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**Soil quality — Detection of water  
soluble chromium(VI) using a ready-  
to-use test-kit method**

*Qualité du sol — Détection du chrome(VI) soluble dans l'eau en  
utilisant un kit d'essai prêt à l'emploi*

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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](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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## Introduction

A test-kit method uses reagents stored in plastic or glass vials prepared for colorimetric detection of specific compounds soluble with water. There is a lot of varieties of test-kits developed mainly for the compounds in water samples based on well-known reactions with commonly-used and chemically stable reagents indicating constant magnitudes of intensities of the signals from target compounds. The common reactions employed for the test-kit usually give colour, by which the user can recognize the concentration level of the compound without absorption spectrophotometers even in the situation when such instruments are not available. Thus, the test-kit determination of the compound in water samples can be used to screen specific compounds in soil samples, if they are easily dissolved into water.

The reaction applied depends on the target compounds. For chromium(VI), a reaction is used, where 1,5-diphenylcarbazine is used, its isomer or derivatives. When other chemicals or derivatives are used, the performance is intended to be similar to that obtained with the original reagents. The test-kit consisting of the reagents has to be identified by confirming the spectrum including intensities at a specific wavelength. The spectrum shape should be similar to each other between those from test-kits and bulk reagents commonly used for absorptiometry in laboratories. The intensities and varieties of interferences to the test-kit method should be within those taking place to absorptiometry test methods with bulk reagents. The quality or purity of the reagents packed in test-kits should keep constant with a deviation around that for the bulk reagents for absorptiometry. The detection limit and working curves available with test-kits should be confirmed in prior of the use.

When screening contaminated sites for Cr(VI) by test-kit detection, Cr(VI) is extracted from soil into water. The recovery of extracted Cr(VI) would be slightly lower than that when extracting with alkali solution; however, the repeatability of test-kit detection for Cr(VI) is high enough to apply the detection manner to contaminated sites as a screening method. Basically, laboratory analysis provides with precise concentration values but takes longer time and higher costs than those of test-kit detection. Furthermore, under the conditions that survey time and the total costs are restricted, test-kit detection is more useful and practical as it can cover more inspection spots, resulting in precise information on the pollution at investigation sites, compared with a conventional manner applying samples to laboratory analysis that costs and limits the numbers of samples to be analysed due to a lean budget for financial reasons.

The two investigation manners, or the conventional methods and the screening work, have their specific advantages and characters. Then, it is quite natural to choose one of the two options. In short, one gives precise concentration values at selected spots within a limited number but calling for high costs while the other provides concentration levels at spots with no limits in number but showing good cost performance.

This Technical Report describes the procedure to screen soil for Cr(VI) using a method with test-kits developed for water samples.

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# Soil quality — Detection of water soluble chromium(VI) using a ready-to-use test-kit method

## 1 Scope

This Technical Report describes the procedure to screen soil samples to detect Cr(VI) using test-kits based on water extraction of Cr(VI) in soil. The test-kit approach in this method is designed to quickly screen soil samples using calibration to indicate the concentration level. A batch test at a liquid to solid ratio of 10 l/kg, designated by ISO/TS 21268-2, has been used in Japan for over 30 years to evaluate the risk of Cr(VI) contamination in soil. The Cr(VI) detection by a ready-to-use test-kit method follows ISO/TS 21268-2.

This screening technique is applicable in laboratories but can also be applied for site screening in the field. The standard system detection covers the range mg/kg to % for Cr(VI).

This method is expected as a screening technique because of the significant colour change given by 1,5-diphenylcarbazide in the existence of Cr(VI). The method may be especially useful in quickly detecting Cr(VI) where a site is assumed to have no Cr(VI) contamination. As with other screening techniques, it is advisable to confirm a certain percentage of both positive and negative test results in another technique, especially when the detected level is near or above a regulatory action limit or when the presence of background or interfering materials is suspected.

Basically, laboratory analysis requires longer time and higher costs than those of test-kit detection. If the same survey time and the costs are allowed with test-kit screening work and conventional investigation methods including laboratory analysis, the former way can work on more inspection spots than the latter. However, test-kit detection should carefully be adopted for site investigation because the recovery of Cr(VI) from soil into water dependent on soil matrices changes the performance of test-kit detection.

## 2 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

#### **ready-to-use method**

analytical method that is ready-made for use, and may be employed in the field

Note 1 to entry: A more familiar name is “field method”.

[SOURCE: ISO 17381:2003, 3.2, modified — definition shortened]

## 4 Principle

Chromium(VI) in solution reacts with 1,5-diphenylcarbazide to form a red-violet complex of chromium-1,5, diphenylcarbazone resulting in colour changes of the solution due to the formation. Spectrometry and colour charts are applied to determine the complex. The test determines Cr(VI)

present in the soil extract as chromate or dichromate ions. The test-kit method to detect the Cr(VI) in soil consists of three basic steps: extraction, filtration and detection.

