

Surface geosciences (pedology)

A spectrophotometric measurement of soil cation exchange capacity based on cobaltihexamine chloride absorbance

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Abstract

Among numerous methods for cation exchange capacity (CEC) determination for soils and sediments, the cobaltihexamine chloride method is frequently used due to its ability to measure CEC at soil pH. After exchange with $\text{Co}(\text{NH}_3)_6^{3+}$ ions, CEC is estimated *via* the measurement of the Co remaining in solution. The modified method proposed allows a more rapid determination of CEC based on the measurement of the absorbance at 472 nm of the cobaltihexamine chloride solution before and after exchange. This method has been applied to various soil's horizons from four sites, selected to cover a wide range of CEC and pH values. The model obtained allows one to calculate CEC from absorbance at 472 nm with 95% confidence intervals. As CEC is of relevant meaning in agronomical and environmental purposes, and more recently in ecotoxicological studies, this modified method can be proposed as a rapid test for CEC evaluation. **To cite this article:** *D. Aran et al., C. R. Geoscience 340 (2008).*

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Résumé

Détermination de la capacité d'échange cationique de sols par la mesure de l'absorbance du chlorure de cobaltihexamine. Parmi les nombreuses techniques de détermination de la capacité d'échange cationique (CEC) des sols et sédiments, la méthode au chlorure de cobaltihexamine est fréquemment employée, en raison de son aptitude à mesurer la CEC à la valeur de pH des sols. Après échange avec l'ion $\text{Co}(\text{NH}_3)_6^{3+}$, l'estimation de la CEC est réalisée par le dosage du Co restant en solution. La méthode modifiée proposée ici permet une détermination plus rapide de la CEC, basée sur une mesure de l'absorbance à 472 nm d'une solution de chlorure de cobaltihexamine avant et après échange. Cette méthode a été appliquée à différents horizons de sols provenant de quatre sites, sélectionnés de façon à couvrir une large gamme de valeurs de CEC et de pH. Le modèle obtenu a permis de déterminer la CEC par calcul à partir de l'absorbance à 472 nm, avec un intervalle de confiance à 95 %. Étant donné l'importance de la CEC dans les domaines agronomiques et environnementaux, et plus récemment dans les études en écotoxicologie, cette méthode modifiée peut être utilisée comme un test rapide d'évaluation de la CEC. **Pour citer cet article :** *D. Aran et al., C. R. Geoscience 340 (2008).*

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1. Introduction

Cation exchange capacity (CEC) is a physicochemical characteristic of soils that gives insight about the ability of a soil to bind cations and release them in soil solution at the soil pH. This cation holding capacity is related to the amount of soil colloids with negative charges, like humus and clay.

CEC determination is of considerable importance for agronomical topics. As CEC reflects soil ability to retain cations available for plants, CEC is used, among other soil's parameters like colloid and nutrient content, to discuss soil fertility [13]. CEC evaluation is also relevant for environmental purposes. For instance, trace element exchangeable fraction in polluted soils or sediments is known to be part of the most labile fraction, can be more or less bioavailable, and may also indicate trace element origin [29].

Soil CEC has recently received attention in ecotoxicological studies. For Rieuwerts et al. [22], CEC is a predictive factor of metal extractability in soils but with little consistency when compared to soil pH. Predictability of U concentration in soil solutions has been demonstrated to be based on multiple parameters. On the opposite, a backward stepwise multiple regression analysis excluded CEC from being an explanatory variable [30]. Beside this, Zn, Fe and Cu accumulation in the leguminoseae plant *Trigonella foenum-graecum* was positively correlated to the CEC values of six experimental soils [27]. In a related study, but comprising only three soils, Gupta and Sinha [12] observed a similar trend for the accumulation of Ni, Cr and Pb in *Chenopodium album*. The bioavailability of ^{137}Cs to various plant species was shown to be dependent on soil CEC for only one of a three-year study [14]. On the contrary, Sauras Yera et al. [25] showed that soil CEC was not a good predictor for ^{137}Cs root uptake while it was the case for ^{90}Sr . In invertebrates, heavy metals accumulated in enchytraeids and earthworms as a function of total metal soil concentrations and CEC values [31]. In *Enchytraeus crypticus*, Peijnenburg et al. [21] demonstrated that soil CEC together with pH were the most important parameters for heavy metal uptake rate constants and bioaccumulation factors. CEC was also one of the most important parameters determining Cd, Zn and Cu accumulation in the ground-dwelling spider *Pirata piraticus* [11].

