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DETERMINATION OF NONCRYSTALLINE SOIL COMPONENTS BY WEIGHT DIFFERENCE AFTER SELECTIVE DISSOLUTION

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Abstract—A procedure based on loss of weight after selective dissolution analysis (SDA) and washing with $(NH_4)_2CO_3$ was developed for estimating the noncrystalline material content of soils derived from widely different parent materials. After extracting with 0.2 N ammonium-oxalate or boiling 0.5 N NaOH solutions, samples were washed with 1 N $(NH_4)_2CO_3$ to remove excess dissolution agents and to prevent sample dispersion. The amount of noncrystalline material removed from the sample by the extracting solution was estimated by weighing the leached products dried to constant weight at 110°C. The results match closely with those obtained by chemical analyses of the dissolution product and assignment of the appropriate water. The proposed weight-loss method is less time-consuming than the chemical method, and no assumptions need be made concerning sample homogeneity or water content of the noncrystalline material.

Extractions of whole soil and dispersed clay fractions indicated that noncrystalline material determinations on the clay fractions underestimated the noncrystalline material content for whole soils from 0 to 34%. Acid ammonium oxalate was found to be a much more selective extractant for noncrystalline materials than NaOH.

Key Words—Allophane, Noncrystalline, Quantification, Selective dissolution, Soil, Weight loss.

INTRODUCTION

Although most soils consist essentially of crystalline minerals, many contain appreciable amounts of noncrystalline,[1] inorganic material. Noncrystalline materials in soils derived from volcanic ash and weathered pumice have been extensively studied, but comparatively little is known about the noncrystalline materials in soils derived from other parent materials. Some Scottish soil clays derived from nonvolcanic ash materials were found to contain small but significant amounts of 0.5 N Na_2CO_3-soluble noncrystalline materials (Mitchell and Farmer, 1962), primarily as coatings and films on minerals (Follett *et al.*, 1965a). Various paracrystalline or noncrystalline forms of iron oxides in soils have also been investigated (Follett *et al.*, 1965b; McKeague and Day, 1966; Schwertmann, 1964, 1973; Schwertmann and Fisher, 1973; Blume and Schwertmann, 1969; Segalen, 1968). These small amounts of noncrystalline materials may contribute significantly to the physical and chemical properties of these soils since they may have: (1) high cation-exchange capacity which may be influenced by pH (Wada and Ataka, 1958; Aomine and Jackson, 1959); (2) high surface area; and (3) high reactivity with phosphate (Saunders, 1964). Thus, it is desirable to characterize and quantify the amount of noncrystalline materials present in soils as well as the amount of the crystalline components.

Traditional methods of estimating the crystalline mineral content of soils, such as X-ray powder diffraction (XRD), differential thermal analysis (DTA), and thermogravimetric analysis (TGA), have severe limitations for quantifying the noncrystalline material content in polymineralic soil systems. These limitations result either from a lack of well-defined parameters, low detection limits, or coincidence with parameters of crystalline minerals which are also present. Selective dissolution analysis (SDA) has been extensively used in the study of the noncrystalline material content of soils and sediments. There are limitations, however, which must be considered in using SDA: First, a continuum of crystalline order exists, ranging from no long-range order to paracrystalline to poorly crystalline to well crystalline (Follett *et al.*, 1965a). It is difficult to assess adequately the portion of this continuum which is extracted by a particular reagent. As has previously been demonstrated, the use of boiling 0.5 N NaOH as a dissolution reagent for noncrystalline soil materials may result also in removal of gibbsite and significant amounts of kaolinite, halloysite, free silica, and montmorillonite (Langston and Jenne, 1964; Wada and Greenland, 1970; Tokashiki and Wada, 1972; Higashi and Ikeda, 1974; Fey and LeRoux, 1977; Wilke *et al.*, 1978). Acid ammonium oxalate in darkness has been shown to be a more selective reagent for dissolution of noncrystalline materials (Schwertmann, 1964; Higashi and Ikeda, 1974; Fey and LeRoux, 1977), although it

[1] The term noncrystalline as used in this paper follows the recommendation of Wada (1977) in describing materials having only local and nonrepetitive structure and includes paracrystalline materials (having one-dimensional structural units) such as imogolite.

