic States, have established or are in the process of establishing reference laboratories.

3.4.1. Tasks

To comply with the conditions for reference laboratories currently being applied in Europe, a reference laboratory shall in general conduct its activities, taking into account the resources available, with the aim to ensure the comparability of data provided by a community of laboratories, and that the quality of the data is sufficient for environmental purposes. The appointment of a reference laboratory is very much needed to obtain a quality structure within the regional and other environmental laboratories.

To achieve these objectives, a reference laboratory must be capable of providing the following functions:

- evaluation and validation of methods for sampling, sample handling, analysis and data treatment, to provide information on method performance to authorities;
- participation in drafting and evaluation of new standards for methods, e.g., participation in standardisation activities;
- assistance to relevant authorities in preparation of requirements for data quality;
- organisation, co-ordination and conducting of interlaboratory comparisons;
- service as a link between the laboratory community and laboratories in other countries, including participation in interlaboratory comparison studies arranged by international bodies or foreign countries;
- development of reference materials or quality control samples for use in the laboratories, or provision of information on availability of Certified Reference Materials for environmental laboratories;
- provision of information on all reference laboratory activities to laboratories and institutions functioning within the defined scope of work; and
- training of laboratory personnel in activities to ensure the needed quality of results.

The reference laboratory shall conduct the activities mentioned above on behalf of the authorities to ensure the quality of sampling techniques, analyses, tests, and measurements.

It is normally necessary to establish several reference laboratories in order to cover all parameters, concentration ranges and matrices. There should, however, only be one reference laboratory for each type of measurement, analysis, or test. The division of tasks between several reference laboratories may be based on matrices, e.g., water, air, solid waste, or on technical areas, e.g., microbiology, chemistry and ecotoxicology.

3.4.2. Criteria

On the basis of requirements for national meteorological institutes, the criteria for the reference laboratories can be defined as described below:

The laboratory shall have the necessary capabilities and the capacity in terms of equipment and staff to allow it to function as a national reference laboratory within the defined scope. The needed capabilities and capacities are as follows:

- The reference laboratory shall have the necessary facilities, equipment, and experience to carry out sampling, measurements, analyses, and tests of relevance to environmental management;
- The reference laboratory shall fulfil requirements set up in internationally accepted documents (for example the ISO Standard 17025);
- The documented quality system of the reference laboratory shall cover measurements, analyses, and tests as well as reference laboratory activities, such as performance of interlaboratory comparisons, etc.;
- The staff of the reference laboratory shall be competent concerning the measurements, tests, and analyses performed and the reference laboratory activities and have general knowledge of relevant legislation;
- The reference laboratory shall be aware that it is acting on behalf of the environmental authorities when reference laboratory activities are being performed, and the reference laboratory shall give these activities high priority and perform the specified activities as agreed.

3.5. Structural and Institutional Arrangement

The establishment of structural and institutional arrangement for environmental laboratories should be based on internationally agreed principles and tailored to Caspian conditions.

3.5.1. Laboratories Involved in Environmental Monitoring

It is proposed that the system of laboratories involved in environmental monitoring should consist of some Reference Laboratories (with different specialities) and a number of Regional Laboratories. Both the Reference Laboratories and the Regional Laboratories are expected to conduct routine measurements, tests, and analyses. In addition, the Reference Laboratories will perform reference laboratory activities. One of the Reference laboratories will be responsible for general QA/QC-activities, such as the drafting of QA Manual requirements, auditing of laboratory quality systems and similar.

The establishment of reference laboratory activities will require additional training of staff and expansion of the capacity and the capability of the proposed laboratories, in order to allow them to function as the Reference Laboratories in accordance with the criteria stipulated above in section 4.

The Regional Laboratories are already established and located in areas where their routine measurements, tests and analyses will be performed. For more details on the physical

upgrading of both Reference and Regional laboratories, see “Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs”.

In addition to the publicly funded laboratories it is possible that a number of private and industrial laboratories will eventually be involved in environmental management. This is, however, not within the scope of this phase of the project.

3.5.2. Structure for Environmental Laboratories

It is proposed that both a physical and a quality structure are established, and that establishment of the structure is based on the principles for such structure described in section 3.3.

All laboratories should be able to demonstrate that the work conducted is in compliance with the requirements.

Physical Structure

A physical structure should be established for all involved laboratories, and the establishment of the physical structure should create the necessary background to avoid duplication of work and work capacity, and allow resources to be allocated where they are of most benefit.

It is proposed that the physical structure is established based on an evaluation, and decisions about which activities in terms of sampling, measurement, testing, and analysis each laboratory should carry out. The decisions taken should be documented, and it is suggested that documentation follow the guidance given in the document EAL-P10 mentioned in section 3.5.

If the suggestion is followed then a document should be prepared and made available for each laboratory. Such a document should specify testing fields covered and types of sample analyses performed in the laboratory. For each testing field and for each sample type, a list of measurements, tests and analyses to be done routinely at the laboratory should be prepared.

Re-evaluation of the scope of the activities of the laboratories should be repeated at appropriate intervals.

Quality Structure

In addition to a physical structure a quality structure should also be established for all laboratories involved in environmental management.

The establishment of a quality structure requires that all laboratories involved in environmental monitoring shall fulfil relevant requirements in ISO Standard 17025 “General requirements for the competence of calibration and testing laboratories” sufficient to ensure reliable data.

In addition all environmental laboratories shall fulfil quality requirements set up by the environmental authorities having the necessary power to issue such requirements.

It is suggested that the environmental authorities begin to formulate such requirements (see "Defining Laboratory Capacities".

3.5.3. The Reference Group

The reference laboratory activities will be co-ordinated, planned and evaluated by a group consisting of:

- a representative from each reference laboratory
- a representative from a central monitoring laboratory in any country not having a reference laboratory (if any)
- a representative from the CPU

The Reference Group will be collectively responsible for discussions, evaluations and decisions of the reference laboratory activities. They should meet regularly, twice per year is suggested, to discuss the following matters:

- The choice of methods for sampling and analysis
- Needs for development of, or validation of, new methods
- Plans for performance of proficiency testing
- Evaluation of earlier proficiency testing
- Quality control issues
- Mutual data reporting formats

The Reference Laboratory for general QA/QC is responsible for:

- Invitations to the meetings
- Drafting and circulating agendas
- Chairmanship of the meetings
- Writing reports from the meetings, including an action list
- Circulating the meeting reports, with decisions and other important matters highlighted, to all CEP laboratories and to the CPU

3.5.4. Fee Structure for Analytical Services

The laboratories may offer their expertise and services to other (non-government) clients for example sampling, measurement, testing, and analysis could be provided on a profit or cost recovery basis.

The Reference Laboratory may also offer reference laboratory activities to other clients.

However, clients should cover the costs of such activities. It is suggested that terms of such activities be laid down in an agreement before the work is carried out. Representatives of the laboratory and the client shall sign the agreements.

Finally it is proposed the laboratories requiring services of the Reference Laboratory should pay. Fees could cover, at least partly, procurement of reference materials, and participation in interlaboratory comparisons or training arrangements to be held by the Reference Laboratory.

3.5.5. Self-monitoring of Industries

Though not within the scope of this phase of the project, it is obvious that in the future the activities carried out at industrial and/or private laboratories should undergo a similar process as the major laboratories are proposed to do in this phase. It will thereby be possible to considerably increase the number of useful data available to the environmental authorities. The fulfilment of both:

- relevant requirements in ISO Standard 17025 “General requirements for the competence of calibration and testing laboratories”; and
- quality requirements set up by the environmental authorities of a private or industrial laboratory creates the necessary background for production of reliable data at these non-governmental laboratories.

However the acceptance of data provided by private/industrial laboratories should be based on clear evidence that all relevant requirements are met.

Therefore it is proposed that in the future private/industrial laboratories should apply for acceptance of produced data and declare that the requirements will be met and that the laboratory will accept inspection of its laboratory’s facilities, equipment, required documentation etc. In addition the scope of laboratory activities for which the laboratory’s data should be accepted should be defined using the principles mentioned above.

A competent body needs to be established to handle applications and carry out the inspections. The Reference Laboratory for general QA/QC is a natural candidate, though measures must be taken to ensure that it has the necessary independent status to be such a body. In those countries where a national accreditation body is in function, experts from that body can be used for these inspections.

3.6. Recommendations and Proposed Reference Laboratories

It is recommended that the authorities in the Caspian Sea riparian states are aware of the difficulties related to provision of reliable data for environmental management.

It is also recommended that the authorities in said countries are aware of the criteria for laboratory activities laid down in internationally accepted documents such as the European Standard EN 45001 "General Criteria for the Operation of Testing Laboratories" and the International Standard ISO/IEC 17025 “General requirements for the competence of calibration and testing laboratories”.

In addition it is recommended that the requirements in the mentioned documents and necessary additional requirements set up by the relevant authorities be introduced during the

upgrading of the laboratory structure. In the end all laboratory activities conducted for the Caspian Environment Programme should be carried out in compliance with these requirements.

The management of the environment requires that the laboratories should be capable of handling a high number of analyses and a number of sample materials, which again will require investment in facilities, equipment, glassware etc.