Moreover, several works related the ecotoxicity of metal-contaminated soils to CEC values. When studying the influence of soil properties on Ni toxicity to barley root and tomato shoot growth using 16 European

soils, Rooney et al. [23] identified soil CEC as the best single predictor for toxicity. Similarly, the toxicity of Zn and Cd toward the enchytraeid *Enchytraeus albidus*, exposed in artificial soil with different organic matter and clay types, were correctly predicted from soil CEC [16]. For non-oligochaete species, similar trends were suggested by Lock et al. [17] who reported on the ecotoxicity of Co on the springtail *Folsomia candida*. On a functional viewpoint, microbial cellulose decomposition in metal polluted soils was related to chemical parameters such as CEC, or readily oxidizable carbon rather than to total metal content [8].

Finally, beside metal studies, organic compound fate in soil has been related to CEC: phenanthrene and atrazine sequestration was indeed correlated to soil organic C content, nanoporosity and CEC values [9].

Even if these results do not absolutely concluded to identify CEC as a systematic key variable of metal fate and availability in soils, they strongly suggest to quantify its value when dealing with metal (and probably some organic compounds) fate, transfer and availability in soils.

Operationally, CEC is defined as the sum of the ion amounts released when mixing a fixed mass of soil with a fixed volume of reactant solution. Methods for CEC determination consist in the complete saturation of soil exchange complex by a mono-ionic solution. The CEC is quantified by measuring the difference between initial and remaining added ion concentration. Alternatively, the added ion adsorbed to soil can be removed and subsequently quantified. Another way is to sum up the soil cations removed from exchange sites.

Different methods have been already published for the determination of CEC. They differ by the reactant used, the variables measured, and the pH conditions during extraction (Table 1). In all cases, the CEC is generally expressed as milliequivalent per 100 g of soil

Table 1
Common methods for CEC determination.

Tableau 1
Méthodes courantes pour la détermination de la CEC.

Reagent	pH conditions	Reference
Ammonium acetate	Buffered	Metson [19]
Barium chloride	Buffered	Mehlich [18], Bascomb [6]
Silver-thiourea	Buffered	Chhabra et al. [7]
Ammonium acetate	Buffered	AFNOR [2]
Cobalthexamine chloride	Soil pH	
Ammonium oxalate	Buffered	

dried at 105 °C (meq 100 g⁻¹), that equals SI unit centimoles of positive charge per kilogramme of soil dry weight (cmol⁺ kg⁻¹). Orsini and Rémy [20] proposed to use Co(NH₃)₆³⁺ as the exchange ion and to quantify the CEC through the measurement of the concentration of Co remaining in solution. This method applies to a large range of soils due to the use of a dilute reagent simulating field soil solution conditions. Furthermore, exchange takes place at similar pH to soil pH as the cobaltihexamine chloride is a non-buffered reagent. This method is thus appropriate for soils with large amount of pH-dependent charges.

As the cobaltihexamine chloride solution is coloured and absorbs at 472 nm, the objective of the present work was to assess the possibility to derive soil CEC values more simply from the absorbance of the extractive solution.

2. Material and methods

2.1. Soil samples

Soils were sampled from four sites located in the surroundings of the city of Nancy (France). They were selected with regard to their various colloid contents and different soil pH in order to cover a wide range of experimental conditions for CEC determination. Soils were collected as follows:

- three samples in the A, AB and C horizon of a Rendzic Leptosol [15] developed on a limestone parent material;
- two samples in the A, E and two others in the Bt horizon of a Luvisol [15] developed in a silty deposit parent material;
- three samples in the A, B and C horizons of a Vertic Cambisol [15] developed on a marl;
- four samples in the A, E, Bs and C horizons of a Podzol [15] developed on a sandstone parent material (Table 2).

The main characteristics of these four soils are compiled in Table 2 and were obtained by standard methods. Particle size analysis and carbonate content were performed by INRA's national soil analysis laboratory. After organic matter dissolution and, for Rendzic Leptosol only, carbonate removing, fractions below 50 µm were determined by the pipette method [1]. Carbonate content was estimated by volumetric method [3]. Total carbon was determined by dry combustion [4] and organic carbon was derived by difference with mineral carbon calculated from

carbonate content. Soil pH in the supernatant of a soil–water suspension was measured [5].

