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

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National foreword

This British Standard is the UK implementation of EN ISO 12404:2021. It is identical to ISO 12404:2021. It supersedes BS EN ISO 12404:2013 and BS EN 16123:2013, which are withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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European foreword

This document (EN ISO 12404:2021) has been prepared by Technical Committee ISO/TC 190 "Soil quality" in collaboration with Technical Committee CEN/TC 444 "Environmental characterization of solid matrices" the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2021, and conflicting national standards shall be withdrawn at the latest by December 2021.

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

This document supersedes EN 16123:2013 and EN ISO 12404:2015.

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Endorsement notice

The text of ISO 12404:2021 has been approved by CEN as EN ISO 12404:2021 without any modification.

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, SC 3, *Chemical and physical characterization*.

This second edition cancels and replaces the first edition (ISO 12404:2011), which has been technically revised.

The main changes compared to the previous edition are as follows:

— The contents of ISO 12404:2011 and EN 16123:2013 were merged;

— The scope was widened to include waste;

— The document was developed parallel with CEN according to the Vienna Agreement;

— The text was editorially revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document provides guidance on the use of screening methods for soil, soil-like materials and waste characterization. Most of the following clauses are applicable to all matrices mentioned. However, a few subclauses are specific to either waste or soil, including soil-like material, only.

One field of application of screening methods is "on-site verification" as recommended in the European Landfill Directive (1999/31/EC) and the Landfill Decision (2003/33/EC).

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 parameter such as a concentration or value, or generate a semi-quantitative result.

Screening methods are applicable for processes such as entrance control at waste disposal sites in conjunction with standardized methods, because they allow fast verification of the documented waste characteristics. They can also be used in similar way when soil or soil-like materials are to be reused in accordance with the guidance in ISO 15176.

Regarding soil, 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-based) 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. In waste investigation, the location of samples is limited to an area where waste is dumped but confirmation of the spatial distribution is still one of the investigation purposes, especially when investigating soil-like material.

The use of screening methods usually increases the efficiency of a site investigation. Generally, many more samples can be analysed or checked and screened for target parameters and results generated faster than using conventional laboratory-based reference methods. Additionally, 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. Any required performance criteria prescribed for a parameter or group of parameters need to be known; this should include an estimate of the uncertainty of the results.

NOTE Although soil screening methods are most commonly used to determine contaminants (pollutants) in soils, for example in investigations of potentially contaminated sites, they can also be used to determine parameters in uncontaminated soils (e.g. agricultural soils). Thus, the word "contaminant" in this document can be construed to apply in any particular context to any relevant soil parameter (e.g. chemical, physical, biological).

INTERNATIONAL STANDARD ISO 12404:2021

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

1 Scope

This document provides guidance on the selection and application of screening methods for assessing soil quality and waste characterization, including distribution of target parameters in soil and soil-like material. The aim of this document is to set out criteria as to when the different kind of screening methods can be applied for the analysis of a certain parameter in soil, including soil-like material, and waste, and which steps are required to prove their suitability.

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

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
screening
application of any analytical semi-quantitative method for exploratory analysis

3.2
screening method
method which is used (often on-site) to quickly explore a given area including target parameter distribution or to test a set of samples and obtain data on sample characteristics

Note 1 to entry: It is not necessarily directly comparable with reference methods.

3.3
reference method
method which is performed in accordance with national or international standards

3.4
on-site verification
inspection to ensure that the waste accepted at a landfill is the same as described in the accompanying documents and that it is in accordance with the basic characterization and/or compliance testing

Note 1 to entry: Procedures can be found in the European Landfill Directive (1999/31/EC) and the Landfill Decision (2003/33/EC).

4 Principles

This document specifies 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.

5 Typical areas for application of screening methods

5.1 General

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

5.2 Support of sampling/sample preparation processes

Screening methods can be used for:

— selection of the most suitable analytical method (concentration range, interferences, specificity, robustness);

— pre-selection of samples for analysis in the laboratory;

— provision of information about accompanying compounds relevant for sample preparation.

5.3 On-site verification

Characteristics of sampled waste are verified, e.g. during transport or at the entrance of waste treatment plants and landfills.

5.4 Monitoring of processes

Screening methods can be used:

— to monitor and control processes (e.g. success of treatment or remediation);

— to perform quality control on a treatment plant.

5.5 Identification of homogeneity/heterogeneity of bulk material

Screening methods can be applied to measure "target compounds" in large amounts of waste as well as soil and soil-like material to check the degree of homogeneity.

5.6 Survey of contaminated sites (hot-spot identification)

Screening methods are useful to identify contaminated areas in contamination-suspected sites. Examples for the application to contaminated sites are given in Annex A (flowchart) and Annex B (hot spot detection).