However, it is not realistic to procure equipment and implement analytical techniques for all parameters and all sample types at all laboratories. It is, therefore, recommended that:

- One laboratory takes on the role as CEP Reference Laboratory for general quality issues. It is proposed that State Inspectorate in Baku, Azerbaijan, is appointed reference laboratory for general QA/QC (Table 1)
- A number of reference laboratories become responsible for the groups of parameters defined in Section 2 above (Table 1)
- One laboratory is designated reference laboratory for sampling and sample handling procedures
- All laboratories, including the Reference Laboratories, are equipped with a set of basic equipment, to be able to mainly perform input and local effects monitoring.
- Some laboratories will perform ambient monitoring of the open sea, and will require some additional equipment for this (see “Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs”).
- The Reference Laboratories will, in addition and where needed, be equipped with certain additional equipment in order to be able to perform their tasks as reference laboratories (see “Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs”).
- For more advanced and/or expensive techniques, and where samples can be stabilised and transported, only the reference laboratory, or the reference laboratory and a limited number of expert laboratories, will perform the analyses (see “Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs”).
- Representatives of the Reference Group meet regularly to discuss analytical methods, quality control measures, proficiency testing and data format (Section 5.3)

It can be foreseen that the introduction of the internationally accepted requirements, new equipment and new techniques will require upgrading of the competence of the staff at the laboratory. It is, therefore, recommended that training of the staff should be an integrated part of the upgrading activities.

Based on the recommendations listed above for the upgrading of laboratories the upgrading could consist of the following:

- Introduction of and finally fulfilment of internationally accepted requirements for laboratories

- Upgrading of the laboratory facilities
- Procurement and installation of equipment, glassware etc.
- Introduction and implementation of analytical techniques
- Training of staff

Finally it is recommended that an overall plan be prepared for the whole upgrading as well as a detailed plan for the upgrading activities for the coming year.

Proposed Reference Laboratories

Laboratory	Responsibilities as Reference Laboratory
State Inspectorate, Baku, Az	Reference Laboratory for general QA/QC, such as: • Support to laboratories in introducing ISO 17025 • Auditing the activities of the other reference laboratories • Auditing the activities of the regional laboratories • Drafting minimum requirements for a QA manual • Drafting a manual for conduction of i/c exercises • Initiating meeting and leading the Reference Group • Training of laboratories in QA/QC
Central DOE, Tehran, Ir	Reference Laboratory for Basic Physical-Chemical Parameters (group 1-7)
State Inspectorate, Dagestan, Ru	Reference Laboratory for inorganic trace elements in soil and sediments and biota (group 8)
Food and Drug, Tehran, Ir	Reference Laboratory for Organic Compounds in biota (and sediments, if included) (group 9)
Sanitary and Epidemic Station, Atyrau, Kz	Reference Laboratory for Radioisotopes (group 10) and for Microbiological parameters (group 12)
Caspian Fisheries, Ru	Reference Laboratory for Hydrobiological Parameters (group 11)
State Inspectorate, Baku, Az	Reference Laboratory for Ecotoxicilogical tests (group 13-14)
State Committee for Ecology Lab in Baku (Azerekolab), Az	Reference Laboratory for sampling and sample handling procedures

Chapter 4. Training Needs

4.1. Introduction

When upgrading laboratories involved in environmental monitoring the ultimate goal is, quite naturally, to provide decision-makers at different levels with more reliable and useful information on the state of the environment. There is no doubt that the chances of making the correct decisions concerning environmental protection, legislation and allowable discharges will increase if the data provided by the laboratories is of the correct and expected quality. However, experience and earlier investigations show that the produced and reported data is far from always of the correct quality for its purpose. This could mean, for example, that the data made available by the laboratories is of incorrect or unknown accuracy, has been sampled at inappropriate locations or with inappropriate frequency, or that the inappropriate parameters have been monitored. There are several possible reasons for why data of incorrect (usually too low) quality is being produced, despite efforts to avoid this. Some of the most frequently identified reasons are (not in prioritised order):

- The laboratory does not have the necessary equipment
- The laboratory facilities are inadequate
- The laboratory does not have access to chemicals and standards of the required quality
- There are no systematic quality assurance or quality control activities
- The staff does not know how to handle their equipment
- The samples are not sampled and/or treated correctly
- The methods used are not suitable for the matrix or the concentration range or the available equipment
- The purpose of the monitoring has not been sufficiently established by the authorities
- The required data quality has not been defined (or communicated) by the data users

Even though the importance of the “hardware” (equipment, facilities, etc.) cannot be underestimated, it has to be stressed that the “software” (training, QA system, quality requirements, etc.) is of equal, if not higher, importance to the ultimate quality and usefulness of the produced data. Moreover, it is very easy to consider a laboratory as upgraded when new equipment has been purchased and installed. However, no laboratory should be considered upgraded and running before it has been made absolutely clear that the staff is not only capable of handling the equipment properly, but that the laboratory has a documented system for procedures such as sampling and sample treatment, internal quality control, analysis and method validation. The international standard ISO 17025 “General requirements for the competence of testing and calibration laboratories” is an excellent source of useful requirements. If such procedures do not exist, there is a significant risk that the new equipment will not lead to improvement in the data quality at all.

This document describes in some detail the background to different training needs that have been identified for the laboratories in the Caspian Environment Programme. The proposed training is linked to the suggested upgrading of equipment and to the proposed structure for Reference Laboratories. The estimated costs for training (and equipment) are summarised in "Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs".

4.2. Training in the Handling of Basic Monitoring Equipment

In "Requirements for Laboratories in the Caspian Environment Programme" a Reference Laboratory structure has been outlined. According to the proposed structure, a few laboratories will act as Reference Laboratories for different types of monitoring (specified parameter groups) in the Caspian Environment Programme, whereas the other laboratories will perform monitoring of a set of basic parameters at sources, in rivers and in coastal waters. The proposed basic monitoring laboratories are in some cases poorly equipped today, whereas some are quite well equipped and will perform other kinds of analyses as well, for other reasons than the CEP. It is, however, proposed that for monitoring of the Caspian Sea they will only perform some basic parameters, whereas more advanced and/or expensive analyses will be carried out by a few laboratories only.

It is furthermore proposed that the basic monitoring laboratories are equipped with a set of equipment, which should be identical for all these laboratories. The suggested equipment consists of basic measuring tools like pH-meters, and as far as possible test-kit type equipment for determination of parameters such as the nutrients. Such test-kit analyses involve the use of pre-fabricated reagents in disposable test tubes, and simple purpose-made spectrophotometers for reading the results. This will not only reduce the danger of contamination of samples and reagents during the analyses, but will also significantly decrease the need for preparation and storage of reagents, stock solutions etc. in an (potentially) unsuitable environment using (possibly unsuitable) pure chemicals.

The intention is that the provided equipment should be possible to handle also for staff with little experience in modern analytical methods, thus making it possible to use all laboratories for the basic monitoring. Providing a large number of laboratories with identical test-kit based equipment should also drastically increase the possibility of producing comparable data, even at laboratories where the facilities in principle are inadequate for most kinds of laboratory work.

Even though the equipment is meant to be fairly easy to handle, it will be necessary to provide training for some staff from each laboratory, as most of them will be totally unfamiliar with these new tools. It is proposed that at least two persons from each laboratory (one technician and one chemist or laboratory manager is proposed) receive training by the manufacturer or the agent when the new equipment is installed. Training sessions should if possible be arranged for several laboratories on the same occasion, not only to reduce the costs but also to give the staff a well-needed opportunity to meet and discuss with staff from other monitoring laboratories.

Recommendation: Two persons from each laboratory performing basic monitoring (approximately 18 laboratories) should receive training in the handling of the new equipment

4.3. Training in Advanced Methods and Equipment

The laboratories that have been proposed as Reference Laboratories in "Requirements for Laboratories in the Caspian Environment Programme" have been selected because they already have significant knowledge within their Reference Laboratory field. They have also been selected because they already possess certain pieces of necessary (and sometimes expensive) equipment, thereby reducing the needs for both hardware upgrading and specific equipment training. Whereas the exact needs for upgrading of equipment has to be discussed with the separate laboratories once the Reference Laboratory structure has been agreed, it is nevertheless possible to at least estimate the need for training that the hardware upgrading will result in.

Apart from equipment such as atomic absorption spectrometers, gas chromatographs and ion chromatographs, funding will be spent on improving the sampling, sample treatment and sample work-up facilities at the Reference Laboratories. The steps performed prior to the actual chemical analysis of the sample at the laboratory are often neglected parts of the analytical process, and adequate equipment and facilities for this has to be provided to the Reference Laboratories.

Another crucial but often sadly ignored part of the analysis of biota and sediments is the determination of the so-called co-factors (for example water and lipid content of biota, particle size distribution and organic carbon content of sediments). In order to be able to compare the data for inorganic and organic components between samples of slightly different characteristics, the results are normalised against selected co-factors. When normalisation has been carried out, the analytical data in for example fish of different age classes and size, and sediments from different locations, can be compared with much greater accuracy. The correct determination of co-factors is thus of equal importance to the usefulness of the resulting data as is the measurement of the actual determinant. Experience shows, however, that many laboratories have great difficulties in determining something as seemingly simple as the water content of fish tissue, and that the determination of co-factors in general is given much lower priority and less resources than the determination of the actual pollutants.

We can thus anticipate training needs for the Reference Laboratories, not only in handling new equipment, but, for the laboratories handling solid samples, also in sample treatment and work-up and in the correct determination of certain co-factors.