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Table 1. Description of soils.

Sample number	Depth (cm)	Parent material	Location (soil name)	Major clay minerals[1]	Classification
1041	30–75	Volcanic ash	Kumamoto, Japan (Choyo)	A ≫ Im, V-C	Inceptisol
905	25–55	Volcanic ash	Kumamoto, Japan (Uemara)	Im, A, V-C	Inceptisol
1	0–15	Alluvial volcanic materials	Los Diamantes, Puerto Rico	V-C, A, K, Mi	Entisol
2	23–61	Diabase and gabbro	Prince William Co., Virginia	M > K > Mi	Alfisol
3	76–91	Coastal Plain sediments	Leon Co., Florida (Dothan)	K, V-C, G	Ultisol
4	36–51	Basic lava flows, gneiss and schist	Orange Co., Virginia (Davidson)	K > V-C, G	Ultisol
5	150–200	Serpentinized peridotite	Western Puerto Rico (Nipe)	G > K > V-C	Oxisol

[1] A = allophane; Im = imogolite; V-C = hydroxy interlayer vermiculites or smectites; K = kaolinite; G = gibbsite; Mi = soil mica; M = montmorillonite.

may partially dissolve magnetite (Baril and Bitton, 1969) and perhaps trioctahedral layer silicates (Arshad *et al.*, 1972), which occur in soils as the exception rather than the rule (Schwertmann, 1973). Noncrystalline silica minerals are not dissolved by acid ammonium oxalate treatment (Wada, 1977).

A second problem in using SDA is the assignment of water content to the oxides of Si, Al, and Fe determined by chemical analysis. Noncrystalline materials are noted for their variable chemical composition (van Olphen, 1971) and thus may be expected to have variable water contents. A constant water content has often been assumed for the noncrystalline components, resulting in assignment of these values without regard for sample composition (Jackson, 1964; Alexiades and Jackson, 1966; de Villiers, 1971). However, SiO_2:Al_2O_3 molar ratios have also been used to assign water contents within specified ranges (Jackson, 1974). Fey and LeRoux (1976) assigned water content by comparing the SiO_2:Al_2O_3 ratio of the sample with those of noncrystalline, synthetic aluminosilicate gels of known water content. This method assumes that synthetic materials are adequate models for predicting the characteristics of natural materials. A more satisfactory method would be to measure the weight loss of a given sample after SDA. In spite of the above noted limitations, SDA presently offers the best approach for both chemical characterization and quantification of the noncrystalline material content of soils.

Table 2. Descriptions of standard minerals.

Mineral	Source	Particle size
Gibbsite	Reynolds Aluminum Company (ARD-74-319)	<100 μm
Kaolinite	Washington Co., Georgia (Ga-1)	<2 μm
Montmorillonite	Crook Co., Wyoming (Wy-1)	<2 μm
Vermiculite	Zonalite, Travelers Rest, South Carolina	<2 μm

The objective of this study was to develop a selective dissolution method for the routine determination of the chemical composition and the quantity of noncrystalline materials in soils. To this end, a procedure was tested based upon the loss in weight of a sample after it had been selectively dissolved by ammonium oxalate or sodium hydroxide as a measure of the noncrystalline material content of the sample.

MATERIALS AND METHODS

Brief descriptions of the soils and standard minerals used in this study are given in Tables 1 and 2. Samples were selected to represent a wide range of noncrystalline material content, parent material, and degree of weathering. Samples 1041 and 905 are from Japanese soils derived from volcanic ash deposits and have significant amounts of allophane (Yoshinaga and Aomine, 1962a) or imogolite (Yoshinaga and Aomine, 1962b) in their respective clay fractions.