5.7 Identification of sources of contamination

Screening methods can be useful to identify the source of a contaminant (hot spot detection) and its distribution or contamination variability in a material stream or stock-pile.

5.8 Monitoring of large areas

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

5.9 Safety issues

Screening methods can be used to detect potentially toxic compounds (e.g. gases, radioactivity, explosives) which could be hazardous to the personnel taking and processing samples.

6 Selection of a screening method

6.1 General objectives

Before the screening of a site can be carried out, a thorough planning phase is necessary.

First, all information available about the site should be evaluated, often by conducting a preliminary investigation such as desk study and site reconnaissance following, for example, ISO 18400-202. This may include historical records or data available from previous investigations. Essential prerequisites for the suitable preparation of a screening investigation is information about the hydrogeological situation, the kind of contaminants and/or 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 might need to be taken into consideration.

NOTE 1 Further additional steps can be considered:

— development of a conceptual site model (see e.g. ISO 21365);

— development of a suitable sampling strategy (see e.g. ISO 18400-104, and ISO 18400-203 or ISO 18400-205, as appropriate);

— preparation of a sampling plan (see e.g. ISO 18400-101).

NOTE 2 Further information relevant to development of sampling strategies can be found in e.g. ISO 11504, ISO 15175, ISO 15176, ISO 15799, ISO 15800 and ISO 19258.

When the target field is waste, a similar approach is advised. Information about sampling strategies and planning can be found in e.g. CEN/TR 15310, Part 1-5.

NOTE 3 For full titles of the documents listed above, see Bibliography.

Taking into account this background information, data quality objectives should be defined that determine the applicability of the screening method. Only with these preliminary steps the selection of screening methods is 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 (see 6.2 for the details);

— parameter values known, expected or already found on-site;

— statistical probability;

— ease of sampling;

— site facilities;

— site area;

— number of results per time unit;

— health and safety considerations.

An example for the selection and application process of screening methods to soil contamination is given in the informative Annex A.

An example for typical results on finding hot spots by screening methods is given in the informative Annex B.

6.2 Data quality objectives (DQOs)

NOTE The data quality objectives (DQOs) process refers to a systematic planning procedure for environmental data collection so that the data can be defensibly interpreted, and statistically analysed where appropriate, to address specified objectives. It includes appropriate sampling design and sampling plans, as well as the analytical strategy and setting analytical data objectives[21]. The concept applies not just to numerical data, but also to the sufficiency of all relevant information in terms of quantity, quality and type.

In land contamination investigations there are typically DQOs that require laboratory analytical data to be meaningfully comparable to a variety of risk-based or other quality criteria.

DQOs should be set for all investigations. They should be defined for the specific purpose of the site investigation phase or activity, e.g. risk-based assessment (human health and controlled waters), remediation, validation or waste classification.

When setting DQOs, account should be taken of the type, quantity and quality of the data required to inform subsequent decisions based on the data and other available information.

DQOs should be set having regard to QA/QC (Quality Assurance/Control) requirements and how comparison with risk-based or other criteria is to be effectively supported by the site data and information as well as the laboratory analytical data collected.

Review points should be identified at key stages throughout the investigation, assessment and remediation design phases to ensure that DQOs remain aligned to the project requirements. The review should include assessment of the continuing validity of the conceptual site model, data consistency, emerging data gaps and levels of uncertainty. A written record of the review should be maintained and incorporated in the assessment and design process and include a statement on whether the DQOs have been met and any shortfalls within the assessment.

6.3 Selection criteria

6.3.1 General

The following criteria should be taken into consideration when selecting the appropriate screening method. The different criteria should be weighted depending on the intended application. The decision-making process and the results should be documented by the user (see flowchart in Annex C and documentation aid in Annex D).

Prerequisites are:

— one known parameter or a set of known parameters;

— aim of determination;

— matrix (soil, soil-like materials, solid or liquid waste).

Whenever a sample is collected from waste, the sample source of waste is obvious. In the case of a sample collected from the ground, if the sample has no link with information on sampling location, screening application has no meaning. Even with soil-like material, samples are to be linked with sampling location in the same way.

6.3.2 Sampling/sample pre-treatment/preparation

Sampling/sample pre-treatment/preparation can include:

— direct measurement [e.g. (handheld) x-ray-fluorescence systems allow direct measurement with limited sampling/sample pre-treatment/preparation];

— pre-treatment/preparation (e.g. extraction, separation);

— particle size and homogeneity.

Most screening methods require the provision of the analyte in an extract/eluate, which therefore requires sample pre-treatment. Pre-treatment needs to be carried out in accordance with the relevant standards.