Recommendation: The Reference Laboratories receiving new equipment for analysis should be given adequate training, from the manufacturer or any other competent body. Furthermore, training should be provided, for the laboratories that analyse biota and sediments, in sample handling and in the determination of co-factors. This training can only be provided by experts within the field, and should preferably be carried out at a selected laboratory in a western country.

4.4. Training in Internal Quality Control

Internal quality assurance and quality control are the perhaps most important tools to ensure internal comparability between the produced data from day to day in a laboratory. The fundamental tool for internal quality control is the use of so called control samples, and the corresponding use of control charts for plotting the results. A control sample is a sample which has a known and well-defined content of the determinant, and which is used to make sure that the analytical equipment gives the correct answer (i.e., that it has been calibrated properly and that the calibration is still valid). Control samples can be either Certified Reference Materials provided by a competent manufacturer, or in-house material which has been verified against external standards (CRMs). It is crucial that the control samples have similar characteristics as the environmental samples that are to be analysed, i.e. similar concentration range and matrix.

Before any series of sample measurements is commenced, a control sample is analysed. The result is plotted in a control chart, which gives a convenient overview of the control sample results over time. By the use of simple statistics it is possible to say, objectively, if the determination is under statistical control or not. If it is, the samples can be analysed. If not, the cause for the deviating control sample value has to be established and a new control sample analysed and deemed to be under statistical control, before the series of environmental samples can be analysed. The principles and the statistics are outlined in detail in the ELA/WELAC document "Guide to Analytical Quality Control" *(check exact title and reference)*. Note that control samples can be used not only to verify the analytical step. By using e.g. CRM sediments or certified biota materials, the whole chain of sample work-up steps can be included in the verification.

During the visits to the laboratories it was revealed that a majority of the laboratories use inadequate methods for internal quality control, thus increasing the risk of not discovering temporary or permanent analytical problems. In most cases the internal quality control was described as the occasional determination of double samples, or that a second person checks the data. These are important steps in assuring the data quality, but they do not cover all aspects of what is expected from modern internal quality control. The staff at all involved laboratories, both basic monitoring laboratories and Reference Laboratories, should thus be trained in adequate methods for internal quality control. The consultant should suggest and organise a short (3-5 days) course. The course should include training on how to prepare samples for internal quality control, on how to apply the samples in practice, as well as on how to interpret and use the results in different types of control charts as a means for internal quality control in a laboratory. The course, furthermore, should include training on the statistical background for the application and use of control charts in internal quality control. The consultant shall, furthermore, select an appropriate software package for the laboratories, to be used for their internal quality control.

<u>Recommendation</u>: Training for at least two persons at all laboratories in internal quality control, including training on how to handle a selected software package.

4.5. Training in Conducting Inter-comparison Exercises

One of the key roles for the proposed Reference Laboratories will be to improve the comparability between data produced by the different laboratories involved in the Caspian Environment Programme through organising and conducting inter-comparison exercises (a form of external quality control). In an inter-comparison exercise, identical samples are distributed to the participating laboratories. The laboratories are asked to analyse the samples for defined parameters and return the answers to the organiser. The concentrations of the samples have to be unknown to the participants (though they will normally be informed of an expected range), but known to a sufficient degree by the organiser. The organiser writes a report on the outcome of the exercise, which is then distributed to all participants. In this way, the single laboratory can get a picture of how well it performs in comparison to other laboratories within the same field, and will also be made aware of its own results that deviate from the mean or assigned value of the distributed samples. The latter information should then be used for improving the analytical performance.

It is vital that the organisers of inter-comparison exercises are capable of:

- producing a homogenous sample batch, with a known uncertainty and within the desired concentration range
- defining the assigned ("true") concentration of the samples, either based on their own measurements or on the results reported by the laboratories
- producing and distributing samples that are stable throughout the test period
- receiving data from the participants and handling these data
- defining and documenting a system for maintaining confidentiality
- producing a report which contains the results and some vital statistics
- supporting laboratories in interpreting results and explaining the statistics used

In order to make a system with frequent inter-comparison exercises practical and useful for the participating monitoring laboratories, it is important that the exercises arranged by any Reference Laboratory follow the same routines, and that the bulleted requirements above are fulfilled. The staff responsible for conducting these exercises should thus receive training on how to perform inter-comparison exercises and on how to evaluate and present the data according to relevant international standards. It is suggested that a few key-persons from each Reference Laboratory participate in a course arranged by the consultants. The consultants should also assist in selecting and providing some of the Reference Laboratories with appropriate software for evaluation and presentation of results from inter-comparison exercises according to the statistics defined in the international standard ISO 5275. The course should be 6-8 days long, incorporate both theoretical and practical aspects, and cover all the basic requirements for organisers of inter-comparison exercises mentioned above.

<u>Recommendation</u>: Staff at the Reference Laboratories should be given training on how to plan, conduct and report inter-comparison exercises, including how to handle the required software for statistical interpretation and presentation of the results.

4.6. Recommendations

It is strongly recommended that the upgrading of laboratory capacity is seen not only as upgrading the equipment and facilities, but that upgrading to a very large extent also is a question of education and training of the staff involved.

It is also recommended that the whole analytical chain be considered in the planning of training activities, so that not only the actual analytical part is covered. Of equal importance is training on sampling, sample handling, pre-treatment and the determination of various co-factors used for interpreting the results.

It is furthermore recommended that all laboratories receive training on methods for internal quality control, and that the Reference Laboratories receive training on conducting inter-comparison exercises (external quality control) for the monitoring laboratories.

The estimated cost for the proposed training is summarised in “Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs”.

Chapter 5. Monitoring Parameters and Laboratory Upgrading Costs

5.1. Introduction

In order to provide authorities with laboratory services related to the management of the environment, the present laboratory capabilities in the Caspian Environment Programme must be upgraded. The capability upgrading requires decisions on

- structural and institutional arrangement to be implemented for the laboratories;
- laboratories to be upgraded (both with equipment and training of the staff).

Decisions on the upgrading of laboratories should be carried out based on detailed investigations of:

- facilities to be used and capabilities of the facilities available;
- future tasks for the laboratories (as Reference Laboratories and in monitoring)
- equipment and capacity of the equipment available;
- possibilities for upgrading existing equipment; and
- staff capacity and capability already available.

The document in hand summarises the need for upgrading of facilities in the CEP in order to provide the Caspian Region authorities with laboratory capabilities for the most needed activities for the Reference Laboratories and for compliance and ambient monitoring.

In addition the document sets up cost proposals for activities to be implemented

- for enhancing the current range of monitored media
- of staff with limited expertise in analytical chemistry, and
- at laboratories where the facilities are inadequate for advanced analyses
- to strengthen the quality systems of the monitoring laboratories

The needed monitoring activities cover both background monitoring of relatively undisturbed areas, monitoring of wastewater effluents from industry (compliance monitoring), monitoring of riverine input and ambient monitoring (of open sea water) in the Caspian Sea Area.

Background material and detailed justification for proposed activities and purchases are given in the following documents:

- Note on Existing Laboratory Capacity
- Suggested Monitoring Parameters and Estimated Laboratory Upgrading Costs
- Note on Training Needs in Connection with the Implementation of a Reference Laboratory Structure

5.2. Required Monitoring Activities

Suggestions for monitoring activities and upgrading needs are based on information and observations obtained during a field trip to the regional CIS laboratories by Latifa Guseynova (see Annex A to "Existing Laboratory Capacities" in June-July 2000, to Iranian laboratories by Mohammad Saeed Abaee late summer 2000 (see Annex B to "Existing Laboratory Capacities"), and visits to some Azerbaijani laboratories by Mikael Krysell in October-November 2000.

An overview on different possible monitoring activities, their goal and general implications for the character of the monitoring activities is given in Table 1. The table has been used as reference when outlining the different types of future monitoring activities for the Caspian Environment Programme.

Typical objectives and characteristics of monitoring activities

Type of monitoring	Focus	Station density and location	Sampling frequency	Number of variables	Duration
Multipurpose monitoring	Space and time distribution of water quality in general	Medium	Medium (12 per year)	Medium	Medium (> 5 years)
Background monitoring	Level of natural water quality	Low	Low (< 6 per year)	Low to high	Variable
Trend monitoring	Long-term evolution of concentrations and loads	Low: major uses and boundaries	High (> 18 per year)	Low for single objective High for multi objectives	Long (> 10 years)
Compliance monitoring	Industrial pollution output	At the industrial outlet	High	Specific for each industry	Continuous
Input monitoring	Pollution input from point sources	River mouths, industrial sites	High enough to resolve seasonal variations	High	Long
Impact surveys	Effects of pollution sources on WQ	Limited to the pollution sources	Medium	Specific (according to source)	Variable
Operational surveillance	WQ for specific uses	Low: at specific uses	Medium	Specific	Variable

Type of monitoring	Focus	Station density and location	Sampling frequency	Number of variables	Duration
Modelling survey	Collection of data for water quality model	Specific	High	Low	Short: calibration and verification
Early warning	Protect critical water use locations such as drinking water intake	Very limited	Continuous	Very limited	Unlimited

5.2.1. Monitoring Medium

Generally, three principal media can be used for monitoring of an aquatic system: water, particulate matter and living organisms. Each medium has its own set of characteristics for monitoring purposes, such as

- applicability to water bodies,
- specificity to given pollutants,
- possibility of quantification such as flux determination,
- sensitivity to pollution, and
- time-integration of the information received (ranging from instantaneous for water samples to integrate for biotic samples)

Water is by far the most common monitoring medium used to date (including in the current CEP activities). The main reason for this is that water samples are comparatively easy and cheap to analyse (given the necessary equipment and analytical reagents are available) with standardised methods, and easy to compare to water quality objectives, which are usually defined as concentrations in water samples. However, for many substances such as hydrophobic organic micro-pollutants and certain heavy metals, water is not the most appropriate medium to monitor because the water-solubility of these substances is very low and hence they concentrate in particulate matter and biota. In water containing suspended solids at concentration ranges of 10-100 mg/l (a range found in rivers or near coasts), the suspended fraction typically accounts for more than 50% for many pesticides. For metals such as Pb, Cr, Fe, Zn, Al and Hg the fraction carried by particulate matter can be more than 99%. Also, the analytical detection limit for some of the substances is of the same order of magnitude asthe water quality requirements for various uses, making it a very hard task to adequately determine the substances in the water phase. Another obstacle is that inadequate sampling equipment or sample handling routines easily will contaminate water samples.

