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Soil quality — Guidance on the selection and application of screening methods

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National foreword

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Soil quality — Guidance on the selection and application of screening methods

*Qualité du sol — Lignes directrices pour la sélection et l'application des
méthodes de diagnostic rapide*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12404 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Introduction

Screening methods, which can be chemical, physical or biochemical in nature, can often be applied in a quick and simple manner. Performance of quick and simple tests can be used in the field (i.e. on-site) and, in some cases, are also applicable for laboratory use. They can indicate the presence or absence of an analyte, or provide a qualitative estimate of a concentration or value, or generate a quantitative result. They can also be used to produce a spatial distribution of concentrations or values within a site, which can be supported by subsequent reference (laboratory-base) analysis. When used in this way, the purpose is generally to obtain information on target parameters or groups of parameters and the location of unusual concentrations, possibly prior to undertaking a more detailed study or investigation. For these purposes, the bias and precision of these methods need not be to the same level as conventional laboratory reference methods, for example as demonstrated by International Standards, as the initial objective of their use is to obtain as much information as possible in a relatively short period of time on the presence or absence, or range of concentrations likely to be determined for a particular site. It may be more important to obtain a result quickly or with an improved spatial resolution as an indication of the magnitude and likely concentration, rather than precise and unbiased values.

Typically, for measurement techniques, a result may be obtained in one of three ways. Firstly, as a qualitative presence or absence result. Secondly, as a semi-quantitative result expressed within a relatively wide range of values, and thirdly, as a result with an accompanying uncertainty of measurement with a significantly smaller range of values that might be expected. (The third option is usually a result generated using a laboratory reference method, with the uncertainty of measurement of laboratory reference methods generally being smaller than that of screening methods.) Whichever result is generated depends on the nature and type of the screening method used, as well as the technology on which the screening method is based.

The use of screening methods usually increases the efficiency of a site investigation, while providing as much information as that obtained in situations where only laboratory reference methods are used. Whilst the use of these rapid measurement techniques at a particular site should not replace conventional analysis, their use greatly facilitates the investigation in a complementary role. Generally, many more samples can be analysed and results generated faster than determined by more conventional testing of laboratory reference methods. This enables areas, for example, those with very high levels of concentrations, or where very low concentrations exist, to be identified much more quickly and efficiently. If too few samples are taken and analysed by more costly laboratory reference methods, there is a risk that these areas might not be identified and could easily be missed. This process then allows more effort to be directed on those areas where high or unusual levels are likely to be present, for example, by employing conventional laboratory reference method analysis. This can save time, money and resources, especially when cost-effective screening methods are applied to a large number of samples and supportive conventional reference method analysis is also undertaken, where relevant.

The use of screening methods, particularly if carried out on-site, can offer an immediate decision-making opportunity which enables staff to direct their efforts more effectively to those areas where a more thorough investigation might need to be undertaken. The guidance in this International Standard describes the application of screening methods, and how they might be used for assessing soil quality. Notwithstanding some of the issues raised, screening methods can generate robust and reliable results which can be used with confidence.

NOTE Although soil screening methods are most commonly used to determine contaminants (pollutants) in soils, for example in site investigations, they can also be used to determine parameters in uncontaminated soils (e.g. agricultural soils.) The use of the word “contaminant” in this International Standard can equally apply to any relevant soil parameter.

Soil quality — Guidance on the selection and application of screening methods

1 Scope

This International Standard provides guidance on the selection and application of screening methods for assessing soil quality. Guidance is given to choose an appropriate screening method for a specific parameter and defines the conditions under which they can be used.

This International Standard does not recommend any particular screening method, but confirms the principles of their selection and application.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11074, *Soil quality — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11074 and the following apply.

3.1

screening method

method which is used (often on-site) to quickly explore a given area or test a set of samples and obtain data on soil quality

3.2

laboratory reference method

laboratory-based method which is performed in accordance with National or International Standards and is not necessarily comparable with screening methods

NOTE A laboratory reference method may be recognized nationally or internationally and is performed within a qualified laboratory. Its results are not necessarily comparable with those of screening methods.

4 General

4.1 Introduction

This International Standard describes a framework for selection and application of screening methods.