6.4 Checks for the selection of candidate methods

6.4.1 General

A candidate screening method should meet predefined requirements in terms of:

— parameter definition;

— field of application;

— method characteristics;

— boundary conditions.

6.4.2 Parameter definition

Possible parameter definitions include:

— total content (e.g. chromium, benzene);

— individual species (e.g. Cr^{3+}, Cr^{6+}, Fe , Fe^{3+}, volatile organic compounds);

— group parameters [e.g. total organic carbon (TOC), adsorbable organically bound halogens (AOX)].

In the case of on-site verification, the parameters are typically defined by declaration or based on the experience of the staff.

6.4.3 Field of application

Fields of application are as follows:

— specified decision value (e.g. limit value, target value);

— concentration range;

— matrix;

— method limitations/interferences.

6.4.4 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.

6.4.5 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 should be weighted differently depending on the intended application.

6.5 Fit-for-purpose test

In a second step, after passing the selection steps in 6.3, the selected method needs to pass a fit-for-purpose test as described in Clause 8.

In case of frequently repeated tasks, the most suitable screening method should be identified and applied, the necessary equipment kept ready and the procedure documented in a standard operating procedure. Selection and fit-for-purpose testing should therefore be performed only once.

6.6 Quality targets

The general quality target of analytical questions is its ability to establish the relationship between the analytical result and its confidence interval on the one hand, and the decision values on the other. This relationship with the decision values means that the analytical method to be used is subject to requirements regarding the quality of the analytical results. These requirements are task-related and should be defined before the screening method is applied. The definition of these quality targets forms the basis for the selection of the appropriate method.

A documented procedure for the application of a screening method and for all associated quality-control measurements should be available to the person undertaking the test. Only after tests have been carried out 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.

7 Applicability conditions for screening methods

7.1 General

Availability of complete information on a screening method of choice is a prerequisite to a decision about its applicability for a particular project, i.e. whether the method is fit for the purpose.

This clause provides guidance on the information required in order to judge potential applicability. This information can be contained in documentation accompanying a method (e.g. supplied by the manufacture of an instrument) or in other documentation. 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 by carrying out appropriate investigations (i.e. fit for the intended purpose).

All information, either supplied or separately obtainable (enclosed leaflet, application documents, etc.) should be easily comprehensible and written in clearly understandable language avoiding use of unnecessary jargon.

NOTE 1 Selection, validation and assessing the applicability might be separate processes. However, these processes can also be carried out in parallel, i.e. for some applications, it can be necessary that further validation be required and that the selection depends on the results of the validation tests for a certain application.

NOTE 2 The performance of a screening method is usually established under typically ideal conditions. However, during routine operation, performance can be affected by the test conditions under which the method is used and also by the capability and experience of the person using the screening method. For detailed guidance, see 7.3.1.

7.2 Performance requirements

Judgement about the applicability of a screening method in a particular situation requires:

— knowledge about why the results are needed, i.e. the purpose of the investigation;

— a set of performance requirements including data quality objectives (6.2).

It is necessary to:

a) have a clear and unambiguous definition of the parameter, group of parameters or property to be determined;

b) have 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) know the matrices or field situations which can be tested using the screening method and procedures for the handling and reporting of extraneous material found during the sampling process;

d) know the required limit of detection, if appropriate, and whether the screening method can always achieve this requirement (however, for contaminated areas where high levels of contaminants are detected, this may not be an issue);

e) know the critical level of interest for each parameter or group of parameters before any analysis is undertaken, irrespective of whether this is a concentration value or a presence or absence requirement;

f) know any required performance criteria prescribed for the parameter or group of parameters (this will include an estimate of the result uncertainty);

g) know the major sources of potential interferences that affect the use of the screening method (therefore, selectivity should be addressed during the decision process. See Clause 6);

h) have a clear concept about how the screening data acquired are to be integrated into the overall assessment process.

Detailed information about the requirements in terms of a range of aspects about which information is required relating to the applicability and application of screening methods is provided in 7.3.2 to 7.3.9.

NOTE As far as the requirements are related to chemical screening methods, many of these factors also apply to laboratory reference methods.

7.3 Screening method applicability

7.3.1 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 should, upon request, provide some data on method performance. However, these data could relate to matrices that are not relevant to the specific application or site investigation. In these cases, the users of the method should demonstrate that the screening method is suitable for the matrix being analysed, and that they can use it in a satisfactory manner to 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 can be used should be known, and should be suitable for the expected concentrations likely to be determined for the site under investigation.

The aspects in 7.3.2 to 7.3.9 should be considered.

7.3.2 Measurement conditions

The following conditions should be confirmed for the measurement:

— 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.