For these reasons many of the 'Not Detectable' or very low values of these substances reported in monitoring data in various programmes may not be a representation of the true presence or absence of the substances, but rather a reflection of an inappropriate medium being monitored. Unfiltered samples are analysed, but the sensitivity of monitoring would be highly increased by analysing particulate matter or biological material.

So far relatively limited experience exists in the Caspian Sea of other than water phase monitoring, but it is recommended to introduce monitoring of particulate matter and biota (including bioassays/toxicity tests) on a more regular basis. Some laboratories are monitoring sediments and biota to some degree already, but there is certainly no overall CEP strategy behind these activities.

5.2.2. Monitoring of Industrial Water Effluents and Polluted Rivers

Compliance Monitoring and Basic Input Monitoring

The publication "National Environmental Action Plan" (State Committee on Ecology, 1998) and earlier information gathered during the first and second phases of this project contain information about parameters to be monitored in wastewater from industries in the Caspian Area. A summary of parameters relevant for monitoring is found in annex A.

Typical parameters for compliance monitoring of wastewater effluents are; temperature increase, pH, total suspended solids, chlorine, sulphide, ammonia, nitrate, biochemical oxygen demand, chemical oxygen demand, and cyanide, oil and grease, phenol, coliform bacteria, and trace elements. Ecotoxicological tests should be applied to estimate the toxicity of the effluent waters (see "monitoring of sediment and biota" below). In addition, certain industrial sites will require the monitoring of specific parameters, depending on their activities.

Measurements, Tests, and Analyses

A table showing proposed measurements, tests, and analyses to be conducted *in situ*, at Regional Laboratories and at the Reference Laboratories is provided in Annex B. In addition, suggested analytical techniques or equipment to be used are provided in Annex B.

The suggestions for which measurements, tests, and analyses should be carried out *in situ* and at Regional Laboratories were made based on the stability of the parameter and on the type of equipment needed.

Many of the parameters can be determined by the use of relatively simple equipment or test kits such as Merck Spectroquant, Dr Lange and Hach equipment. Such test kits consist of prefabricated reagents and measurement instruments. Staff with no or limited expertise in analytical chemistry are able to use such kits after some training.

As mentioned trace organic and inorganic components are included in the list of parameters (Annex A). Such analyses are to be performed at the respective Reference Laboratory, as

the analyses require suitable facilities, considerable analytical expertise and heavy equipment. Furthermore, these parameters are stable (after preservation of the samples), making it possible to collect samples at different locations around the Caspian Sea and send them to the Reference Laboratory for analysis.

Proposal for Monitoring of Water Effluents

A proposal listing which monitoring activities should be given highest priority is found in Annex A.

The basic parameters are: temperature increase, pH, total suspended solids, ammonia, nitrate, phosphorous, biochemical oxygen demand, chemical oxygen demand, oil and grease, and phenol. In addition, river input of metals and organic compounds bound to particles should be monitored.

Other parameters will have to be chosen at certain locations and for certain laboratories, depending on the character of the wastewater or river water discharged in that area.

Sampling of Water Effluent from Industry

Samples to be analysed should be representative of the effluent, discharged by a facility. The samples can be taken from a container into which the entire volume of wastewater has been collected. Alternatively, the samples can be collected directly from the effluent stream. It is important to ensure that the wastewater is completely mixed when samples are collected. The total volume of effluent discharged should also be measured in order to calculate the total amount of the parameter of concern has been released into the environment.

Advanced sampling equipment is available for collection of representative samples from effluents. Such equipment may collect samples automatically during a fixed time period. In addition the equipment may measure flow, pH, conductivity etc. continuously.

For the time being, it has been suggested that both the collection of samples and measurement effluent volume be done manually if no other equipment is available.

5.2.3. Monitoring of Ambient Water

There is a considerable interest to establish routine ambient monitoring of seawater in the Caspian region. The primary objective of such monitoring is to establish the degree of large-scale pollution and to identify areas where the water quality has decreased due to pollution. Such a monitoring net should also include background monitoring stations, where the natural or normal background values of different parameters can be established. The latter monitoring is normally carried out at remote areas, such as points on rivers far upstream or far from pollution sources, and at rather low frequencies.

The parameters to be measured for background monitoring include general physical chemical parameters such as

- indicators for general water quality: temperature, pH,
- gases: dissolved oxygen
- dissolved salts: sodium, potassium, calcium, magnesium, chloride, and sulphate
- nutrients: nitrogen and phosphorus containing compounds, such as ammonia, nitrate, orthophosphate, and organic compounds containing nitrogen and phosphorus.

Other parameters may also be of interest. However, for many parameters the concentrations are very low and therefore relatively advanced analytical techniques and equipment are required. The use of such methods requires considerable expertise in analytical chemistry. Such expertise has to be available at one Reference Laboratory at least before all parameters can be included in the monitoring activities.

The parameters to be measured for ambient monitoring (near coastal and open sea monitoring) include general physical chemical parameters such as:

- temperature and salinity
- indicators for general water quality: temperature, pH, suspended solids
- gases: dissolved oxygen and sulphide
- nutrients: nitrogen and phosphorus containing compounds, such as ammonia, nitrate, orthophosphate, and organic compounds containing nitrogen of phosphorus
- COD/BOD (or TOC if concentrations are not high enough)
- oil hydrocarbons
- chlorophyll

Due to the instability of many of the above-mentioned parameters, it will be necessary to ensure that a few “ambient monitoring” (sea-going) laboratories have the capacity to measure these parameters, as most of them cannot be sampled, preserved and transported with a sufficient degree of reliability.

5.2.4. Monitoring of Sediments and Biota

Sediment Monitoring

Particulate matter could be in the form of deposited bed sediment and suspended solids, normally both should be monitored.

The objectives of the sediment monitoring would be, as examples:

- To establish a better knowledge of the integrated pollution in selected areas of the Caspian Sea, and
- To be able to assess the retention of pollutants in the river sediment, and thereby assess the significance of sediment in river pollution transport.

Relevant aspects to cover would be sedimentation rates, particle size distribution, total nitrogen and phosphorus, organic content and oxygen consumption of the sediment. Also concentrations of trace elements and hydrophobic organic micro-pollutants in sediments would be relevant to measure, including some background locations as reference for the more polluted sites. The toxicity of the sediments can also be monitored using ecotoxicological tests. Sediment monitoring, requiring some specialised analytical equipment and skills, should be performed by a few selected laboratories only.

Biological Monitoring

Biological monitoring in the Caspian Sea and its tributaries would have a good potential for detection of presence and assessment of effects of pollution, as a supplement to the traditional physic-chemical water monitoring. The general advantage of biological monitoring is that it provides an integrated measure of the pollution load over time (hence, no problem with low sampling frequencies). Two options are considered potentially relevant for the CEP. Both would require substantial planning and preliminary surveys to establish detailed methodologies, train personnel, determine reference conditions, etc.

- Monitoring of pollution effects on ecosystems by indicator organisms (bioassays):

By monitoring the absence/presence/abundance of certain indicator species (e.g. benthic macroinvertebrates) a pollution index may be determined for a location. Usually this method is used to assess organic pollution, which would be highly relevant. The usefulness of this approach can only be determined after a preliminary survey to identify appropriate indicator organisms.

The advantage of this kind of monitoring is its simplicity, once a baseline has been established. It is relatively cheap compared to chemical monitoring and requires no special equipment or facilities. The disadvantage is the requirement for knowledge of taxonomy and occurrence of native species in the local environment (baseline). This is a clear task for one or two specialised laboratories with the properly trained staff.

- Monitoring by chemical analysis of biotic samples, such as fish, shellfish or plants:

this might provide an opportunity to monitor presence and effects of bio-accumulating toxic substances that might otherwise be beyond the scope of determination by local or regional laboratories, due to excessive costs of chemical analyses of such substances. Monitoring of biota would primarily take place in the form of specific surveys at recurrent intervals. The advantages of this kind of monitoring are that it is relevant to human health, and requires less advanced facilities than for determination of the same substances in the water phase, where the concentrations will be much lower. The disadvantage is the higher costs compared with the indicator organisms monitoring.