It defines the whole process, from the selection of the screening method, the applicability and fit-for-purpose testing, the fulfilling of the acceptance criteria, the quality control of the applied method, to the documentation of measurement results.

The suitability of any particular screening method depends on the parameter or group of parameters requiring determination and on the technical nature of the method.

Screening methods can be classified in various categories as outlined below.

4.2 Typical areas for application of screening methods

Screening methods may complement a laboratory reference method, but may also be used alone where a definitive decision can be made using the screening method. Screening methods can be used to set priorities for a site investigation or facilitate the design of a sampling plan. Even allowing for the higher uncertainties of measurements generally associated with such methods, they may be used to quickly obtain an indication of, for example, the quality of soil or the concentration of parameters of interest present on a site, including those areas showing very high levels, and those areas showing low levels. Knowledge of these results can be used quickly and decisions taken directly after the measurements are made to identify specific site locations where further sampling and analysis may be required, or to prioritize those samples that may need to be analysed using laboratory reference methods.

Screening methods are often used to aid selection of those samples that are to be determined in a laboratory and those that are not. In addition, they can be used as an indicator that may suggest those locations where further investigation or follow-up action needs to be taken. Screening method analysis can be carried out in one of three ways:

- a) at or very close to the sampling location on-site in the field;
- b) at or very close to the sampling location inside a dedicated test room or an area equipped with basic services such as electricity and water, for example, where non-portable equipment needs to be used;
- c) in a conventional laboratory.

Screening methods carried out in a dedicated test room, or an area equipped with basic services, usually result in better quality than those carried out on-site. In addition, any unusual or unexpected result can quickly be repeated, if necessary.

Screening methods constitute a useful addition to standard procedures in the following areas.

4.2.1 Support of sampling/sample preparation processes

Screening methods may be used for the following:

- preselection of samples for analysis in the laboratory;
- selection of the most suitable analytical method (working range, specificity, robustness);
- provision of information relevant for sample preparation.

4.2.2 Monitoring of processes

Screening methods may be used:

- to monitor and control processes (e.g. remediation);
- to perform quality control on the operation of a soil remediation treatment plant.

4.2.3 Identification of homogeneity/heterogeneity of bulk material

Screening methods may be applied to measure “target compounds” in large amounts of soil and soil-like material to check the degree of homogeneity.

4.2.4 Survey of contaminated sites (hot-spot identification)

Screening methods are useful to identify contaminated areas in contamination-suspected sites. An example for contaminated sites is given in Annex A, Figure A.1.

4.2.5 Monitoring of large areas

Screening methods may be used for determination of the distribution of key parameters, e.g. nutrients in agricultural land.

4.2.6 Safety issue

Screening methods can be used to detect potentially hazardous compounds (e.g. gases, explosives) which may be harmful to the individuals taking and processing the samples.

5 Selection of a screening method

5.1 General objectives

Before the screening of a site (see 4.2) can be conducted, a thorough planning phase is necessary.

First, all information available about the site has to be evaluated. This may be historical records or data available from previous investigations. Essential prerequisites for the suitable preparation of a screening investigation is information about the hydrogeologic situation, the kind of contaminants and parameters of interest and the concentrations or values likely to be expected, as well as any information about the locality, including the former use of the site.

Furthermore, the infrastructure of the site and the accessibility may need to be taken into consideration.

With this background information, data quality objectives have to be defined, that determine the applicability of the screening method. Only with these preliminary steps is the selection of screening methods possible.

Some examples of detailed questions are listed below. This list is not exhaustive and not all might be relevant for a specific site:

- parameters and analytes of interest;
- matrices of interest and condition and variability of matrix;
- data quality objectives;
- parameter values known, expected or already found on-site;
- general spatial extent of relevant parameters;
- ease of sampling;
- site facilities;
- site area;
- number of results per time unit;
- health and safety considerations.

5.2 Selection criteria

The following criteria should be taken into consideration when selecting the appropriate analytical method. The decision-making process and the results have to be documented by the user.

