

A SCREENING METHOD FOR DETECTION OF HEXAVALENT CHROMIUM LEVELS IN SOILS

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A rapid and low cost method to determine Cr(VI) in soils based upon alkaline metal extraction at room temperature is proposed as a semi-quantitative procedure to be performed in the field. A color comparison with standards with contents of Cr(VI) in the range of 10 to 150 mg kg⁻¹ was used throughout. For the different types of soils studied, more than 75% of the fortified soluble Cr(VI) were recovered for all levels of spike tested for both the proposed and standard methods. Recoveries of 83 and 99% were obtained for the proposed and the standard methods, respectively, taking into account the analysis of a heavily contaminated soil sample.

Keywords: hexavalent chromium; screening methods; soil.

INTRODUCTION

Several industrial processes including metallurgy, electroplating, leather tanning, polishing, painting, pigment manufacture, wood treatment, and electricity generation produce significant quantities of chromium wastes. Because of its widespread, long-term use, chromium contamination of soils and groundwater has been detected in thousands of sites worldwide. The identification of the two most common species in the environment, Cr(III) and Cr(VI), is an important issue for the environmental protection and remediation of contaminated areas. While Cr(VI) is relatively mobile in the environment and acutely toxic, mutagenic, teratogenic and carcinogenic, Cr(III) is relatively immobile under moderately alkaline to slightly acidic conditions, and has a relatively low toxicity.¹⁻⁴

Analysis for total chromium is straightforward, however, successful analysis for Cr(VI) in soil matrices can be more complex. An optimal extraction procedure should completely release Cr(VI) species without disturbing the species distribution.^{3,4} Because Cr(VI) is a strong oxidizer, there are certain conditions in the environment that preclude chromium from existing in the hexavalent state. Under highly reducing conditions Cr(VI) is rapidly reduced to Cr(III).⁵

Considering that sampling uncertainty is the main component responsible for the final uncertainty of chemical analysis of soils in contaminated areas, the use of alternative methods to be carried out *in situ* is highly desirable.⁶⁻¹⁰ Even if they are less precise than the classical ones, *in situ* methods minimize the uncertainties inherent to the heterogeneous distribution of metals in the soil of contaminated areas as they allow a larger amount of data to be collected.

In the characterization and clean-up of contaminated sites, analytical data are often needed immediately and on-site, a requirement that conventional laboratory methods do not fulfill. The development of a rapid test to provide a fast, portable, easy to use analytical tool, that is, at the same time, capable of providing trustworthy information

in essentially real time is highly desirable.¹¹ The use of analytical kits to perform screening tests *on site* or even in the lab allows the investigation to focus on highly contaminated areas and helps guide additional analyses during a single sampling event. The amount of data available as a result of rapid tests allows faster management of uncertainties in decision-making about contaminated sites by the technical team.¹²

Considering the value of implementing more dynamic and realistic approaches to streamline assessment and cleanup activities at hazardous waste sites, this study proposes a field screening method for analysis of Cr(VI) in soils.^{13,14} In order to check its effectiveness, this rapid test was applied to different kinds of soil samples and spiked materials, using as reference method hot carbonate-hydroxide extraction followed by UV-visible spectrometric detection.^{15,16} In order to guide and simplify decision-making about sites evaluated with the field test, four Cr(VI) content domains were considered for the different degrees of soil contamination – (1) low: <10; (2) intermediate: 10 to 40; (3) high: 40 to 150; and (4) very high: >150 mg kg⁻¹, corresponding respectively to differing management alternatives: (1) no immediate action is necessary; (2,3) a confident decision depends on additional analysis of soils by more rigorous analytical methods; and (4) remedial action is undoubtedly needed for agricultural use of soil.¹⁷

The choice of the intervals for the Cr(VI) contents selected in this work was based on the guidelines supplied by Companhia de Saneamento Ambiental do Estado de São Paulo (Environmental State Agency) for total chromium in soils from contaminated areas, which, in the absence of specific legislation for Cr(VI), was used as a starting point to develop this field test.¹⁷

EXPERIMENTAL

Reagents and standard solutions

A potassium dichromate stock solution (50 mg Cr(VI) L⁻¹) was

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used to prepare all Cr(VI) standard solutions and to fortify the soil matrices evaluated. Deionized water (18.2 MΩ cm) obtained from a Milli-Q ultrapure water system (Millipore Corp.) was used to prepare solutions and to dilute sample extracts. Reagents of analytical grade or higher were used throughout the experiments: sodium hydroxide (NaOH - Synth), nitric acid (HNO₃ - Merck), sulphuric acid (H₂SO₄ - Synth); sodium carbonate (Na₂CO₃ - Vetec); potassium dichromate (K₂Cr₂O₇ - Vetec); chromium oxide (Cr₂O₃ - Vetec); 1-5-diphenylcarbazide (C₆H₅NHNHCONHNHC₆H₅ - Vetec).