Developing the Monitoring Capability of Sediments and Biota

The fact-finding mission revealed that very little activity and scarce experience exist for monitoring of other types of media than water samples. It is assessed that monitoring of sediments and biological samples has the potential to contribute valuable information about the water quality. In initiating such activities, experiences from the earlier and on-going monitoring of sediments and biota should be taken into account.

The activities are to be seen as a pilot project for introducing sediment and biological monitoring as integral parts of the routine water quality monitoring programme in the future. The following should be considered:

Planning. It is suggested that the point of departure be taken at the laboratories, where sediment monitoring and biological monitoring, including toxicological tests using bioassays, has already been carried out on a (relatively) regular basis. This means that these laboratories have both certain experience in this field, and also that they have at least some of the necessary sampling and analytical equipment. It is proposed that biological monitoring focus on both chemical analysis of biotic samples, such as fish, shellfish or plants, and bioassays using indicator organisms. The equipment procured for upgrading of the laboratories will be used to carry out the necessary analyses. The group of Reference Laboratories will be given the task of defining the pilot project. The same group will also decide on the equipment and methodology to apply.

Activities will include: (1) collection and summarising of current and past experiences with sediment and biological monitoring, (2) identification of locations, species, parameters, and frequency to monitor in the pilot project, (3) definition of methodology (4) identification of necessary equipment for sampling, sample preparation and analysis and (5) identification of suitable species and methods for ecotoxicological tests.

Implementation. The duration of the pilot study will be at least one annual cycle in order to allow for detection of possible seasonal variations. The most significant equipment in need of upgrading for this component are sediment samplers and equipment for sample preparation and work-up. The group of Reference Laboratories will identify the necessary training in sampling, handling, preparation and preservation of samples, which can be given by the consultant in co-operation with experienced local staff. It is envisaged that two persons from each institution will be trained as part of the pilot project. Note that although the Reference Laboratories for organic and inorganic components in sediments and biota will carry out the analyses, sampling will be performed by a much larger number of laboratories.

Activities will include: (1) procurement of equipment for sampling, sample treatment and analysis, (2) testing of equipment, (3) sampling in the field according to pilot project plan (incl. on-the-job training), (4) laboratory analysis and ecotoxicological tests (incl. training in sample preparation in lab), (5) 2-3 meetings for exchange of experience and evaluation of programme, (6) analysis of results and (7) recommendations for a permanent programme.

5.3. Equipment and Staff Requirement

The equipment required at each laboratory depends upon the parameters to be measured, the methods to be applied, and the number of samples to be analysed.

5.3.1. Need for Equipment

Basic Input Monitoring

The proposed basic input monitoring, which all regional laboratories should be able to carry out, requires equipment for measurements of the following parameters:

- Temperature (increase)
- pH
- Total suspended solids
- Oil and grease
- Ammonia, Nitrate, Phosphorous
- Total-N and Total-P
- Biochemical Oxygen Demand
- Chemical Oxygen Demand
- Dissolved Oxygen
- Fluoride, cyanide
- Phenol
- Total and faecal coliforms

The two first parameters only require simple measuring instruments, The third and fourth require basic laboratory equipment (filtration equipment, oven and balance). As mentioned it is proposed that the remaining parameters be analysed by use of test kits (coliforms using the tube method).

A rough estimate of cost for such the equipment is found in Annex C. The total cost for a complete set of basic equipment is approximately US$ 80 000. The cost will be reduced for those laboratories where already available instruments can be used. A rough estimate of the running costs for a maximum of 500 samples per year gives approximately US$ 10 000.

Special Parameters

The reference laboratories, and some other specialised laboratories, will be required to carry out measurements of other constituents, in addition to the basic set of parameters mentioned above (Annex A). Some of these laboratories already have the required equipment, but it will be necessary to upgrade the equipment at all of them to a greater or lesser degree.

The cost will have to be individually assessed for each laboratory. The cost estimate for specialised parameter monitoring is given in Annex C.

5.3.2. Staff Requirement and Training

It is expected that two trained technicians are needed to carry out sampling and analyses for basic parameters. For the specialised parameters, the needs will be individual for each laboratory. Training of staff, both for analytical work and for sampling, is a most important part of the laboratory upgrading.

It is estimated that the following training activities will be necessary:

- Training of two technicians from each laboratory receiving new equipment for the basic parameters
- Training in modern methods for internal quality control for two persons from each laboratory
- Training in conduction of inter-comparison exercises for staff from the Reference Laboratories
- Tailored training activities for staff from Reference Laboratories receiving new equipment

The costs are outlined in Annex C.

5.3.3. Choice of Analytical Methods

As far as possible all analytical methods used should be international standard methods. These exist for most of the parameters that are mentioned as candidate monitoring objects. Usually the harmonised use of standard methods is the best start when trying to reach comparable results in a group of laboratories. References to existing standard methods are given in the table in Annex A.

Some standard methods are less fit for ambient water monitoring, as they can be targeted at sewage water or similar waters of relatively high concentrations of the determinant in question. In such cases, or when the appropriate standard method does not exist, it is the responsibility of the reference laboratory for that parameter to issue instructions on how the determination should be performed. These instructions could be in the form of a simple addendum to the standard method, explaining what steps have to be taken to modify the method for use in the CEP, reference to some existing method or an entire method description. If possible, reference could simply be made to analytical methods developed for monitoring purposes by one of the international monitoring commissions and organisations (e.g. the Helsinki Commission, HELCOM, or the International Council for the Exploration of the Seas, ICES).

The reference laboratory for QA/QC will be responsible for drafting and maintaining a list of the recommended methods, including additions, changes and other method descriptions.

The test kit methods all use a standardised set of chemicals and equipment, and instructions on how to handle the analysis. For these analyses, the need for standardisation is thus covered more or less by the very definition of the test kit concept. The test kit methods do not follow the international standard methods in detail, but in most cases they use exactly the

same principle. Method comparisons between these test kit methods and standard methods have been performed on numerous occasions, and these results show that the modern test kit methods give data which are highly comparable, and of the same quality, as do the standard methods. It has even been demonstrated that for laboratories where the staff is less experienced or the facilities are limited, the test kit methods give more reliable results.

5.4. Recommendation

It is recommended that establishing or upgrading of environmental laboratory capabilities in the CEP is carried out on basis of decisions of the environmental authorities on

- structural and institutional arrangement to be implemented for the laboratories
- laboratories to be established/upgraded

In addition it is recommended that detailed investigations of existing facilities, equipment and possibilities for upgrading of available equipment be carried out.

Furthermore it is recommended that a group is established to plan and implement all needed activities. It is suggested that representatives from the proposed Reference Laboratories should have a co-ordinating role during the establishment/upgrading of all laboratory capabilities.

Recommendations for parameters, and estimated upgrading costs, are given in the annexes below.

Chapter 6. Decision Process for Defining Laboratory Capabilities

6.1. Introduction

All laboratories need from time to time to adjust the scope of activities (measurements, tests and analyses) the laboratory offers.

New parameters may be introduced and others may be excluded from the laboratory's list of measurements, tests, and analyses. In addition, new analytical techniques may replace older or perhaps more expensive techniques.

Therefore the environmental authorities and the laboratories have to establish tools for how the scope of activities offered to clients can be kept updated and how laboratories can avoid the use of measurements, tests, and analysis of limited value from a regulatory perspective, or which may be unnecessarily expensive.

The document in hand describes briefly how such decisions can be made. The decision process supports the use of resources where most needed.

6.2. Performance Characteristics

The important operational characteristics of a method are called *performance characteristics* and they provide the basis for selection of an appropriate methodology. Performance characteristics are not constant for a methodology since analytical values generated tend to be dependent on or influenced by the user.

The most common performance characteristics needed to describe a method are

- limit of detection
- accuracy (systematic error)
- precision (random error)

The limit of detection has been defined in several ways in the past, but most international organisations now tend to use the following definition. The *limit of detection* is the lowest concentration that with a given level of confidence can be detected.

The *accuracy* of a method is the closeness of the obtained value to the true value. Accuracy is related to a single analytical result and therefore represents a combination of systematic and random error. The systematic error is the difference between the mean of a large number of results and the true value.

The *precision* of a method is a statement of the closeness of agreement between mutually independent results and is usually stated in terms of standard deviation. It may be stated in different ways depending on the conditions under which it is measured. *Repeatability* is a type of precision relating to measurements made under repeatable conditions, e.g. same

method, same material, same operator, same laboratory, narrow time period. *Reproducibility* is a concept of precision relating to measurements made under reproducible conditions, e.g. same method, different operator, different laboratories, different equipment, long time period.