5.2.1 Sampling/sample pretreatment/preparation

Sampling/sample pretreatment/preparation:

- direct measurement [e.g. handheld (X-ray-fluorescence systems) allow direct measurement with limited sampling/sample pretreatment/preparation];

- pretreatment/preparation (e.g. extraction, separation);
- particle size and homogeneity.

5.2.2 Parameter definition

Parameter definitions are as follows:

- total content (e.g. chromium, benzene);
- individual species [e.g. Cr^{3+} , Cr^{6+} , Fe^{2+} , Fe^{3+} , volatile organic carbon (VOC)];
- sum parameters [e.g. total organic carbon (TOC), adsorbable organically bound halogens (AOX)].

5.2.3 Method characteristics

Method characteristics are as follows:

- sensitivity, selectivity, accuracy value (e.g. limit value, target value);
- working range;
- limit of detection;
- matrix interferences;
- method limitations/interferences.

5.2.4 Boundary conditions

Boundary conditions are as follows:

- rapidity (in relation to aim of determination);
- mobility;
- costs;
- quality target of analysis;
- frequency of use (continuous, once only);
- competence of staff;
- legal requirements;
- availability and/or ease of acquisition of the necessary equipment;
- infrastructural conditions.

The criteria shall be weighted differently depending on the intended application.

5.3 Fit-for-purpose test

In a second step, after passing the selection steps in 5.2, the selected method has to pass a fit-for-purpose test as described in Clause 7.

5.4 Quality targets

A documented procedure for the application of a screening method and for all associated quality-control measurements shall be available to the person undertaking the test. Only after such tests have been carried out and which demonstrate the intended sensitivity and stability, should field measurements be undertaken.

Applying control charts to the results of such tests, over a long period of time, should demonstrate the performance of the screening method and indicate whether it is acceptable or not.

6 Application and applicability of a selected screening method

6.1 General

This clause deals firstly with the requirements of applying a screening method, secondly, with the quality assurance aspects during application, and finally with the applicability of a screening method.

After a screening method has been selected and validated, it has to be decided if the method is fit for the purpose and can be applied for a certain project.

NOTE Selection, validation and assessing the applicability might be separate processes. However, these processes might also be carried out in parallel, i.e. for some applications, it might be necessary that further validation be required and that the selection depends on the results of the validation tests for a certain application.

Where there is little or no useful information on the application of a particular screening method for a particular on-site investigation, it is necessary to demonstrate that the screening method is suitable for use on the site.

6.2 Screening method requirements

Before use, a decision needs to be made regarding why and for what purpose the results need to be generated. The following factors should be considered and taken into account. As far as the requirements are related to chemical screening methods, many of these factors also apply to laboratory reference methods.

- a) A clear and unambiguous definition of the parameter, group of parameters or property being determined by the screening method needs to be available.
- b) A clear description of the response measured, and, if necessary, why and when this result can be used to give an estimate of a particular parameter concentration.
- c) The matrices or field situations which can be tested using the screening method shall be documented, and procedures on the handling and reporting of extraneous material found during the sampling process shall also be documented.
- d) The required limit of detection, if appropriate, and whether the screening method can achieve this requirement shall be defined. For contaminated areas where high levels of contaminants are detected, this may not be an issue.
- e) The critical level of interest for each parameter or group of parameters needs to be known before any analysis is undertaken, irrespective of whether this is a concentration value or a presence or absence requirement.
- f) Any required performance criteria prescribed for the parameter or group of parameters needs to be known; this will include an estimate of the result uncertainty.
- g) The major sources of potential interferences that affect the use of the screening method. Therefore, selectivity should be addressed during the validation process, see Clause 5.
- h) A clear concept should be worked out on how the screening data acquired are integrated into the overall assessment process.

6.3 Screening method applicability

6.3.1 General

Usually, the performance of a screening method is established under typically ideal conditions. However, during routine operation, performance may be affected by the test conditions under which the method is used. This includes for example, the environmental site conditions, such as temperature, humidity, and other

extreme weather conditions. The performance of the method is also affected by the capability and experience of the person using the screening method. As a consequence, it may be very difficult to achieve “typical” performance data. Test-kit screening method manufacturers shall, upon request, provide some data on method performance. However, these data may relate to matrices that are not relevant to the specific application or site investigation. In these cases, the users of the method shall demonstrate that the screening method is suitable for the matrix being analysed, that they can use it in a satisfactory manner and produce results of acceptable quality, if necessary by testing the method using appropriate reference samples.