After air drying (24 h at ambient temperature, this procedure was previously shown to result in dry weight values that differed by less than 4% to the ones derived from drying the soils at 105 °C), these 14 samples were sieved on 2 mm stainless steel mesh and then kept in the dark at ambient temperature before CEC analysis. For clayey horizons, samples were hand-crushed prior to sieving.

2.2. CEC measurements

For CEC determination, 2 g dry weight (d.w.) soil were placed in 50 ml polypropylene tubes and 40 ml of 0.05 N cobaltihexamine chloride solution were added. After continuous shaking for one hour (duration previously shown appropriate to reach equilibrium) on a rotating device (60 rpm), tubes were centrifuged at 7000 g for 10 min and supernatants filtered on 0.22 µm Millex GP filters. Absorbencies at 472 nm (A472) and pH were immediately measured. The A472 of the 0.05 N cobaltihexamine chloride solution was also determined. Then, Co concentration in supernatants was quantified by flame atomic absorption spectroscopy (Perkin-Elmer AAnalyst 100) together with Fe³⁺, Ca²⁺, Mg²⁺ and Mn²⁺. Na⁺ and K⁺ concentrations were measured by flame atomic emission spectroscopy. At last, Al³⁺ was determined by furnace AAS (Varian spectra-300) and H⁺ was estimated from pH measurement (pH 3000 pH meter WTW) of the supernatant. Certified solutions from Panreac Quimica SA were used to obtain calibration curves.

The whole experiment was repeated twice, the 0.05 N solution of cobaltihexamine chloride (CAS 10534-89-1, sigma H7891) being freshly prepared before each CEC determination.

2.3. Data analysis

According to Orsini and Rémy [20], CEC can be derived from the concentration of Co remaining in the supernatant, determined by flame emission or atomic absorption spectrometry, as

$$\text{CEC}_{\text{Co}} = \left[[\text{Co}]_{0.05\text{N}} - [\text{Co}]_{\text{assay}} \right] \times 3/58.93 \times V/m \\ \times 10^3 \times 100$$

with the following notation: CEC_{Co}: cation exchange capacity (meq 100 g⁻¹ or cmol⁺ kg⁻¹) in terms of the concentration of Co in the supernatant; [Co]_{0.05N} and [Co]_{assay}: Co concentration in 0.05 N cobaltihexamine

Table 2

General characteristics of the four soil profiles.

Tableau 2

Caractères généraux des quatre profils étudiés.

Soil type	Horizon	Depth (cm)	Particle size fractions (%)			CaCO ₃ (%)	Organic C (g kg ⁻¹)	pH
			Clay	Silt	Sand			
Rendzic Leptosol	A	0–7	26.4	24.1	8.8	40.7	106.2	7.5
	AB	7–25	8.0	28.9	6.8	56.3	38.8	7.9
	C	> 25	14.0	17.4	8.0	60.6	9.6	8.4
Luvisol	A	0–8	23.3	61.1	15.6	0	33.8	5.4
	E	8–20	21.5	62.0	16.5	0	11.4	4.8
	Bt1	20–60	57.2	32.4	10.4	0	4.5	4.9
	Bt2	> 60	69.0	23.3	7.7	0	4.2	4.8
Vertic Cambisol	A	0–10	58.4	33.4	8.2	0	14.5	6.6
	B	10–30	43.6	43.2	13.2	0	22.3	6.5
	C	> 30	49.8	38.5	11.7	0	7.4	7.0
Podzol	A	0–5	4.9	11.3	83.8	0	27.0	3.8
	E	5–30	4.0	9.4	86.6	0	8.0	4.0
	Bs	30–50	12.7	13.3	74.0	0	30.2	4.4
	C	> 50	10.7	11.3	78.0	0	2.8	4.6

chloride solution and sample supernatant, respectively, in g l⁻¹; V: volume in l of 0.05 N cobaltihexamine chloride solution added to soil sample (0.04 l); m: soil dry mass used (2 g).

As cobaltihexamine chloride solution absorbs at 472 nm, Co concentration in solution can be determined from A472 measurements. Considering the A472 of a solution of a given normality (i.e. 0.05 N), a CEC_{A472} (meq 100 g⁻¹ or cmol⁺ kg⁻¹) can be calculated as:

$$CEC_{A472} = \left[\frac{(A472_{0.05N} - A472_{assay})}{A472_{0.05N}} \right] \times 50 \times V/m \times 100$$

where A472_{0.05N} and A472_{assay} correspond respectively to the absorbencies at 472 nm of 0.05 N (= 50 meq l⁻¹) cobaltihexamine chloride solution and of sample supernatant; V: volume in l of 0.05 N cobaltihexamine chloride solution added to soil sample (0.04 l); m: soil dry mass used (2 g).