Samples and sample preparation

The work was carried out with different types of soil samples (S₁, S₂, S₃, S₄ and S₅) presenting different physicochemical properties and metal contents (Table 1). The commercially available sand (mesh 100), denote as S₁, in the absence of organic matter and Cr(VI), was used in order to represent an inert soil. Sample S₄ presenting a low content of Cr(VI) was collected in a former wood treatment facility, whereas the sample S₅ presenting a high content of Cr(VI), was collected in an industrial landfill located in a former chromite mining unit used to process different chromium salts.^{18,19} Samples S₂ and S₃ were obtained from different non-contaminated areas located in the wood treatment facility and in the industrial landfill mentioned above, respectively. These samples were used in the present work as the background soil samples (absence of contamination). Before to conduct each analysis, the several different soil samples were dried in air (~ 25 °C) and then passed through a 2x2 mm stainless steel sieve.

Table 1. Physicochemical characteristics of the soil samples used in this work

Variables	S ₁	S ₂	S ₃	S ₄	S ₅
pH ^a	-	4.77	6.72	7.16	7.60
Organic matter (%) ^b	< 1.0	3.7	8.5	2.9	4.3
Fe (%) ^c	-	2.27	6.29	1.69	3.36
Mn (mg kg ⁻¹) ^c	-	102	155	152	542
Cr (mg kg ⁻¹) ^c	78.0	78.9	53.0	159	2537
Cr(VI) (mg kg ⁻¹) ^d	<1.0	<1.0	<1.0	2.0	450.0
Grain size distribution ^e					
Pebble (%)	0	0.5	0	1.2	0
Sand (%)	100	52.2	33.8	57.4	61.6
Silt (%)	0	25.9	31.2	25.2	17.6
Clay (%)	0	21.5	35.0	16.2	20.8
Texture	Sand	Sand with mud	Mud with sand	Sand with mud	Sand with mud

^aSoil:solution ratio - 1:1 in deionized water; ^bGravimetric analysis - 360 °C; 2 h^{20,21}; ^cTotal content by XRF analysis; ^dMethod of extraction - USEPA3060A¹⁶; method of analysis - USEPA 7196A²²; ^eAnalysis by sieving and sedimentation techniques.

Fortification of the background soil samples with K₂Cr₂O₇ and Cr₂O₃

For each type of background soil, S₁ (sand), S₂ and S₃, 2.5 g of sample were weighed into erlenmeyer flasks to perform the standard extraction procedure, and in parallel, 1.5 g were weighed into the centrifuge tubes to perform the proposed procedure for Cr(VI). All the experiments were conducted in duplicate.

Spike of K₂Cr₂O₇

The background soil samples were fortified with different contents of Cr(VI), as shown in Table 2. After the mixture of solutions, the flasks were vigorously agitated and putted into rest for 10 min.

This range was used to evaluate how the various types of soil, with low, medium and high contamination of Cr(VI), would behave in order to define the working range for the proposed method. The soil sample with low (S₄) and high contamination (S₅) were also fortified, to generate contents of 4 and 1000 mg kg⁻¹ Cr(VI) in soil, respectively. These concentrations were chosen because they represent approximately double the original quantity of Cr(VI) present in each of these samples.

Table 2. Volumes of the standard solution of 50 mg L⁻¹ Cr(VI) added to fortify the different types of soil studied: S₁ (sand), S₂ and S₃

Final fortified content of Cr(VI) mg kg ⁻¹	Standard method 2.5 g soil V _{added} (μL)	Proposed method 1.5 g soil V _{added} (μL)
10	500	300
40	2000	1200
150	7500	4500
500	25000	15000

Spike of Cr₂O₃ and K₂Cr₂O₇

In order to evaluate the recovery/interconversion of both chromium species, 10 mg of Cr₂O₃ were weighed and added to erlenmeyers and then centrifuged in tubes. In the case of the standard method, 7500 μL were added in 2.5 g of soil and in the case of the field test were added 4500 μL in 1.5 g of soil of the standard solution of Cr(VI) (50 mg L⁻¹).