The range of applications over which a particular screening method may be used shall be known, and shall be suitable for the expected concentrations likely to be determined for the site under investigation.

The following criteria have to be considered:

- parameters and analytes (e.g. oxidation state of ion);
- measurement range/graduation; “zero” may not be stated for the lower limit of the operating range;
- matrix;
- matrix interferences, measures to be taken for their prevention or elimination;
- temperature range, pH range, other physical conditions;
- storage and shelf-life of the reagents.

6.3.2 Principle of the measurement

Chemical reaction or physical concept.

6.3.3 Instruction for method setup

The instructions for method setup are as follows:

- description of supplied reagents (e.g. composition, indications of danger);
- description of supplied equipment, such as test vessel, metering device or colour scale;
- description of how and with which measuring instrument the evaluation may be performed;
- additional reagents required for the application (e.g. acid for pH adjustment);
- additional equipment required for the application (e.g. thermo-reactor for chemical oxygen demand).

6.3.4 Sampling and samples

A description of the following shall be given:

- description of sampling and of sample preparation;
- description of sample quantity and volume.

6.3.5 Performing the measurement

The following measurement steps shall be considered:

- health and safety precautions;
- handling;
- step-by-step (pictogram);
- introduction of the sample into the test equipment;

- reaction time (interval);
- ascertainment of results;
- cleaning and maintenance .

6.3.6 Statement of results

The following statement of results shall be considered:

- number of figures after the decimal point;
- precision/accuracy;
- conversion table;
- conversion factors.

6.3.7 Sample and reagent disposal instructions

The following disposal instructions should be considered:

- waste, waste water, hazardous waste;
- spent reagents employed;
- return of the spent reagents and any associated residual sample to the kit/reagent manufacturer for suitable clean up, disposal and potential recycling.

6.3.8 Characteristic data of the method

Characteristic data of the method are as follows:

- sensitivity, specificity, robustness, accuracy, linearity of calibration, working range;
- certified reference materials with certificates, other reference products like in-house materials, control standards, interlaboratory comparison samples.

6.3.9 Literature references

The literature references for the following should be given:

- description of procedure;
- additional information, examples of possible applications.

7 Fit-for-purpose evaluation

7.1 General

In general, fit-for-purpose evaluation means providing information about whether a method of choice gives results that are related to the corresponding reference method. The intensity and type of fit-for-purpose testing depends on the quality targets defined according to 5.4 and the type of screening technique used.

Three modules of testing may be applicable:

- evaluation of accuracy, trueness and precision (see ISO 5725-2^[4]);
- avoiding false negative results;
- evaluation of individual comparability to reference methods.

These three modules can be combined depending on the quality target. Reproducibility testing is always required the first time a special test application is introduced.

Reproducibility testing can easily be combined with the testing of individual comparability.

In case the manufacturer of the screening test provides data or other users' published data on successful fit-for-purpose tests under comparable conditions are available, these data may be referred to and may reduce the effort of the current test.

7.2 Accuracy evaluation

One (or more) typical, homogenized samples (preferably certified reference materials or measured by a reference method, containing known amounts of analytes) is analysed repeatedly (at least three times) to access the precision and trueness of the screening method. The data set is evaluated according to the quality target. The result of this testing can be used to express the precision of the method.

In the case of screening methods which give only a concentration range or yes/no-results, the test should provide information about whether replicate measurements of the screening method always give the same range or the same yes/no-information.

7.3 Exclusion of false negative results

In many cases of application, the screening method is used to preselect samples. It is then important that the screening method does not give false negative results. False positives are not critical as, in these cases, a control by a reference method provides clarification.

In order to test the probability of false negative results, a test scenario according to the quality targets shall be designed.

Firstly, the accuracy shall be derived from recent measurements of reference samples (see 7.2). Alternatively, a typical set of samples covering the expected range of application is prepared and measured using the reference method and the screening method. The data are evaluated to derive the accuracy and the probability of false negative response.

