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REFERENCE HANDBOOK ON ENVIRONMENTAL COMPLIANCE AND ENFORCEMENT IN THE MEDITERRANEAN REGION

Part IV

SAMPLING

In cooperation with

WHO

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IN THE MEDITERRANEAN REGION

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PREFACE

Within the framework of the MED POL Programme Phase III for the Assessment and Control of Marine Pollution in the Mediterranean adopted in 1996, special reference is made on the pollution control component to assist countries to fulfill the provisions of the Protocol for the Prevention of Pollution from Land-based Sources and Activities (LBS Protocol). In fact, Article 6 of the Protocol, which was signed in 1980 and revised in 1996, calls for the strengthening and/or establishment of systems of inspection related to land-based pollution.

Among the activities for the promotion of the environmental inspections, a workshop of experts on Compliance and Enforcement of Legislation in the Mediterranean for Control of Pollution resulting from Land-based Sources and Activities, was convened in Sorrento, Italy in 2001, to review progress in that field and discuss future activities. As a result, it was recommended that guidelines on compliance and enforcement be developed, indicating the general lines to be followed rather than going into detailed recommendations.

These guidelines have been prepared, reviewed and commented upon by the National MED POL Coordinators and the final text provides the framework for the enhancement and strengthening of the environmental inspection systems in the Mediterranean. The countries may use them to specify their own code of conduct and practices to be followed by their Inspectorates.

Following the preparation of the said guidelines, it was felt that more information was needed on a number of technical issues, so that reference information developed adequately could better assist the implementation of the guidelines. As a result, the Handbook containing more detailed information was produced, under the technical supervision of WHO/MED POL and with the assistance of a team of five experts.

The purpose of the Handbook is to raise the level of performance of the environmental inspectors and support the above mentioned guidelines by providing details on assessing, developing, implementing and sustaining a viable inspection programme.

All aspects of an inspection programme are covered, including planning and designing enforcement programmes, international cooperation, non-point sources of pollution and compliance strategies, enforceability of permits, self-compliance, environmental negotiations, public participation, voluntary agreements, profiles of inspectors, inspection policies and planning, sampling, inspection techniques and training. To address those elements of comprehensive inspection programmes, the Reference Handbook includes the following:

- Organization issues
- General procedural issues
- Human infrastructure
- Sampling

The above structure appears in the four volumes, each one presenting a specific subject related to environmental inspections. The experts team is composed by professionals with long-standing experience on inspectorates in their countries. The texts reflect the authors experience from different angles and different philosophies that enrich the contents. It may happen that some issues are mentioned in more volumes. This is due to the fact that repeated issues provided another perspective and/or are needed for the complete understanding of the specific volume. The experts team is composed by the following scientists:

Mr Yasser Sherif is a former Head of the Environmental Inspection Unit in the Egyptian Environmental Affairs Agency (EEAA). He was responsible for preparing Part I related to "Organizational issues".

Mr Rani Amir is the Director of Marine and Coastal Environment Division in the Israeli Ministry of Environment. He was responsible for preparing Part II related to "General procedural issues".

Mr Allan Duncan is former Chief Inspector of Her Majesty's Inspectorate for Pollution (HMIP) in the UK. He was responsible for preparing Part III related to "Human infrastructure".

Mr Robert Kramers is a specialist in the Dutch Information Centre for Environmental Licensing and Enforcement. He was responsible for preparing Part IV related to "Sampling".

Mr Robert Glazer is former Head of a regional inspectorate for the Ministry of the Environment in the Netherlands and coordinator of the European Network for the Implementation and Enforcement of Environmental Law (IMPEL). He was responsible for preparing the Guidelines on compliance and enforcement and acted as a coordinator and reviewer for all four parts of the Reference Handbook.

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1. Introduction

Taking representative samples requires special skills and experience. This part of the Reference Manual document should there for not be seen as the manual for inspectors to perform all these measurements them self. For example, taking air-samples special expertise in this field is required, of course in other fields less expertise could be necessary. The aim of this part is to give the inspector more understanding in problems and difficulties that will come with environmental sampling.

1.1. Role of the sample

Emissions and pollutions are very different all over the world. Not only the sorts of emissions and pollutions, but also the quantities, often depending on geographical, economical and social circumstances. Sometimes countries have well developed systems of industry's self monitoring. In licenses or sometimes in the law itself prescriptions can be found how to monitor. In more and more cases industries themselves have their own environmental management system of which monitoring programmes are part. Sometimes however these systems are completely absent.

Several reasons exist to monitor. Analysing trends, determining fate and transport of pollutants, defining critical areas or measure the effectiveness of conservation practices. But also to evaluate program effectiveness, to make waste load allocations, to model validation and calibration, to define an environmental problem or to conduct research.

In this toolkit however we will not focus on these reasons. Our reason will be sampling as a form of compliance assessment. Of course there will be overlaps between monitoring activities for other reasons. The results of this monitoring, the way in which it was done, the trust authorities have in the monitoring, etc. may and probably will be taken into account in deciding whether or not to assess compliance through sampling. Nevertheless sampling will never be the exclusive territory of situation where no monitoring at all takes place. Also in situations where monitoring is done, it will be of importance to assess compliance every now and then. Not only to check whether the monitoring results of industries themselves are correct, but also to prevent that forms of self-monitoring will cause that the responsible people in industry may feel free as they like. This might easily lead to abuse.

Monitoring programmes, either by industries of by government(s) may cover:

- Controlled emissions of waste gases and airborne particulate to air via chimney stacks;
- Controlled discharges of waste water via sewers to and from effluent treatment plants, directly to receiving waters such as the sea, lakes, rivers and streams and to land via septic tanks and soakaways;
- Controlled disposal of solid waste to landfill sites;
- Controlled disposal of solid and liquid wastes, including organics, to incinerators;
- Process raw material inputs (e.g. trace contaminants) and operating conditions (e.g. process temperature, pressure and flowrate);
- Fugitive releases to air, water and land; being those that are not coming from a defined point but rather from a number of widespread points;
- Energy efficiency and water consumption;
- Noise and odour nuisances;
- In addition, receiving environments (e.g. ambient air, grass, soil surface and ground waters) may be monitored.

Monitoring programmes may be a routine exercise of sampling at regular intervals or may be short-term campaigns with a concrete objective. These programmes include stages of planning, preparation, implementation, data analysis and reporting. In this toolkit we will only discuss these aspects against the background of compliance checking and enforcement.

Sampling is a step in the overall compliance checking and enforcement process. It is a key element. Samples collected during an inspection must be of known quality and collected following sound technical procedures to ensure their reliability for use as evidence. Effective communication with the laboratory is a critical component in the process.

Official samples, as well as records and other information obtained during sampling, must always be collected and treated as though the material will be used as evidence in an enforcement action. This involves ensuring that each sample is properly collected, prepared, and documented. The primary factors to consider when collecting a sample are:

- Communicating effectively with the receiving laboratory;
- Ensuring that an adequate amount and representative portion of a batch of the product is collected for analysis;
- Assuring that no cross-contamination occurs during sampling of large containers and that proper containers and caps are utilized;
- Assuring that all official samples are properly identified, preferably with a unique numbering or other identification system;
- Recording the inspector's observations of the sampled batch, such as photographs, copies of records, data, correspondence, and/or results of interviews;
- Obtaining any labelling and literature that pertain to the sampled product or batch;
- Obtaining signed statements or sworn affidavits from persons who may potentially serve as witnesses.

Duplicate samples are collected on the request of the person (or establishment) that has a direct (legal) interest in them. In general national legislation will determine who is entitled to be given these kinds of samples. These samples should be collected, identified and sealed using the same equipment, techniques and sampling protocols used to collect official samples. This could be identified as an equal amount of the product taken in the same manner from the same site, stack, container, etc. It might even be an equal portion acquired by subdividing the samples taken by the inspector. This will only be possible in case the sampled portions were of sufficient size to do so. Subdividing small-size units may not be preferable, because:

- The integrity of the sample as evidence is more difficult to maintain and defend
- Cross-contamination is minimised
- The possibility of exposure during sampling is minimised
- The laboratory can determine the net contents, if necessary

Convenience, accessibility, and safety should also be considered when selecting a sample site.

1.2. Accuracy and precision

To serve as evidence most legal systems will demand a high decree of accuracy and precision. Not only the sampling itself but also the way in which the analysis is carried out will be crucial for the trust in the results. According to chemistry definitions *'accuracy'* denotes the nearness of a measurement to its accepted true value and is expressed in terms of error.

'Precision' refers to the reproducibility of results. It is the agreement between the numerical value of two or more measurements that have been made in an identical fashion.

In the discussion on accuracy and precision variables such as samples technique, calibration of field and/or laboratory equipment, inspector's skills in technical and chemical sampling, quantity of target material, etc. play a role.

Laboratory reports frequently mention a confidence factor which may be measured in plus or minus percentiles. Other chemicals may disturb the picture of the target chemical. In that kind of situation it is good to question whether the data can still be used.

Putting the right quality of manpower on sampling and following the appropriate sampling protocols is to state the obvious. Even a lager team of qualified persons may be necessary to get accurate and precise results.

All the characteristics of the sampling methodology (see 1.4) should be written in a label attached to the sample, in order to perfectly identify the sample. This label should also include:

- A unique sample identification number assigned from a sequentially number register
- Date and time of sampling
- Sample preservation (if applicable)
- Process relevant details
- References to measurements made at the time when the sample is taken.

1.3. Quality considerations

A sample's quality is co-determined by the method used. This method should be determined by the what and why of the sampling activity. It should be assured that the right method is selected and documented properly. The one who is leading the sampling (which may be the individual inspector on his own) carries responsibility for the sample's quality. He must control and insure that proper methods and procedures are followed throughout the entire process from acquiring clean sample containers and other tools, transportation of these containers (without contaminating them), taking the samples, transporting them back to the laboratory without contaminating them, determining the method of analysis, what chemicals to analize for and how small of a concentration they should look for.

Hints

Use clean sampling containers and tools
Don't contaminate them during transport
Determine the method of analysis
What chemicals to analyse
What concentration to look for
Don't take samples of which the quality cannot be guaranteed!!!

If you cannot guarantee the quality of the samples you should not take them at all!

The integrity of a sample and the continuity of evidence must be maintained so that if the sample does show non-compliance with the law, it can be used as evidence of the violation. Continuity of evidence is the control of evidence from the time it was taken to the time it was analysed. This means documenting anyone who had contact with, or had care of the evidence.

In case of necessary transfer of evidence to another person or move it to another location, personal delivery by the person already in possession of the evidence is the best practice. Secure methods of mail or shipment may also be used. Seal the evidence and be sure it is adequately identified. Ensure that the package is locked or securely fastened. Retain original bills of lading and other shipping documents. Contact the recipient to arrange for pick-up or receipt. Tell the recipient how the evidence is sealed and marked. In this way the recipient can determine whether the evidence has been disturbed.

Issuing a license it is considered best practice to include quality considerations in the monitoring requirements with the relevant limits. This should help the measurements to be reliable, consistent and auditable. The main quality considerations to be recognized can be found in the table 1.

Table 1

Quality considerations	
Calibration, maintenance and certification	The monitoring system should regularly be calibrated and maintained, and relevant instruments, personnel and analytical laboratories should be certified under recognised schemes
Updating of monitoring requirements	The monitoring programme should regularly be reviewed and updated to take account of: changes in limits the latest compliance situation of the process new monitoring techniques
Off-scale situations	Under some temporary process situations monitoring equipment may go off-scale e.g. during abnormal conditions or during start-up or shut-down. In such cases it is important that the permit states how long the monitoring is allowed to be off-scale before emissions are judged to be non-compliant
Availability and breakdown of monitoring equipment	The permit should state if/how long a process is allowed to continue operating in the event of a breakdown of monitoring equipment. Consideration should be given to specifying requirements for data capture, off-line maintenance/calibration periods and back-up monitoring (e.g. taking of occasional spot samples while continuous monitors are available)

1.4. Representativeness and methodology

Regardless of the procedure all samples must be representative of the material or event. Together with methodology, representativeness is a prime issue.

Does your sample represent a specific waste stream, site, event or activity? Is it representative for what you needed to evaluate for compliance?

Representativeness includes that the sampling is carried out according to a relevant instruction or standard. Sampling is a complex operation that strongly affects the analytical results and the conclusions derived from them.

The sample must be representative in the time and also in the space. This means that if the discharge from an industry is being analysed, the sample taken to the laboratory should represent all that it has been discharging during, e.g. a day of work (*time representativeness*); or if a material is being monitored, the portion of sample should represent the thousands of tonnes that are being introduced in the plant (*space representativeness*). Samples should be collected from a location that is representative of the facility's discharge. If the facility has more than one discharge point it may be necessary to collect samples from several locations in order to adequately characterize the facility's entire discharge.

The sampling should be carried out with no change in the composition of the sample. In fact, there are parameters in a sample that should be determined, or somehow preserved, in situ as their value may change with time, e.g. the pH and the content of oxygen of a wastewater sample. If we are sampling lime or NaOH we have to preserve it immediately to prevent contact with air as they react with the air to give carbonates.

Of course complexity increases as the sample to analyse may range from a few grams up to thousands of tonnes, and the substances to be analysed can vary widely in their nature. The material may be homogeneous, in which case sampling at a single point is enough. But there are few homogeneous materials. Generally the materials are heterogeneous, and several samples from different points will be required in order to have a spatially representative sample.

On the one hand, solid, gaseous or liquid samples can be found; which in turn can be mineral, vegetable, or animal samples; on the other hand the concentration of what is being monitored may range from trace levels upwards. Therefore it can be concluded that it is difficult to provide general rules for sampling all type of substances of materials.

A number of factors determine the methodology. Methodology can be defined wide or narrow. In this toolkit we will use the wide approach.. The following factors are part of this approach.

- The ***location*** at which the samples are to be taken should be such that the material is well mixed, and it should be taken always from the same defined points. In order to have homogeneity in composition and temperature the sampling should be carried out far enough from mixing points. When monitoring particles it is also important that samples are taken from points far enough from flow disturbances, such as bends, which could make particles to be distributed non-uniformly in the bulk to be sampled.
- The ***frequency*** at which the samples are to be taken is usually decided on a risk basis, taking into account the variability of the flow, its composition, and the magnitude of the variability with respect to limit unacceptable values.
- The ***sampling 'method'*** and/or ***equipment***
- The ***size*** of individual samples and bulking arrangements to provide composite samples
- The ***type of sample*** e.g. sample for single or multiple determinant analysis
- The ***personnel*** in charge of taking the samples should be skilled.

2. Preparing the sampling

2.1. When, why and how to take a sample

Sampling can be an expensive activity. As such this is a reason to question why to take the sample. But let's assume that not all sampling is expensive and that we will only sample if useful and cost-effective.

In general the environmental inspector will take a decision on the questions 'when' and 'why' to sample.

When to sample is determined by the best chance to obtain a representative sample. The amount of the product(s) to be collected depends largely on the amount of material required for the anticipated laboratory analysis and to assure representativeness, including the quantity required for quality control purposes (i.e. splits, repeat examinations and replicates). Considering these sampling needs, the sample size is to be kept to a minimum to reduce the burden of disposal of the unused sample portion and to mitigate potential human and/or environmental exposure.

Why a sample is taken is more subjective. It is initiated if there is a lack of confidence in available data or because of incomplete data at the facility or home office. Sampling may be required by the law or a license. But also data needed to document an event can be a reason. In this toolkit the 'why' will focus on compliance checking and enforcement.

Sampling procedures include designation of sample types, volumes, containers, and preservation methods to be used for each pollutant parameter as well as sample identification and documentation procedures. These are general procedures. Specific information on each facility should also be developed. This may include pollutant parameters to be sampled, sampling location and safety concerns. Obtaining this information before the sampling trip will allow the sampler to bring the proper equipment, know where to sample and what pollutants to sample for, and be familiar with necessary safety precautions.

Hints

- The sample must be representative
- Use the appropriate standard operating procedure
- Identify the analysis method in your site specific quality assurance plan
- Notify the laboratory of the specificity and chemical concentration required in the analytical report

Sampling (analysis) will be needed to clarify whether there is compliance or non-compliance. The most fundamental types of samples are composite samples[1] and grab

[1] Composite samples are collected over time (either by continuous sampling or by combining individual grab samples) and reflect the average characteristics of the sampled material during the sampling period. Composite samples are either 'flow proportional' or 'time composite'. Composite samples may be needed to determine the average characteristics of the sampled material, particularly if the material has a highly variable pollutant concentration or flow rate. Composite samples should be collected during the entire period the facility is operating and discharging, e.g. 12 hour a day in case of 12 hours of discharge that day.

samples[2]. Both types of samples can be either planned or samples of opportunity[3]. Standard operating procedures should be kept on file at the inspectorate, insuring defensible repeatability and consistency and a written record of what was done. Deviations from standard procedures must be thoroughly documented. Several years after the event it may be necessary to refer to the sampling. Only laboratories should be used that adhere to written standard operating procedures.

Standard operating procedures supplement site specific procedures as e.g. laid down in a license or in a sampling plan as part of a company's environmental management system.

2.2. Information sources

To place the decision to sample, what to sample, where and how to sample, in the right context, sufficient basic information is necessary. This information can be drawn from different sources. It can be information in written or spoken form, but also visual observations, electronic information, etc. Written information will very often turn out to be the most important information source. And as we will see in this paragraph there can be very many of them.

The most common written information sources will be the license of the company, other legal provisions and the documents that are in the company's file at the office of the inspectorate. The license and these documents probably contain information on the processes in the company, telling the substances that may or may not be used, the emission limits, the obligation to monitor emissions, the registration of emissions and waste streams, the allowed size of the storage of these substances and the conditions under which this is allowed.

But also the company's floor plans are very much worth to study to get knowledge of the position of different activities, about the electric system, the sewer system, safety routes, etc.

Documents with monitoring information can not only tell whether there was compliance with existing rules, but also about substances and their concentrations present at the company's premises. Process flow charts can be important in calculating streams of incoming and outgoing materials and the possible exceeding of
licensed emissions.