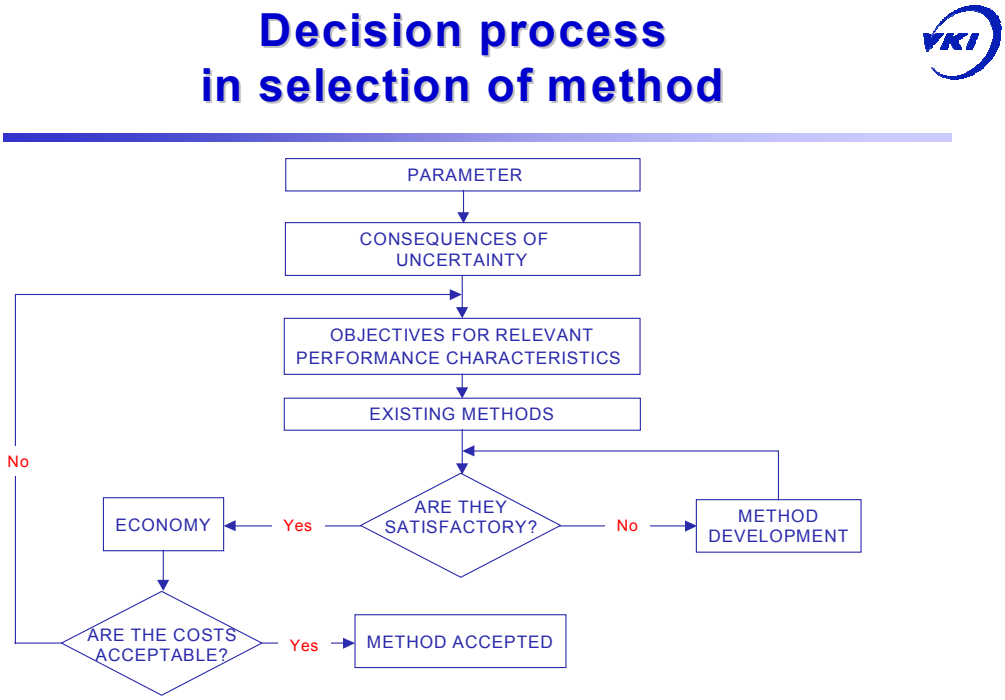
6.3. Selection of Method

The choice of method will depend on several factors, the most important being the performance characteristics mentioned above. A stepwise procedure for making the choice is shown schematically in the figure below.

First the parameter to be determined is selected and the possible consequences of uncertainty are evaluated. Based on this, the relevant *performance characteristics and the target values* are selected*: the quality requirements (or quality objectives)* are established for the measurement, testing or the analytical process.

Existing methods for the determination of the parameter are evaluated, based on performance characteristics either from literature or previously established in the laboratory. If a method which meets the data quality requirements and which has a reasonable cost is found, the choice can be made and the analytical work can start.

If existing methods are insufficient, a new method must be developed or the strategy for the investigation can be changed, resulting in a change in data quality objectives.



6.4. Recommendation

It is recommended that the environmental authorities and laboratories involved in activities related to management of the environment at appropriate intervals evaluate if the applied analytical methods fulfil required quality requirements.

In addition it is recommended that the environmental authorities and the laboratories at appropriate intervals should evaluate if the applied analytical methods could be replaced by methods that have better performance characteristics and may be also cheaper to perform.

It should be the responsibility of the Reference Laboratory to take the leading role in such activities, which could be initiated by e.g. changes in legislation, changes in the water quality or development of new analytical or data evaluation tools. Regardless of such changes, a plan for regular re-evaluation of the performance characteristics must be constructed.

It is further recommended that the laboratories follow the decision process in the selection of methods to be used for parameters that have not been measured, tested or analysed before.

ANNEX A: Proposed list of Water Quality Variables

Type of variable	Type of monitoring					Reference to international standard method
	Back-ground	Input monitoring (rivers, sewage water, industries)	Near coastal and ambient water (open sea)	Sediments and river-borne particulate material	Biota	
General:						
Transparency			X			--
Temperature	X	X	X			--
PH	X	X	X			ISO 10523
Susp.Solids	X	X	X			ISO 11923
DO	X	X	X	X		ISO 5813, 5814
Conductivity	X	X				ISO 7888
Salinity			X			(Grasshoff*)
Total hardness	X					ISO 7980
Chlorophyll-a			X			DIN 38412-16
Nutrients:						
Ammonia	X	X	X			ISO 7150
Nitrate/nitrite	X	X	X			ISO 13395
Total nitrogen		X	X	X		ISO 11905
Phosphorus		X	X			ISO 6878, EN 1189
Total phosphorous		X	X	X		EN 1189

Type of variable	Type of monitoring					Reference to international standard method
	Back-ground	Input monitoring (rivers, sewage water, industries)	Near coastal and ambient water (open sea)	Sediments and river-borne particulate material	Biota	
Organic matter:						
BOD_5	X	X	X			EN 1899
COD_{Cr}	X	X	X			ISO 6060
Major ions: (Na, K, Ca, Mg, F, Cl, SO_4)	X					EN/ISO 10304
Other inorganic: Cyanide		X				ISO 6703
Trace elements: Trace Elements (Notably As, Cd, Cu, Cr, Pb, Hg, Ni, Zn)				X	X	ISO 16590 (Hg) ISO 5666 (Hg) ISO 15586
Organic toxics:						
Oil hydrocarbons			X			ISO 10301
Oil and Grease		X				--
Phenols		X	X			ISO 6439, 14402

Type of variable	Type of monitoring					Reference to international standard method
	Back-ground	Input monitoring (rivers, sewage water, industries)	Near coastal and ambient water (open sea)	Sediments and river-borne particulate material	Biota	
Organic contaminants:						
Pesticides				X	X	ISO 6468
PCBs, HCBs etc				X	X	
Bioassays:		X		X		ISO 6341, 12890, 10706, 14669
Ecotoxicology						
Microbiological:						
Faecal coliforms		X				ISO 4831, 4832
Total coliforms		(X)				

Note 1In addition to variables listed here, compliance monitoring will have to consider relevant variables associated with special pollution sources .

Note 2: A comprehensive list of priority pollutants is given in the report “Development of Priority List of Chemical Pollutants” (November 2001), and should be consulted when choosing the exact parameters for trace elements (inorganic pollutants) and organic pollutants at different locations.

Grasshoff = “Methods of Seawater analysis”, ed. Grasshoff et al., 1999 (Verlag Chemie, Germany)

ANNEX B: Recommended Measurements, Tests and Analyses to be Conducted *in situ*, at the Regional Laboratories (basic input monitoring) and at the Reference Laboratories/ Ambient Monitoring laboratories

Parameter	On site measurement Technique or equipment to be used for onsite measurements	Basic input Laboratory Techniques or equipment to be used	Reference Laboratory and ambient monitoring Techniques or equipment to be used
Indicators for general water quality			
Temperature increase	Portable equipment	(Calibrated thermom.)	(Calibrated thermom.)
PH	Portable equipment	pH-meter	pH-meter
Total suspended solids		Filtration equipment, drying and weighing	Filtration equipment, drying and weighing
Dissolved Oxygen	Portable Oxygen probe	Oxygen probe	Oxygen probe or Winkler titration
Conductivity		Conductometer/probe	Conductometer/probe
Salinity	CTD probe		Salinometer
Total hardness		EDTA Titration	EDTA Titration
Chlorophyll-a			Spectrophotometry after extraction

Nutrients			
Ammonia, nitrite/nitrate, total nitrogen, phosphorous, total phosphorous		Test kit	Spectrophotometry after pretreatment of the sample
Organic matter			
Biochemical oxygen demand (BOD)		Test kit	Test kit or standardised method and subsequently titration or use of electrode to estimate the oxygen consumption
Chemical oxygen demand (COD)		Test kit	Test kit
Major ions			
Sodium, potassium, Calcium, Magnesium, Fluoride, Chloride, Sulphate			Ion Chromatography
Other inorganic			
Cyanide			Titrimetric method

Trace Elements			
Arsenic, Cadmium, Copper, Chromium (tot), Iron, Lead, Nickel, Zink			Atomic absorption spectrophotometer and hydride-generation instrument after digestion of the sample
Mercury			Mercury analyser after pre-treatment of the sample
Organic toxins			
Oil hydrocarbons			Fluorescence or Gas chromatography
Oil and grease		Extraction, evaporation and weighing	Extraction, evaporation and weighing
Phenol		Test kit	Test kit or alternatively spectrophotometry after pre-treatment of the sample
Organic contaminants			
Pesticides and PCB:s			GC-MS after pre-treatment of the sample
Microbiological parameters			
Coliform bacteria (faecal and total)		Incubation, tube method	Incubation, colony forming or tube method

Test kits such as Merck Spectroquant, Dr. Lange and Hach consist of ready-made equipment such as test tubes containing prefabricated reagents and measuring instruments. The test kits may be of use -toof staff with no or limited expertise in analytical chemistry.