7.3.3 Principle of the measurement

The measurement principle of the screening method should be either based on a chemical reaction or a physical concept.

7.3.4 Instruction for method setup

The following should be confirmed for instruction of method setup:

— supplied reagents (e.g. composition, indication of hazards);

— supplied equipment, such as test vessels, metering devices or colour scales;

— how and with which measuring instrument the evaluation can be performed;

— additional reagents required for the application (e.g. acid for pH adjustment);

— additional equipment required for the application (e.g. thermo reactors for chemical oxygen demand).

7.3.5 Sampling and samples

The following information on sampling and samples should be confirmed:

— sampling including the location, especially in soil and soil-like material investigation, and of sample preparation;

— sample quantity and volume.

7.3.6 Measurement steps

The following measurement steps should be considered:

— health and safety precautions;

— handling; step-by-step (pictogram), introduction, training;

— introduction of the sample into the test equipment;

— reaction time (interval);

— ascertainment of results;

— cleaning and maintenance instructions.

7.3.7 Statement of results

The following should be considered for the selected method:

— number of figures after the decimal point;

— precision/accuracy;

— conversion table/factors;

— recommended methods for assessment of results.

7.3.8 Sample and reagent disposal

The following should be considered:

— the form of the waste (e.g. waste water, hazardous waste);

— what spent reagents there will be;

— the potential for return of the spent reagents and any associated residual sample to the kit/reagent manufacturer for suitable clean up, disposal and potential recycling.

7.3.9 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.

8 Fit-for-purpose evaluation

8.1 General

In general, fit-for-purpose evaluation means proving whether a method of choice provides results that are sufficiently related to the corresponding reference method in the context of use. The intensity and type of fit-for-purpose testing depends on the quality targets defined according to 6.2 and the type of screening technique used.

Three aspects of testing are relevant:

— accuracy testing;

— exclusion of 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.

If the manufacturer of the screening test provides site-relevant data or other users' published data on successful fit for purpose tests under comparable conditions, these data may be referred to and thus reduce the amount of application-specific testing required.

If screening methods, rather than delivering discrete values, provide a concentration range or yes/no-results, the reproducibility and false negatives should be evaluated.

8.2 Accuracy testing

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.

8.3 Exclusion of false negative results

In many cases of application, the screening method is used to pre-select samples. In these cases, it is important that the screening method does not give false negative results. False positives are not critical as in these cases a control by reference method delivers clarification.

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

Firstly, the precision needs to be defined or derived from the actual precision (e.g. based on 8.2).

A set of typical samples covering the expected range of application is prepared (homogenized) and characterized by the reference method. All samples are also analysed by use of the screening method.

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

The number of samples analysed depends on the required accuracy of the test. Ten typical samples are considered the minimum.

In the case of screening methods that only give concentration range or yes/no-results, the test should deliver information regarding the number of false negatives of repeated measurements of the screening method.

8.4 Testing of individual comparability

One (or more) typical homogenized samples are prepared and analysed both by the screening method and the reference method (six replicates).

The results are statistically evaluated according to the statistical procedure described in Annex E.

9 Analytical acceptance criteria

9.1 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, 9.2); others also to be continuously checked during the use of the method.

9.2 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 10) and clearly documented.

9.3 Continuous criteria

Continuous monitoring of quality acceptance criteria.

Quality acceptance criteria should be continuously monitored. If deviations from the quality criteria appear, measures need to be taken and documented. Use of the method may continue after re-covering quality criteria.

10 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.

— All staff applying the screening methods should be suitably trained. Details of the training need to be documented.

— Details of the screening method need to 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 pre-treatment carried out. These details should be available to the person undertaking the analysis using the screening method. The level of validation needs to be documented to show the performance of the screening method for providing qualitative, semi-quantitative or quantitative data.

— The type and number of quality control samples, that should be analysed to demonstrate that the analysis remains under control, needs to be fully documented together with the acceptable range of measurement. The form of these control samples will depend on 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., could depend on specific applications and their availability. 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 an instruments' performance.

— 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.

— 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.

— A written procedure for the user of the method should 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 needs to be checked using appropriate materials.

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

11 Documentation

The application of this document 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 needs to 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:

— presentation of decision criteria in accordance with Clause 6;

— documentation of the qualifications of decision makers and personnel performing analysis;

— documentation of individual quality assurance measures;

— documentation of continuous quality assurance measures;

— sampling record;

— written report of the analysis, including:

  — indication of measured values with clear identification of samples;

  — indication of the equipment used;

  — deviations from the operating procedure, if applicable;

  — assessment of results;

  — pre-treatment.