An historical examination of the company may indicate which substances are at stake. This is important in order to minimise risks and take effective measures and have the proper sample analyses done. It may be possible to determine the origin of certain wastes, which can give indication on its nature. Will it be dangerous or will it not? Questions that have to be answered in these matters are:

- which are/were the company processes, the products, the raw and auxiliary materials used for that and the waste coming from it;
- what does the inspection and enforcement history of the company look like;
- what is in the company's license;
- which transporting company's worked for the company;

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[2] Grab samples are individual samples collected over a period of time representing the sampled material under the conditions only at the time the sample is collected. Grab samples are usually taken manually but can be collected using an automatic sampler.

[3] Samples of opportunity are events that were not generally anticipated. They may be required because of a new process or expansion or the may be necessary because of a spill or discharge.

- how about the quantity and composition of the industrial waste removed from the company's premises;
- how about the way in which the company's administration is run;
- what about the date on the company's buying and removing;
- what do other inspections organisations or colleagues know of the company;
- what can the license, the Chamber of Commerce of citizens/NGO's tell about the way the company works;
- have complaints or offences of the company been reported.

Also topographical information, information in e.g. municipal files on the composition of the soil or ground water streams, electric cables, underground sewer systems , pipelines etc. at the company's premises can be useful to know.

Also labels on packaging can provide an indication, although one always has to be careful not to take the label as a guarantee! There may be something different in the packaging!

Another source may be found in the files of the police in case the company has been prosecuted and/or convicted before, because of acting against the law. This information may include the reliability of the persons that are in the lead of the company and thus of possible offences against the law. Dependent on the ruling national legislation this information may or may not be used as a source for environmental inspectors.

Complaining citizens or non-governmental organisations (NGO's) may provide useful information on forbidden activities. This can occur through personal contacts with inspectors, but also through a special telephone (or internet) service provided by local, regional or national authorities in order to take citizens complaints seriously into account. In many countries environmental inspectors consider complainers as allies. Of course an inspector will always take care to verify the information of any complainer or witness before taking it as the truth. The higher the consciousness of citizens on environmental problems, the better the quality and quantity of their information supply towards the authorities.

Observing a company may also supply an inspector with valuable information, e.g. the number of trucks entering or leaving the company grounds with raw materials, waste or end products, the colour of the surface waters in nearby streams, the colour the nearby soil, the state of the vegetation nearby the company, near surface water borders or following the dominant direction of the wind. But also the 'smell' that hangs around a company.
These observations can be supported by taking pictures, not only from the landside but – if available- from the air, if possible even with infrared photography. This can provide very useful information about the spots of possible (illegal) emission sources.

The internet becomes more and more an important source of information, also for environmental inspectors. It may provide a lot of technical information on a company's processes, not only because an individual company may have an internet-website, but also by searching information on the web on issues found in written files. Sometimes even a comparison can be made in case of company's that work in more than one country and present themselves on the web. This may reveal difference in approach in different countries. It can be interesting to investigate why this is.

2.3. Qualification and training of inspectors

Taking into account the accuracy and precision, the quality considerations and all safety precautions, getting a good sample in safe way is not an easy job. So far as known there are no special educations or certifications needed to take samples, which can also be used as evidence. For example in The Netherlands any inspector could take samples as long as the accepted guidelines for sampling are followed.

Nevertheless it's of great importance an inspector is to practice his or her skills on a regular basis. In practice it happens some inspectors take samples on an almost daily basis while others only take a few samples a year or even less. Recommended is to have a yearly training. This could be done within the organisation itself (for example by the sample coordinator (if present) or by a third party.

The training should provide inspectors with proper sampling skills for a variety of media, i.e. solid, liquid or gas phase. It should include: safe techniques; laboratory requirements; sampling protocol; location selection; and, other related topics. The training should also be concentrated on the presentation of demonstrated "best available technologies" related to field techniques, laboratory expectations, standard operating procedures as defined within published standards/regulations

2.4. Inspection attendees

An inspector may carry out the sampling on his own. Certainly not always this will be possible. This may be because of the complexity of the sampling in a technical way (for example: taking representative air samples should always be taken by special experts within this field) or in a physical way. But also because of safety reasons. For that reason it is good practice that inspectors are attended by others.

The need may be determined by things like the place where the site is located (rocky area, swamp, industrial area, stack, hostile area, etc.), when the sampling will take place (daylight, night, snow, rain etc.), the kind of sample (air, water, noise, soil etc.), technical complexity.

Depending on the specifics of these circumstances the inspector may be attended by:

- Another inspector;
- Someone from the ministry of environment;
- Local or regional officials;
- A technologist from a laboratory;
- An expert (or experts);
- A (or more) police-officer(s).

To avoid misunderstandings and thus to safeguard the sampling quality it is recommended to conduct the sampling by two people at least. Of course in all cases it has to be established who is in the lead. This has to be determined before arriving at the site. This person is responsible for the coordination of the inspection and the on-the-spot decisions.

2.5. Making a sample plan

Prior to conducting a sampling a sample plan has to be developed. This plan will serve as a guide in performing the sampling. The amount of detail necessary depends on the purpose of the sampling and to whom the results will be submitted. As a minimum, the plan should include the following.

1. Introduction

<u>Reason</u>: define the reason for sampling.

Purpose or aim: define the purpose or aim of sampling. The aim is leading for the strategy that will be followed. The purpose can be:

- Determination of the chemical substances in water, soil, air or waste. The strategy should be aimed for representative samples. Important in this is the amount of sample points, the location, depth of sampling and the question if composition samples should be made.
- Determination of the source of the pollutants. The strategy should be aimed on the relation between pollutants and emissions. Important in this is describing the situation, and to support this with spot samples.
- Determination of the dimension of the pollution. Important in this is to spread the sample points as good as possible over the sample area.

Describing the location: where is the location situated, what kind of surface and activities are taking place, orientation in space (the background).

2. Describing object

How does the non-compliance record regarding permits and legal obligations looks like. Are there any suspicions, complaints or statements about illegal activities.

Description strategy: describe in the plan the components that will be sampled and the strategy that has to be followed.

Note: If is a criminal investigation in progress, if so the sample strategy will have to follow the criminal investigation strategy.

The characteristics of the substance to be sampled: during the sampling a pollution of the substances has to be assumed. Possible pollutants has to be identified and safety regulations will have to be taken.

Register and report: in most cases it is recommended to take photos or videos of the situation before the sampling start (before getting the technical evidence). A situations sketch should be made which will include locations, sample points and measurements. Further, sample registration forms should be used to describe the composition, the amount and the method of sampling.

Note: if any product (like pesticides) are being sampled, make sure to make all necessary arrangements for confiscating (chain-to-custody).

3. Sampling equipment

Define the necessary equipment; like sampling materials, registration material and tools.

4. Packaging and transport

Packaging: for every kind of sample the package material should be defined.
Transport: describe by whom, how and when the samples will be transported. Further if transfer to the laboratory will take place right away or if the samples will first be stored.
Storage: define the storage conditions, don’t forget the contra-samples

5. Co-ordination

Co-ordination of the sampling: address the function of co-ordinator. He will be responsible for the progress of the sampling, the safety of the people taking the samples, and when necessary to call-in help from experts.

Contacts with participants: the co-ordinator will maintain the contacts with the suspect and other people involved.

6. Safety

The characteristics of the substances: define the hazardous substances that will be encountered and the protective equipment will have to be used during sampling.

Measurements: describe when, which and how measurement will take place.

Personal protective equipment: describe the personal protective equipment and other safety materials. Describe how used clothing will be handled afterwards.

Responsibilities: describe who is responsible for safety. This can be the sample co-ordinator like described above or a special safety co-ordinator.

Note: The plan should also mention that any instructions from the (safety) co-ordinator will have to be followed at once.

7. Composition of the team

Describe the composition of the sampling teams (names, function or duty) and describe the workload, the way of communication and the estimated time path.

2.6. Measurement Methods

Monitoring must be based on recognised and validated methods, which are generally termed “standard” methods, where they are available. Standard methods are produced by CEN, ISO and the national standards organisations in the different states. Two key issues in relation to standard methods are:

- who chooses, proposes or specifies the standard method for use in a given situation;
- how is this method judged to be acceptable.

Standard methods may be chosen, proposed or specified for use in a compliance monitoring programme by:

- the competent authority - this is the usual procedure;
- the operator – this is usually a proposal which still needs approval by the authority;
- an expert – this is usually an independent consultant who may propose on behalf of the operator; this proposal still needs approval by the authority.

When deciding whether to approve the use of a method the competent authority is generally responsible for deciding if the method is acceptable, based on the following considerations:

- fitness for purpose - is the method suited to the original reason for monitoring as shown for example by the limits and performance criteria for an installation;
- legal requirements;
- facilities and expertise – are the facilities and expertise available for monitoring adequate for the proposed method e.g. technical equipment, staff experience.

The choice of measurement method may be constrained and/or informed if it is:

- defined in legislation;
- recommended in published technical guidance.

2.7. Duration of the monitoring

The total duration of a monitoring programme is often aligned to the operating life of a process, particularly when the timeframe(s) of any harmful effects are short compared to the operating life. However, monitoring may sometimes need to start before a process has begun operating (e.g. to establish baseline ambient concentrations before any extra impacts from the process). Similarly, monitoring may sometimes need to continue after a process has ceased operating if its harmful effects are more long-lived (e.g. monitoring of groundwater after closure of fuel depots, landfill sites or nuclear installations).

2.8. Frequency of the monitoring

The frequency of monitoring refers to the time between individual measurements or groups of measurements at a process or in a receiving environment. It can vary widely between different situations (e.g. from one sample/year to on-line measurements covering 24-hours/day). Monitoring frequencies can be divided into two main categories:

- continuous;
- non-continuous.

Non-continuous monitoring can be further divided into four sub-categories:

- periodic;
- response;
- reactive;
- campaign.

Descriptions of the possible approaches, which should be considered, are noted below:

- <u>Continuous monitoring</u>: This involves an ongoing series of measurements that provide data with a high time resolution (e.g. continual readings from rapid-response instruments). The data are often available in real time (e.g. as instrumental read-outs or electronic displays) and so are useful for short-term process control purposes. Continuous monitoring may be relatively expensive compared to non-continuous monitoring depending on the required frequency of periodic measurements. Also, it may not be an option for some pollutants / situations. This may be because appropriate continuous instruments have not yet been developed, or detection limits are too high to allow measurements without pre-concentration of samples, so that pollutant samples must be accumulated over a period in order to be detectable;
- <u>Non- continuous periodic monitoring</u>: This involves measurements made at regular intervals in order to cover a defined part of the operating time of a process. It may involve spot measurements made at regular intervals, analysis of samples accumulated over regular periods, or instrumental data obtained at regular intervals during operation

of the process. The periods of monitoring should be specified in advance (e.g. in a permit or legislation) and designed to be representative of the total operation;

- Non- continuous response monitoring: This involves measurements made in response to special events which are foreseeable but cannot be precisely scheduled (e.g. start-up and shut-down conditions, low and high utilisation conditions). The monitoring is done at irregular intervals. It is “routine” because the events to be measured can be anticipated but not their timing;
- Non- continuous reactive monitoring: This involves measurements made in reaction to special events such as exceeding of limits, which cannot be foreseen. The work is therefore devised on an ad-hoc basis rather then being specified in advance, and is done at irregular intervals. Because of the nature of this monitoring it may not be possible to specify the measurement methods in advance;
- Non- continuous campaign monitoring: This involves measurements made in response to a need or interest in obtaining more fundamental information than routine, day-by-day monitoring normally provides. The types of events which may trigger campaigns include evidence of epidemiological effects, and permit applications for new processes where baseline monitoring is needed to aid assessments. Campaign monitoring usually involves measurements that are relatively detailed, extensive and expensive, so that they cannot be justified on a regular basis. Examples are: sampling of dioxins in soil around incinerators; detailed speciation of volatile organic compounds for odour or other investigations, studies to verify more conventional measurements and estimate uncertainties, eco-toxicological surveys, and fundamental research studies.

3. Sampling tools

3.1. Sampling equipment

The laboratory can provide information on the types and volume of samples needed for particular pollutant parameters, sample preservation methods and holding times, and shipping instructions. They may also provide equipment, such as samplers, pH meters, sample containers, chain-of-custody forms, sample labels, tags and seals.

Prior to the sampling trip, any required sampling and safety equipment should be assembled, cleaned, and checked to ensure that it is in proper working order. All necessary paperwork should also be prepared prior to the trip. This may include assembling and marking, as possible, the required sample container labels or tags, forms an lab request sheets. Sampling and field analytical equipment such as pH meters should be calibrated.

Equipment and materials:

- Certificate of designation, identity card or badge
- Camera/video camera
- Notebook
- Watch
- Explosion-proof flashlight
- Checklists
- Pens
- Important telephone numbers
- Site file
- Spare batteries
- Soil sample drills
- Sealing materials
- Spark-free vessel key
- Stainless steel spade
- Glass pipette
- Plastic container (chemically resistant)
- Glass container (for oil, grease, phenol, organic samples)
- Amber glass containers (for iron cyanide)
- Containers with Teflon lined lids (for volatile organics)
- Electronic thermometer
- Electronic pH/C thermometer
- Plastic bags that can be sealed
- Aluminium dip sticks
- Storage crate
- Measuring tape (roll-up)
- Compass
- Cool box

3.2. Clean tools / equipment

Sample tools can easily be contaminated. Cleaning them is crucial under all circumstances. In case of oil or greases it can be clever to put and transport them in sealed plastic bags. The best way to act is to clean sampling tools and protective clothing immediately after use and to store them separately where necessary. The laboratory that will be performing the sample analyses may be contacted for specific cleaning instructions. Some laboratories may provide pre-cleaned sample containers.

To clean sampling materials and protective clothing the following cleaning products may be used:

- green soap
- benzine
- nitric acid (0,1 M)

- water
- (soft) brushes
- cleaning cloths
- tube brush

Try to remove the contamination with cold or hot water using brushes, non-perfumed tissues and cleaning cloths.

Inorganic contaminations can be removed with water with some acid. Organic contaminations can be cleaned with benzine, denatured alcohol, green soap or another phosphate free detergent. Equipment of stainless steel and Teflon may be cleaned in a dish-washer. High-pressure spraying pistols and steam cleaners are very good to clean drill pipes. In all cases the equipment should finally be rinsed out with water. Let the equipment dry in warm or cold flow of air and store it dry and dust free.

In case it is no longer possible to clean the equipment or in case deficiencies on the equipment are discovered, the equipment should be replaced as soon as possible. Some equipment has only a limited life-time and has to be replaced anyhow. For example gas tube filters, gas detections tubes, safety helmets.

Other means (of detection), like the pH-meter, need calibration from time to time. Take care that you have an administrative system to take care that this happens in time.

Inspectors should be aware of all requirements regarding the proper disposal of sampling equipment. Where equipment has been damaged or contaminated to the point that it is no longer usable, such equipment should be properly cleaned, sealed, and deposited in the appropriate waste containment facility. Broken glass bottles or jars and glass thieves require wrapping in multiple layers of newspaper prior to placing in bags. If contaminated, this broken glassware will require depositing in an approved hazardous waste storage or handling facility. If it is necessary to reuse glass or metal sampling equipment, inspectors will need to decontaminate these implements after each sample. As described before, decontamination normally requires washing and rinsing, which creates a waste that should be disposed of in accordance with the applicable regulations.

4. Taking the samples

4.1. Air

We started this part of the Reference Manual document by explaining the aim of this manual "Environmental sampling". Again we would like to emphasize this chapter is give the inspector more understanding in problems and difficulties that come with waste gas monitoring and thus better understanding in interpretation of the measurements results, and not to perform all these measurements them self.

4.1.1. General aspects of waste gas sampling

Emissions from a process include both gaseous and particulate pollutants and the sampling and measurement techniques required to provide an accurate measurement of each are entirely different.

Particulates are an inhomogeneous suspension in the gas stream and rather more complex sampling procedures are applied to obtain a representative sample. In contrast the gaseous pollutants can be regarded as being a relatively homogeneous mixture in the stack gases and it is a fairly simple matter to obtain a representative sample. The main problems in sampling gaseous pollutants occur with fairly reactive gases where sample transport and conditioning is of paramount importance.

Continuous and periodic (discontinuous) monitoring

Both particulate and gaseous pollutants can be monitored continuous or periodic. The basic differences between continuous and periodic monitoring are:

- Periodic sampling, carried out by manual methods, is generally labour intensive and requires relatively well trained staff to obtain representative results;
- Periodic monitoring does not give a continuous record of the process emission conditions. Many separate measurements have to be made to provide data which will stand up to statistical analysis;
- Periodic manual sampling provides more accurate results from the point of view of being more easily traceable and therefore such methods are often used for calibration of continuous monitors;
- Continuous monitoring is more capital intensive but, with a service contract for maintenance and calibration, may be comparable with manual sampling in the long term;
- Whether continuous monitoring is justified may depend on the size of the process, the emission limits which have been set and the availability of suitable continuous monitoring equipment;
- Continuous monitoring equipment is only available for a limited number of pollutants although most pollutants can be monitored continuously at a price;
- Continuous monitors give a continuous record of the emissions from the process and provide instant indications of problems with emission control equipment. For this reason they are favoured by regulatory authorities wherever instrumentation of reasonable cost is available e.g. NOx;
- Continuous monitors may be less accurate in an absolute sense than manual standard methods but they are amenable to statistical analysis because of the vast numbers of data points obtained;
- Continuous monitors require periodic calibration against standard manual methods.

As a general rule manual standard methods are required to verify the results from continuous monitors but continuous monitors, provided that they are properly calibrated and maintained provide better information for regulatory authorities and plant operators alike. Cost is likely to be the deciding factor in many cases.

In situ and extractive monitoring

Continuous monitoring can be subdivided into in-situ monitoring where the duct is (in effect, the measuring cell) and extractive where the sample is extracted along a sampling line to a measurement station which may be quite remote from the duct. If a sample has to be withdrawn from the duct, handled and passed on to a measurement system, errors are introduced. Therefore, if the measurements can be carried out in-situ, without the need to extract the sample and handle it in any way, the errors are much reduced.

However, it is more difficult to standardise or calibrate in-situ instruments. On the other hand, in extractive systems great care is required in designing the gas handling system in order to preserve sample integrity but calibration of the instrumentation with standard gases is relatively easy.