The methods and analyses for the content of inorganic and organic trace constituents are expensive and require considerable expertise in analytical chemistry, and should thus only be carried out by a very limited number of laboratories

ANNEX C: Cost Estimates for Equipment and Training

Please note that all costs are rough estimates, not based on quotations on specific equipment from specific suppliers

I: Estimation of the cost, Basic Input Monitoring Laboratory

Item	Cost (US$)
Facilities It is expected that the existing facilities can be used. However, some costs for upgrading and repair have to be expected.	0
Cost of sampling equipment Hand-held samplers (Ruttner type), 3 per laboratory	3000
Equipment for basic monitoring Temperature (increase) Thermometer to be used for calibration of work thermometers and a number of work thermometers	850
pH One precision pH meter to be installed at a laboratory And two pH meters for field measurements	3000
Total suspended solids Pumps and filtration units (3 systems)	1500
Dissolved Oxygen	3000
Conductivity	3000
Total hardness	2000
Chlorophyll-a	5000
Oil and grease	0
Coliforms, tube method	5000

Item	Cost (US$)
The remaining parameters Nutrients Biochemical Oxygen Demand Chemical Oxygen Demand Phenol Are all suggested to be determined by use of test kits such as Hach, Merck Spectroquant or alternatively Dr Lange Analysis System. The equipment consists of e.g.spectrophotometer and portable laboratory photometers, accumulators, printers, stands, pipette. Additional equipment for destruction of samples: thermoreactor, thermostats, BOD dilution water set, etc. Total for the remaining parameters	30 000
In addition to the above mentioned items, the laboratory has to be equipped with materials as sample bottles, pipettes, glassware, refrigerators, water purification system, balances, furnaces, extraction equipment etc. A rough estimate suggests	20 000
Total for a set of basic monitoring equipment, one laboratory:	**80 000 US$**

The cost will be reduced if some of the above mentioned items are already available at the laboratory.

II: Examples of estimated cost, additional Reference Laboratory and ambient monitoring equipment

Item	Cost (US$)
Cost of facilities	
In order to facilitate for trace analyses, new laboratory benches, clean-air benches etc. may have to be installed. Any costs for major laboratory refurbishment have to be estimated if needed.	15 000
Cost of equipment	
Graphite oven AAS	65 000
Autoanalyser for nutrients	100 000
Spectrophotometer	12 000
Ion Chromatograph	40 000
GC-MS	150 000
Fluorimeter	10 000
Costs for sample preparation and treatment	
For organic or inorganic parameters	15 000
Cost of sampling equipment	
Sediment samplers	10 000
Cost of software	
Software for Internal Quality Control	12 000
Software for the conduction, interpretation and reporting of inter-comparison exercises according to ISO 5725	12 000

The cost will be reduced if some of the above mentioned items are already available at the laboratory.

III: Estimation of the cost for training activities

Assumption: 18 laboratories, of these 6 Reference Laboratories

The cost for training courses include teaching, course material, travel and accommodation, and time

For backgound and details, see "Note on Training Needs in Connection with the Implementation of a Reference Laboratory Structure"

Training Activity	Conducted by	Participants	Cost in US$, total
Training in handling the new basic equipment	Equipment manufacturer	2 from each laboratory	90 000
Training in internal quality assurance	Consultant	2 from each laboratory	75 000
Training in conducting inter-comparison exercises	Consultant	2 from each Reference Lab.	75 000
Training on determination of co-factors	Consultant or Expert Laboratory	Staff from biota and sediment Ref Lab	50 000
Training on special equipment	Equipment manufacturer	Laboratories with new equipment	150 000
Total for training:			**440 000**

ANNEX D: Estimation of Equipment Cost per Laboratory

(Selection of Reference Laboratories and laboratories for ambient monitoring is based on site visits, questionnaires and response from the respective countries on the original suggestion, and is motivated in “Requirements for Laboratories in the Caspian Environment Programme”)

Laboratory	Will carry out	Basic equipment	Other laboratory equipment	Total cost for laboratory	Existing equipment assumed to be used
Kazakhstan					
Ministry lab, Atyrau	Basic monitoring	80 000		80 000	
Mangystau Regional lab	Basic monitoring	80 000		80 000	
Atyrau Sanitary and Epidemic Station	Reference Laboratory for Microbiology and radioisotopes Basic monitoring	80 000	Microscopes, incubators, etc	140 000	Most equipment for microbiology exists but will be modernised
TOO Service Project, Aktau	Basic monitoring	80 000		80 000	
TOO Monitoring, Atyrau Old Airport	Basic monitoring Sampling/preservation for inorganic trace elements Ambient monitoring	80 000	Sample preparation equipment Fluorimeter Spectrophotometer Sampling equipment	160 000	New AAS exists (graphite??) Sampling equipment exists

Turkmenistan					
Control and Inspection, Turkmenbashi (Kaspekokontrol)	Basic monitoring Ambient monitoring	80 000	Sampling equipment Spectrophotometer Fluorimeter Chemicals, scale	140 000	
Russia					
Agrochemical Service Stn, Kalmykia	Basic monitoring	80 000		80 000	
Inspectorate, Dagestan	Reference Laboratory for Inorganic trace elements in soil, sediments, biota and water Basic monitoring Ambient monitoring	80 000	Graphite AAS Fluorimeter Spectrophotometer Sample preparation equipment Sediment sampler	220 000	
Caspian Fisheries Research Institute	Reference Laboratory for Hydrobiology Ambient monitoring		Spectrophotomer	50 000	

Azerbaijan					
State Inspectorate, Baku	Reference Laboratory for General QA/QC Reference Laboratory for Ecotoxicological Tests	80 000	Sample preparation equipment Scale	110 000	AAS
Azerekolab, Ecology, Baku	Reference Laboratory for Sampling Ambient monitoring Background monitoring	80 000	Sampling equipment Fluorimeter Spectrophotometer Ion chromatograph	140 000	Sampling equipment
Sumgayit State Ecology	Basic monitoring	80 000		80 000	
Hydrometeoro-logy State Monitoring	Basic monitoring	80 000		80 000	

I.R. Iran					
Central DOE lab	Reference laboratory for Basic Physical-Chemical parameters Background monitoring Ambient monitoring	80 000	Spectrophotometer Fluorimeter	140 000	CTD and salinometers Sampling equipment
Gorgan DOE lab	Basic monitoring	80 000		80 000	
Sari DOE lab	Basic monitoring	80 000		80 000	
Chaloos DOE lab	Basic monitoring	80 000		80 000	
Anzali DOE lab	Basic monitoring	80 000		80 000	
Research Institute of Petroleum Industry	Basic monitoring	80 000		80 000	
Water and wastewater, Tehran	Basic monitoring	80 000		80 000	AAS
Fisheries Research, Anzali	Basic monitoring	80 000		80 000	
Food and drug, Tehran	Reference laboratory for Organic Compounds in Biota		Sample preparation equipment Standards, chemicals	50 000	GCs, GC-MS

Chemical Engineering Research Centre, Tehran	Should be used for special studies and method development/verification only			no basic costs	
Total cost for CEP				**1 365 000**	

ANNEX E: Total Cost Summary

Investments	Cost, US$	Comments
Equipment for 6 Reference Laboratories	700 000	
Equipment for the 17 other laboratories	1 650 000	
Consultant support for arranging tenders, purchases, starting up Ref Lab activities etc.	210 000	12 m/m estimated
PCs for approx. 15 laboratories	50 000	Some labs lack PCs
Software for Internal QC	252 000	21 laboratories assumed
Software for inter-comparisons	48 000	4 Reference Laboratories
Training	440 000	
Grand total:	**3 350 000**	

Note that cost for local staff is not included

Running costs per year*	Cost, US$	Comments
Basic monitoring, 21 labs	210 000	
Special monitoring, 8 labs	120 000	
Inter-comparison exercises	30 000	
Certified Reference Materials	25 000	
Software upgrading	20 000	
Grand total:	**405 000**	

** "Running costs per year" is an approximation which does not include sampling costs (ship-time, travelling etc) or staff, only chemicals, gases, reagents, consumables etc for the analytical work.*

It may be necessary to break down the upgrading project in several smaller projects, so that the laboratory upgrading can be carried out step by step. Below the upgrading is divided into a number of smaller projects. Please note that the estimated total cost becomes slightly higher for a number of smaller projects.

Project	Cost, US $	Comments
Training of laboratories in QA/QC and start-up of reference laboratory activities	410 000	Including certain software, PC:s etc.
Upgrading of reference laboratories	760 000	
Upgrading of laboratories in Kazakhstan	450 000	
Upgrading of laboratories in Turkmenistan	190 000	
Upgrading of laboratories in Russia	290 000	
Upgrading of laboratories in Azerbaijan	210 000	
Upgrading of laboratories in I.R. Iran	610 000	
Implementation of reference laboratory activities	410 000	Including support to implement proficiency testing
Total:	3 650 000	

ANNEX F: Mission Report on Analytical and Monitoring Laboratories and completed questionnaires for 13 laboratories in Kazakhstan, Turkmenistan, Russia and Azerbaijan

F.1. Introduction

This is the mission Report on a series of visits to Kazakhstan, Turkmenistan, Russia and Azerbaijan within the Environmental Laboratories Assessment Project. The visited institutions and people are listed below. The mission was executed from 26 June to 13 July.

F.1.1. Kazakhstan

- The Laboratory of Atyrau Regional Department of Ministry of Natural Resources and Environment
- The Sanitary and Epidemic Laboratory
- The Non-Governmental Laboratory TOO Monitoring
- Mr. A.B.Palymbetov, Deputy Akim Atyrau Region
- Mr. Iv Van Low, UN Volunteer, Caspian Region Development Support Programme Coordinator, Atyrau
- The Laboratory of Mangystau Regional Department of Ministry of Natural Resources and Environment
- The Sanitary and Epidemic Laboratory
- The Non-Governmental Laboratory TOO Caspian Service Project
- Mr. K.B.Shalgimbayev, Deputy Akim Mangystau Region, Aktau
- The Laboratory of Almaaty Regional Department of Ministry of Natural Resources and Environment
- The Sanitary and Epidemic Laboratory
- The Scientific Laboratory of Zoology Institute
- The Scientific Laboratory of Chemical Scientific Research Institute
- The Plague Scientific Research Institute in Almaaty, Kazakhstan (personal contacts with: Acad. A.M.Aykimbayev, Dr. of Biology, Prof. R.A.Burdelov, Dr. of Biology A.B.Bekenov, etc.)