Annex A
(informative)

Example for the selection and application process of screening methods to soil contamination

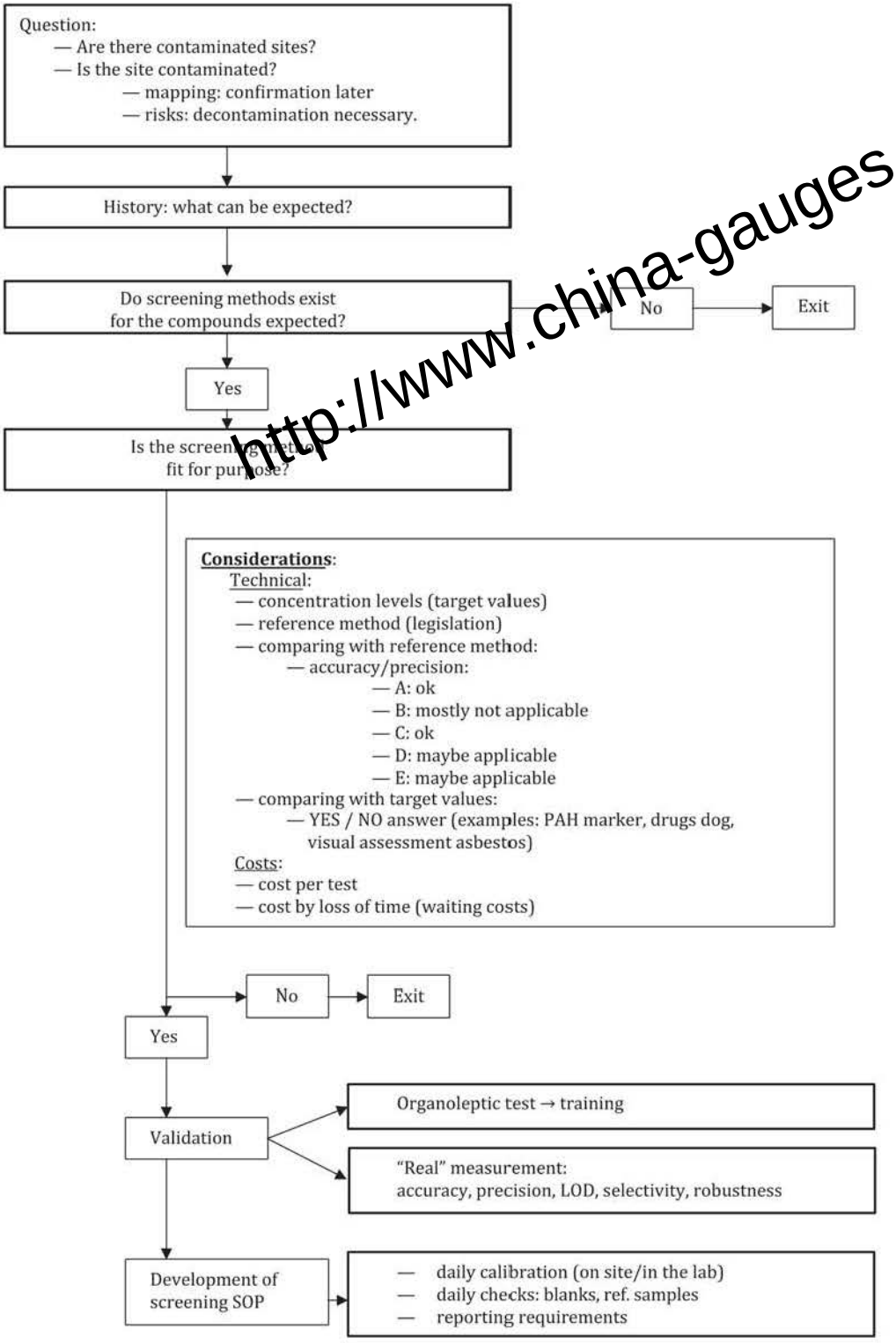
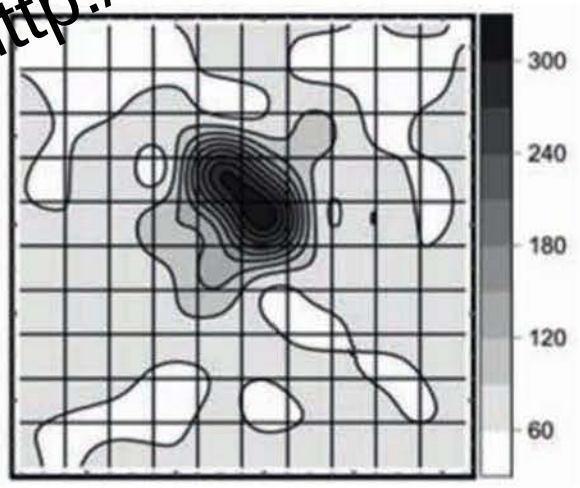