Extraction methods

Standard method

The analytical procedure was conducted according to the USEPA (1996) with the modifications suggested by James *et al.*^{15,16} Using a semi-analytical balance, 2.5 g of the dried and powdered sample were weighed in a 250 mL erlenmeyer flask. The spike material (when used) was added directly to the sample aliquot at this point. Next, 50.0 g of the liquid digestion solution (0.28 mol L⁻¹ NaOH + 0.5 mol L⁻¹ Na₂CO₃) were carefully weighed and added to the flask, and the flask was heated to 90-95 °C. Samples were maintained at this temperature for 60 min, with sporadic shaking. The temperature was monitored in a blank containing only the digesting solution. After this heating period, the flasks were cooled to room temperature and weighed again, when losses were corrected by adding deionized water to a final mass of 100.0 g. An aliquot of approximately 45 mL of each suspension was centrifuged for 10 min at 3600 rpm. After centrifugation, the sample was ready to be analyzed by the USEPA 7196A method.²²

Field screening method

For the procedure proposed to be carried out in the field, 50 mL conical graduated polyethylene centrifuge tubes were used, allowing volumes to be measured directly in the flask. A mass of 1.5 g of the previously dried and sieved soil was weighed into these tubes. In most cases, this mass corresponds to the 2 mL-tube mark for naturally humid soils. The same extracting solution used in the standard method was added to the 20 mL mark and the suspension was vigorously shaken by hand and left to settle for 10 min. After this period, the suspension was shaken again for approximately 1 min, the volume was adjusted to 40 mL with distilled water, and the tube was again shaken for approximately 1 min. Depending on the soil characteristics, the suspension was left to settle for approximately 10 to 30 min.

Development of color and chromium quantification

Standard method

The centrifuged solutions were analyzed using the USEPA 7196A Method.²² For each solution an appropriate dilution was performed, which varied from 1:1 to 1:40, depending on the extract content of Cr(VI). After appropriate dilution, 45 mL of the solution were weighed on a semi-analytical balance using a 100 mL beaker. The pH was adjusted to 2.0 ± 0.5 , with a 40% (v/v) sulphuric acid solution. Then 1.0 mL of 0.5% (m/v) diphenylcarbazide solution was added. The mixture was homogenized and transferred to a 50 mL volumetric flask where the volume was adjusted using deionized water. After that, a portion of the colored solution was transferred to a cuvette ($b = 1.0$ cm) and the absorbance was measured after 10 min at 540 nm. A model 6405 spectrophotometer from Jenway was used throughout. The blank consisted of distilled water with all other reagents added. In the case of colored and/or turbid samples, a blank was prepared using the same dilution without addition of diphenylcarbazide.

Preparing the analytical curve

The analytical curve was prepared from dilutions of a 5 mg L⁻¹ solution of Cr(VI) prepared with K₂Cr₂O₇ from 0.1 to 1.0 mg L⁻¹. The standards were treated in the same way as the samples. The spectrophotometric linear regression of the curve was $Abs (in 540\text{ nm}) = 0.005 + 0.72 C_{Cr(VI)} / (mg\text{ L}^{-1})$.

Field screening method

After measuring 5 mL of the extracted solution directly in the graduated 15 mL tube, 200 μ L of 40% sulphuric acid (v/v) were added followed by 100 μ L of the 0.5% diphenylcarbazide solution (m/v). The tube was closed and the solution was gently mixed, watching for gas being evolved. After 5 to 10 min, the color developed was visually compared to standards that received the same treatment. This procedure allows classifying the samples into four categories: (1) < 10 mg kg⁻¹; (2) between 10 and 40 mg kg⁻¹; (3) between 40 and 150 mg kg⁻¹, and (4) > 150 mg kg⁻¹. The visual comparison analysis for Cr(VI) that could be used in the field (see Supplementary Material), taking into consideration the fact that the State Environmental Agency of São Paulo does not prescribe standards for Cr(VI) in soils.¹⁷ Therefore, the evaluation of contaminated sites generally compares the results obtained for Cr(VI) in soil samples with the guidelines given for total chromium. The background and alert values for total chromium in soils are 40 and 75 mg kg⁻¹, respectively, and the maximum values allowed for agricultural, urban and industrial activities are 150, 300 e 400 mg kg⁻¹, respectively. Thus, if the soil sample investigated with the suggested field test shows a Cr(VI) content higher than 150 mg kg⁻¹, the content of total chromium will automatically have surpassed the first value of intervention established in the guidelines, corresponding to agricultural soils.