F.1.2. Turkmenistan

- The Laboratory of the State Turkmenistan Fisheries Committee and Regional Department of Ministry of Natural Resources and Environment, Ashgabat, Turkmenistan

F.1.3. Azerbaijan

- The Laboratory of The State Caspian Protection Inspectorate, Azerbaijan
- The Laboratory of Baku State Committee of Ecology, Azerbaijan
- The Laboratory of Sumgayit State Committee of Ecology, Azerbaijan
- The Laboratory of The State Hydrometeorology Committee, Azerbaijan

F.2. Kazakhstan

F.2.1. The Laboratories of the Ministry of Natural Resources and Environment in Atyrau (1), Aktau (1), Almaty (2)

The Laboratory premises more or less do not meet hygienic requirements. The Laboratories are not appropriately equipped (no AAS, Gas Chromatography, Liquid Chromatography, potable water, air, soil, sediment, radionuclide analysers, water or air flow measuring equipment). Ecotoxicology studies are not implemented. The majority of the staff are top qualified specialists well skilled in laboratory work.

Being financed by the Government the laboratories are experiencing difficulties with chemical reagents supply, uniform, laboratory furniture, equipment etc.

F.2.2. The Sanitary and Epidemic Laboratories of the Health Ministry in Atyrau and Aktau

These laboratories meet hygienic requirements. They provide hygienic studies of water, air and soil. Radiology, bacteriology and toxicology studies are also provided. The laboratories are sufficiently equipped to deal with above-mentioned analysis.

F.2.3. The Laboratories of Academy Institutions

Zoology Institute

The premises are old. Although equipment still works, it does not meet the modern standards. Implemented analyses: ion concentration of water, heavy metals, pesticides, toxicology studies on cold-blooded and warm-blooded organisms.

Chemical Institute

The premises and equipment meet modern requirements. Analyses are carried out on a contract basis by order. Implemented analyses: water and soil (air and ground are possible), no ecotoxicology studies.

F.2.4. Non-Governmental Institutions

TOO Caspian Service Project

The laboratory has cramped premises. Implemented analyses: water, air and soil, heavy metals, organic substances, physical parameters. Equipment does not meet modern standards (no AAS, Gas Chromatography, Liquid Chromatography, potable water, air, soil, sediment, radionuclide analysers, water or air flow measuring equipment). Ecotoxicology studies are not implemented.

TOO Monitoring

The laboratory has cramped premises. However, a new laboratory building is currently under construction . Implemented analysis: water, air and soil. Equipment is old (no AAS, Gas Chromatography, Liquid Chromatography, potable water, air, soil, sediment, radionuclide analysers, water or air flow measuring equipment). Ecotoxicology studies are implemented.

The Hydrometeorology Department of Kazakhstan does not have its own laboratory.

F.3. Turkmenistan

F.3.1. The Laboratories of the Ministry of Natural Resources and Environment in Ashgabat (1), Turkmenbashi (1)

The Laboratory premises (2 rooms) do not meet hygienic requirements. The Laboratory is not appropriately equipped (no AAS, Gas Chromatography, Liquid Chromatography, potable water, air, soil, sediment, radionuclide analysers, water or air flow measuring equipment). It does not seem that equipment is appropriately maintained. Implemented analyses: ion concentration of water, physical parameters. Neither toxicology studies, nor heavy metals analyses are implemented.

Being financed by the Government (the Government has not invested in the laboratory for 5 years) the laboratories are experiencing difficulties with chemical reagents supply, uniform, laboratory furniture, equipment etc.

F.3.2. The State Committee of Turkmenistan for Fisheries

This institution does not have a laboratory.

F.4. Russia

F.4.1. The Special Inspection for State Control and Analysis of the State Committee for the Environment, Astrakhan

The Inspection is one of the major environmental institutions. The analyses implemented by the laboratory are listed below:

- natural water – 34 parameters
- wastewater – 27 parameters
- soil – 21 parameters
- air – 7 parameters
- sediment – 12 parameters
- active sludge – 4 parameters

The premises meet hygienic standards. The laboratory is appropriately equipped.

F.4.2. Kalmytskaya - The State Agrochemical Service Station Laboratory

The premises meet hygienic standards. Implemented analyses: water, soil, plants. However, neither air emissions, nor biology and toxicology parameters are analysed. The laboratory deals mostly with agricultural chemistry.

F.4.3. The Accredited Laboratory of the State Inspection and Analyses of the Ministry of Natural Resources and Environment, Dagestan

The premises meet hygienic standards. The laboratory is appropriately equipped. The implemented analyses are listed below:

- natural water, ground water and water from melted snow – 29 parameters
- wastewater – 21 parameters
- soil, ground, sediments – 8 parameters
- air – 4 parameters

F.5. Azerbaijan

F.5.1. The State Caspian Inspectorate Laboratory of the State Committee for Ecology

The laboratory is one of the major environmental institutions. The laboratory is sufficiently equipped (AAS, Gas Chromatography, Liquid Chromatography, air, harmful substances analyser etc). The implemented analyses are listed below:

- natural water and wastewater – 64 parameters
- soil – 21 parameters

- air – 8 parameters
- sediment – 12 parameters
- active sludge – 4 parameters

Biology and ecotoxicology studies can also be implemented. The premises meet hygienic standards. The equipment is appropriately distributed to the relevant rooms and and is checked by a physical engineer.

F.5.2. Baku State Committee of Ecology Laboratory

The laboratory is equipped with AAS, Gas Chromatography, Liquid Chromatography, air harmful substances analyser, etc. A series of analyses is implemented: water, soil, air, sediment. However, neither biology, nor ecotoxicology studies are done. There are no specialised laboratory premises.

F.5.3. Sumgayit State Committee of Ecology Laboratory

The laboratory is one of the major environmental institutions. The laboratory is sufficiently equipped to analyse water, air and soil. However, there is not modern equipment (AAS, Gas Chromatography, Liquid Chromatography, potable water or air analyser). Implemented analyses are listed below:

- natural water, wastewater – 35 parameters
- air – 17 parameters
- sediment – 12 parameters

Neither biology, nor ecotoxicology studies are implemented. The premises do not meet hygienic standards.

F.5.4. The State Hydrometeorology Committee Laboratory

The Laboratory is equipped to analyse pollutants in water, air and soil. Implemented analyses are listed below:

- natural water, wastewater, water from melted snow – 53 parameters
- air – 18 parameters
- sediment and soil – 32 parameters

Ecotoxicology studies are not carried out. The laboratory does not have modern equipment (AAS, Gas Chromatography, Liquid Chromatography, potable water or air analyser). The premises do not meet hygienic standards.

F.6. Conclusions

The following problems and lacking items were revealed during the current assessment of the state regional laboratories:

- financial problems*
- equipment upgrade
- laboratory furniture upgrade*
- modern methods*
- uniform*
- safety equipment*
- chemical reagents and standards (Azerbaijan, Kazakhstan, Turkmenistan)
- maintenance* of premises
- stable power, heating, water and gas supply
- potable water, air, soil and sediment analysers*
- measuring equipment for water and air flow*
- CTD*
- ultrasonic bath
- rotation evaporator
- water ion exchanger (production of pure water)
- motor boats*
- special vehicles
- portable laboratories
- complex laboratories for water, air and soil
- 10-15 l deep water and surface bathometers
- Van-veen sediment sampler with covered area range (preferably box core sampler)
- Electronic balance
- PCs and digital equipment
- equipment and materials for ecotoxicology studies
- data, reports and information are not in computer format; most of the laboratories do not have a database.
- *in all laboratories

F.7. Recommendations

The current study recommends the following:

Establishment of national laboratories in each literal country equipped to a common standard:

- identical laboratory equipment, furniture, PCs, etc.

- assessment and upgrade of the old methods of pollutants measurements in natural water, wastewater, ground water and water from melted snow; air and air emissions; soil and sediment; active sludge in biological WWTP

Establishment of the regional reference laboratory – **The Central Audit Laboratory** with the following departments:

- Audit and Methodology Department
- Analytical Department (water, air, soil, sediment)
- Biology Department
- Ecotoxicology Department
- Assistance Department (sampling group, conservation and processing of samples)
- Department for Ecotoxicology Studies of Flora and Fauna in the Caspian Sea and Microbiology and Bacteriology Studies

The Central Audit Laboratory should have the following responsibilities:

- establishment of a common reporting format and testing scheme
- external audit
- training for laboratory employees
- upgrade courses and training for laboratory employees on the Caspian environment
- database establishment for new laboratories
- participation in standards elaboration
- participation in environmental project documents elaboration (EIA)

F.8. Outcome

Reliable quality and quantity load data on:

- pollutant discharges (river, municipal, industrial, agricultural, radioactive, etc.)
- emissions (organised and disorganised)
- rainfall
- other contaminants polluting the Caspian Sea environment

Real data on the Caspian Sea:

- water quality
- air quality
- sediment quality
- flora and fauna, incl. seals.

Establishment of a reliable database on the Caspian Sea