Figure A.1 — Flowchart of the selection and application process of screening methods

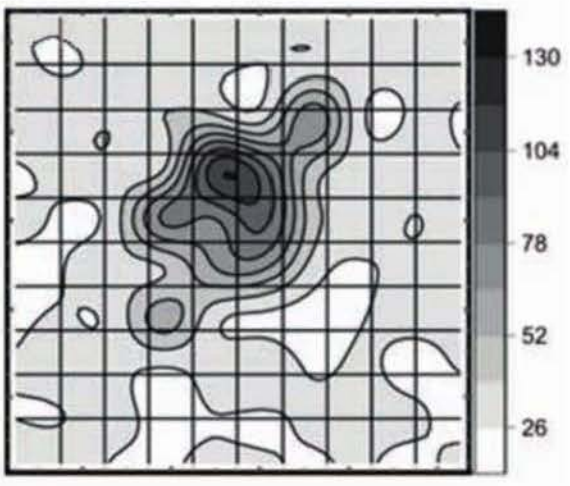
Annex B
(informative)

Typical results on finding hot spots by screening methods

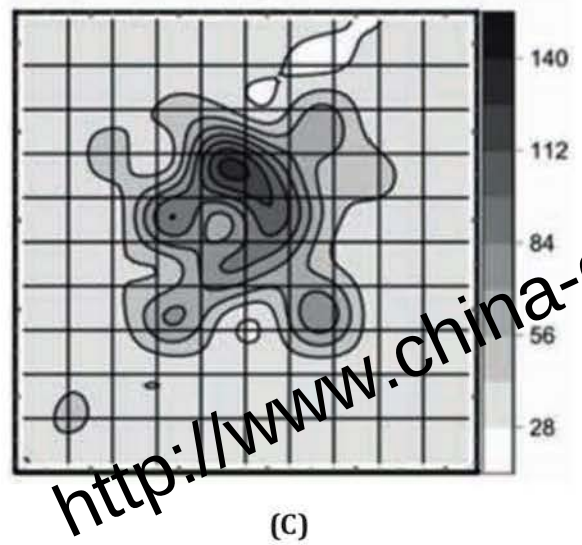
To track the source of a pollutant — in this example chromium — at a site, screening methods were applied in three detection ways, which are portable XRF (B) and hand-held XRF (C) (ISO 13196) as well as test-kit photometric detection (D) (ISO/TR 18105). Contour maps show the distributions of the total chromium (B and C) and water extractable chromium(VI) (D). The screening results were confirmed by a reference method (ISO 18227) using bench-top XRF indicated in A.



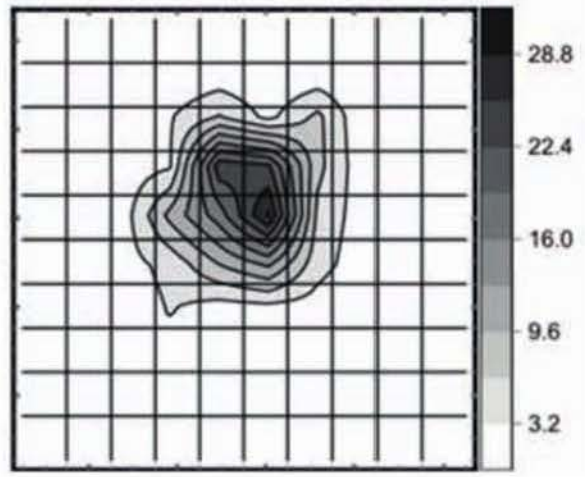
(A)



(B)



(C)



(D)

Figure B.1 — Results on screening soil to find the source of chromium at a site by applying screening methods, which were confirmed by a reference method

Annex C
(informative)