RESULTS AND DISCUSSION

Extraction of Cr(VI) from the contaminated samples and recovery of fortified soluble Cr(VI)

Both the standard procedure used in laboratory as well as the screening method for field detection were used to extract Cr(VI) in the contaminated samples. The basic difference between the standard and the proposed method is that the former uses heating for approximately 1 h, whereas the proposed extraction is performed approximately in 20 min at ambient temperature. Figure 1 shows the results obtained for both extraction methods tested in two samples of contaminated soil evaluated with different Cr(VI) contents.

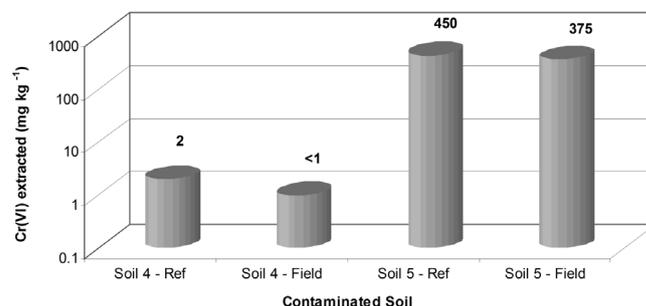


Figure 1. Cr(VI) contents extracted from the contaminated soil samples. Ref stands for the reference method, and field is the denomination used for the proposed field procedure

Sample S₄ has a content of ~ 2 mg kg⁻¹ Cr(VI), which is below the reference value of 10 mg kg⁻¹. Even presenting a 45% recovery of Cr(VI) in the sample, the proposed method would be able to indicate in the field that the soil evaluated is contaminated with Cr(VI) with a content below 10 mg kg⁻¹. In the case of soil S₅, which is highly contaminated (450 mg kg⁻¹), an extraction of about 85% of the Cr(VI) was observed using the proposed method. This result agrees with those obtained by James *et al.* for cold extraction,¹⁵ when a recovery of about 82% was obtained, compared to the application of the same method under heat. The use of the proposed field method would indicate that the sample presents a Cr(VI) content above 150 mg kg⁻¹, thus triggering remediation actions. It can be emphasized that in both cases is possible to perform a comparative visual inspection of the soil samples using colored standard solutions (see the Supplementary Material).

In order to evaluate the effect of the matrix contaminated with Cr(VI), a fortification of samples S₄ and S₅ was performed to double their original content. Figure 2 shows the recovery of soluble Cr(VI) in the fortified samples. It can be seen that the recoveries from sample S₄, using the standard method and the field test were very similar, about 78 and 74%, respectively. On the other hand, for soil S₅ a recovery of 98 and 79% was observed for the standard and the proposed methods, respectively. A lower recovery obtained with the field test is expected, due to the conditions of cold extraction. For both soils, recoveries were obtained in the acceptable range of 75-125%, showing that the proposed method can be used for both soil matrices.¹⁶

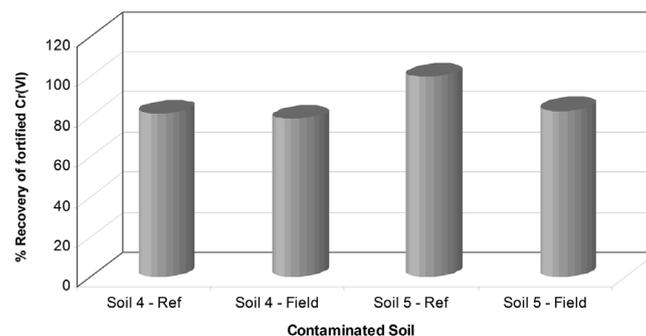


Figure 2. Recovery obtained for Cr(VI) after soil fortification

Research has shown that samples of soils or residues rich in reducing agents (for instance, organic matter, sulfides, ferrous iron) interfere in the determination of Cr(VI), due to the possibility of chromium reduction in this environment. Low recoveries in fortified matrices (<75%) are actually expected for this type of soil, not due to errors, but due to the fact that Cr(VI) cannot exist under reducing conditions.^{15,16,23} Thus, it is inappropriate to attribute a poor efficiency

of the method, or an error in the recovery of Cr(VI) in the fortified matrices, to values outside the traditional range of acceptance (e.g. recovery between 75 and 125%).¹⁶