A wide range of techniques are involved not all equally applicable to each measurement situation. In making measurements to test regulatory compliance, instrumentation needs to be of a high standard and some countries, for example Germany, operate an approval procedure. The typical approval procedure is designed to check instrumentation under laboratory and operating conditions to ensure satisfactory performance and reliability both from the point of view of the measurements made and with respect to safety and robustness. As a result of this procedure it is possible for the German Government to produce a manual on continuous emission measurement which contains lists of manufacturers and equipment which is approved for regulatory compliance measurements.

4.1.2 Gaseous Pollutants

When sampling any gas, care is necessary to ensure that the integrity of the sample is preserved between the sampling point and the collector or analyser. In in-stack techniques this requirement is obviated because the sampling stage is avoided. For most gases it is necessary to sample the gases through a heated line constructed from an inert material. The heated line is necessary to prevent condensation of water and subsequent loss of water soluble gases from the sample. In addition, volatile organic compounds can be lost by condensation. An alternative to extraction using heated lines is a dilution probe system. This technique involves the addition of an inert, stable zero gas (such as dry nitrogen or synthetic air) at the probe to reduce the dew point of the sample gases thus eliminating the problem of condensation.

It is necessary to construct the sampling lines, and other surfaces exposed to the sampled gases, of inert materials, otherwise losses of reactive components will occur. For example sulphur dioxide and oxides of nitrogen both undergo reactions with stainless steel. Hydrogen fluoride reacts with almost everything and only PTFE (which can only be used below its temperature of decomposition) has been shown to be an adequate material for the sampling lines.

Periodic monitoring of gaseous pollutants

There are several aspects that must be taken into account while caring out the monitoring. A general idea of the compounds that are present is necessary, in order to choose a suitable analysis method in which interferences are reduced to a minimum.

Sample time: Generally sample must be collected at least until it is detectable, or at least make sure that the concentration is less than a certain value. If the time for sampling is too short it is necessary to have a more sensitive analytical method, which may be more expensive. If the sampling time is too long it might be necessary to dilute the sample to have a concentration in the optimum range of detection of the analytical method.

Sampling time / contamination level: When the mean value of the concentration of a pollutant is the desired result, the longer the sampling time the closer we are to that mean value. On the other hand, when what is wanted are the peak values the sampling time should be short enough so that peak values are not hidden by averaging in time.

Flow rate-control: A way to shorten the sampling time could be to increase the flow rate; but this flow rate must be adequate. If too high the efficiency of the collector is decreased, the filter could break, and preferential lines in the collectors could appear.

Stability of the sample: Frequently, the pollutant to be analysed can be altered, decomposed, or transformed into other compound during the sampling process itself and the period before analysis. Some pollutants may be absorbed onto the walls of the container and onto the sampling equipment; consequently it is recommended that the sample is not stored for a long period. In order to avoid these problems the sample should be kept in darkness, at a low temperature (typically less than 4 degrees) and occasionally some chemical reactants could be added for preserving the properties of the sample unit analysis. In general, the materials that are in contact with the sample are made of borosilicate glass or PTFE.

Parameters to measure while sampling: The personnel of the field should provide also some additional data of the conditions of the sampling point, particular the pressure and the temperature.

Equipment for periodic monitoring of gaseous pollutants

The methods for periodic monitoring generally include the use of sampling trains, which may be included in the same apparatus as the particle-sampling device. This equipment should consist of at least three devices:

- A flow measurement device to calculate the volume of flow to sample. It should be exact and properly calibrated;
- A pump to extract the sample, it is advisable that the pump is of constant flow type since the volume of air extracted can then be more easily calculated;
- A sample collector, whose mission is to collect the gaseous compounds to be determined, it is desirable that this collector has both high efficiency and known efficiency for the gas that is being sampled. There are several methods in use:

    - *Absorption by solutions*: usually called impingers. The most widely used. The gas is bubbled through a suitable solution and is trapped in the solution by direct solutilization or by chemical reaction.
    - *Adsorption on fine solids*: some gases can be adsorbed onto finely divided solids

- *Cooling techniques*: they consists of containers that are kept at temperatures increasingly lower, so that in each container there is the condensation of those gases whose boiling point is higher than the temperature in that container.
- *Detector tubes*: the simplest manual method for monitoring gaseous pollutants. Detector tubes consist of a glass tube packed with a chemical reagent which changes colour on contact with the specified gas. The length of stained reagent is proportional to the concentration of the gas in a measured volume of sample. These methods are semi-quantitative in that the samples have to be taken in exact accordance with the manufacturer's instructions, they suffer from interference problems, the confidence limits are wide and they were developed for workplace atmosphere monitoring rather than source monitoring. Nevertheless under certain circumstances, where it has been shown that there is reasonable agreement between the detector tube and a standard method, tubes may be useful for an approximate indication of pollutant emissions.
- Other simplified methods: such as sample bags or personal sampler pumps can be used if appropriate.

Continuous monitoring of gaseous pollutants

Extractive systems are those in which the gas is extracted from the stack continuously along a sampling line, transported and conditioned before entering the analyser unit. The sampling point should be selected so that it is representative of the gas stream.

Great care should be taken in the transportation and conditioning of the gases. The sampling lines must be made of material that ensures that no reaction occurs with the compounds of the gas, the compound to be measured is not absorbed/adsorbed onto the line, and materials from the sampling line is not added to the gas.

Condensation of water in the sampling lines must be avoided since there could be losses of gases that are dissolved in the water; in addition condensation of other gases should be avoided. There are two usual procedures to avoid this, the first is by heating the lines to maintain the gases above their dew point, and the second by diluting the gas with an inert gas (for example with nitrogen).

Extractive systems should be calibrated as complete systems to avoid problems caused by absorption/adsorption within the system prior to calibration.

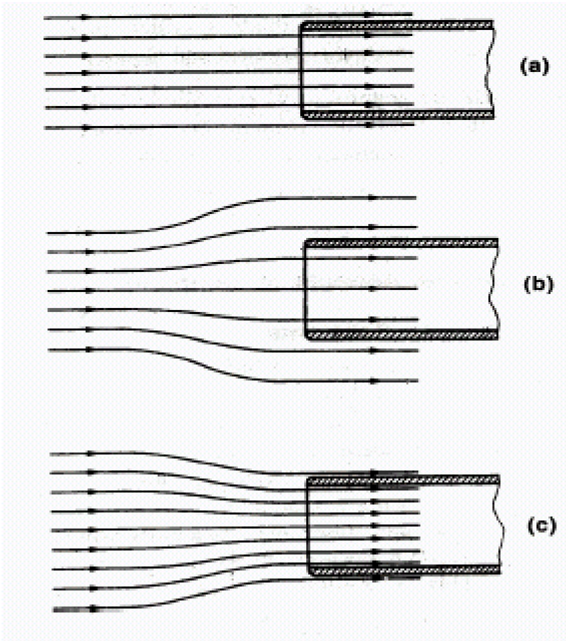
In-situ systems are those in which the measuring cell is the duct itself; they are based on a beam of a certain wavelength that crosses the duct and it is attenuated proportionally to the concentration of the compound. As there is no extraction of the sample the problems related to its handling are not discussed further. The window through which the beam passes must not absorb radiation at the selected wavelength and should be maintained perfectly clean.

4.1.3 Particulate Pollutants

Periodic monitoring of particulate pollutants

In order to ensure representativeness of the sample, providing the wide range of particle size, it is necessary to sample isokinetically; this is that the velocity at which the sample enters the sampling nozzle is the same as the gas velocity in the duct. This is necessary because the particles of small size (say less than 5μm) follow the flow lines, as in picture a, and if the sample is not taken isokinetically there will be increase or a decrease of the particles sampled

If the sample velocity is less than the gas velocity in the duct, as in picture b, there will be a reduction of small particles captured and, consequently, the percentage of large particles (larger than about 5µm) in the sample will be higher. If the sample velocity is higher than the gas velocity in the duct, as in picture c, there will be an increase in particles captured and the percentage of small particles in the sample will be higher.



Picture 1 (Illustration of the Principle of Isokinetic Sampling)

Because of the importance of isokinetic sampling in particulate sampling one of the most important facets is the measurement of flue gas flow profiles within the duct at the point of sampling. The relationship between the highest, lowest and mean pitot-static readings are used to decide whether a particular sampling point is suitable for making particulate measurements.

It should be emphasised that an isokinetic sampling does not cause a sharp cut off at a particular particle size. At any particular sampling rate, some particle sizes will be sampled totally, others will not be sampled at all. Particle sizes in between will be partially affected and the magnitude of the effect will be dependent on the degree of divergence from the isokinetic condition. The choice of 5µm to illustrate what is a small particle is fairly arbitrary and is used simply as a guideline.

Equipment for periodic monitoring of particulate pollutants

The equipment for sampling particulates include a sharp sampling nozzle, carefully designed, a sampling flow rate measurer, and a particle separator, which can be located inside or outside the duct. If it is outside the duct, provision to avoid condensation of compounds should be made by heating the probe and the particle separator. The separator can be a cyclone, a filter, or both. An adequate filter should comply with the following:

- The material of the filter should be selected so that it does not react with the material of the particles and dissolve it.
- The filter should be temperature resistant, according to the level of temperature of the particles.
- Where the composition of the sample is required care must be taken so that the filter does not add or change any compound of the sample
- The capture efficiency of the particle size that we want to sample should be known.

The sampling point for periodic monitoring of particulate pollutants

The sampling point should be located, if possible, at least four pipe diameters downstream and two upstream of any obstruction or change in flow direction. For rectangular pipes, the hydraulic diameter is used instead of the diameter. In any case a minimum distance of not less than one pipe diameter and preferably upstream from any disturbance should be used. If particles could be large enough to have an appreciable settling velocity the location of sampling point should be in a vertical duct.

A preliminary test should be done to check the suitability of the sampling plane. Velocity and temperature are measured in a number of points equally distributed across the sampling plane; if the temperature differs more than 10% from the adjacent points the location is not suitable and another location where the flow is more homogeneous should be sought.

Once the sampling plane has been chosen the number of sampling points and the distribution of them across the sampling plane can be selected from the following table. It's recommended that the number of points is not less than 4. See table 2.

Table 2

Recommended number of sampling points	
duct diameter (in meter)	**number**
0 - 0,8	4
0,8 - 1,5	8
1,5 - 2,2	12
2,2 - 3,1	18
3,1 - 4,2	24
4,2 - 5,5	32
5,5 - 7,0	40
>7,0	>40

The duration of the sampling depends on a number of factors, including the concentration of particles and the accuracy of the weighting. Under ideal conditions in the duct, the inaccuracy is about 10% of the particle concentration.

Continuous monitoring of particulate pollutants

There exist several types of continuous particle monitors; these are in-situ equipment, where the duct is, in fact, the measuring cell. Since they make the measurement through the whole stack cross section, the location of these instruments is not critical; but it should be noted that these methods are calibrated against standard manual methods which should be placed as close to the in-stack monitor as possible and complying the basic provisions stated above.

Equipment for continuous monitoring of particulate pollutants

Continuous particle monitors should be of a high standard and approved by the competent authority. They include the use of:

- Transmissometers, which are based on the transmittance of light through the absorbing gas stream. They are the most frequent used. Range from 10 mg/m^3 to 2000 $mg/m^{3.}$
- Beta attenuation monitors, which are based on the attenuation of a beam of Beta rays. Range from 2 mg/m^3 to 2000 $mg/m^{3.}$
- Light Scattering instruments, which detect under 15-degrees-scattered light. For low concentration up to 1 $mg/m^{3.}$ No standard manual methods are available for calibrations
- Tribo-electric systems, which measure the current created when a particle collides with a rod, which depends on the particle size distribution and composition. For concentrations less than 1mg/m^3. This system needs careful calibration.

However all these methods are only as accurate as the calibration undertaken by way of an extractive system, which is about 10%.

4.2 Water

There are two basic types of samples: grab and composite samples. Both types can be used in open en closed wastewater systems.

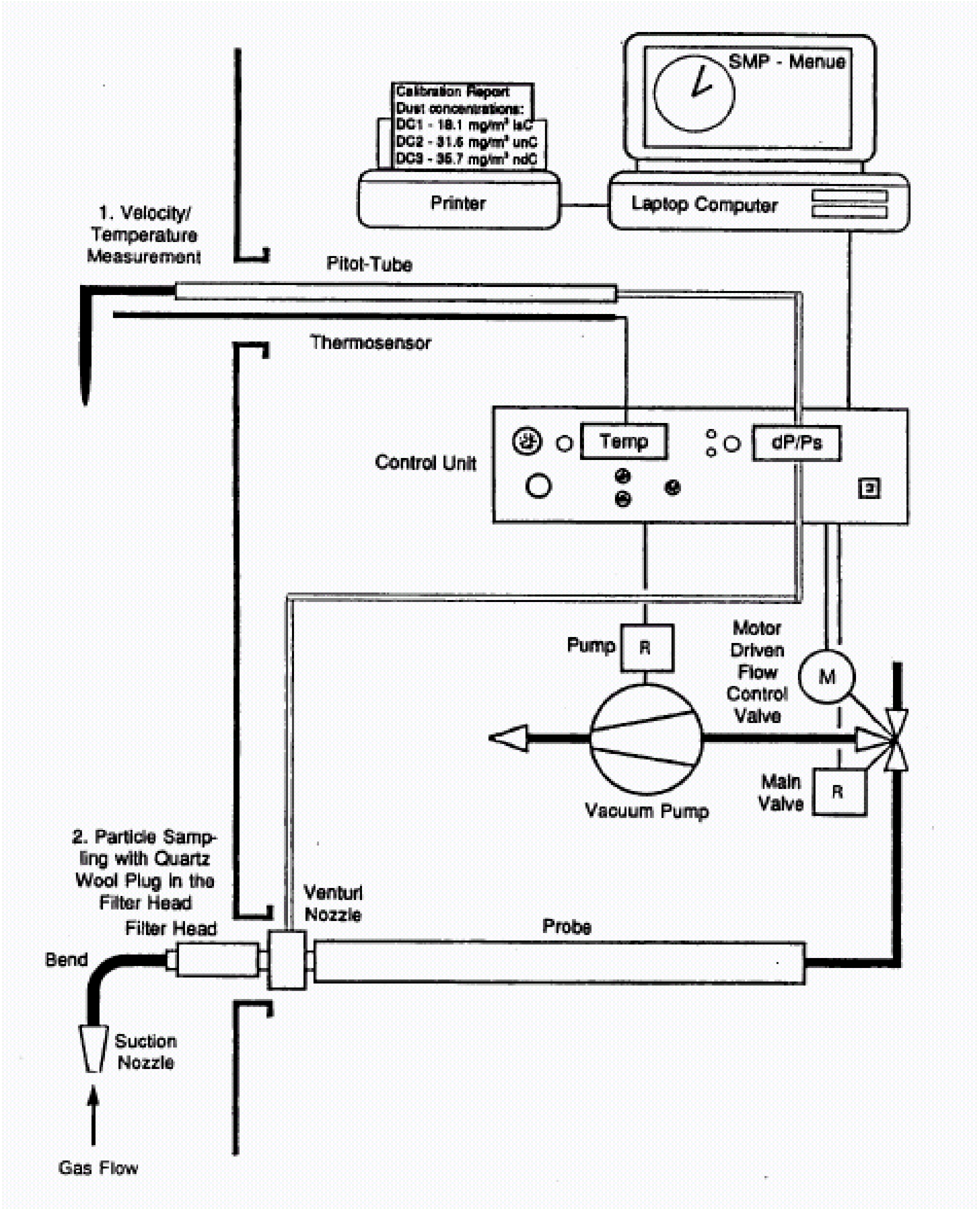
4.2.1 Grab samples

Grab samples are individual samples collected over a period of time not exceeding 15 minutes and represent waste stream conditions only at the time the sample is collected. Grab samples are usually taken manually but can be collected using an automatic sampler. Grab samplers may be appropriate for batch discharges, constant waste conditions, to screen the discharge to see if particular pollutants are present, or if extreme conditions such as high pH are characteristic of the discharge.

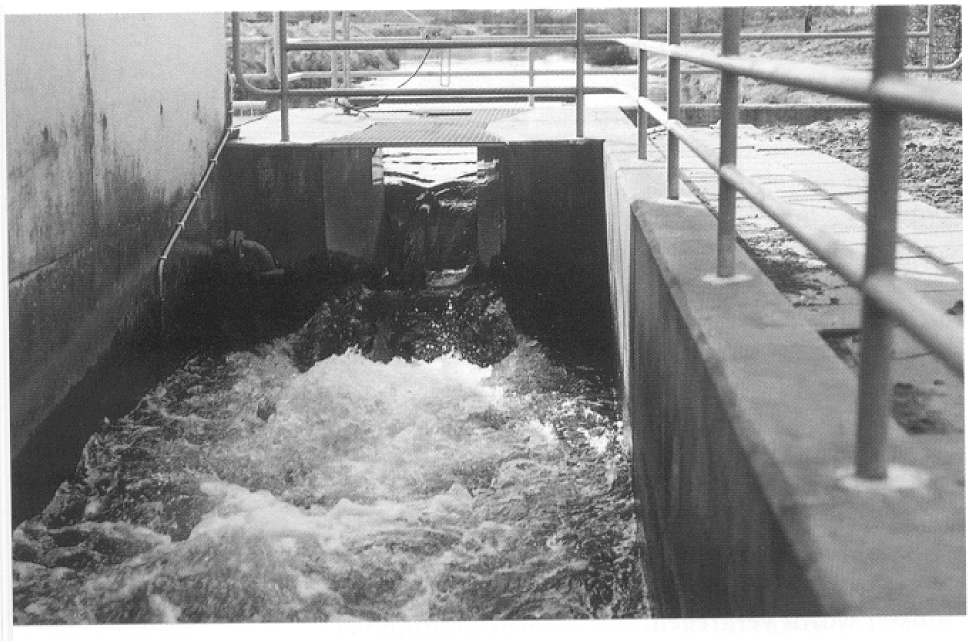
Further, grab samples should be collected for pollutants that tend to change or decompose during the composition period such as pH, cyanide, total phenols, and volatile organics. Also, grab samples should be collected for oil and grease samples since oil and grease tends to adhere to sampling equipment.

Location

Grab samples should be taken in the turbulent and well mixed area of the flow, where the chance of solids settling is minimal. This. with exception of the samples that have to be analysed volatile compounds and dissolved gasses. When sampling, the surface should not be skimmed nor should the channel bottom be dragged. Samples should not be collected from stagnant areas containing immiscible liquids or suspended samples.