Decision making process

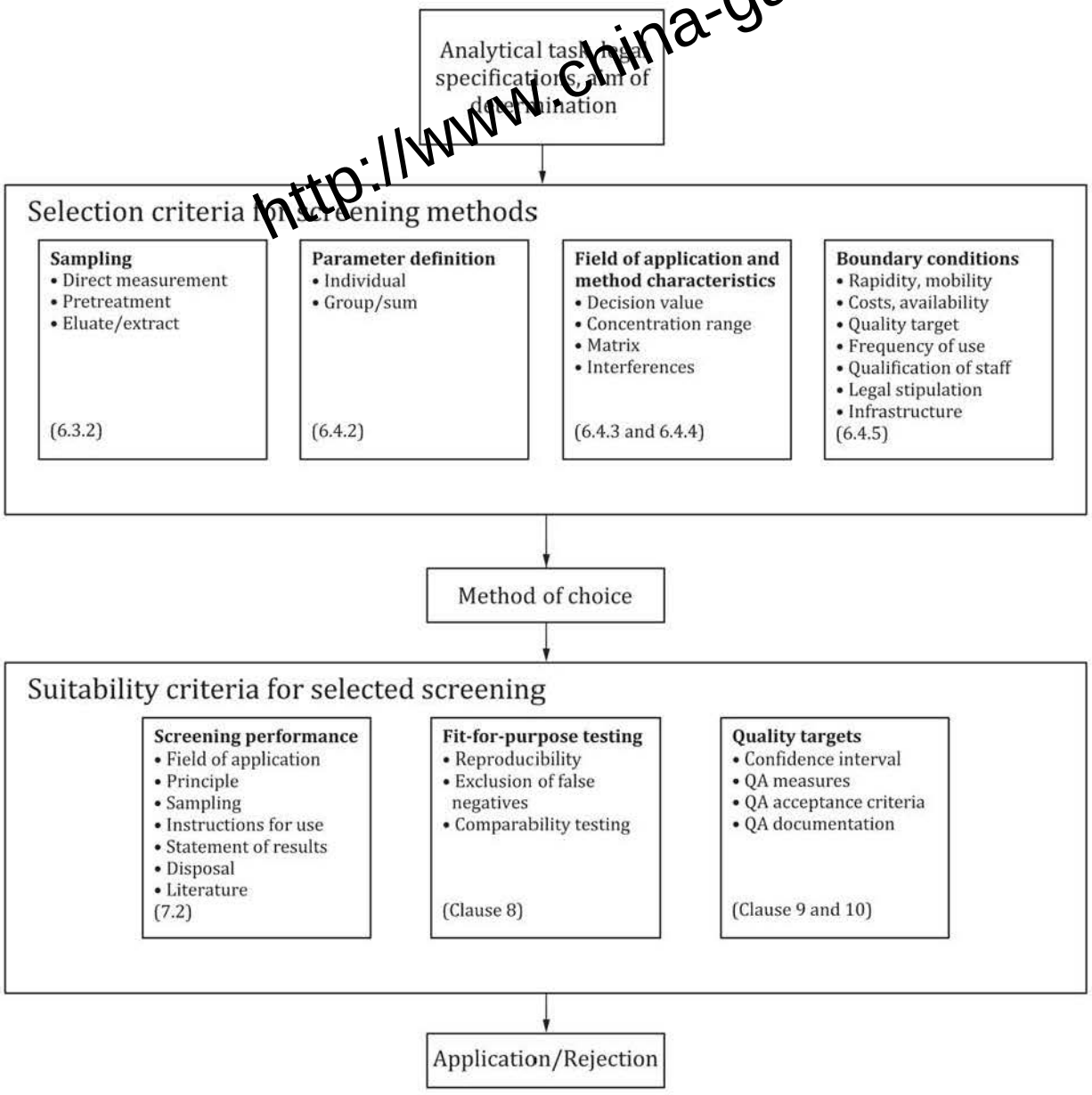


Figure C.1 — Flowchart of the decision-making process

Annex D
(informative)

Example of documentation aid/check list

Short description of analytical task: **Definition of the quality target:**

Selection criteria

Parameter definition

Relevance please tick	**Aspect**	**Applicability to method of choice**
☐	Specified analyte:	☐
☐	Group or sum parameter:	☐

Field of Application

☐	Decision value:	☐
☐	Concentration range:	☐
☐	Matrix:	☐
☐	Expected interferences:	☐
☐	Method limitations:	☐

Sampling, sample pre-treatment

☐	Direct measurement:	☐
☐	Sampling required:	☐
☐	Sample pre-treatment required:	☐
☐	Eluate/Extract required:	☐

Boundary conditions

☐	Rapidity (in relation to aim of determination)	☐
☐	Mobility	☐
☐	Costs	☐
☐	Quality target of analysis	☐
☐	Frequency of use (continuous, once only)	☐
☐	Qualification of staff	☐
☐	Legal stipulations	☐
☐	Availability and/or ease of acquisition	☐
☐	Infrastructural conditions	☐
☐	Others:	☐

Applicability conditions

relevant	condition	applied
☐	Temperature range, pH range, other physical conditions	☐
☐	Storage and shelf life of the reagents	☐
☐	Chemical reaction or physical concept	☐
☐	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	☐
☐	Description of sampling and of sample preparation	☐
☐	Description of sample quantity	☐
☐	Health and safety precautions	☐
☐	Step-by-step (pictogram), introduction, training	☐
☐	Reaction time (interval)	☐
☐	Ascertainment of results	☐
☐	Maintenance instructions	☐
☐	Number of figures after decimal point	☐
☐	Precision/accuracy	☐
☐	Conversion table, conversion factors	☐
☐	Recommended methods for assessment of results	☐
☐	Waste, waste water, hazardous waste	☐
☐	Return to the manufacturer	☐
☐	Calibration	☐
☐	Available certificate of analysis	☐
☐	Description of procedure	☐
☐	Additional information, examples of possible applications	☐