For each experiment, we plotted CEC_{A472} versus CEC_{Co} values and run a regression analysis. All calculations were made according to published methods [26,32].

Moreover, we calculated CEC considering the sum of the soil cations determined in supernatants (Fe³⁺, Ca²⁺, Mg²⁺, Mn²⁺, Na⁺, K⁺, Al³⁺ and H⁺). In this case, the CEC_{sum} (meq 100 g⁻¹ or cmol⁺ kg⁻¹) can be derived as follows:

$$CEC_{sum} = \sum_i [M_i] \times v_i \times V/m \times 10^3 \times 100$$

where [M_i] stands for individual cation concentration corrected for cobaltihexamine chloride solution values (mol l⁻¹) and v_i for the ion valence, respectively. V and m were already defined above.

Then, we studied the relationship between CEC_{A472} and CEC_{sum} as described previously.

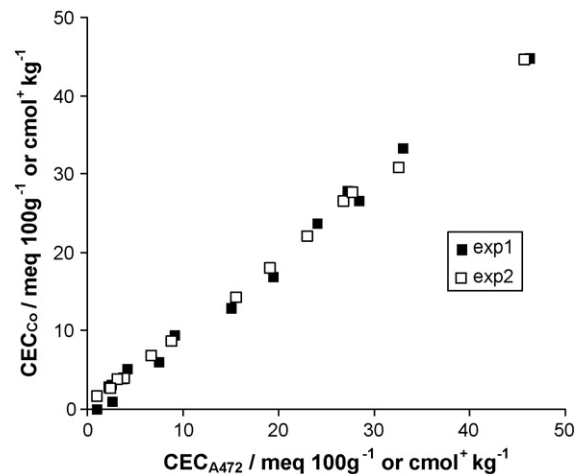


Fig. 1. Scatter plot of CEC_{A472} versus CEC_{Co} in the two independent experiments (exp1 and exp2). CEC_{A472} and CEC_{Co} correspond respectively to CEC values derived from 472 nm absorbencies and Co concentrations of the cobaltihexamine solution after exchange.

Fig. 1. Graphe de la CEC_{A472} versus CEC_{Co} pour deux expériences indépendantes (exp1 et exp2). CEC_{A472} et CEC_{Co} correspondent respectivement aux valeurs de CEC calculées à partir des absorbances à 472 nm et des concentrations en Co de la solution de cobaltihexamine après échange.

3. Results

CEC_{A472} and CEC_{Co} values determined from the 14 horizons in two independent experiments are plotted in Fig. 1. The slopes and intercepts resulting from simple linear regression analyses were tested by using a two-sided Student's *t* test with 12 degrees of freedom (Table 3).

Thus, the tests on both slopes were highly significant ($P < 10^{-11}$), whereas the corresponding intercepts did not significantly differ from zero ($P = 0.417$ and 0.257 , for experiment 1 and 2, respectively). Moreover, a test of comparison performed on the two slopes showed that they were not significantly different (Student's *t* test with 24 df; $P = 0.257$), suggesting that data from experiment 1 and 2 could be pooled together to derive a unique final model. To this end, and considering the above-mentioned results, a regression line crossing the origin was further fitted to the entire set of pooled data. The fitted model ($r^2 = 0.99$) was as follows (Fig. 2):

$$CEC_{Co} = 0.969 \times CEC_{A472}$$

As the slope of this common model was significantly different from unity (two-sided Student's *t* test with 27 df; $P = 6.7 \times 10^{-4}$), CEC_{A472} cannot be considered as a direct (i.e. with no correction) estimate of CEC_{Co}. The corrective factor 0.969 should therefore not be omitted, to prevent from a 3% overestimation of CEC_{Co} value. However, the meaning of this factor is limited. Indeed, if a classical regression was run, the slope would be slightly higher (0.972) and would differ significantly from 1 to a lesser extent ($p = 0.039$) and, at the same time, the *Y*-intercept would not differ significantly from 0. We preferred a regression through the origin analysis (RTO) since no *Y*-intercept at all was justified in our model.