Picture 2 (automatic Isokinetic Sampler)



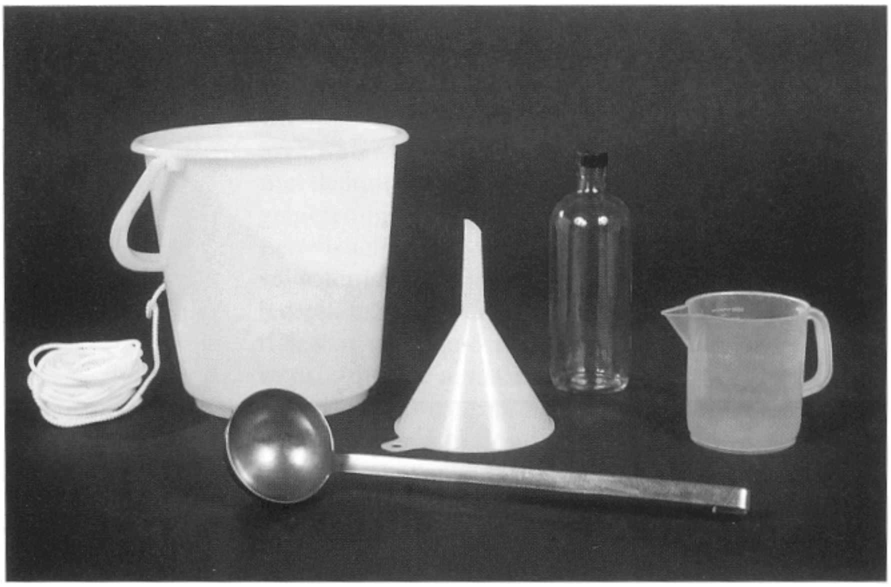
Picture 3 (open water system)

Method

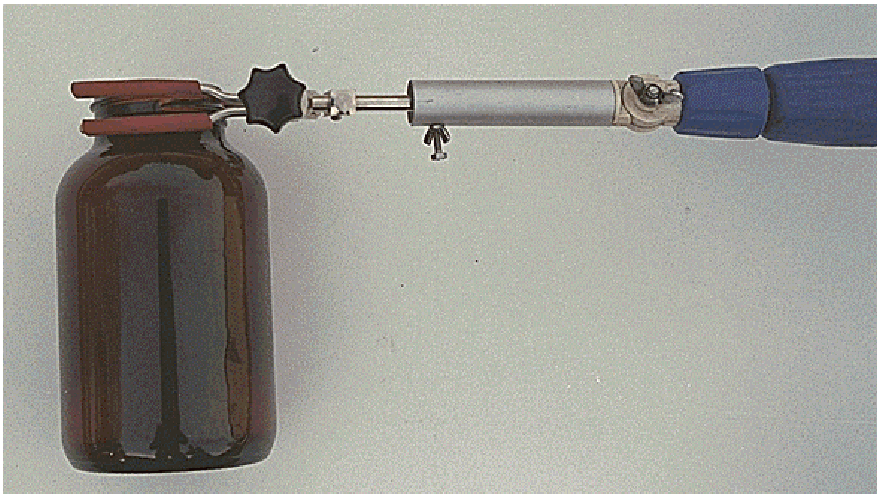
Wastewater streams can be distinguished in open and closed systems

Samples from an open system (for example a channel) can be collected by:

- sample bowl: with a simple sampling bowl it's possible to take an amount of water from the stream, A funnel can be used to pour the water in a sample bottle;
- bucket: if the sample should be analysed on more parameters, and there for more bottles should be filled, it is to be recommended to use a bucket to take an amount of water from the stream. With the sample bowl water can be taken from the bucket using again the funnel to fill the bottles;
- sample bottle with stick: the bottle (which can be connected to a stick) should be hold down in the waste water stream to fill it up.



Picture 4 (simple sampling tools)



Picture 5 (bottle connected to a stick)

Samples from a closed system (for example a pipe) can only be taken if facilities for sampling are installed. A sampling facility could be:

- Automatic sampling device: which has the possibility to take manually a grab sample on any chosen time;
- Connection with a tap or valve: before taking the sample this part of the system should be flushed.

The tools should be made of inert material which will not influence the later to perform analysis. Actions like shaking and pouring should be minimized to the most necessary. During the sampling contamination of the sample should be avoided.

4.2.2 Composite samples

Composite samples are collected over time and reflect the average characteristics of wastewater during sampling period. Composite samples can be taken either by continuous sampling (automatic samplers) or by combining individual grab samples (manually).Composite samples are either flow proportional or time composite:

- In flow proportional sampling, the volume of sample collected is proportional to waste flow at the time of sampling. Flow proportional samples can be obtained by collecting various sample volumes at equal time intervals in proportion to flow or by collecting a constant sample volume per unit of wastewater flow. In case of automatic sampling the sampler will be controlled a flow indicator;
- Time composite samples consist of constant volume sample aliquots collected in one container at equal time intervals. For example, 500 millilitres of sample collected every 15 minutes over a 24-hours period. In case of automatic sampling the sampler will be controlled by a clock.

Composite samples may be needed to determine the average characteristics of waste streams, particularly if the waste stream has a highly variable pollutant concentration or flow rate. Composite samples should be collected during the entire period the facility is operating and discharging. For example, if the facility has processed that discharge 16 hours a day, samples should be collected during the entire 16-hours period.

Methods for automatic sampling

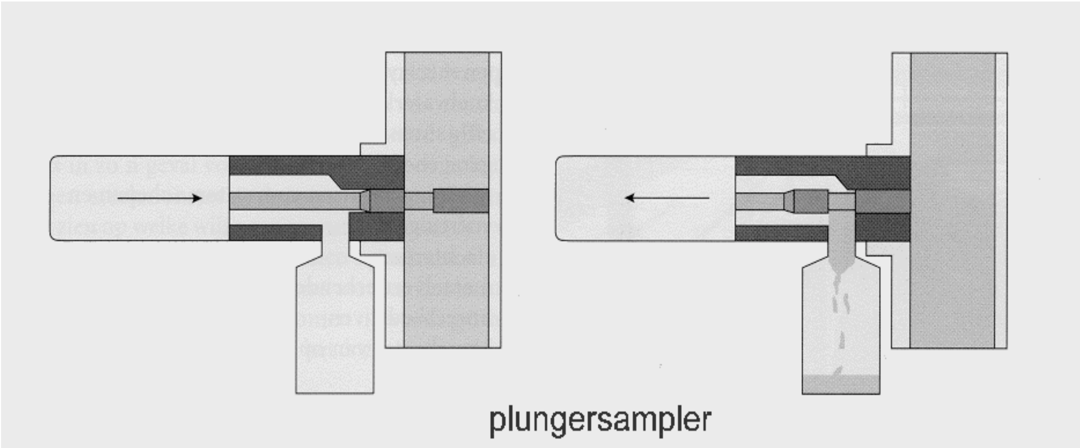
In this part a short survey of equipment will be given that can be used for automatic sampling of waste water streams in open and closed systems.

Open systems:

- Vacuum sampler: this is the most common method for open systems. With this equipment samples are collected with a vacuum pump through an intake nozzle (or suction pipe) to a sample container;
- Double check valve with pump: with this equipment part of the waste water stream is continuously pumped around through a by-pass. Samples are taken through the double check valve at an estimated time to a sample container (this system is also suitable for taking samples from pressure pipes);
- Hose pump sampler: with this equipment samples are being sucked by a hose pump and directed to the sample container.

Closed systems:

- Double check valve: with this equipment samples with a fixed volume are collected from a by-pass to a sample container;
- Plunger sampler: with this equipment samples with a fixed volume are collected with the use of a plunger from the main pipe or a by-pass to sample container.



Picture 6 (plunger sampler for closed water systems)

Location

Composite samples should be taken on a place were there is turbulence. With automatic sampling equipment in open systems the suction point should be located as close as possible downstream from an obstruction. With automatic sampling in a closed system the sampling point should not be located in a bend or a constriction. If wastewater is discharge by a pump the sampling location should be on the pressure side of the pump.

If using a vacuum sampler in combination with a closed system, the suction point of this equipment should be located on the position where the closed system is discharging into an open discharge system. With vacuum sampling the suction point should always be subsurface.

Distance between sampling location and sampling equipment

With vacuum sampling the suction hose or pipe should always be under a slope and as short as possible. The suction hose should be protected against freezing and direct sunlight, and should not have any twists or unnecessary bends.

Diameter pipes

To prevent any blockage in the sampling equipment the diameter of all streaming parts (from suction point to the outlet in the sample container) should be at least 13 mm.
Further, out of research a significant difference is shown in the chemical oxygen demand (COD) in samples taken with equipment using 9,5 mm pipes and equipment using 13 mm pipes

Suction speed (vacuum and hose samplers)

The average suction speed should be at least 0,3 m/s.

However, out of research there is no significant difference in COD results with speed of 0,3 m/s or 0,6 m/s.

Sampling interval and volume of sample

The sampling interval should be estimated in a way that a twenty-four hours sample should at least consist out of 100 small samples.

The sample volume needed for analysis depends on the type and number of analyses to be preformed.

> **HINT**
> **The sampler should contact the person of the laboratory that will perform the analysis to determine sample volumes needed for a particular sampling event.**

Sample containers

Sample containers should be made of chemically resistant materials that will not affect the nature or concentration of pollutants being measured. Containers must be large enough to hold the required sample volume.

- <u>Glass containers</u> should be used for oil and grease, phenol, and organics samples.
- <u>Amber glass</u> sample containers should be used for pollutants such as iron cyanide that oxidize when exposed to sunlight.
- <u>Containers with Teflon lined lids</u> should be used when collecting volatile organics

In general, plastic containers are easier to handle and less likely to break, so this may be the best type of container if glass is not needed. Sample containers should be properly cleaned prior to use.

> **HINT**
> **The laboratory that will perform the sample analysis should be contacted for specific cleaning instructions. Some laboratories may provide pre-cleaned sample containers.**

At the discharge of the sample to the sample container it should be prevented to let air into the container. If the composite sample is to be analysed on volatile organics the discharge of the sample into the sample container should be in a way that loss of volatile components is prevented.

4.2.3 Preservation and holding time of the sample

Many pollutants are unstable and may alter in composition prior to analysis. There for to ensure that samples remain representative, they should be analysed as soon as possible after collection. If immediate analysis is not possible, samples should preserved to minimize the changes in pollutant concentration between collection and analysis. There are three basic types of preservation:

- cooling
- pH adjustment

- chemical fixation

The sample (sample container) should be stored at a temperature between 0ºC en 4ºC. This temperature should be reached as soon as possible after collecting the sample. Freezing of the sample should be prevented.

Out of research is significant difference is shown in COD results from samples stored at a temperature lower than 4 ºC and samples stored at a temperature higher than 15 ºC. Cooling suppresses biological activity and volatilization of gases and organic substances. Table 3 shows the holding time for a few parameters.

Even with proper preservation, samples should be analysed within certain recommended holding times. These holding times are the maximum times allowed between the time the sample is collected and when it is analysed. If composite samples are collected, the holding time limitations begin when the last aliquote is added to the sample. Performing sample analysis within the allowable holding times helps ensure that the analytical results are valid and representative of the wastewater. Certain pollutant parameters such as pH have no standard method of preservation and should be analysed immediately.

Table 3

Parameter	**Temperature during storage**	**Chemical fixation**	**Max. storage life**
Biochemical Oxygen Demand if suspected BOD < 50 mg/l	between $0^{o}C$ en $4^{o}C$	-	24 hours
Biochemical Oxygen Demand if suspected BOD ≥ 50 mg/l	between $0^{o}C$ en $4^{o}C$	-	24 hours
	≤ $-18^{o}C$	-	72 hours
Chemical Oxygen Demand (COD)	between $0^{o}C$ en $4^{o}C$	-	48 hours
		adjust pH to < 2 with concentrated H_2SO_4 (18M)	5 days
	≤ $-18^{o}C$	-	5 days
Kjeldahlnitrogen	between $0^{o}C$ en $4^{o}C$	-	48 hours
		adjust pH to < 2 with concentrated H_2SO_4 (18M)	5 days
	≤ $-18^{o}C$	-	5 days

Parameter	Temperature during storage	Chemical fixation	Max. storage life
Heavy metals (excluded Hg)	between 0^oC en 4^oC	adjust pH to < 2 with HNO_3 (15M)	1 month
Hg	between 0^oC en 4^oC	adjust pH to < 2 with HNO_3 (15M) and sublement min. 0,5 g $K_2CR_2O_7$ / ltr	1 month

4.3 (Hazardous) waste

Sampling is generally conducted to verify the identity of a waste or to identify potential releases of hazardous wastes or constituents to the environment.

In most cases, sampling will not be performed during routine inspections. However, the inspector should be aware of, and identify, potential sampling requirements that may need to be fulfilled in future inspections, particularly in cases where the inspector has identified potentially non-complying conditions or criminal activity during the course of the inspection.

Reasons for sampling

There are many possible conditions or activities which may lead the inspector to determine that sampling will probably be necessary.

Examples of some of these conditions include situations in which:

- The owner/operator is handling a potentially hazardous waste as a non-hazardous waste. (Sampling may be required to verify that the waste is hazardous or non-hazardous.)
- In-plant waste handling practices indicate that mislabeling/misidentification of waste is likely to occur, or that wastes may vary significantly in characteristic over time and be mismanaged as a result. (Sampling may be required to demonstrate that the facility is mislabeling or misidentifying wastes.)
- There is visible or other observable evidence of possible releases of hazardous wastes from waste management units, satellite storage areas, waste generating areas, etc.(Sampling media and wastes may be required to demonstrate that a release has occurred or is occurring.)
- Wastes may be being managed improperly, i.e., in an inappropriate treatment or disposal unit. (Sampling may be required to verify that the correct wastes are being managed in the facilities various waste management units.)

4.3.1 Procedures for sampling liquid waste

Liquid waste stored in small unit

A small unit is defined as a package-unit smaller than 200 dm^3 (200 litre), like barrels and tins etc.

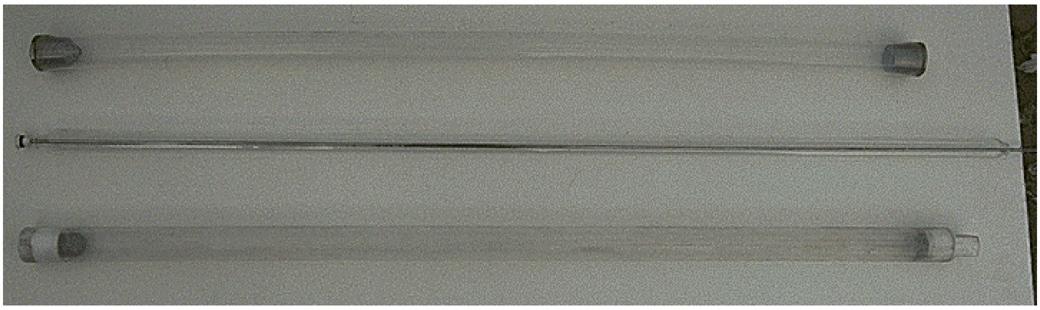
Defining the sample area

If more package-units are present, every single package-unit has to be considered as a sample area. Depending the aim of the investigation all package-units or a few package-units will be picked out for sampling. The samples from the different package-units should not be mixed.

Sampling method

Make sure the cap or the lid of package-unit is on the top side and let the content of the unit come to rest. Provide a connecting bond between the package unit (if this is made out of metal) and a ground probe to prevent any discharging or accumulation of static electricity while sampling. Wear footwear with adequate conductivity. If the use of tools are necessary to release the cap make sure they are made of material which will not cause sparks. Copper is often used in this. Release the cap from the packaging-unit very slowly so pressure or underpressure can disappear. Remove the cap.

- Sampling siphons: Lower a sampling siphons into the package-unit and take a sample of the whole content (0,35 tot 1 dm^3). The complete content should be placed in the transport sample bottle. This method is often used for cylindrical barrels and other small package-units (see picture 7).

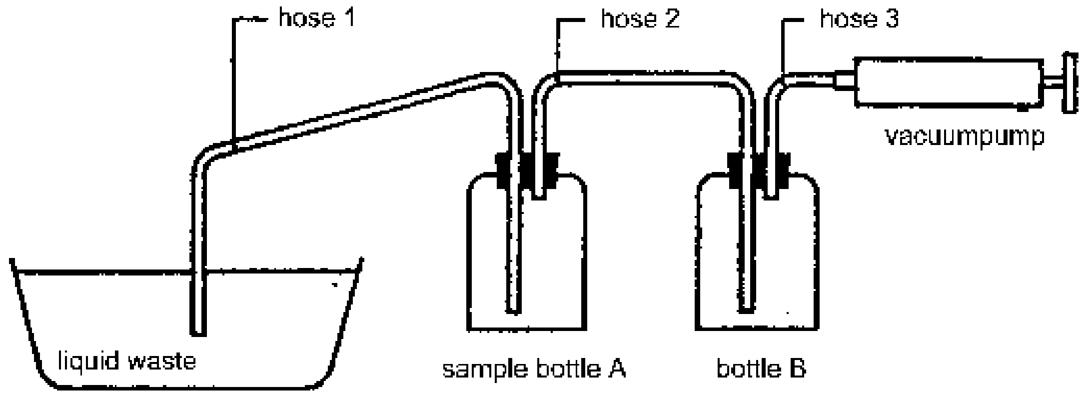


Picture 7 (sampling siphons)

- Pump or sample bottle: Take with a pump or with a sample bottle, samples from the middle of the top layer, the middle layer and the bottom layer of the package-unit. Work from the top to the bottom to prevent any turbulence of sediment in the lower layer. The volume of the samples should be 0,35 to 1 dm^3. The samples should be added to make the composite samples. This composite sample can be divided in to the transport sample bottles. Make an estimation of the volume of the different layers. In a later stage it could be important to calculate the average concentration from volume and the concentration of the different layers.

Liquid waste stored in cylindrical tanks

A cylindrical tank is defined as a lying, round package unit with a volume around 20 m^3, like a tank (lorry), tank wagon or stationary tanks.