Samples were buffered at pH 5.0 using a 1 N sodium acetate-acetic acid solution and treated with 30% H_2O_2 to remove organic matter. Oxidation products were removed by several washings with 1 N NaCl. The clay fractions (<2 μm) were dispersed in distilled water by successive ultrasonic treatments. It was necessary to add first dilute NaOH (pH 10) and then dilute HCl (pH 4) to the volcanic ash soils to aid dispersion. The clay fractions were collected by decantation following centrifugation. Samples of soils, soil clays, and standard minerals were K-saturated using 1 N KCl, except for vermiculite which was Na-saturated, washed successively with water, 1:1 water:methanol, 1:1 methanol:acetone, and acetone until free of chloride as

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determined by the $AgNO_3$ test. Samples were then air dried, lightly crushed, and placed in a desiccator over P_2O_5 for storage at constant relative humidity. After 3 days of storage, 300-mg aliquots were dried overnight at 110°C and weighed to determine sample moisture contents.

Noncrystalline materials were extracted with either (1) 50 ml of 0.2 N ammonium oxalate (pH 3.0) for 2 hr on a reciprocating shaker in darkness according to the procedure of Schwertmann (1964) and Fey and LeRoux (1977), or (2) 250 ml of boiling 0.5 N NaOH for 2.5 min in a Ni beaker according to the procedure of Hashimoto and Jackson (1960). Triplicate samples of approximately 250 mg were weighed into preweighed, pretreated Nalgene centrifuge tubes or Ni beakers. A set of blank tubes was carried through each of the procedures to account for any weight loss by the tubes upon heating. Following treatment, the residue and supernatant solutions from the Ni beakers were transferred to preweighed, pretreated centrifuge tubes. Supernatant solutions were collected by decantation after centrifugation and immediately analyzed for Si, Al, and Fe by atomic absorption spectroscopy (AA). The residues were washed three times with 1 N $(NH_4)_2CO_3$ and once with an equal volume of distilled water to remove remaining dissolution-treatment chemicals. The residues were dried in the tubes overnight at 110°C, resulting in volatilization of excess $(NH_4)_2CO_3$ as NH_3, CO_2, and H_2O. Preliminary studies indicated that complete volatilization of 1 N $(NH_4)_2CO_3$ solutions in centrifuge tubes resulted when the solutions were heated at 110°C overnight. Inspection of oven-dry samples following SDA furthermore indicated no visible salt residues. Tubes were allowed to cool in a desiccator over P_2O_5 prior to weighing. A wire hook attached to the frame of the balance pan was used to hold tubes in a vertical position to reduce weighing errors resulting from variable orientation of the tubes on the balance pan.

In addition to removing free iron oxides, a dithionite-citrate-bicarbonate (DCB) treatment may remove significant quantities of noncrystalline Fe, Si, and Al (Wada and Greenland, 1970; Tokashiki and Wada, 1972; Follett *et al.*, 1965; Fey and LeRoux, 1976, 1977). All samples were initially extracted without prior DCB treatment to avoid this possible source of error. Due to the high Fe contents of some samples, rapid re-precipitation of materials dissolved by NaOH occurred before separation of the solid and liquid phases could be completed. Therefore, NaOH extraction was also carried out on deferrated samples. The DCB-extractable Fe, Si, and Al was determined by AA. The total weight loss of all samples following SDA was calculated on the basis of the oven-dry weight (110°C) of undeferrated samples.

Dehydroxylation endotherm peak areas at approximately 280° and 530°C, obtained using a DuPont 990 Thermal Analyzer with a differential scanning calorimeter (DSC) cell, were used to estimate the amounts of gibbsite and kaolinite, respectively, present in samples before and after SDA. This was accomplished using regression equations developed by Sampath and Zelazny (1977) relating peak areas to the weight of gibbsite or kaolinite present in a sample. By analyzing a sample before and after dissolution treatments, reasonably accurate differences in mineral content were obtained, and errors resulting from differences in crystallinity, particle size, and slight variations in thermal properties of soil and standard minerals were minimized. All weight-loss and chemical data were corrected for gibbsite and kaolinite dissolved by SDA using formulae of $Al(OH)_3$ and $Al_2Si_2O_5(OH)_4$, respectively.