## 5 Reagents

- 5.1 **Water**, de-ionized, as an extraction solvent to extract Cr(VI) from soil samples.
- 5.2 **Test-kit reagent**, i.e. 1,5-diphenylcarbazide.

## 6 Interferences

Arsenic, cobalt, iron(II), iron(III), tin, vanadium, lead and phenol may give colour changes by reacting with 1,5-diphenylcarbazide. Reducing agents like iron(II) and ascorbic acid effect to reduce Cr(VI) to Cr(III). Thus, they also interfere with a chromium(VI)/1,5-diphenylcarbazone reaction.

## 7 Apparatus

- 7.1 **Spectrometer**, hand-held or portable photometer working at a wavelength of 540 nm.
- 7.2 **Colour chart**, with colour bars indicating a value on a reference concentration.
- 7.3 **Spoon**, non-metal to take soil samples.
- 7.4 **Sample tube and vial**, e.g. plastic or glass tube for the extraction operation by shaking and a transparent glass vial for the test-kit observation.

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## 8 Procedure

### 8.1 Calibration

When using a photometer for photometric observation of Cr(VI) in the sample solution, prepare a calibration curve using blank and working standards. The working standards should be prepared by spiking an appropriate amount of Cr(VI) solution into de-ionized water.

When using a colour chart for the detection of Cr(VI), the calibration process is not necessary.

### 8.2 Sample detection

Weigh out 1 g of a soil sample and put into 10 ml of water. Shake the mixture for 1 min and then filtrate using a 0,45 µm pored membrane filter. Dilute 1 ml of the filtrate with water to give at least 10 ml, when the colour of the sample is out of the range of the colour chart supplied by the manufacturer due to too high concentration of Cr(VI) in the filtrate.

### 8.3 Measurement

Introduce a certain volume of the diluted sample (7.2) into the test-kit apparatus. Measure the absorbance of the solution at a wavelength of 540 nm using a suitable cell with a 10 mm optical path length or observe the colour comparing with the colour chart.



## 9 Quality assurance (QA) and quality control (QC)

A program of field QA/QC should include a minimum of periodic soil blanks, equipment blanks, soil spikes and duplicates. Other procedures should be implemented depending on the specific requirements of each site.

To ensure accurate quantification and repeatable results, it is recommended that a recalibration be run at a certain interval or at least daily.

The analytical results contain uncertainty arising from numerous sources, heterogeneity of soil sample, difficulty in using matrix matched reference samples, etc. The expanded uncertainty of measurement can be estimated from the uncertainty budget with standard uncertainties. See ISO/IEC Guide 98 for the guidance on the estimation of expanded uncertainty.

## 10 Test report

The test report (see ISO 12404) should contain at least the following information:

- a) a reference to this Technical Report;
- b) complete identification of samples;
- c) the results of the determination;
- d) any details not mentioned in this Technical Report or which are optional, as well as any factor which may have affected the results.

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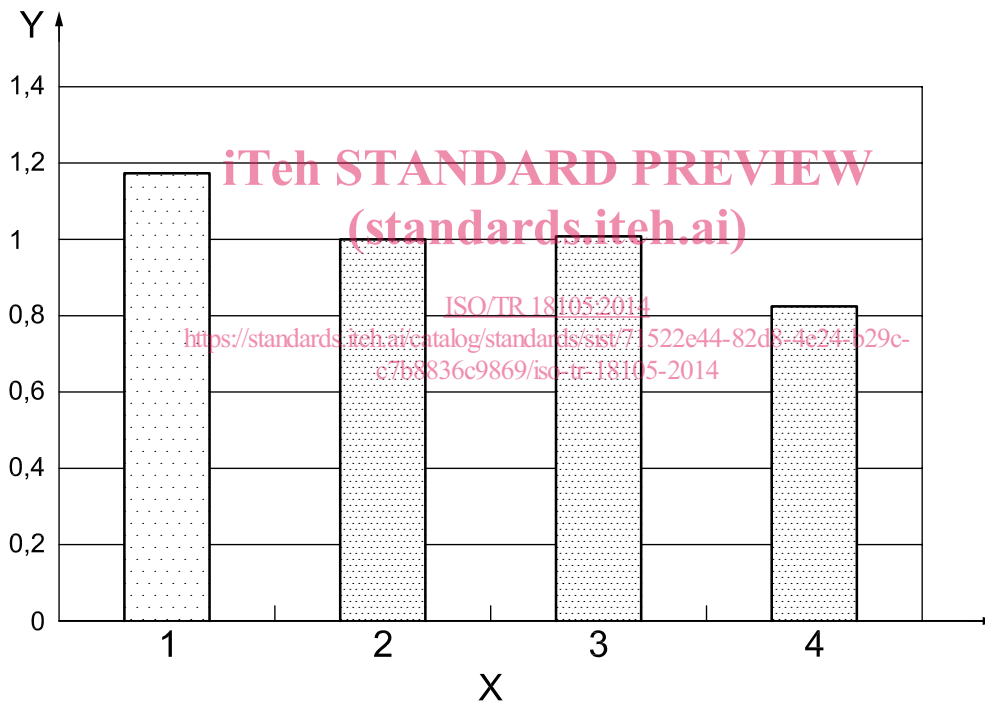
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## Annex A (informative)