Picture 8 (pump with bottle)

Defining the sample area

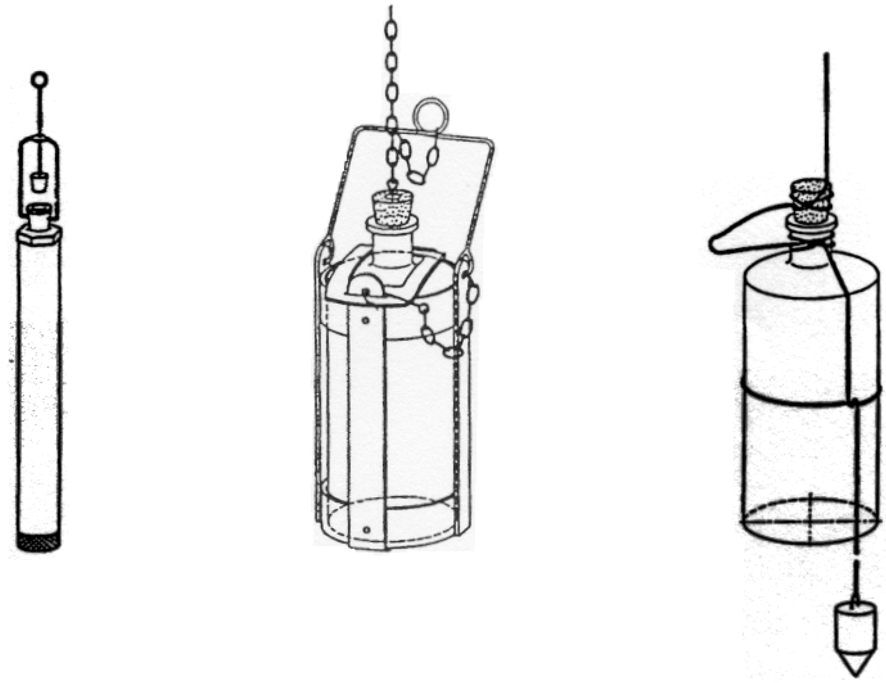
Any compartment within a tank should be considered as one sample area. If the tank is non-compartmentalised, the tank itself is to be considered as one big compartment. All compartments should be sampled individually. The samples from the different compartments should not be mixed.

Sampling method

Open the lid of the tank very slowly and carefully so any pressure or underpressure can escape. Caution: because of pressure in the tank the lid can lift itself with great force. Make sure to wear protective clothing and respiratory protection.

- Sample jug or sample bottle: depending on the level height of the liquid a top, a middle and a bottom sample, or only a middle and a bottom sample or just a bottom sample should be taken. See table 4. Work from the top to the bottom to prevent any turbulence of sediment in the lower layer The volume of the samples should be 0,35 to 1 dm^3. The samples should be added, in the volume percentage given in table 4, to make the composite samples. This composite sample can be divided in to the transport sample bottles. Make an estimation of the volume of the different layers. In a later stage it could be important to calculate the average concentration from volume and the concentration of the different layers. See picture 9.

- During unloading: It's also possible to take samples during unloading of the liquid. Be aware of static electricity; a truck or wagon should be connected to a ground probe. Take samples of 1 dm^3 after 20%, 50% en 80% of unloading. Before taking the sample the tap should be well flushed. See method sample jug or sample bottle for preparing the composite sample.



Picture 9 (different types of sample jugs / bottles)

Table 4

height of **liquid level in %**	**sampling level *in* % from bottom to the top**			***proportion of the composition sample in %***		
	top	**middle**	**bottom**	**top**	**middle**	**bottom**
100	*80*	*50*	*20*	*30*	*40*	*30*
90	*75*	*50*	*20*	*30*	*40*	*30*
80	*70*	*50*	*20*	*20*	*50*	*30*
70		*50*	*20*		*60*	*40*
60		*50*	*20*		*50*	*50*
50		*40*	*20*		*40*	*60*
40			*20*			*100*
30			*15*			*100*
20			*10*			*100*
10			*5*			*100*

Liquid waste stored in large unit

A large unit is defined as a package-unit larger than 20 m^3, like storage tanks on land and sea ships.

Defining the sample area

Every compartment within these sea ships and storage tanks should be considered as one sample area. If the sea ship or storage tank is non-compartmentalised, the ship or tank itself is to be considered as one big compartment. All compartments should be sampled individually. The samples from the different compartments should not be mixed.

Sampling method

Open the lid of the tank very slowly and carefully so any pressure or underpressure can escape. Caution: because of pressure in the tank the lid can lift itself with great force. Make sure to wear protective clothing and respiratory protection.

- Sample jug or sample bottle: Take with the jug or bottle a top, a middle and a bottom sample. Work from the top to the bottom to prevent any turbulence of sediment in the lower layer The volume of the samples should be 0,35 to 1 dm^3. The samples should be added to make the composite samples. This composite sample can be divided in to the transport sample bottles. Make an estimation of the volume of the different layers. In a later stage it could be important to calculate the average concentration from volume and the concentration of the different layers.
- Taps on tank: If the tanks are equipped with taps at different levels these can be used for taking samples. Before taking the sample the taps should be well flushed. See method sample jug or sample bottle for preparing the composite sample etc.
- Running sample or all-level sample: taking a representative running-sample requires a lot of experience. The idea is to lower the sampler in the liquid and after reaching the bottom raising it again in a constant speed. The sampling is correct if the sampler isn't filled completely (not 100%). The complete content of the sampler should be placed in the transport sample bottle.
- During unloading: It's also possible to take samples during unloading of the liquid. Be aware of static electricity; a sea ship should be connected to a ground probe. Take samples of 1 dm^3 after 20%, 50% en 80% of unloading. Before taking the sample the tap should be well flushed. See method sample jug or sample bottle for preparing the composite sample.

4.3.2 Procedures for sampling solid waste

Solid waste stored in small unit

A small unit is defined as a package-unit smaller than 200 dm^3, like barrels, tins and bags etc.

Defining the sample area

If more package-units are present, every single package-unit has to be considered as a sample area. Depending the aim of the investigation all package-units or a few will be picked out for sampling. The samples from the different package-unit should not be mixed.

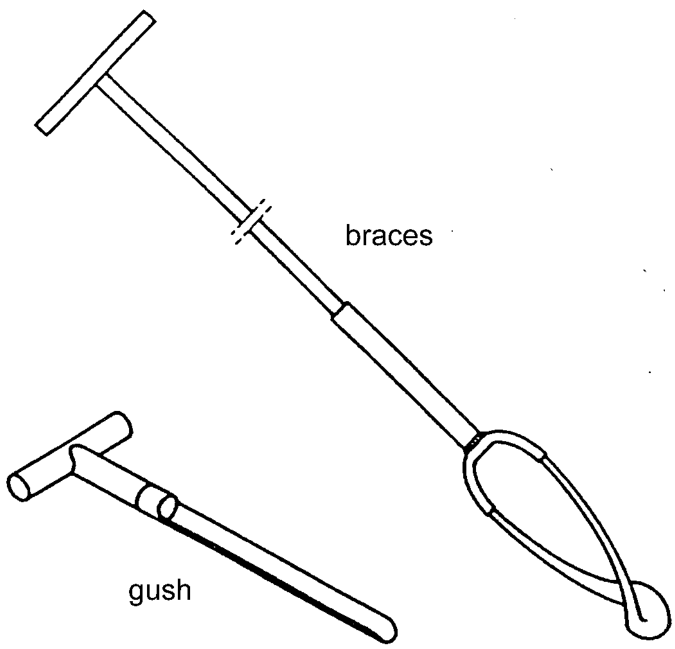
Sampling method

Make sure the cap or the lid of package-unit is on the top side or the bag is standing up right. Release the cap or lid from the package-unit carefully or make a hole in the bag.

- Gush : Depending on the waste material it's possible to use the Gush. Take a sample over the complete column (depth) of sample area. The volume of the sample and thus the size of the sampler depends on the size of structure of the waste. See table 5. The complete content should be placed in the transport sample bottle.
- Braces: If taking a sample Over the complete column (depth) with the Gush isn't possible, the Braces is a good alternative. With the Braces it's possible to take a top, middle and bottom sample from the sample area. For the size of the samples see table 5. The samples should be added to make the composite samples. This composite sample can be divided into the transport sample bottles. See picture 10.

Table 5

Max. size in mm of structure	**Min. diameter in mm of “guts”**	**Min. volume of sample and transport sample**
5	15	1
10	30	2
20	60	5
40	120	10
80	use heavy equipment	40
> 100		100



Picture 10 (Gush and Braces)

Solid waste stored in large units

A large unit is defined as a package-unit larger than 200 dm^3, like trucks, containers, ships etc.

Defining the sample area

Every compartment within these trucks, containers or ships should be considered as one sample area. If the truck, container or ship is non-compartmentalised, the truck, container or ship itself is to be considered as one big compartment (sample area). At the samplers discretion a few compartments should be picked out for sampling. The samples taken from these compartments should not be mixed. When sampling a compartment is practically not possible a sample area within this compartment is to be defined. This sample area should have at least a volume of 5 m^3 with a depth of 1 meter (if the package unit allows this).

Sampling method

Make an estimation of the volume of the compartment (sample area). Define the number of sample point according to table 5 and formula below.

SP = 5 + (S / AD)

where:
SP = min. number of samples points (to be round up)
S = number of samples according to table 3
AD = average depth of the compartment or the sample area

Divide the sample points equally over the surface of the container or sample area (use a raster if necessary). Take a samples of the waste at each sample point over the complete depth of the container or the sample area that was defined.

- Gush and Braces: depending the structure of the waste material the Gush or the Braces should be used. The complete sample can be obtained by taking sub-samples at different depths until the column (the complete depth of the container) is completely drilled through. The volume of the sample and thus the size of the sampler depends on the size of structure of the waste. See table 5.

- During unloading: It's also possible to take samples during unloading of the waste. For example when unloading by conveyer belt. The min. amount of samples to be taken depends on the total volume of the waste. Divide the period of unloading in time intervals using the formula below and table 6.

TI = U / S

where:
TI = number of time intervals in hours to take a sample
U = period of unloading in hours
S = number of samples according to table 3

Add all the sampled materials (described in the methods above) to one composition sample. Divide the composition sample into one or more transport samples.

HINT
When sampling during unloading, make sure to take enough sample material since the sampling method cannot be repeated.

Table 6

total volume of compartment or sample area (in m^3)	**min. number of samples**
0 to 1	1
1 to 2	2
2 to 5	4
5 to 10	6
10 to 15	8
15 to 20	10
20 to 50	20
50 to 100	30
100 to 150	40
150 to 500	50
500 to 1000	60
> 1000	$60 * \sqrt{}$ (volume) / 1000

In case the compartment could not be sampled completely, and there for a sample area had to be defined, it's recommended to take some indicative samples from the rest of the compartment. These indicative samples can be added to one composite sample. The results of this indicative sample can be used to support the results of the samples taken in the sample area.

Solid waste not stored in units

Waste not stored in units is defined as waste dumped on a pile or spread over the ground.

Defining the sample area

Every separate amount of dumped waste which can be identified as a separate unit should be defined as a sample area. Investigate if within this sample area the type of waste is the same. If more types of waste can be identified, more sample areas has to be defined.

Sampling method

The sample area that has been defined should be sampled according to the method described for "solid waste stored in large units".

4.3.3 Sediment, slurry and sludge

Sediment, slurry and sludge is defined as a mixture of water and not or hardly solved solid materials, without a liquid and solid layer that can be identified.

Defining the sample area

Sediment, slurry and sludge are to be sampled in the same way as described for liquid waste. If this is not possible the sampling method for solid waste should be used.

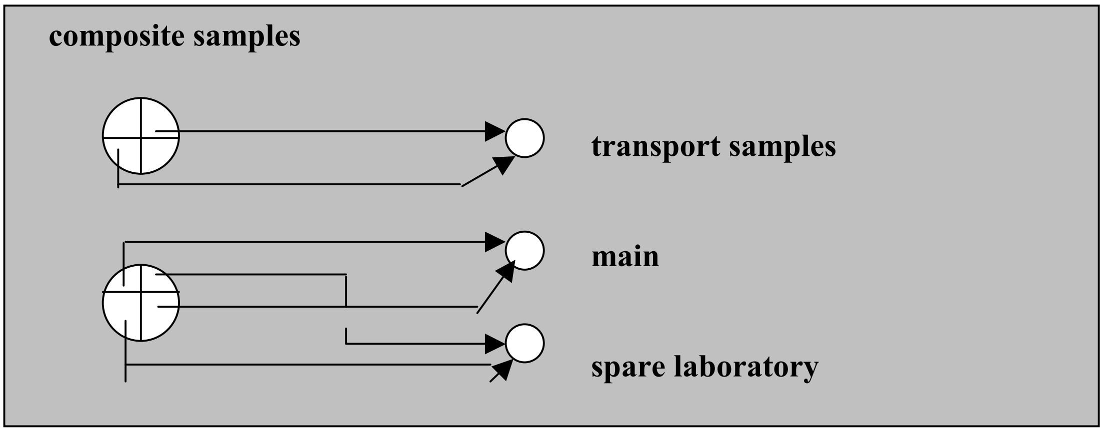
4.3.4 Transport samples

As described in previous chapters, samples should be added to make a composition sample from which transport samples can be made.

Number of transport samples

Depending the aim of the investigation it is to be recommend to take one spare sample for the laboratory analyses and one spare sample for contra expertise. This makes the total of three transport samples for every sample point.

Dividing the composition sample into transport samples in case of solid waste the next method can be followed.



Picture 11 (dividing the composition samples into transport samples)

HINT
mixing the different samples into a composite sample can be done at the sampling area itself out of practical consideration, but it is to be recommended to do this at the laboratory.

Packaging

The most common used package unit for transport sample units is the bottle or jar made out of dark glass with screw cap. The dark glass will protect the material against the influence of light. If the material consists hydrogen fluoride or very strong acids glass can be affected and synthetic bottles have to be used .

- When transport sample consist of large amount of solid waste it’s possible to us plastic bags.
- When transporting volatile organic materials the bottle or jar should be filled completely and should be closed gasproof.
- When transporting material that can develop gasses like sludge from a sewage treatment plant the bottle or jar should be filled for 75 %.

HINT
Out of safety precaution it's recommended to put the bottle or jar into a plastic bag.

4.4 Soil and groundwater

This chapter describes the sampling of soil and groundwater (or soil water). For sampling soil there are similarities with sampling Solid Waste. To avoid duplication references to this chapter are made when necessary. In case of groundwater references will are made to the chapter Water.

Reasons for sampling

There can be many different reasons for sampling soil or groundwater, but the reasons for an inspector are most always connected to the knowledge or suspicion of contamination. This can be because an incident took place, a malfunction of an installation, the way a storage facility is operated or because of other clear visible indications (like color of the soil).

In case of groundwater there can be an other important reason next to determining the level and the substances of a contamination. If a contamination has been located or if there is a potential risk an operation could contamined the soil or groundwater (for example a landfill) it could be important to see if there is no leaching into the groundwater. In this case sampling becomes monitoring. Effective water monitoring will provide early warning of water pollution and should allow corrective action to be taken in good time.

4.4.1 Procedures for sampling soil

Soil dumped on a pile or spread over ground (max. 1000 m^3)

- Make an estimation of the volume of the sample area.
- Determine the amount of sample points according to table 7

Table 7

Volume (m^3)	<150	150-500	500-1000
number of samples	40	50	60

- Determine the amount of sample point by dividing the amount of samples by 5 and the average depth (in meters) of the sample area.

SP = S / (5 * AD)

where:
SP = amount of sample points
S = amount of samples according to table 1
AD = average depth of the sample area

- Divide the sample point equally over the sample area. Use if necessary a raster.
- Take on every sample point a column of soil over the whole depth of the sample area. Use a Braces (see waste). Every sample should be about 150 gram soil. Is same as volume of Braces.
- Samples from the same sample area should be added to make the composition sample.
- Make transport samples out of the composition sample (see waste).
- Determine amount of transport samples according to table 8.

Table 8

Volume (m^3)	<400	400-1000
Number of transport samples	1	2

- Label the samples package units
- Send the samples to the laboratory

Make contra expertise sample for the owner ground.

Soil in a container

- Make an estimation of the volume of the sample area
- The amount of soil within a container will always be less than 150 m^3. The minimum amount of samples to be taken is there for always 40.
- The amount of sample point for a container is always 8.
- Divide the sample point equally over the sample area. Use if necessary a raster.
- Take on every sample point a column of soil over the whole depth of the sample area. Use a Braces. (see waste). Every sample should be about 150 gram soil. Is same as volume of Braces.
- Samples from the same sample area should be added to make the composition sample.
- Make transport samples out of the composition sample (see waste)
- Label the samples package units
- Send the samples to the laboratory
- Make a contra expertise sample for owner ground.

Transport and packaging

See chapter 4.3.4, Waste.

4.4.2 Procedures for sampling groundwater

To sample groundwater first a borehole should be drilled. The selected depth and diameter of the borehole depends on the hydrogeological conditions and the physical characteristicson of soil and groundwater. Every site setting is unique.

Drilling

The choice of drilling method and equipment should therefor be made on a site-specific basis whilst considering the following.

- The depth and diameter of drilling required and likely depth of first water strike.
- The ability to penetrate the formations anticipated.
- The degree of contamination anticipated.
- The ability to obtain samples and identify different formations.
- The ability to identify groundwater inflows.
- The extent of disturbance to ground materials during drilling.
- The impact of drilling technique on groundwater quality.
- Ability to install sampling or monitoring equipment.

For drilling boreholes for monitoring purposes a competent professional should undertake specification of drilling position and depths. The following knowledge should be taken into account:

- the depth and lateral extent of the groundwater system to be monitored. If this lies below perched or other groundwater systems, steps need to be taken to ensure a seal is maintained between systems both during drilling and following installation of the monitoring point;
- the likely depth and seasonal variation in water table in unconfined groundwater systems. Normally drilling should continue below the lowest level of seasonal water table variation, to a depth sufficient to allow adequate purging and sampling;
- the most likely depth of contamination arising. This will vary depending on factors such as where exactly contamination enters the groundwater system, how far down-gradient of the site the monitoring point is located and the hydraulic characteristics of the groundwater system. For example in a flood plain there will be a component of groundwater movement vertically upwards which will be the result of discharges to surface water so that monitoring points can probably be designed to relatively shallow depths. Conversely, a location on a hill top may require deeper monitoring points due to the tendency for groundwater to move vertically downwards;
- the vertical distribution of contamination. This may require the provision of multi-level, nested or clustered boreholes.