As shown in Table 1, Cr(VI) was not detected in samples of S_1 (sand), S_2 and S_3 . In order to evaluate the behavior of the method in relation to different levels of soil contamination, different fortifications were performed, from 10 to 500 mg kg⁻¹ of Cr(VI) and 10 mg Cr₂O₃ in the different types of soil studied. Figure 3 shows the percentages of soluble Cr(VI) recovered for the samples S_1 (sand), S_2 and S_3 , using the standard method and the proposed procedure.

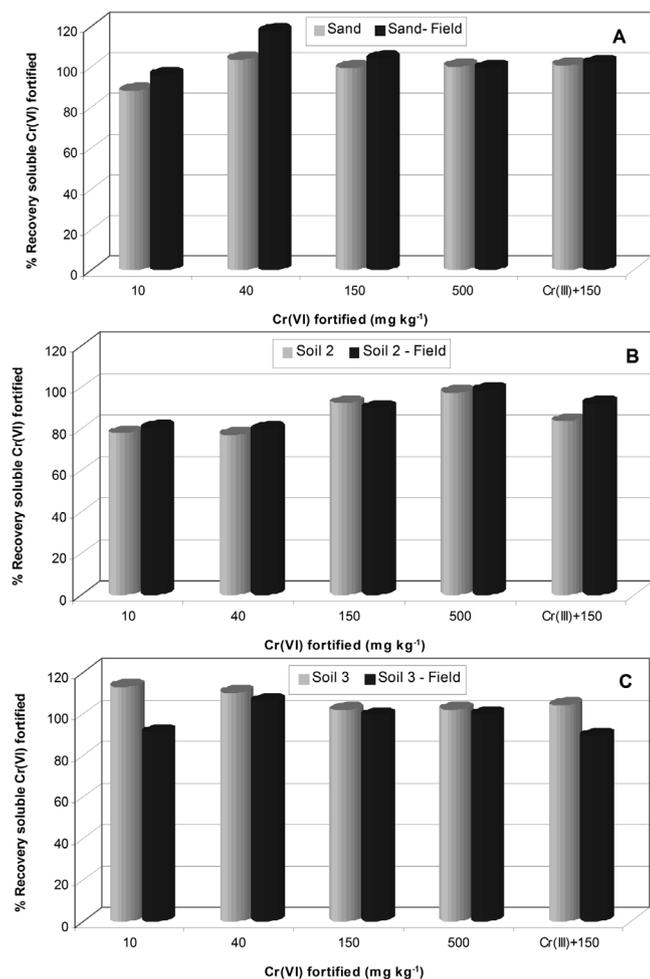


Figure 3. Recovery of soluble Cr(VI) in S_1 (sand), S_2 and S_3 spiked soil samples using both methods

According to the results, it can be observed that low recoveries were obtained for the spike of 10 mg kg⁻¹ Cr(VI), especially for S_2 . This result is expected due to the possibility of matrix interference in this range of Cr(VI) content. On the other hand, for the other fortifications, recoveries between 78-110% were found, which are within an acceptable range for the proposed method, indicating that these matrices are not reducing, sorbing or precipitating the added Cr(VI). It should be observed that the method was proposed for an *in situ* screening intended to map the contaminated site rapidly and at low cost.

