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Sakai, Hiroyuki
Railway Technical Research Institute, Japan Railways

Imai, Kazumichi
Hitachi High-Technologies Corporation

Suzuki, Yoshihiro
Sibata Scientific Technology

Wada, Shin-ichiro
Laboratory of Environmental Geochemistry, Division of Bioproduction Environmental Sciences,
Department of Agro-environmental Sciences, Faculty of Agriculture, Kyushu University, Fukuoka

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Application of Test Kits for Water Samples to Screening Contaminated Soils for Chromium (VI)

Hiroyuki SAKAI¹, Kazumichi IMAI², Yoshihiro SUZUKI³ and Shin-Ichiro WADA*

Laboratory of Environmental Geochemistry, Division of Bioproduction Environmental Sciences,
Department of Agro-environmental Sciences, Faculty of Agriculture,
Kyushu University, Fukuoka 812-8581, Japan

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To develop a screening method for chromium (VI) in soils, test kits normally used for water samples were applied to the water extracts from two chromium (VI)-contaminated reference soil samples. The chromium (VI) contents determined by water extraction at an ambient temperature and detection by commercial test kits amounted to 71% and 34% of the certified content for sandy loam and clay soil, respectively. The recovery increased to 82% and 59% for respective soil when the extraction was made with a mixed sodium carbonate-sodium hydroxide solution that is employed in the standard method for soil chromium. These results indicate that the extraction with water or alkaline solution combined with chromium (IV)-detection by test kits works as a rapid screening method for chromium (VI) in contaminated soils.

Keywords: chromium (VI), screening, soil pollution, test kit

INTRODUCTION

Test kit (TK) methods use reagents stored in plastic or glass vials prepared for colorimetric detection of specific compounds soluble in water (ISO Central Secretariat, 2003; British Standards, 2009). There are a variety of TKs 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 TK usually give colors, by which users can recognize the concentration level of the compound without absorption spectrophotometers even in the situation when such instruments are not available. Thus, the TK determination technologies for the compound in water samples can be applied to screening specific compounds in soil samples, in case they are easily dissolved into water.

As mentioned above, TKs have a large variety of applications to water samples for a number of target compounds, especially inorganic ions. The analytical detection system is based on a colorimetric procedure. The reactions used are very common and well known, which were established many years ago and has been used for a long time period as simple analytical technique. Anions of inorganic compounds in soil samples, negatively charged, which can be analyzed with TKs, are basically dissolved into water when the anions are extracted with water as soil particles are negatively charged. Thus, the anions, which are not adsorbed on but isolated from soil particles, must be detected when TKs are applied to the

water samples prepared from soil samples by extraction. Chromium (VI), in other words, chromate or dichromate is expected to be a possible target compound for TK detection. Furthermore, the goal of screening methods, especially for soil quality, is to detect interesting compounds in soil, the concentration of which exceeds the allowable limit in the soil environment. This means that there is less interest in knowing exact concentration values but great curiosity about recognizing the concentration levels of the compounds. Then, the TK for chromium (VI) was applied to the water sample given through the extraction of chromium (VI) from soil samples. Basically, when extracting chromium (VI), hot alkaline solutions are used as a solvent. If the extraction just with water at an ambient temperature still works for the TK application, however, this makes the procedure easy since screening methods are commonly used at outdoor sites. This short report describes the recovery of chromium (VI) at the extraction of the element with water at room temperature, which is determined through the TK and ion-chromatographic separation/conductivity detection, respectively. Based on the results, the possibility of the application of TK to the screening of chromium (VI) in soil samples will be discussed, which was basically developed for water samples.

PRINCIPLE

The reaction applied depends on the target compounds. For chromate and dichromate or chromium (VI), used is a reaction given by 1,5-diphenylcarbazide, its isomer or derivatives. The element easily dissolves in water and the ions react with 1,5-diphenylcarbazide to form a red-violet complex of chromium-1,5-diphenylcarbazone resulting in color changes of the solution due to the formation of the complexes coordinated to chromium (VI). Spectrometry is applied to determine the complex. The test determines chromium (VI) present in the soil extract

¹ Railway Technical Research Institute, Japan Railways, Hikari-cho 2-8-38, Kokubunji, Tokyo 185-8540, Japan

² Hitachi High-Technologies Corporation, Ichige 882, Hitachi-Naka, Ibaraki 312-8504, Japan

³ Sibata Scientific Technology, Nakane 1-1-62, Soka, Saitama 340-0005, Japan

* Corresponding author (E-mail: wadasi@agr.kyushu-u.ac.jp)

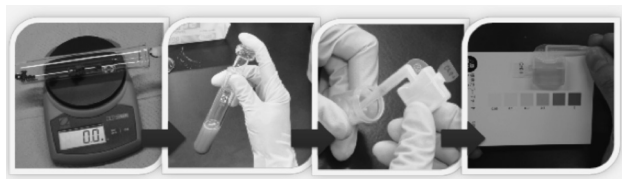


Fig. 1. An example of TK detection for chromium (VI). The extraction procedure is independent of TK manufactures but the colorimetric procedure including photometrical detection or observation with a color chart is specified to the manufacture of the TK used.

as chromate or dichromate ions. The TK method to detect the chromium (VI) in soil consists of three basic steps: extraction, filtration and detection (Fig. 1).