Drillingmethods

The most commonly used drilling methods are:

- conventional rotary drilling;
- cable percussion (shell and auger);
- augers (hollow stem, continuous flight or single flight).

A summary of advantages and disadvantages of conventional drilling methods is presented in table 9.

Addition of water during drilling

It is sometimes necessary and unavoidable for water to be added either as a circulating fluid for rotary drilling or to loosen up unconsolidated materials in percussion drilling. Where water is added it must come from a source of known quality. Where critical, a sample and analysis of the added water should be provided as a reference against water samples recovered from the borehole during drilling or from monitoring installations.

Sample containers

For collecting the samples see chapter 4.2.2 (Water)

Table 9

Drilling Method	Advantages	Disadvantages
Cable Tool	• Inexpensive • easily cleaned • easy to identify lithological changes • and water strikes • bulk and undisturbed samples possible • minimum use of drilling fluids • use of temporary casing allows accurate installation of lining and • annular fill	• slow • cannot penetrate hard rock • can smear sides of borehole
Rotary Auger	• rapid; • inexpensive; • easily cleaned; • hollow stem augers allow continuous; • sampling in unconsolidated materials; • lining can be installed directly into hollow stem augers; • no drilling fluids needed.	• cannot penetrate hard rock; • hollow stem augers cannot penetrate • where cobbles or boulders are present; • sampling depth and water strikes • difficult to identify using solid stem • augers; • solid stem augers cannot be used in • loose ground (hole collapses); • unable to install annular fill and seals in collapsing ground.
Other Rotary Methods	• an be inexpensive; • fast in consolidated materials; • can be adapted to drill all formation types; • continuous samples can be cored in consolidated rock and clay.	• can be expensive; • fluids need to be added (e.g. air, foam, water, mud); • possible introduction of contaminants (including oil from air compressor) with circulating fluid; • recovery of samples can be slow when drilling at great depths; • can smear sides of borehole; • synchronous casing methods in unconsolidated formations only allow installation of narrow diameter lining.

Sampling equipment

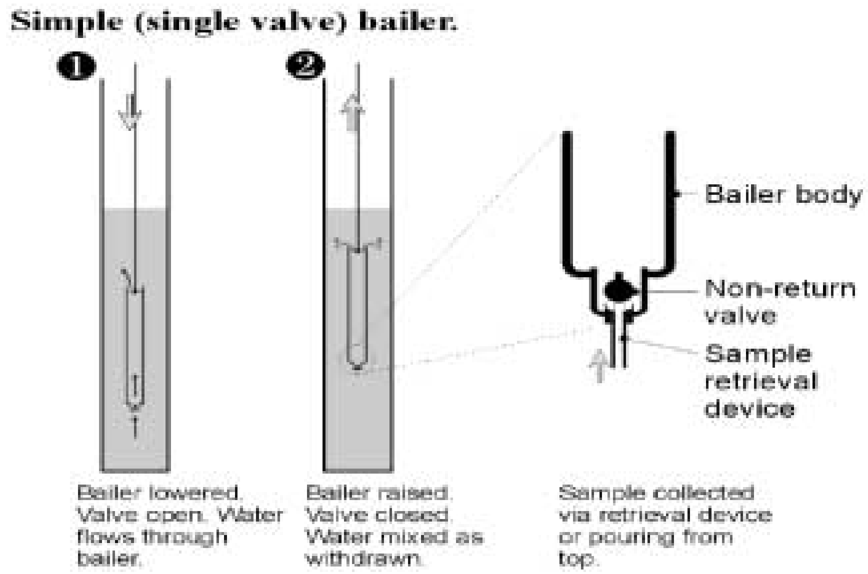
There are many sampling methods and types of sampling device capable of obtaining groundwater samples from boreholes all of which have their advantages and disadvantages. The most common methods and devices currently in use are:

Bailers and depth samplers: they can be obtained for use in sampling and monitoring points over a wide range of diameters, and can be constructed from a wide range of plastics (PVC, polypropylene, PTFE (Teflon) or stainless steel. These devices provide a simple means of obtaining a “grab” sample either from the top of the water column (bailers) or from a specific depth in the water column (depth samplers). Both methods involve manually (or mechanically) lowering the sampling device into the borehole on a rope or wire and then withdrawing the device full of water to ground surface.

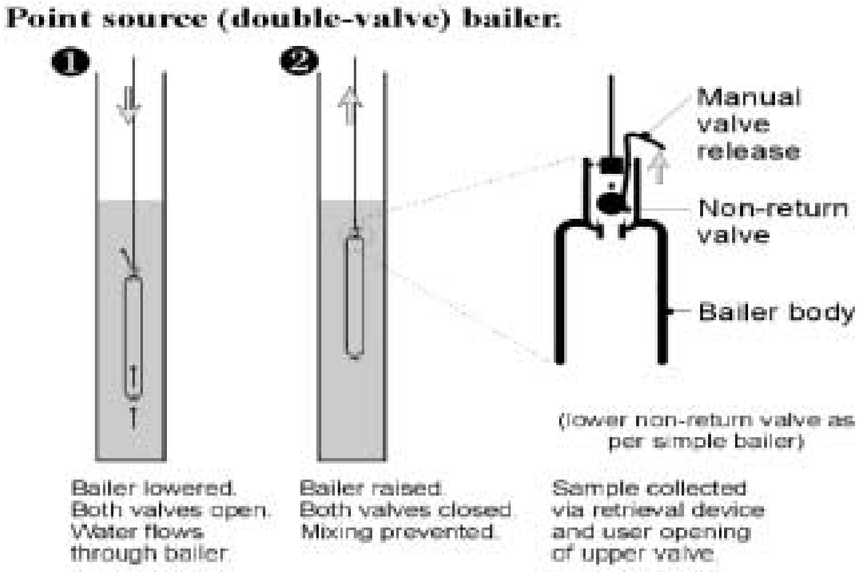
The bailer may be of varying levels of sophistication.

- Bucket type (open top, sealed base).
- Bottom check valve only (see picture 12). A ball and seat arrangement remains open during the sampler’s descent, but closes under the weight of liquid in the sampler during removal.
- Double check valve bailer (see picture 13). Both the upper and lower check valves close once the bailer stops descending through the water column, to collect a point-specific sample. Double check-valve bailers allow depth sampling within the borehole.
-

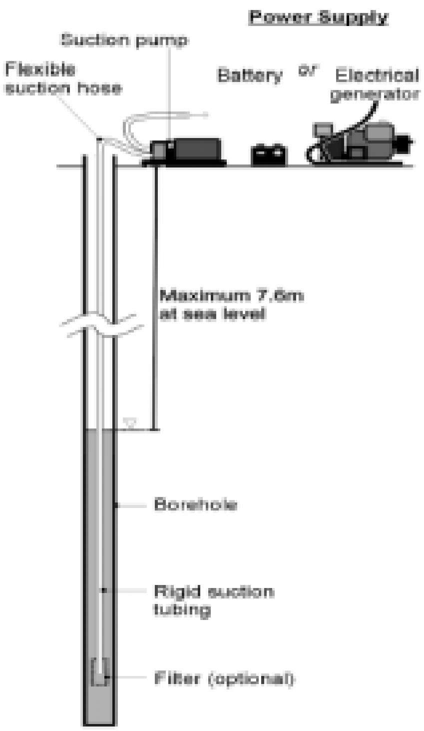
Picture 12



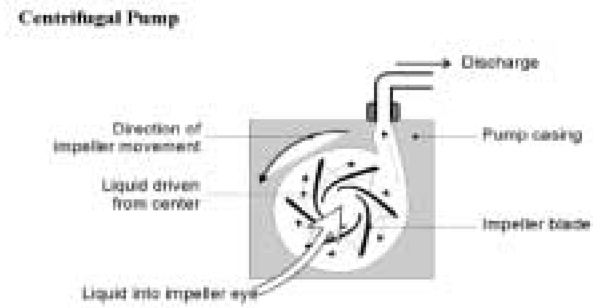
Picture 13



<u>Suctionpumps</u>: these are surface mounted pumps (see picture 14), either electrically, diesel or petrol powered. Due to the practical limit of suction lift of approximately 7.6 m (at sea level), this method of sampling is only practical for shallow water levels. The most commonly employed suction-lift pumps are the surface centrifugal pump (see picture 15) and the peristaltic pump (see picture 16). The centrifugal pumps are capable of very high delivery rates. The peristaltic pump are low-volume vacuum pumps but particularly useful where samples have to be collected from narrow access tubes. Both however are, because of the negative pressure caused by the vacuum, not suited for sampling volatile compounds.

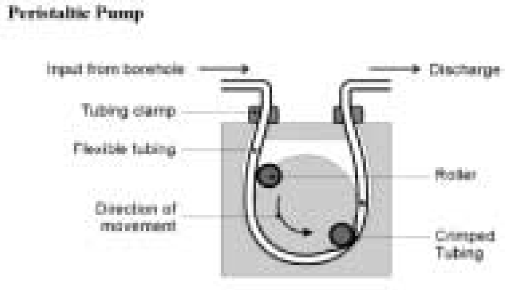


Picture 14



Picture 15

Picture 16



<u>Inertial pumps</u>: these pumps are comparatively cheap and suitable for a wide range of applications. The operating principle of the pump is based on the inertia of a column of water contained within a riser tubing. The pump consists of a foot valve connected by a rigid or semi-rigid rising main that runs to ground level. The whole system is alternately lifted and lowered at a rate sufficient to drive water continuously upwards to discharge at surface. The pump can be operated manually at shallow depths, though is better used with a powered mechanical drive system to achieve greater lifts (e.g. to 60 m in a 50 mm diameter borehole).

<u>Electric submersible pumps:</u> These pumps operate by driving water upwards using helical rotors or gears. Both types of pumps have an electric motor below the pumping mechanism, which draws in water under slight suction, then pressurises it for discharge.

<u>Gas displacement and bladder pumps:</u> These pumps operate on the same principle, using hydrostatic pressure in the water to fill the pump chamber and compressed air to displace the water to the surface.

Monitoring

As mentioned in the paragraph ‘reasons for sampling’ there can also be a reason to monitor the groundwater. In this case it’s important this will be done at least 5 meters from the source. If this is less it will bea question of monitoring (sampling) at the source.

4.5. Pesticides

This chapter is about taking samples from pesticides. While in the four previous chapters samples are being taken to pollutants or the level of pollutants in air, water, soil and waste. In this chapter samples are taken to determine if the product itself is legal or not. The physical sample collection is only a step within the overall-sample collection, which takes place at the pesticide producer or marketplace establishment. The other steps, like planning, reporting etc, will be described in other chapters. process.

Pesticide product samples include formulated materials from companies that manufacture, formulate or otherwise process pesticides (i.e., producers) and from commercial entities that sell wholesale or retail pesticides (i.e., dealers). Sometimes pesticide product samples are collected at the user level or at a port of entry.

4.5.1. Sample collection

The overall sample collection and analyses process includes:

- Sample collection planning
- Sample collection (including documentation)
- Sample identification
- Transfer to the laboratory
- Analysis of the sample(s)
- Reporting of analytical results
- Evaluation of analytical results
- Case preparation, enforcement action, and final disposition

4.5.2. Quantity of Sample to be Collected

The amount of each product to be collected depends largely on the amount of material required for the anticipated laboratory analysis and to assure representativeness, including the quantity required for quality control purposes (i.e., splits, repeat examinations, and replicates). Considering these sampling needs, the sample size is to be kept to a minimum to reduce the burden of disposal of the unused sample portion and to mitigate potential human and/or environmental exposure.

4.5.3. Sample units

Small-size Sample Units (Retail)

Small-size sample units are those that contain 5 litre or less of liquid or 10 kg or less of solids. These units are characterized by market-ready packages typically intended for retail distribution. Samples of these pesticide products will consist of an entire container (or group of containers) that is purchased from the producer, dealer, or user. Samples are preferably taken from original, previously unopened transport cases.

Large-size Sample Units

Large size sample units, or bulk samples, are those that contain more than 5 litre of liquid or more than 10 kg of solids. These are also units that are typically supplied to marketplace establishments for direct commercial use. When sampling these large-size units, it is recommended that smaller samples be removed from these larger containers in the field. This will reduce costs for larger amounts, make handling during sampling and subsequent analysis easier, and reduce the amount of material to be disposed of following testing.

In some situations, such as granular or pelletized lawn-care products the entire retail unit should always be collected and submitted to the laboratory. Experience has shown that these granular products are almost always non-uniform in nature and it is much easier for the laboratory to obtain an adequate portion for analysis than for the inspector to try and obtain a small representative sample in the field.

4.5.4. Equipment and techniques

Sampling from Containers

When sampling from large containers in the field, care should be given to the selection of sampling equipment and sample container composition. Physical samples should not be placed in direct contact with incompatible materials. Organic materials, such as plasticisers, may leach from rubber and some plastics into the pesticide sample. Similarly, some pesticides can leach through plastic containers. To minimize these problems, glass or stainless steel construction materials are recommended; however, plastic tubing, scoops, etc. may be used for sampling where the pesticide material is in direct contact for only a short time. A chemical substance should never be placed directly into a plastic bag or plastic bottle for storage or transport.

Some formulations, such as aqueous-based ant-microbial products are often packaged in plastic; these original containers are suitable for storage and transport as long as there is no sign of deterioration. If deterioration or other leakage is noted the sample should be transferred to a glass container to assure adequate containment during transport to the laboratory.

Sampling from large-size Solid Units

When planning the inspection for sampling of solid unit material, equipment such as a grain trier, or a disposable plastic tube or scoop should be included in the equipment list. Additional sampling tools should be taken on the inspection to avoid having to decontaminate sampling equipment in the field. Samples should be taken from the predominant batch or lot. To avoid contamination, a new or cleaned sampling tool should be used for each sample taken. Glass containers should be used to store and transport samples. Lid liners should preferably be made of Teflon however, polyethylene has also proven suitable for most formulations; aluminium foil or latex liners should not be used. Following sampling, non-disposable grain samplers should be cleaned with soap and water and solvent (e.g., acetone) and dried prior to reuse. Plastic tubes and scoops are to be disposed of following the method identified on the product label for empty containers or other authorized procedure.

Equipment needed to collect samples of dry materials includes the following:

- Protective gloves and eye wear;
- A grain sampler. This item can be found in several forms:
- Wide-mouth glass jars with Teflon-lined lids;
- Data sheets, identification tags, 4 mil plastic bags (various sizes), official seals, and chain of-custody forms (if necessary).

A recommended procedure for collecting dry samples from bags is as follows:

1. If possible, turn the bag of pesticide product over several times, both horizontally and vertically, and then lay the bag on its side on piece of protective paper or other disposable covering.
2. Through the opened seam or a tear in one of the top corners of the bag, insert the closed grain sampler (with the outer sleeve opening facing up) or plastic tube in a diagonal direction into the bag.
3. If a grain thief is employed, push the closed grain sampler to the opposite corner of the bag, then open and close the grain sampler to collect the sample. The intent is to

collect a representative sample of the material from different portions of the bag. If a plastic sampling tube is used simply push it to the opposite corner of the bag.

4. Withdraw the sampling device containing the sample, being careful to avoid spilling and puffing the contents into the air. The plastic sampling tube can be tilted slightly downward (covering the open end of the tube with the thumb) while withdrawing the tube so as to fully retain the sampled material.
5. Transfer the material from the sampler by tilting the tube into a glass jar; label, officially seal, complete chain-of-custody form, and prepare for transport to the laboratory.
6. If more sample is needed, take a similar sample from the other top corner of the bag.
7. Samples from the same unit may be composite; samples from different units of the same batch are to be placed in separate containers; however, the same sampling tube may be used without decontaminating. Samples from different batches of the same product require use of new or decontaminated sampling equipment.

These procedures for collecting samples of solid material from bags can be modified as needed to collect solids from other types of containers. By carefully following a sampling protocol, cross -contamination during sampling can be minimized. If plastic tubes are used to collect samples, it is recommended that they be destroyed after each use by cutting them up and disposing of the pieces in an authorized manner.

Sampling from large-size Liquid Units

Samples should be collected in the field from producer establishment and marketplace liquid pesticide products in containers in excess of 1 gallon in size. Liquid units should be sampled using a siphon device with unused disposable type plastic tubing, or a new or decontaminated reusable glass thief for each batch or lot to be sampled. If the label directions indicate that the product to be sampled should be mixed before use, the unit should be agitated prior to sampling. Glass containers should be used to store and transport all samples. Teflon lid liners are preferred; however, polyethylene may also be used in most cases. Rubber, aluminium foil, or paper-lined lids must not be used. Plastic tubes should be disposed of by the method identified on the product label for disposal of empty containers or other authorized procedure.

Equipment needed to collect sub-samples from liquid units include the following:

- A new or decontaminated glass thief or siphon device with disposable tygon tubing
- glass bottle with Teflon-lined lid
- Identification labels, plastic bags, and official seals
- Labelled transport container(s) with appropriate packing material and other office supplies, transport forms, official seals, and chain-of-custody forms.

The following is a recommended procedure for collecting samples from larger units in the field:

1. Thoroughly agitate the material to be sampled, if required, by shaking or rolling the unit before sampling.
2. Insert siphon hose or glass tube (thief) through the access port in the bulk container being sampled.
3. Collect a composite sample from three depths: near the bottom, the middle, and the top of the liquid level. If a glass thief is employed, a representative sample may be obtained by slowly lowering the thief to the bottom of the container, allowing liquid to fill the glass tubing as it is submerged.
4. Cover the top of the thief tightly with the thumb and carefully transfer the sample into the glass sample container. If sediment, layering, or phase separation is observed,

collect a duplicate sample using the same thief or tubing; decontamination is usually not necessary.

5. Label, officially seal, and package the sample container for transport.
6. Properly dispose of the hose and other disposable sampling equipment. Clean other contaminated sampling equipment with soap and water, rinsing with water and acetone, and allowing to dry before using to collect additional samples.