RESULTS AND DISCUSSION

Comparison of weight-loss and chemical determination

Chemical and weight-loss data for the noncrystalline material content of the several soil clays examined are shown in Table 3, after correction for the loss of gibbsite and kaolinite as determined by DSC. Weight-loss values were reproducible over a wide range of noncrystalline material contents, with standard deviations of less than 1%. For a comparison of weight-loss and chemical determinations, water contents were assigned on the basis of the SiO_2:Al_2O_3 molar ratio, according to the procedure of Jackson (1974). As would be expected from the heterogeneous compositions, the values are not in complete agreement, although they are similar for both dissolution reagents. Regression equations were calculated to determine the degree of correlation of weight-loss data with chemical data. The appropriate equations for ammonium oxalate and NaOH are:

$$\%\ \text{weight loss} = 0.58 + 1.00\ [\text{sum of oxides, ammonium oxalate + assigned } H_2O]$$

$$r^2 = 0.996$$

and

$$\%\ \text{weight loss} = 2.08 + 0.95\ [\text{sum of oxides, NaOH + assigned } H_2O]$$

$$r^2 = 0.998.$$

The high correlation coefficients (r^2), the intercepts being close to zero, and the slopes being close to one indicate that the proposed weight-loss technique yields results very similar to those obtained by standard SDA techniques. It therefore seems possible to quantify the noncrystalline material content of soils without time-consuming chemical determinations or assuming sample homogeneity and water content. Used in conjunction with chemical analysis, more accurate char-

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Table 3. Chemical and weight-loss determinations of noncrystalline materials from soil clays following selective dissolution analysis.

Sample number	Selective dissolution analysis[1] (%)						DSC analysis[3] (%)	
	SiO_2	Al_2O_3	Fe_2O_3	Sum of oxides	Sum of oxides + H_2O[2]	Weight loss	Gibbsite lost	Kaolinite lost
				Ammonium oxalate				
1041	22.5	29.5	4.8	56.8	71.9	72.2 ± 1.2	0.0	0.0
905	23.4	38.1	8.4	69.9	88.5	85.7 ± 0.8	0.0	0.0
1	2.5	6.8	7.6	16.9	21.4	23.4 ± 0.8	0.0	3.1
2	0.1	0.0	0.5	0.6	0.7	1.2 ± 0.1	0.0	1.9
3	1.0	0.5	0.3	1.8	2.0	2.0 ± 0.0	0.0	0.0
4	0.0	0.0	0.5	0.5	0.6	0.7 ± 0.1	0.0	1.3
5	0.5	0.4	1.5	1.5	1.8	1.7 ± 0.6	0.0	0.3
				NaOH[4]				
1041	30.6	40.6	0.1	71.3	90.3	88.0 ± 1.3	0.0	0.0
905	21.9	34.3	1.1	57.3	72.3	72.1 ± 3.9	0.0	0.0
1	14.6	19.1	1.6	35.3	44.7	42.6 ± 0.5	0.3	13.7
2	5.8	0.0	0.4	6.2	7.8	9.1 ± 0.2	0.0	19.6
3	2.1	1.6	0.1	3.8	4.5	9.3 ± 0.2	2.0	21.6
4	4.6	3.2	0.2	8.0	8.7	10.0 ± 0.2	0.2	17.0
5	0.1	8.3	0.1	8.5	9.2	9.3 ± 0.2	21.5	6.4

[1] All data corrected for loss of gibbsite and kaolinite.
[2] Assignment of water according to the procedure of Jackson (1974) on the basis of SiO_2:Al_2O_3 ratio.
[3] Differential scanning calorimetry.
[4] Extractions on deferrated samples, corrected for removal of Fe_2O_3.

acterization seems possible in that the water content may be measured, instead of estimated.