Similar results were observed after fortifications with Cr(III), where no interferences were found in detecting Cr(VI), nor conversion between species. According to the literature, extraction procedures using NaOH/Na₂CO₃ solution, both using heat and at ambient temperature, do not induce either Cr(VI) reduction, or oxidation of Cr(III).^{15,16,23}

Before the extractant solutions is discharged, it is advisable to use

a solution of concentrated ascorbic acid to reduce the Cr(VI) present in solution, followed by the recovery of Cr(OH)₃ after adjusting the pH to 8.^{19,24}

CONCLUSIONS

A simple, quick, low cost method to screen hexavalent chromium in the field is proposed. The method is based on the metal extraction at room temperature with manual shaking. Quantitative recoveries of soluble Cr(VI) were obtained in the fortified samples at all levels of contamination studied (10 to 500 mg kg⁻¹). Interconversion of chromium species (VI) and (III) were not observed during the proposed procedure. A comparison of Cr(VI) results obtained using the proposed field method with the standard method, which uses the same extracting solution without heating, did not show significant differences. Thus, it can be concluded that a comparative visual inspection of the soil samples can indeed be carried out using the colored standard solutions considered in the present work. This type of comparison permits to take a reliable decision about the contaminated site, mainly for cases where the contamination level is low (< 10 mg kg⁻¹) or very high (> 150 mg kg⁻¹). For cases where the contamination level is moderate (10 to 40 mg kg⁻¹) or high (40 to 150 mg kg⁻¹) a reliable action concerning the site requires additional soil analysis using more rigorous analytical methods. The main advantages of the proposed procedure are: minimized consumption of solvent; shorter extraction time; lower energy expenditure; possibility of handling several samples at the same time; easy execution; low cost.

SUPPLEMENTARY MATERIAL

Available at <http://quimicanova.sbg.org.br>, in PDF file, with free access.

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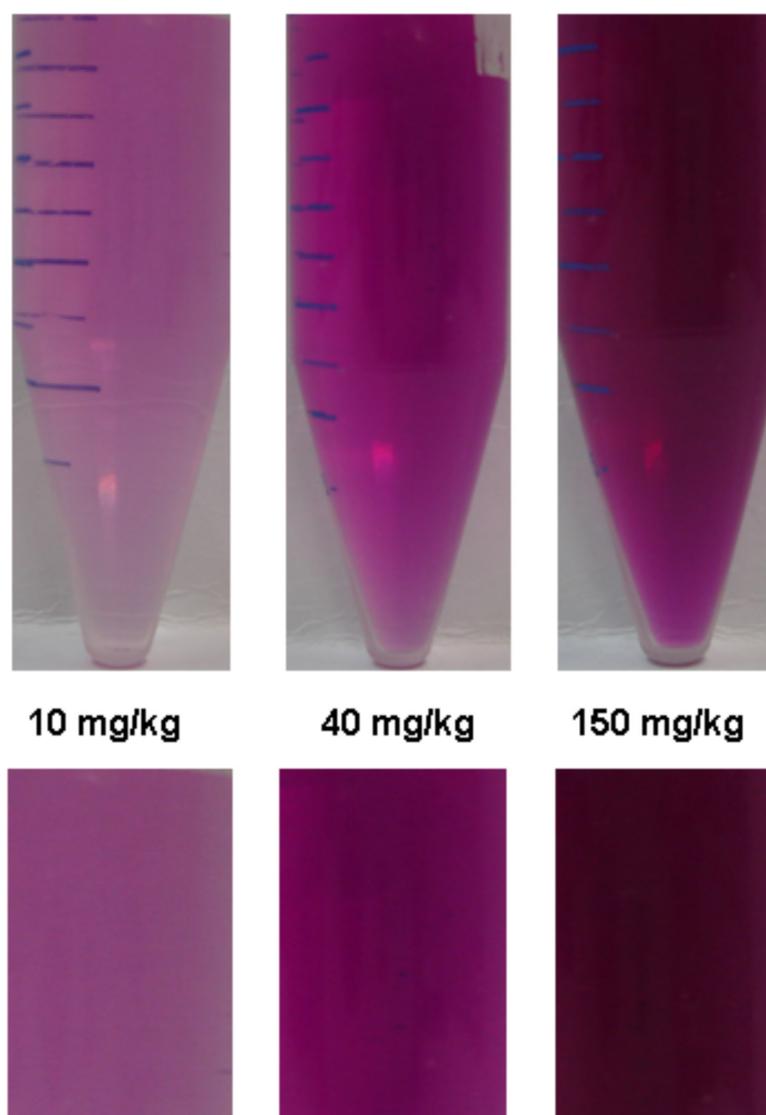


Figure 1. Visual comparison standard proposed for field analysis of Cr(VI) in soils