Sampling from tanks

Techniques for collecting liquid samples from tanks, such as those utilized in bulk repackaging operations, have not been well established or standardized. At a minimum, the inspector should discuss with laboratory personnel and their supervisor the proposed method for sampling bulk repackaging tanks or other large storage units. In such cases it may be desirable to consult with hazardous waste inspectional staff to assure the collection of a representative sample in a safe manner. If the unit does not look secure, sampling should not be attempted without supervisory approval. The inspector should also be aware that a sample collected from a release valve or discharge spigot may not be representative of the contents of the entire tank.

4.5.5. Duplicate Samples

Duplicate samples are collected only at the pesticide product establishments request; however, the inspector must make the offer. These samples should be collected, identified, and sealed using the same equipment, techniques, and sampling methods. Duplicate samples can be defined as an equal amount of the product taken in the same manner from the same container.

While a firm may request that duplicate samples from small-size units be prepared by dividing the contents of these units into equal portions, small-size units are not to be subdivided. The reasons for this are: (1) the integrity of the sample as evidence is more difficult to maintain and defend; (2) cross-contamination is minimized; (3) the possibility of exposure during sampling is minimized; and (4) the laboratory can determine the net contents, if necessary.

4.5.6. Safety Practices

This chapter will address the basic issues associated with pesticide product inspections. For general safety precautions see chapter 6.

It is the responsibility of each inspector to protect themselves and others during pesticide handling and sampling activities associated with an inspection. The inspector can best achieve this by staying current with all required health and safety training. To minimize risks during sampling, appropriate protective clothing and safety equipment must be used. Protective gear must be adequate to prevent accidental exposure to pesticides through the eyes, nose, mouth, and skin. At a minimum, the inspector should follow the safety precautions specified on the label of the product being sampled. The inspector should be aware of the establishments safety requirements and basic emergency treatment. While on-site, it is advisable that the inspector be familiar with the location of medical assistance in the event of an emergency.

Selecting the appropriate safety equipment for sampling depends on the type and volume of pesticide to be sampled. Collecting small-size samples from marketplace establishments will usually require only a minimum of safety procedures and equipment. When taking samples from larger-sized containers at producer, dealer, or user sites, inspectors must heed the handling precautions found on pesticide labels. Similarly, instructions for the use of safety equipment (i.e., compatibility warnings) must be followed. At a minimum, the following safety equipment should be used during sampling:

- Hand protection - organic liquid-proof gloves, preferably of latex or synthetic rubber, long enough to protect the wrist;
- Eye protection - goggles or face shield;
- Protective footwear in the form of rubber soled, non-skid, metal-toed shoes and plastic disposable shoe covers, or rubber/neoprene boots;
- Hard hat;
- Coveralls made of closely woven fabric or Tyvek or long rubber apron;
- Respiratory protective device when sampling toxic materials from large size units.

Various cartridges must be used to protect against different chemical vapours and gases. The inspector should be careful to select the appropriate cartridge for the product being sampled. Note: The use of respiratory protective devices requires appropriate training and fit-testing. Before using this type of equipment, the inspector should ensure that their training and fit-testing is current.

Before sampling any pesticide, the precautionary statements on the label should be read to determine whether the pesticide is toxic through dermal absorption, inhalation, or ingestion. Some pesticides may be injurious through all three routes. After determining which exposure routes should be avoided for the particular pesticide to be sampled, the proper safety equipment may be selected. The inspector should always be careful when collecting samples, regardless of the declared toxicity of the pesticide. There is always the risk that the pesticide product may be mislabeled or improperly formulated.

When collecting samples, the inspector should remain alert to hazards such as spilled or improperly stacked materials, moving equipment, poor ventilation, and bad lighting. The following general guidelines are provided for the safe handling or sampling of pesticides:

- Read the label and, at a minimum, follow the handling instructions for mixing and loading;
- Use care and the proper tools when opening and closing larger containers;
- Open and sample pesticides in areas where leaks and spills can be cleaned up easily and properly;
- If chemicals are spilled on clothing or directly onto the skin, remove the clothing immediately and wash the exposed dermal area thoroughly with plain water. Always keep a change of clothing on hand for such emergencies;
- Wash hands immediately after sampling;
- Do not use the mouth to siphon and do not put hands near the mouth and eyes during sampling operations;
- Keep a supply of clean water and waterless hand cleanser readily available;
- Know the limitations of the protective equipment being used, especially respirators;
- Have the phone numbers of local hospitals, doctors, or poison control centres available.

4.6. Sample Preparation

Each sample should be identified in the inspector's handwriting with the date, sample number, and the inspector's initials. When more than one sample is collected, each sample should be further identified with an additional number or letter. This identification is normally written on the label or an adhesive strip placed on the bottle, or jar.

In case of large-size pesticide unit, the handwritten label should also provide the following information for the safety of those handling the sampled product:

- Name of the product;
- Principal active ingredients and concentration;
- Company name and address as shown on label;
- Distinguishing marks or code numbers;
- Label should be stamped in red ink with “POISON” and skull and crossbones if the products label was so marked.

Samples should be sealed by placing an inverted plastic bag (4 mil thickness, recommended), tying a knot, and turning the excess amount of bag back over the knot and taping the excess bag below the knot. It is important that the sample label be legible through the plastic bag. It is preferable to have each glass unit sealed in a separate bag, or at a minimum, packaged so that is there no direct contact of glass upon glass

4.7. Chain-to-Custody

A complete and accurate chain-of-custody record is a critical component of official sample documentation. The purpose of an irrefutable chain-of-custody is to trace possession and assure integrity of an official sample from the time it is collected until it is introduced as evidence in a legal proceeding. An accurate, written record of the movement of the sample should be maintained on a chain-of-custody form.

4.8. Temporary Storage and transport to the Laboratory

Following collection, documentation, and initiation of chain-of-custody, the officially sealed samples should be stored in a secure area prior to transfer to the laboratory. All documentation will normally accompany the sample(s) to the laboratory. At a minimum, the laboratory should receive the label, an investigation summary report, any chain-of-custody forms, as well as any correspondence or records related to the products ingredients, stability, or mixing for use. Whatever the method of transfer is , the inspector and/or laboratory personnel must maintain a record of how the sample was transferred, including all transport papers and receipts.

Regardless of which method is employed for sample transfer to the laboratory, several requirements need to be followed:

- Glass containers must never be packaged directly against each other, either within the same plastic bag or within the same transport container;
- The sample label should always be legible through the protective plastic bag;
- Liquid samples should not be packaged with solids in the same transport container;
- A copy of an investigative summary report or equivalent form should be included with the samples, protected in a plastic bag or sleeve;
- The laboratory director or designee should be notified by telephone that the samples are being transported, the mode of transfer, and the expected arrival date.

5. Documentation and reporting

5.1. Sample documentation

The objective of sample documentation is to validate sample integrity. The sample must be uniquely identified continued through all subsequent activities. Additional necessary documentation regarding the nature and circumstances of each sample needed as evidence should be reported. At a minimum, the following items should be documented:

- Sample site identification and/or project number
  - General location (e.g., address of facility)
  - Specific location (of sampled products in facility)
  - Area description and related observations
- Date and time of sampling
- Sample description
  - Container contents (specific material collected)
  - Name of substance for which analysis is needed
  - Reason for collection
  - Quantity of sample collected (volume, number, weight)
  - Identity (sample numbers) of related samples, if any
- Sampling method
  - Composite, grab, or pre-packaged unit
  - Devices and tools used – Pre-cleaning of equipment and decontamination between uses
- Storage and transport
  - Primary container and lid type and any pre-cleaning used
  - Packaging procedure
  - Preservation, if applicable
  - Method of transfer to laboratory (including date and time)
- Other documentation
  - Custody and document control records
  - Books and records information
  - Photographs
  - Statements and affidavits
  - Technical and professional remarks
  - Correspondance, phone logs, notes, etc.

5.2. Sample reporting

The report involves summarising and presenting the sample results. Next to the information which is documented during sampling the following should be reported:

- Tests-results
- Legal requirements
- Comparison and conclusion about compliance

6. Safety precautions

6.1 General caution

Inspectors should arrive at the site well prepared. Not only as far as the sampling or other work is concerned, but also as far as their own safety is. Unless the law says so, it may not be expected that the site-owner will provide the required safety equipment.

When conducting sampling, samplers need to be aware of health and safety hazards and take the proper precautions. Safety requirements can be gathered from file information, personnel that have previously sampled the facility, or by contacting the facility. Samplers need to be properly clothed and have adequate safety equipment available. Entering confined spaces should not be done unless the inspector is properly trained to do so and has the proper equipment such as rescue equipment and respirators. Confined spaces should never be entered unless first tested for sufficient oxygen and lack of toxic and explosive gases. Two persons should be present, one to enter the space and the other one to be outside. The entering person should wear a safety harness attached to a retrieval system. This system can be used to rescue the sampler in the confined space without requiring anybody else to enter.

Safety hints
Study the safety situation before the sampling trip Prepare you safety equipment before the sampling trip DO NOT enter confined spaces, unless properly trained Do regular health and safety trainings Read safety labels Be aware of the facility’s safety requirements and emergency treatment (first aid) Not only have, but USE your safety equipment Be prepared for improperly stacked materials, bad lighting, poor ventilation, moving equipment and spilled materials Always use the proper tools, don’t improvise Have clean water at hand during the sampling Have a cellular telephone and emergency numbers at hand during the sampling

Despite safety precautions an inspector should always be careful during the sampling. There is always the risk of unknown factors, mislabelling of products or improper formulation of documentation or information from other persons. The inspector should remain alert to hazards such as spilled or improperly stacked materials, moving equipment, poor ventilation, and bad lighting. The inspector should always use the proper tools on the one hand to prevent contamination of the sample, on the other hand to safeguard his safety and that of others. During the sampling process it is advised to think about possible accidents, how to prevent them and – in case of occurrence- how to minimise the risks for the inspector and other people as well as for the environment.

Safety precautions and actions during the sampling can be:

- In case of a spill of chemicals on clothing or directly onto the skin, remove the clothing immediately and wash the exposed dermal area thoroughly with plain water. Always keep a change of clothing on hand for such emergencies;
- Wash your hands immediately after sampling (first with your gloves still on);
- Do not use the mouth to siphon;

- Do not put hands near the mouth and eyes during sampling operations;
- Keep a supply of clean water and waterless hand cleanser readily available;
- Know the limitations of the protective equipment being used, especially respirators;
- Don’t smoke and don’t use open fire during sampling;
- Don’t mix substances in case of the possibility of or the uncertainty of a reaction;
- Have the phone numbers of local hospitals, doctors, or poison control centres available.

6.2 How to identify danger

Preventive measures limit the risks, while corrective measures minimise the possible effect of an incident. The use of safety equipment is part of the corrective measures. To find out which preventive or corrective measures could be taken, we will have to determine the dimensions of the risk.

This can be done by analysis per risk-area, meaning that risks are mapped concerning the sample itself and concerning the working circumstances. This analysis can be performed by the inspector himself. The method we will describe here will mainly be useful in case of on-site inspections.

The inspector will start his analysis by writing down the risks. He may use a ‘sampling point form’. For an example see Annex A.

In the form is important:

1. to get an overview of the possible composition of the sample, both under normal circumstances and under calamities. As basic material for this we can use existing documentation on chemical substances and their dangers;
2. that the followed sampling procedure is described in short;
3. that the necessary personal safety equipment is written down.

This form is not only useful for the inspector himself but also for his colleagues that may visit the site later on. He will also fill out a ‘site visit form’. See Annex B for an example. The filled out form will contain the following:

- a short description of the site: what is being produced, what technology is being used. This can be done by using e.g. the license, but also on the basis of a personal talk with company staff;
- a map of the site containing process- and storage rooms with increased risks; escape routes (in case of calamities) and route to the sampling point;
- the necessary personal safety equipment for the visit to this specific company;
- personal hygiene before, during and after the site visit;
- point of attention concerning the visitor’s regulations (e.g. announcement of arrival, alarm points, personal guidance by company staff);
- preliminary and final risk level of sampling at the site.

It can be considered to add a summary of the license to the site visit form.

After the risk analysis the inspector will be able to categorise the different risk-levels, which we could indicate as follows:

low

high

Risk level 1

- There is no or only minor danger for the inspector to be exposed to dangerous concentrations, both in nature as in quantity, and
- The sampling circumstances (quality sampling point, weather conditions, accessibility in case of calamities) are reasonable, and
- In case of on-site inspection: the industrial activities (process of manufacturing, storage of substances) are of low risk level or slightly increased

Risk level 2

- Increased danger for the inspector to be exposed to dangerous concentrations of substances, in nature or in quantity, or
- The sampling conditions are bad, or
- The manufacturing activities have an increased risk level (dangerous manufacturing process, storage of dangerous substances at a distance of more than 15 m, or
- The accessibility for other persons is difficult or impossible

Risk level 3

- High danger for the inspector to be exposed to dangerous concentrations of substances in nature or in quantity, or
- Unknown how high the risk for the inspector in this concern is, or
- The sampling circumstances/facilities are unknown, or
- The storage of dangerous substances at a distance of less than 15 m, or the possibilities of these substances being present in the water, air or soil to be sampled

We can list some examples of the different risk levels. Please note that these are **just examples**, not all situations are mentioned and actual circumstances can make that another risk level is more appropriate!

<u>Risk level 1</u>
- waste water treatment plants
- restaurants, pubs
- routine inspections of ship yards
- waste water sampling at dairy farms
- bilge water under good circumstances
- assisting at environmental inventories
- thermally polluted waste water
- routine inspection at a high altitude (storage tanks)
- suspected soil and waste
- chemical process industry/metallurgical industry with good provisions concerning labour circumstances

<u>Risk level 2</u>
- chemical process industry/metallurgical industry, other than level 1
- investigating locations with suspected dredge
- non-routine inspection at high altitude
- near traffic
- bilge water under unfavourable circumstances
- possibility of dangerous gasses and or aerosols that are given off

- without specific sampling facilities
- surface water sampling from the shore, gangway inspection ship etc.
- during weekends, evening and night time
- near waste water streams with a possible very high pH value (pH ≥ 10) or very low pH value (pH ≤ 3)
- site inspections with possibly increased concentrations of dangerous substances in the streams to be sampled
- manure in storage tanks or ships

<u>Risk level 3</u>
- all non-routine investigations
- unforeseen circumstances, like disturbances in the manufacturing process, fire
- at the discovery of peculiar situations
- chemical process industry/metallurgical industry, other than level 2
- sampling tank trucks or tank boats
- circumstances/working under time pressure

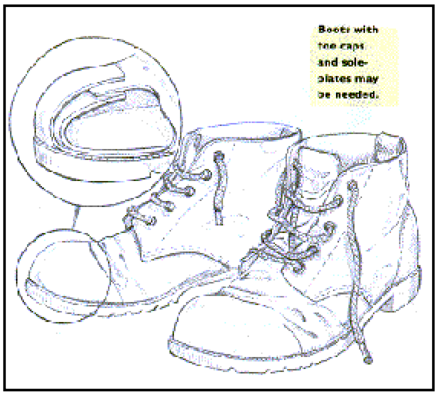
6.3. Protective clothing

It is the responsibility of each inspector to protect themselves and others during the sampling activities associated with an inspection. A regularly repeated health and safety training is important for inspectors to stay sharp with the latest developments in this field. To minimise risks during sampling, appropriate protective clothing and safety equipment must be used. Protective gear must be adequate to prevent accidental exposure to chemicals, pesticides, etc. through the eyes, nose, mouth and skin. At a minimum the inspector should follow the safety precautions specified on the label –if present- of the product being sampled. The inspector should be aware of the establishment's safety requirements and basic emergency treatment. While on-site, it is advisable that the inspector be familiar with the location of medical assistance in the event of an emergency. Selecting the appropriate safety equipment for sampling depends on the type and volume of material to be sampled.

As a minimum the following equipment should be used during sampling:

- Hand protection – organic liquid-proof gloves, preferably of latex or synthetic rubber, long enough to protect the wrist; always fit the sort of glove to the material to be sampled. Don't fold the gloves, this will weaken them. Don't put the gloves away inside out. Wash your gloves (while still on your hands) with water before storing them in a cool and dry spot. Never store them in the sunlight!
- Eye protection – goggles or face shield
- Ear protection
- Protective footwear in the form of rubber soled, non-skid, metal soled and toed shoes and plastic disposable shoe covers, or rubber/neoprene boots. Always pay attention that the sole is resistant against chemicals, oil and grease.
- Hard hat
- Container with plain water
- Swimming jacket (in case of dangerous situation when sampling surface waters)
- Safety line (sampling surface waters)
- Coveralls made of closely woven fabric of Tyvek® or long rubber apron

Picture 17



- Respiratory protective device when sampling toxic materials from large size units. Use the proper cartridge against the specific chemical vapours and gases or dust. Be careful in the selection of the appropriate cartridge[4].

Picture 18 (gas detector)

Picture 19

(Standard) safety equipment
• hard hat • work boots/safety shoes (steel toes and soles, heat resistant, oil resistant, slip resistant) • Safety vest ('Public works department') • Cotton overall • Polyflex trousers • Polyflex coat • Coveralls made of closely woven fabric or Tyvek® or long rubber apron • Fire and acid proof overcoat • Safety gloves • Respirator • Safety harness • Safety glasses (poly-carbonate) • Cover glasses (blanc) • Ear defenders • Explosion risk meter • Compressed air escape unit • First-aid kit • Explosion proof torch • Fire extinguisher • Cellular telephone

[4] Beware: the use of protective devices requires appropriate training and fit-testing. Before using this type of equipment, the inspector should ensure that their training and fit-testing is current. Wrong use can be dangerous!!!