The data are evaluated under consideration of the precision. The probability of false negatives is calculated.

The number of comparison samples analysed depends on the required accuracy of the test.

In the case of screening methods which only give a concentration range or yes/no-results, the test should provide information on how many false negatives the repeated measurements of the screening method results in.

7.4 Testing of individual equivalence

One (or more) typical homogenized samples are prepared and analysed both by the screening method and the reference method (six complete approaches). At least six sample replicates should be analysed.

8 Analytical acceptance criteria

8.1 General

After a screening method has been proven to meet the given acceptance criteria, it may be used for the defined purpose. Some of these criteria are to be checked before using the test (starting criteria, 8.2); others have to be continuously checked during the use of the method.

8.2 Starting criteria

The starting criteria are as follows:

- selection process according to Clause 6 successfully completed and documented;
- evaluation criteria according to Clause 7 successfully evaluated and documented;
- quality targets are defined according to an analytical task and documented;
- individual fit-for-purpose scenario designed, successfully passed and documented;
- quality assurance (QA) measures and corresponding QA-acceptance criteria should be fully defined (Clause 9) and clearly documented.

8.3 Continuous criteria

Periodically, monitoring of QA-acceptance criteria and an appropriate system-suitability check are carried out.

In the case of deviations from the QA-criteria, measures have to be taken and documented. Use of the method may continue after recovering QA-criteria.

9 Quality assurance

In order to provide confidence in the results generated, the following factors should be considered and taken into account. Many of these factors also apply to laboratory reference methods.

- a) All staff applying the screening methods shall be suitably trained. Details of the training shall be documented.
- b) Details of the screening method shall be fully documented and adhered to when carrying out the test, and be relevant to the particular application or site investigation being undertaken. This includes any sampling procedures, and subsequent sample preparation and/or pretreatment carried out. These details shall be available to the person undertaking the analysis using the screening method. The level of validation shall be documented to show the performance of the screening method for providing qualitative, semi-quantitative or quantitative data.
- c) The type and number of quality control samples, that shall be analysed to demonstrate that the analysis remains under control, shall be fully documented together with the acceptable range of measurement. These control samples may depend upon the specific application of the screening method, e.g. whether a presence or absence test is being undertaken, or whether a concentration is being determined. In addition, the type of quality control samples, i.e. whether they comprise known standard solutions, certified reference matrix materials, blank solutions, etc., may depend on specific applications and their availability. The use of quality control charts should be used where possible. Where the analysis is not under control, the cause should be identified and remedial action taken and recorded. Applying control charts for a reference or control material provides good documentation of instruments' performance.
- d) The use and documentation of (certified) reference material measurements is important to demonstrate traceability. If no such material is available, appropriate samples can be prepared in a laboratory and used as control materials in the field. Only when the instrument characteristics are verified in the field, can the results of sample measurements be accepted. Measurements of quality control samples should be documented to ensure traceable results.
- e) The organization performing the screening methods should participate in appropriate and relevant proficiency-testing schemes. However, it is recognized that some screening methods are not suitable for certain proficiency-testing schemes that operate mainly for laboratory reference methods.
- f) A written procedure for the user of the method shall be available, regarding recalibration procedures, verification of analytical signals and results of reference materials. Only after the instrument meets the documented system-suitability checks, can the field measurements be processed. The stability (drift) of the instrument shall be checked using appropriate materials.

- g) Health and safety considerations shall be assessed with respect to the use of the screening method, especially when undertaken on-site. In addition, the disposal of resulting waste material shall be carried out in accordance with documented procedures.

10 Documentation

The application of this International Standard enables a qualified decision to be made regarding the most appropriate method of analysis for the task at hand. At the same time, however, the decision-making process shall be transparent and verifiable. For this reason, thorough documentation is especially important, starting at the beginning of the test and lasting until assessment of the analytical results. Systematic documentation provides objective proof of the quality of analysis.

The minimum requirements for documentation include the following:

- presentation of decision criteria in accordance with Clause 6;
- documentation of quality assurance measures;
- documentation of the qualifications of decision makers and personnel performing the analysis;
- sampling record;
- written report of the analysis, including
 - indication of measured values with clear identification of samples,
 - indication of the product used,
 - deviations from the operating procedure, if applicable,
 - assessment of results, and
 - pretreatment.