When other chemicals or derivatives are used as a reagent besides 1,5-diphenylcarbazide, its isomer or derivatives, the performance shall be the same as those given with the original reagents. The TK consisting of the reagents shall be identified by confirming the spectrum including intensities at specific wavelengths. The spectrum shape shall be similar to each other between that from TKs and bulk reagents commonly used for absorptiometry in laboratories. The intensities and varieties of interferences to the TK method should be within those taking place to absorptiometry test methods with bulk reagents. The quality or purity of the reagents packed in TKs should remain constant with a deviation around that for the bulk reagents for absorptiometry. The detection limit and working curves available with TKs should be

confirmed prior to the use.

MATERIALS AND METHODS

Two commercial reference soil samples were used; a sandy loam and a clay soil containing $241 \pm 9.0 \text{ mg kg}^{-1}$ and $176 \pm 12.9 \text{ mg kg}^{-1}$ of chromium (VI), respectively. One gram portion of the soil sample was weighed into a glass test tube and mixed with 10 mL of water. The tube was shaken for 1 min by hand and the mixture was filtered through a $0.45\text{-}\mu\text{m}$ pore membrane filter. Following the manufacturer's instruction, 2.5 mL of the clear filtrate was introduced into a TK for chromium (VI) (Simple Pack, Sibta Co., Saitama) after dilution with water when necessary (Fig. 1). After color development, the content of the TK was transferred into a quartz cell with a 10-mm optical path and the absorbance at 540 nm was recorded. In a similar way, calibration curve was prepared with a series of standard chromium (VI) solutions. The approximate concentration of chromium (VI) can be directly read by comparing the developed color with the colors printed on the standard chart supplied by the manufacturer. In addition, chromium (VI) was extracted with a mixed sodium carbonate–sodium hydroxide solution that is employed in the standard test method for soil chromium (VI) at ambient temperature and determined with the TKs.

For comparison, chromium (VI) was extracted and determined by the standard method (ISO Central Secretariat, 2010), i. e., extraction with a hot mixed

Table 1. Results of chromium (VI) determination by standard and proposed methods

Extraction method	Detection method	Certified (A) or detected value / mg kg ⁻¹		Repetition
		CRM061–030 Sandy loam	CRM060–030 Clay soil	
		241 ± 9.0, certified	176 ±12.9, certified	
Alkaline at 92°C	Ion– chromatographic detection (<i>n</i> =2)	192	122	8
		245	128	
		average, 218	average, 125	
Alkaline at 92°C	Photometric detection (<i>n</i> =2, ISO 11083) (B)	206	126	8
		206	115	
		average, 206	average, 120	
Alkaline at ambient temperature	TK detection (<i>n</i> =5) (C)	201	103	40
		203	105	
		184	100	
		195	105	
		203	107	
		average, 197	average, 104	
Ratio of C/A, % (D)		82	59	–
CV of C, %		4.0	2.3	–
Water at ambient temperature	TK detection (<i>n</i> =5) (E)	159	63	240
		173	62	
		169	61	
		175	60	
		180	53	
		average, 171	average, 60	
Ratio of E/A, % (F)		71	34	–
CV of E, %		4.6	6.6	–
Ratio of E/B, %		83	50	3,000

sodium carbonate–sodium hydroxide solution and determination by ion chromatography.

RESULTS AND DISCUSSION

At the preliminary evaluation with commercially available Certificated Reference Materials (CRMs), the TK method with water extraction at ambient temperature showed good performance as a screening method (E in Table 1). The recoveries were found high for the sandy loam and the clay soil. There was no significant difference in the concentration value between alkaline and water extraction for each CRM despite ISO 15192: 2010(E) (ISO Central Secretariat, 2010) requires extraction with a hot alkaline solution. The standard deviation (CV) of the detected concentration values by the TK is less than 10% under each condition ($n=5$). The TK method works, therefore, for quick check of samples. The check can be repeated much more than that by the ISO method (B in Table 1).

As stressed earlier, the goal of screening is to detect target compounds in soil samples whose concentrations are over the allowable limits in the soil environment. Thus, there is, ultimately, less interest in obtaining the exact concentration values. Under these circumstances, the TK detection works for the purpose of screening methods even while the recovery of target compounds is poor. What is to be confirmed is the reproducibility of the recovery. If the detection limits of target compounds are higher than the concentrations allowable for the soil

environment while the reproducibility keeps constant even at a low recovery, the detection methods work as screening methods. In the case of chromium (VI) detected by TKs, the recoveries are 34% and 71% for sandy loam and clay soil. But the reproducibilities of the recoveries are only 4.6% and 6.6% as cv., respectively. The detection limit of chromium (VI) is 0.02 mg kg^{-1} in soil samples while the allowable limit in the soil environment is 0.05 mg kg^{-1} in soil. Thus, TK detection is expected as screening methods for its practical use in the field of soil quality.

This report has shown the possibility of TK detection as a screening method. The applicable compounds for soil quality will be all anions, to which TKs developed for water samples have been applied. This suggests that TK detection have a large variety of applications and can cover all of anions, which are commonly checked at chemical pollution inspection in the ground.

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