Then, for any given experimental A472_{assay} value, and considering A472_{0.05N} (typically 0.941 in our experiments), it becomes possible to estimate the corresponding CEC_{A472} and CEC_{Co} values. Moreover, a prediction interval at probability level $(1 - \alpha)$, for any

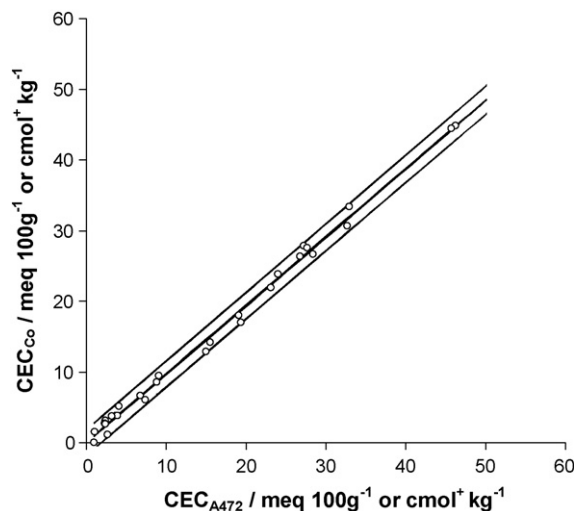


Fig. 2. Regression of CEC_{Co} on CEC_{A472} (data obtained from experiment 1 and 2 were pooled). Plot of fitted regression line (bold) and 95% prediction limits (light). CEC_{A472} and CEC_{Co} correspond respectively to CEC values derived from 472 nm absorbencies and Co concentrations of the cobaltihexamine solution after exchange.

Fig. 2. Régression de la CEC_{Co} en fonction de la CEC_{A472} (les données des expériences 1 et 2 ont été regroupées). La droite de régression est représentée en trait gras, les limites de prédiction à 95 % en traits fins. CEC_{A472} et CEC_{Co} correspondent respectivement aux valeurs de CEC calculées à partir des absorbances à 472 nm et des concentrations en Co de la solution de cobaltihexamine après échange.

CEC_{Co} corresponding to a given value of CEC_{A472}, can be obtained as:

$$PI_{CEC_{Co}}^{1-\alpha} = 0.969 \times CEC_{A472} \pm t_{(\alpha/2; 27 \text{ df})} \times s \times \sqrt{\frac{CEC_{A472}^2}{12076} + 1}$$

where *s* stands for the residual standard deviation, equalling $\sqrt{0.786}$. As the square root term of the equation is, in our case, close to the unit, it can be neglected. Therefore, the expression simplifies to:

$$PI_{CEC_{Co}}^{1-\alpha} = 0.969 \times CEC_{A472} \pm t_{(\alpha/2; 27 \text{ df})} \times s$$

In particular, a 95% prediction interval can be calculated as:

$$PI_{CEC_{Co}}^{0.95} = 0.969 \times CEC_{A472} \pm 1.819$$

The same procedure was followed to study the relationship between CEC_{sum} and CEC_{A472}. The fitted regression model ($r^2 = 0.99$) resulted in the following equation:

$$CEC_{sum} = 0.947 \times CEC_{A472}$$

The slope differed significantly from 1 (two-sided student *t* test with 27 df, $P = 1.9 \times 10^{-5}$) and this could

Table 3
Regression coefficients as obtained from the regression analysis of CEC_{Co} as a linear function of CEC_{A472}, for experiment 1 and 2 data.
Tableau 3
Paramètres de la régression linéaire établie entre la CEC_{co} et la CEC_{A472}, pour les données des expériences 1 et 2.

	Slope (<i>P</i> value)	<i>Y</i> -Intercept (<i>P</i> value)
Experiment 1 (<i>n</i> = 14)	0.987 ($P < 10^{-11}$)	-0.41 ($P = 0.417$)
Experiment 2 (<i>n</i> = 14)	0.957 ($P < 10^{-11}$)	0.248 ($P = 0.257$)

result from an hypothesized release of cations (Fe^{3+} , Ca^{2+} , Mg^{2+} , Mn^{2+} , Na^+ , K^+ , Al^{3+} and H^+) from the soil not due to an exchange with the cobaltihexamine ion.