Annex A (informative)

Example for selection and application process of screening methods

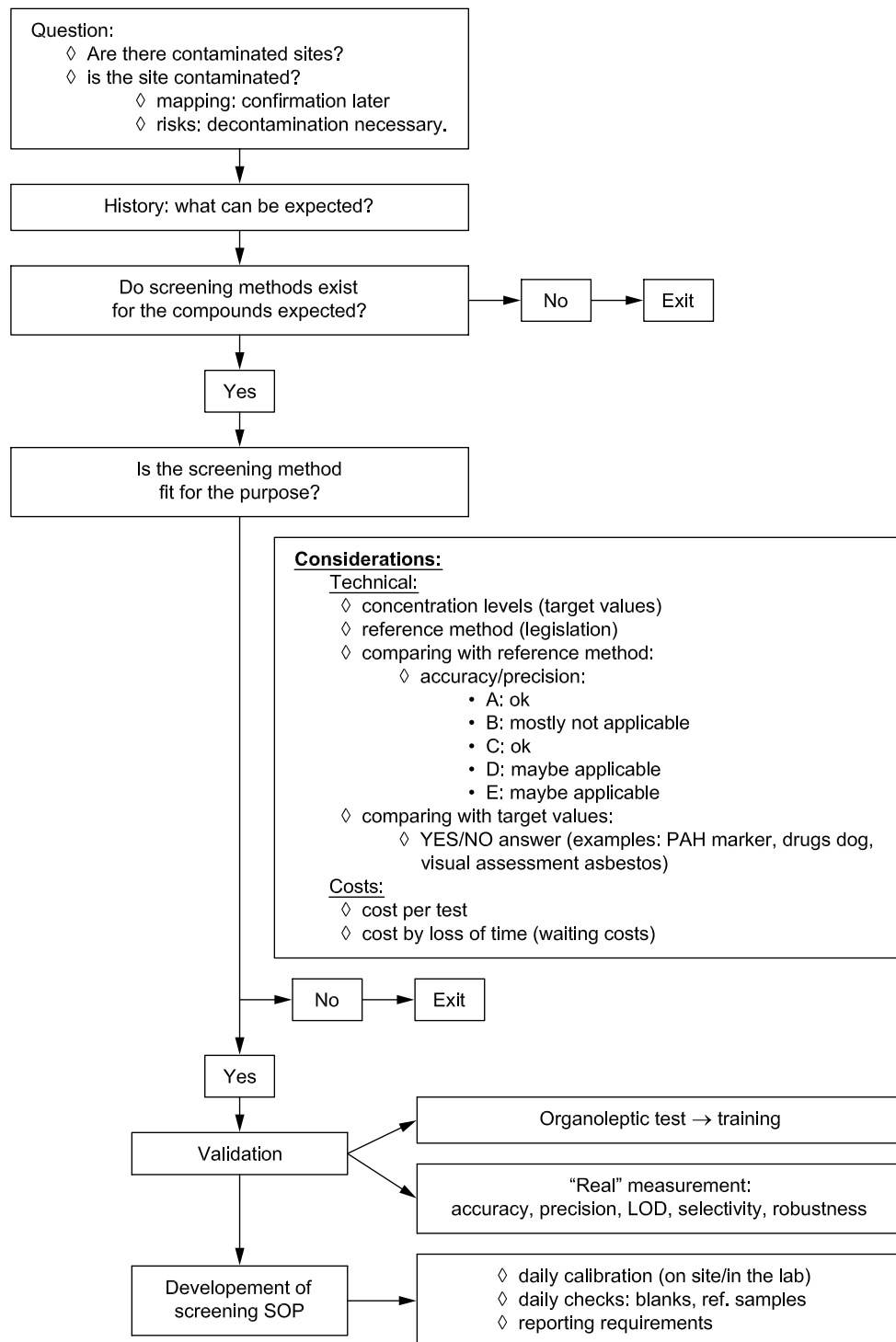


Figure A.1 — Example for selection and application process of screening methods

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- [4] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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