Fit-for-purpose testing

Relevance please tick	Test type	Result summary	Test passed
☐	Reproducibility		☐
☐	False negatives		☐
☐	Comparability		☐

Quality Assurance measures

Apply	Measure	Define measure and quality target:
☐	Measurements of standards and possible reference materials	
☐	Plausibility tests by standard addition	
☐	Comparative tests with reference methods	

Apply	Measure	Define measure and quality target:
☐	Inter-laboratory tests	
☐		
☐		

Acceptance criteria

☐ Selection process according to Clause 6 successfully completed

☐ Application criteria according to Clause 7 successfully evaluated

☐ Quality targets defined according to analytical task

☐ Individual Fit-for-purpose scenario designed, successfully passed

☐ Quality assurance measures and corresponding quality acceptance criteria defined (Clause 10)

Results:

Name of responsible organization: _____

Name of responsible person: _____

Date: _____ Signature: _____

Annex E
(informative)

Statistical tool for individual comparability — Equality of results from reference method and screening method: Mean value t-test for real samples

E.1 General

At least six sub samples of a representative homogenized material are analysed with both methods to be compared under the same conditions.

E.2 Test on outliers

The results of both series are checked according to Grubbs test on outliers. One outlier per series is accepted. The test value (G_p) according to Grubbs is calculated as follows (Formula C.1):

$$G_p = \frac{\left|X^* - \bar{X}\right|}{s} \tag{C.1}$$

where

G_p is the test value;

X^* is the single value (possible outlier);

$\bar{X}$ is the mean value of a series;

s is the standard deviation of a series.

The test value is compared with the tabled value rM ($f = N$; $P = 95$ %). Where the test value is higher than the tabled value, the individual result under observation has been identified as outlier — and shall be eliminated accordingly. Mean value and standard deviation are calculated by the remaining data again and the Grubbs test is repeated. In case of detection of another outlier, the data series is not valid and a new set of data shall be established (minimum number of valid data: 5).

E.3 Homogeneity of variances

The variances of both data series are checked according to the variances-F-test.

The test value is calculated according to Formula C.2:

$$G_p = \frac{(s_S)^2}{(s_R)^2} \tag{C.2}$$

where

G_p is the test value;

s_S is the standard deviation of the screening series;

s_R is the standard deviation of the reference series.

The result is compared with the corresponding tabled value for F-distribution ($f_v = N_v - 1$, $f_R = N_R - 1$; $P = 99$ %).

Where the test value is higher than the tabled value, a significant difference is proven. According to the current variations, no comparability is found. Where the test value is lower or equal to the tabled value, no significant difference is found; i.e. the variation of the results is equal.

NOTE Where the variance of the screening method is significantly lower than the variance of the reference method, the F-test is successfully passed.

E.4 Mean value t-test

After elimination of outliers, the mean values of both data series are compared according to the mean value t-test. The test value is checked by use of Formulas C.3 and C.4:

$$G_p = \frac{|\bar{X}_R - \bar{X}_S|}{s_d} \cdot \sqrt{\frac{N_R \cdot N_S}{N_R + N_S}} \tag{C.3}$$

and

$$s_d = \sqrt{\frac{(N_R - 1)\cdot s_R^2 + (N_S - 1)\cdot s_S^2}{N_R + N_S - 2}} \tag{C.4}$$

where

G_p is the test value;

$\bar{X}_R$ is the mean value of reference series;

$\bar{X}_S$ is the mean value of screening series;

N_R is the number of data in reference series;

N_S is the number of data in screening series;

s_d is the mean standard deviation from both series;

s_R is the standard deviation of the reference series;

s_S is the standard deviation of the screening series.

The mean standard deviation from both series is compared to the tabled value of the t-distribution ($f = N_R + N_S - 2$; $P = 99\ \%$).

Where the test value is higher than the tabled value, no equality of the compared data sets is given. Where the test value is lower or equal to the tabled value, the result of the test is equality. Where the variation of the screening method is significantly lower than the variation of the reference method, it is allowed to use s_R of the reference method instead of s_d in Formula C.3. Otherwise, a false non-equality may result. Depending on the quality target of the analytical task, the user may be willing to change probabilities of the tests mentioned above. In case of deviations, the argumentation shall be plausible and well documented.

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