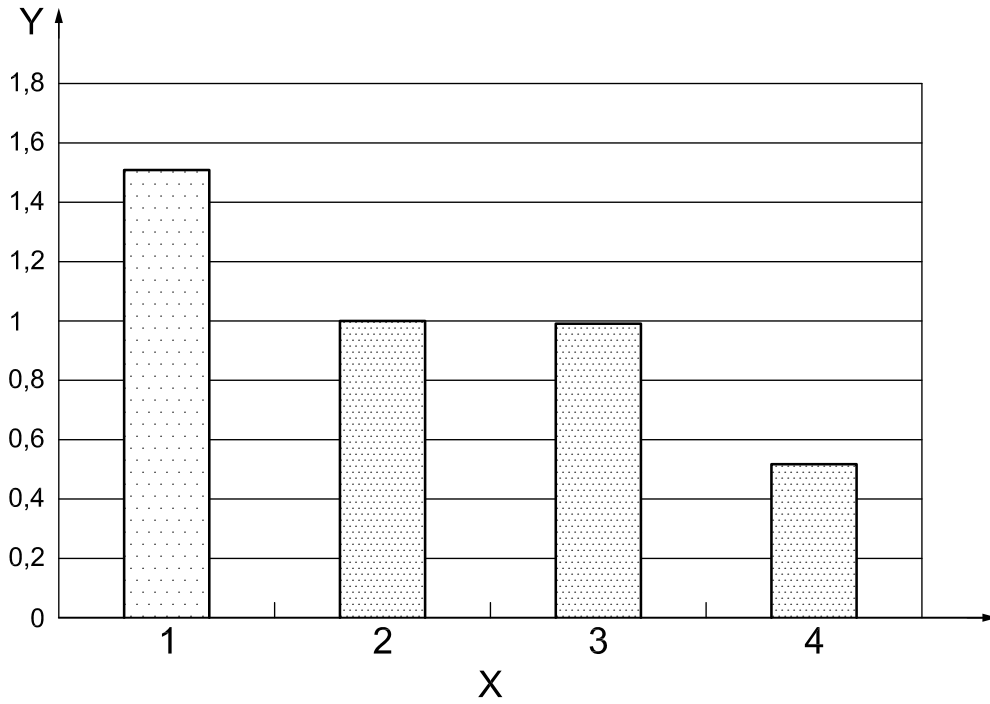
### Extraction of Cr(VI) from soil CRMs

A preliminary evaluation of test-kit detection performance with certified reference materials (CRMs) showed that test-kits effectively work as a screening method. [Figure A.1](#) and [Figure A.2](#) show the relative concentrations of Cr(VI) in sandy loam and clay CRMs when Cr(VI) was extracted with alkaline solution as designated by ISO 15192 and EPA 3060 as well as with water as designated in this Technical Report. The Cr(VI) recovery when applying the test-kits to the detection of Cr(VI) in the CRMs was high for the sandy loam CRM, even when the Cr(VI) extraction was performed with water. With the clay CRM, the recovery by detection with water extraction was half that with alkaline extraction. However, the standard deviation ( $n = 5$ ) is less than 10 % under each condition. [Figure A.3](#) shows the improvement of the recovery achieved by adding salts when extracting Cr(VI) from clayey soil into water. In this case, it was found that addition of  $\text{Na}_2\text{SO}_4$  and  $\text{Al}_2(\text{SO}_4)_3$  improved the Cr(VI) recovery.



- Key**
- Y C/C ISO 15192
  - X 1 certified value
  - 2 by ISO 15192
  - 3 by EPA 3060
  - 4 by TK proposed

**Figure A.1 — Cr(VI) determined in the CRM (sandy loam) — Test-kit (TK)**



**Key**

- Y C/C ISO 15192
- X 1 certified value
- 2 by ISO 15192
- 3 by EPA 3060
- 4 by TK proposed

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**Figure A.2 — Cr(VI) determined in the CRM (clay) — Test-kit (TK)**