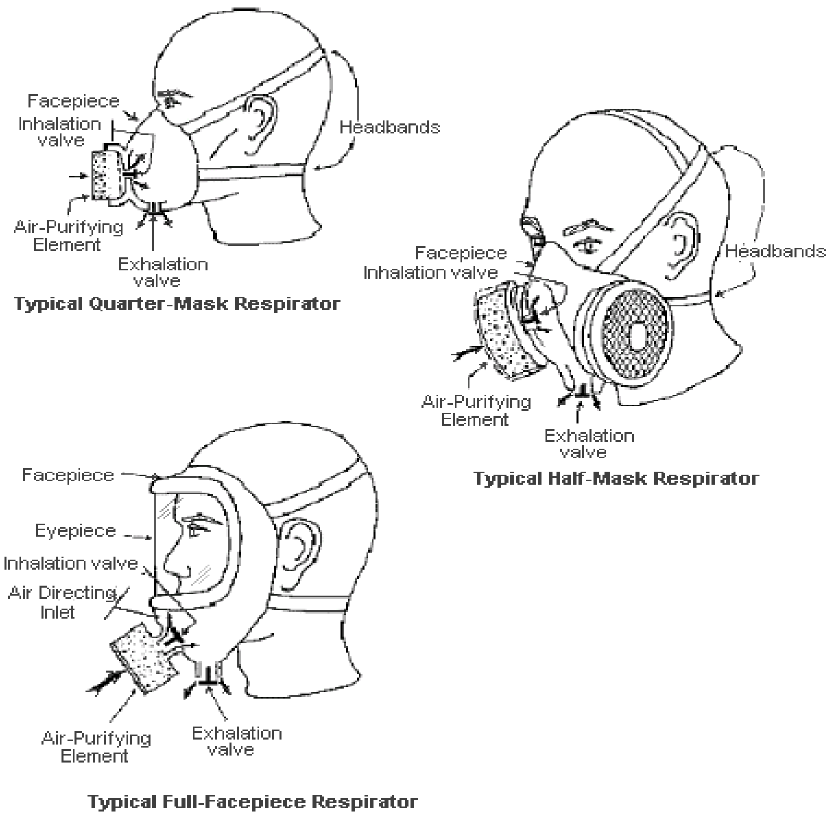
6.4 Respiratory protection

6.4.1 Purpose

The purpose of a respirator is to prevent the inhalation of harmful airborne substances and/or an oxygen-deficient atmosphere.

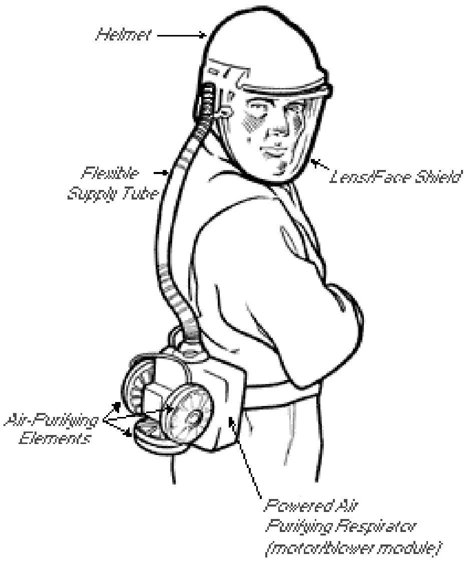
Functionally, a respirator is designed as an enclosure that covers the nose and mouth or the entire face or head. Respirators are of two general "fit" types:

- The tight-fitting respirator is designed to form a seal with the face of the wearer. It is available in three types: quarter mask, half mask, and full face piece. The quarter mask covers the nose and mouth, where the lower sealing surface rests between the chin and the mouth. The half mask covers the nose and mouth and fits under the chin. The full face piece covers the entire face from below the chin to the hairline (see picture 20).



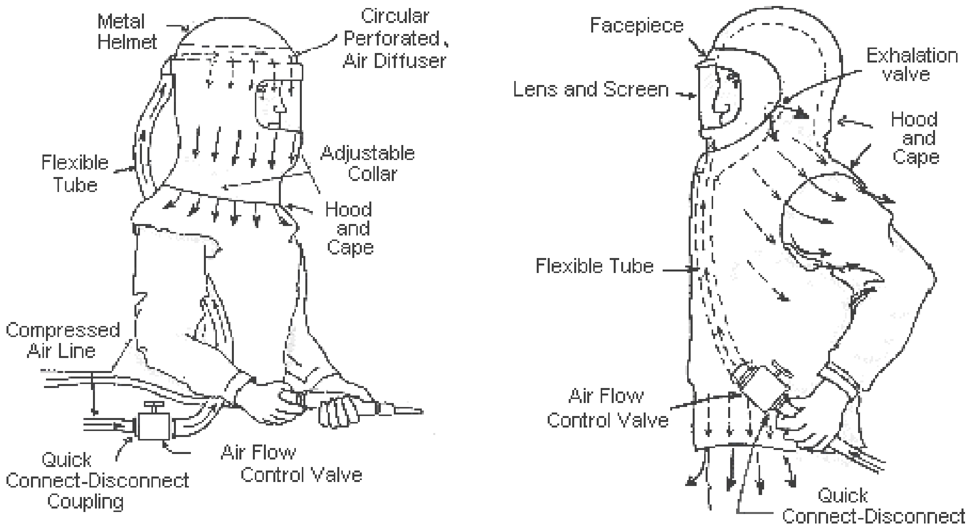
Picture 20 (tight-fitting respirators)

- The loose-fitting respirator has a respiratory inlet covering that is designed to form a partial seal with the face. These include loose-fitting face pieces, as well as hoods, helmets, blouses, or full suits, all of which cover the head completely. The best known loose-fitting respirator is the supplied air hood used by the abrasive blaster. The hood covers the head, neck, and upper torso, and usually includes a neck cuff. Air is delivered by a compressor through a hose leading into the hood. Because the hood is not tight-fitting, it is important that sufficient air is provided to maintain a slight positive-pressure inside the hood relative to the environment immediately outside the hood. In this way, an outward flow of air from the respirator will prevent contaminants from entering the hood (see picture 21).



Loose-Fitting Facepiece

Picture 21a (loose-fitting respirators).



Abrasive Blasting Respirator (Hood Respirator)

Loose-Fitting Hood with Blouse

Picture 21b (loose-fitting respirators).

6.4.2 Airborne (or respiratory) hazards

This may result from either an oxygen deficient atmosphere or breathing air contaminated with toxic particles, vapours, gases, fumes or mists. The proper selection and use of a respirator depend upon an initial determination of the concentration of the hazard or hazards present in the workplace, or the presence of an oxygen deficient atmosphere.

Airborne hazards generally fall into the following basic categories:

- Dusts: particles that are formed or generated from solid organic or inorganic materials by reducing their size through mechanical processes such as crushing, grinding, drilling, abrading, or blasting.
- Fumes: particles formed when a volatilised solid, such as a metal, condenses in cool air. This physical change is often accompanied by a chemical reaction, such as oxidation. Examples are lead oxide fumes from smelting, and iron oxide fumes from arc-welding. A fume can also be formed when a material such as magnesium metal is burned or when welding or gas cutting is done on galvanized metal.
- Mists: a mist is formed when a finely divided liquid is suspended in the air. These suspended liquid droplets can be generated by condensation from the gaseous to the liquid state or by breaking up a liquid into a dispersed state, such as by splashing, foaming, or atomising. Examples are the oil mist produced during cutting and grinding operations, acid mists from electroplating, acid or alkali mists from pickling operations, paint spray mist from spraying operations, and the condensation of water vapour to form a fog or rain.
- Gases: gases are formless fluids that occupy the space or enclosure and which can be changed to the liquid or solid state only by the combined effect of increased pressure and decreased temperature. Examples are welding gases such as acetylene, nitrogen, helium and argon; and carbon monoxide generated from the operation of internal combustion engines. Another example is hydrogen sulphide, which is formed wherever there is decomposition of materials containing sulphur under reducing conditions.
- Vapours: vapours are the gaseous form of substances that are normally in the solid or liquid state at room temperature and pressure. They are formed by evaporation from a liquid or solid, and can be found where parts cleaning and painting takes place and where solvents are used.
- Smoke: smoke consists of carbon or soot particles resulting from the incomplete combustion of carbonaceous materials such as coal or oil. Smoke generally contains droplets as well as dry particles.
- Oxygen deficiency: an oxygen deficient atmosphere has an oxygen content below 19.5% by volume. Oxygen deficiency may occur in confined spaces, which include, but are not limited to, storage tanks, process vessels, towers, drums, tank cars, bins, sewers, septic tanks, underground utility tunnels, manholes, and pits.

6.4.3 Respirator classifications

Respirators provide protection either by removing contaminants from the air before they are inhaled or by supplying an independent source of respirable air. There are two major classifications of respirators:

- Air purifying respirators: devices that remove contaminants from the air.
- Atmosphere-supplying respirators: devices that provide clean breathing air from an uncontaminated source.

Each class of respirator may have tight-fitting and loose-fitting face pieces. An important aspect of respirator operation and classification is the air pressure within the face piece. When the air pressure within the face piece is negative during inhalation with respect to the ambient air pressure, the respirator is termed a negative-pressure respirator. When the pressure is normally positive with respect to ambient air pressure throughout the breathing cycle, the respirator is termed a positive-pressure respirator. The concept of negative and positive pressure operation is important when considering potential contaminant leakage into the respirator.

Air purifying respirators are grouped into three general types: *particulate removing, vapour and gas removing,* and *combination.* Elements that remove particulates are called filters, while vapour and gas removing elements are called either chemical cartridges or canisters. Filters and canisters/cartridges are the functional portion of air-purifying respirators, and they can generally be removed and replaced once their effective life has expired. The exception would be filtering face piece respirators (commonly referred to as "disposable respirators," "dust masks," or "single-use respirators"), which cannot be cleaned, disinfected, or resupplied with an unused filter after use.

- Particulate-removing respirators are designed to reduce inhaled concentrations of nuisance dusts, fumes, mists, toxic dusts, radon daughters, asbestos-containing dusts or fibres, or any combination of these substances, by filtering most of the contaminants from the inhaled air before they enter the breathing zone of the worker. They may have single-use or replaceable filters. These respirators may be non-powered or powered air-purifying. A powered air-purifying respirator (PAPR) uses a blower to force the ambient atmosphere through air purifying elements to the inlet covering.
- Vapour- and gas-removing respirators are designed with sorbent elements (canisters or cartridges) that adsorb and/or absorb the vapours or gases from the contaminated air before they can enter the breathing zone of the worker. *Combination* cartridges and canisters are available to protect against particulates, as well as vapours and gases.

Atmosphere-supplying respirators are respirators that provide air from a source independent of the surrounding atmosphere instead of removing contaminants from the atmosphere. These respirators are classified by the method that is used to supply air and the way in which the air supply is regulated. Basically, these methods are: self-contained breathing apparatus (air or oxygen is carried in a tank on the worker's back, similar to SCUBA gear); supplied-air respirators (compressed air from a stationary source is supplied through a high-pressure hose connected to the respirator); and combination self-contained and supplied-air respirators.

6.4.4 Limitations of respirator use

Not all workers can wear respirators. Individuals with impaired lung function, due to asthma or emphysema for example, may be physically unable to wear a respirator. Individuals who cannot get a good face piece fit, including those individuals whose beards or sideburns interfere with the face piece seal, will be unable to wear tight-fitting respirators. An adequate fit is required for a respirator to be effective. In addition to these problems, respirators may also be associated with communication problems, vision problems, fatigue, and reduced work efficiency.

In principle, respirators usually are capable of providing adequate protection. However, problems associated with selection, fit, and use often render them less effective in actual application; these problems prevent the assurance of consistent and reliable protection, regardless of the theoretical capabilities of the respirator. Occupational safety and health experts have spent considerable effort over the years developing fit-testing procedures and methods of measuring respirator effectiveness, thereby improving protection for those employees required to wear them.

6.4.5 Respirator protection program

Whenever respirators are required to be worn, a written respirator protection program must be developed. The program must consist of worksite-specific procedures governing the selection, use, and care of respirators. The program must be updated as often as necessary to reflect changes in workplace conditions and respirator use.

The respiratory protection program must cover the following basic elements:

- Procedures for selecting respirators for use in the workplace;
- Medical evaluations of employees required to use respirators;
- Fit testing procedures for tight-fitting respirators;
- Use of respirators in routine and reasonably foreseeable emergency situations;
- Procedures and schedules for cleaning, disinfecting, storing, inspecting, repairing, and otherwise maintaining respirators;
- Procedures to ensure adequate air quality, quantity and flow of breathing air for atmosphere-supplying respirators;
- Training of employees in the respiratory hazards to which they are potentially exposed;
- Training of employees in the proper use of respirators, including putting on and removing them, any limitations on their use, and maintenance procedures; and
- Procedures for regularly evaluating the effectiveness of the program

6.4.6 Respirator selection

Respirator selection requires correctly matching the respirator with the hazard, the degree of hazard, and the user. The respirator selected must be adequate to effectively reduce the exposure of the respirator user under all conditions of use, including reasonably foreseeable emergency situations. Proper respirator selection involves choosing a device that fully protects the worker from the respiratory hazards to which he or she may be exposed and permits the worker to perform the job with the least amount of physical burden.

Selection factors

Many factors must be considered carefully in respirator selection. In choosing the appropriate respirator, one must consider the nature and extent of the hazard, work requirements and conditions, and the characteristics and limitations of the respirators available. The following categories of information must be taken into account:

- Nature of the hazard, and the physical and chemical properties of the air contaminant;
- Concentrations of contaminants;
- Relevant permissible exposure limit or other occupational exposure limit;
- Nature of the work operation or process;
- Time period the respirator is worn;
- Work activities and physical/psychological stress;
- Fit testing and;
- Physical characteristics, functional capabilities and limitations of respirators.

6.4.7 Medical aspects

Persons assigned to tasks that require the use of a respirator must be physically able to perform the work while using the respirator. Accordingly, employers have the responsibility of ensuring that employees are medically fit to tolerate the physical and psychological stress imposed by respirator use, as well as the physical stress originating from job and workplace conditions. Employees must be medically evaluated and found eligible to wear the respirator selected for their use prior to fit testing or first-time use of the respirator in the workplace.

6.4.8 Use of respirators

Once the respirator has been properly selected and fitted, it is necessary to ensure that the respirator is used properly in the workplace. The following conditions may compromise the effective use of the respirator and jeopardize worker protection:

- Face piece seal leakage
- Removing the respirator at the wrong times in hazardous atmospheres
- Not properly performing user seal checks
- Not properly repairing defective parts.

In these circumstances, there is the danger that employees may have a false sense of security in feeling that they are protected while they are not.

6.4.9 Maintenance and care

It's strongly emphasizes the importance of a good maintenance program which includes at least:

- Cleaning and disinfecting procedures;
- Proper storage;
- Regular inspections for defects (including leak check); and
- Repair methods.

In addition the manufacturer's instructions for inspection, cleaning, and maintenance of respirators should be consulted to ensure that the respirator continues to function properly.

- **Glossary**

BOD*: Biochemical Oxygen Demand is a measure of the amount of oxygen that bacteria will consume while decomposing organic matter under aerobic conditions.*

COD*: Chemical Oxygen Demand Chemical oxygen demand (COD) is a measure of the total quantity of oxygen required to oxidize all organic material into carbon dioxide and water.*

Compliance monioring: *process to compair actual emissions of pollutants for an installations with permitted emission limit values within a defined degree or confidence*

Emission*: discharge of a quantity of substance, energy or vibration into the environment (air, water, soil ..). The emission can be expressed as a total quantity in absolute or as a rate per a difined period of time.*

Groundwater: *all water which is below the surface of the ground in the saturation zone and in direct contact with the ground or subsoil*

Isokinetically*:* *when the velocity at which the sample enters the sampling nozzle is the same as the velocity in the duct*

Monitoring: a *continuous or regular periodic check to determine the on-going nature of the potential hazard, emissions, conditions along environmental pathways and the environmental impacts*

Sample plane: *Area or section, for example within a pipeline, where the sample points are positioned*

8. References

- Best practice in compliance monitoring, 2001 – IMPEL
- Internet- EPA US
- Technical Guidance Notes (Monitoring) – Environmental Agency UK
- Pesticide product inspections – INECE
- Respiratory protection, Technical manual – OSHA
- Guidance on Monitoring of Landfill Leachate, Groundwater and Surface Water - Environmental Agency UK
- Reference document on general principles of monitoring (draft) – EU commission
- Monsterneming – Commissie Intergraal Waterbeheer

ANNEXES

ANNEX A

SITE VISIT FORM

Name of the enterprise : --

License dated : --

Safety contact (name) : --

Consultation with the company (dates) : --

Description of the company (to be made with/by the company's safety expert)

………

………

………

☐ Map (*) ☐ Flow chart production method (*) ☐ Internal safety prescriptions (*)

Preparation site visit

…………………………………………………………………………….
…………………………………………………………………………….
…………………………………………………………………………….
…………………………………………………………………………….
…………………………………………………………………………….
…………………………………………………………………………….
…………………………………………………………………………….

☐ smoking ban
☐ safety shoes
☐ boots
☐ dust filter
☐ gas filter
☐ special clothing
☐ safety glasses/mask
☐ safety gloves
☐ ear protection

Routing inside the company, including escape routes

……..
……..
……..
……..

☐ Indicated on the map

What to do in case of calamities

……..
……..
……..
……..

Personal hygiene

……..
……..

(*) to be provided for by the company

ANNEX B

SAMPLING POINT FORM

Name of enterprise: --

Date : --------------------- Name inspector : ------------------------------

No. **Description sampling point** (location at site, approach sampling point etc.)

Expected composition of the samples ***(routine wise / in extremes (calamities))***

Substance	Name	Toxic risk level	Fire risk	Remarks
1. --------	------------------------------------	--------------------	------------	------------------
2. --------	------------------------------------	--------------------	------------	------------------
3. --------	------------------------------------	--------------------	------------	------------------
4. --------	------------------------------------	--------------------	------------	------------------

Other remarks

ANNEX C

International symbols of danger

Explosive
Substances that can explode trough contact with fire or are more sensitive to bumping and rubbing than dinitrobenzene

Oxidizing
Substances that can, through contact with other substances, especially flammable substances, react strongly in an exothermal way

Inflammable
Liquids with a flashing point of less than 0° C and a boiling point of 35° C or more, as well as substances that:

- at normal temperature exposed to the air, without supply of energy can rise in temperature and can finally catch fire;
- in solid condition, by short impact of an ignition source, can easily be ignited and after removal of the ignition source continue burning and glowing
- in liquid condition, have a flashing point of less than 21° C
- in gaseous condition under normal pressure, are inflammable with air by contact with water of humid air, develop easily inflammable gasses in a dangerous quantity

Toxic
Substances that by inhalation or by entering through mouth or skin, can cause very serious acute or chronic dangers or even death

Harmful
Substances that by inhalation or by entering through mouth or skin, cause dangers of a limited nature

Corrosive
Substances that, through touch, have a destructive effect on living tissues

Dangerous for the environment
Substances of which the use has or can have an immediate or delayed effect on the environment