Once again, a prediction interval at level $(1 - \alpha)$, for any CEC_{sum} , corresponding to a given value of CEC_{A472} , can be derived as:

$$PI_{\text{CEC}_{\text{sum}}}^{1-\alpha} = 0.947 \times \text{CEC}_{\text{A472}} \pm t_{(\alpha/2; 27 \text{ df})} \times s \times \sqrt{\frac{\text{CEC}_{\text{A472}}^2}{12076} + 1}$$

The value of s in that case being $\sqrt{1.278}$. As previously explained, the expression simplifies to:

$$PI_{\text{CEC}_{\text{sum}}}^{1-\alpha} = 0.947 \times \text{CEC}_{\text{A472}} \pm t_{(\alpha/2; 27 \text{ df})} \times s$$

Notably, a 95% prediction interval is given by:

$$PI_{\text{CEC}_{\text{sum}}}^{0.95} = 0.947 \times \text{CEC}_{\text{A472}} \pm 2.318$$

4. Discussion and conclusions

Two models were derived from our experiments, allowing to obtain estimations of soil CEC values, expressed either as CEC_{Co} or CEC_{sum} . Values will not differ to a large extent but reporting results as CEC_{Co} would be preferable since the model is based on a single determination (the concentration of Co in the medium), whereas the other model was derived from multiple measurements (i.e. Fe^{3+} , Ca^{2+} , Mg^{2+} , Mn^{2+} , Na^+ , K^+ , Al^{3+} and H^+ concentrations), each one being a source of uncertainty. The relationship between CEC values determined by absorbance measurement of cobaltihexamine ion and values issued from the analytical determination of cobalt has already been reported [10]. The authors concluded that both measurements methods were interchangeable but the slope of the regression line was not statistically tested toward the unit and no confidence intervals were calculated. Here, we show that CEC_{Co} and CEC_{A472} values are indeed extremely close one to the other but not interchangeable. Mechanistically, the discrepancy between both values may result from a tight loss of absorbance in the sample extracts which is not paralleled by a loss of Co. A lysis of the cobaltihexamine complex could lead to such phenomenon but it remains unexpected due to its high stability (dissociation constant $K_d = 10^{-35}$). Also, the complex $\text{Co}(\text{NH}_3)_6^{3+}$ may react with solubilized soil components leading to a putative less absorbing compound. However, literature data does not allow to comfort such hypothesis.

As expected from soil selection, results of CEC_{Co} determination cover a large range of values, from 0.0 – 1.0 $\text{cmol}^+ \text{kg}^{-1}$ for the E horizon of the Podzol to 44.8 – 44.3 $\text{cmol}^+ \text{kg}^{-1}$ for the A horizon of the Rendzic Leptosol. Results are consistent with colloids content of the various samples: sandy or clay-poor samples (the whole profile of the Podzol and the illuvial E horizon of the Luvisol) show the lowest CEC, while organic or swelling clay-rich samples (A and AB horizons of the Rendzic Leptosol, and the whole profile of the Vertic Cambisol) exhibit the highest ones.

The null CEC_{Co} value for the E horizon of the Podzol found in experiment 1 (Fig. 1) is due to the extremely low colloids content of this sample. Due to a quartzic sandstone parent material and podzolization process, this profile is sandy-textured with very low clay content. Furthermore, E horizon is depleted in organic colloids as metal-humus complexes migration takes place through this horizon. Therefore, for low CEC samples (below 2.4 $\text{cmol}^+ \text{kg}^{-1}$ in this experiment) the method for CEC determination presented here shows insufficient accuracy.

On account of the significance of the CEC evaluation in the various topics presented in the introductory part, a rapid method for CEC determination, even with low accuracy, is required for screening purposes. The method presented here consists in a short exchange followed by measurement of absorbance after centrifugation and filtration, and therefore is rather not time-consuming and simple to perform. The variability observed between experiment 1 and 2 was tiny and mainly due to the subsampling of soils. It could be possible to reduce it again by increasing the mass of the subsamples. The use of cobaltihexamine chloride reagent proposed by Orsini and Rémy [20] is of great relevance for a large range of soils because this non-buffered reagent provides an exchange at soil's pH. Thereby this method is appropriate for both acid soils and soils with high pH-dependent charges.

Moreover, since a wide range of soil CEC were chosen for deriving the models, these ones may be used as predicting tools for a large variety of soils. For instance, a statistical distribution analysis of the CEC values of the French soil database [24] revealed that the 1st and 9th deciles reached 6.2 and 18.90 $\text{cmol}^+ \text{kg}^{-1}$, respectively, for a data set of approximately 170 000 values. In the same way, the US soil survey database [28] comprises more than 100 000 CEC values from various samples and pedons, and the 1st and 9th deciles were in this case 4.4 and 34.2, respectively. Then, it arises from these distributions of CEC data that the

proposed method would be applicable for most of the soil samples.

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