Comparison of the noncrystalline material content of whole soils and soil clays

Many studies dealing with noncrystalline materials have been conducted using only the clay-size fractions obtained by dispersion procedures. This results from the general assumption that noncrystalline materials and reactive components occur only in this fraction. The relatively few studies of the noncrystalline materials in whole soils (<2 mm) generally deal with CEC measurements or fractionation of Fe materials. Weber and Zelazny (1979) showed that significant amounts of reactive components may occur in the >2-μm fraction of certain Virginia soils. Therefore, it is important to assess the contribution of noncrystalline materials to whole soils as well as soil clays.

Extractions from whole soils and soil clays can be compared if it is assumed that efficient dispersion of the clay fraction resulted by the methods employed and that noncrystalline materials occur only in the clay fraction. Results of this comparison are shown in Table 4. The estimated values are significantly lower than the measured values for whole soils except for the ammonium oxalate extraction of sample 5 in which the estimated value is equal to the measured value. Much of this difference is undoubtedly due to incomplete dispersion of coatings of noncrystalline materials from sand and silt particles. The dissolution of crystalline particles and aggregates may also have contributed to the observed differences, however, Follett *et al.* (1965a) and Jones and Uehara (1973) showed that most noncrystalline soil materials exist as coatings that bind aggregates of minerals together, rather than as separate particles.

Table 4. Comparison of selective dissolution analyses using ammonium oxalate and NaOH for whole soil and clay fractions by weight loss.

Sample number	Clay fraction		Whole soil noncrystalline component	
	Content (%)	Measured noncrystalline component (%)	Estimated (%)[1]	Measured (%)
		Ammonium oxalate		
1041	28.0	72.2	10.2	55.2
905	30.0	85.7	25.7	36.3
1	23.7	23.4	5.5	7.3
2	67.1	1.2	0.8	1.5
3	48.3	2.0	1.0	1.4
4	62.1	0.7	0.4	2.0
5	68.8	1.7	1.2	1.2
		NaOH		
1041	28.0	88.0	24.6	50.4
905	30.0	72.1	21.6	41.4
1	23.7	42.6	10.0	13.6
2	67.1	9.1	6.1	9.0
3	48.3	9.3	4.5	9.1
4	62.1	10.0	6.2	6.9
5	68.8	9.3	6.3	19.2

[1] Estimated whole soil noncrystalline component = (noncrystalline component of clay fraction) × clay content/100.

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Table 5. Effects of extraction with ammonium oxalate and NaOH on pure minerals.

Sample	0.2 N ammonium oxalate		0.5 N NaOH	
	Sum of oxides (%)	Theoretical weight loss (%)	Sum of oxides (%)	Theoretical weight loss (%)
Gibbsite	0.1	0.2	40.8	62.5
Kaolinite	0.0	0.0	4.9	5.7
Montmorillonite	0.2	0.3	2.5	2.9
Vermiculite	0.4	0.5	2.7	4.6

Comparison of NaOH and ammonium oxalate extractants

Several workers, as discussed above, have shown that NaOH is poorly selective as a dissolution reagent for noncrystalline materials. Failure to account for dissolution of crystalline components by NaOH has undoubtedly resulted in overestimation of noncrystalline content in many previous studies. By correcting for the gibbsite and kaolinite dissolved by NaOH and ammonium oxalate, as estimated from DSC analyses, valid comparisons of the effectiveness of the reagents may be attained. The results of dissolution treatments of standard minerals with NaOH and ammonium oxalate are shown in Table 5. Incomplete dissolution (62.5%) of the synthetic gibbsite is likely due to its large particle size and possibly from supersaturation of the extracting solution with respect to gibbsite. The latter alternative was ruled out by repeating the extraction with a 25-mg sample rather than a 250-mg sample (both were extracted with 250 ml of NaOH). The results were similar for both sample sizes, indicating that incomplete dissolution was primarily a result of the large particle size. The NaOH treatment also dissolved 5.7% of the kaolinite, 2.9% of the montmorillonite, and 4.6% of the vermiculite. Extraction of deferrated soil clay materials from sample 5, which is high in both gibbsite and kaolinite, resulted in complete dissolution of gibbsite (51% in the deferrated sample in Table 5, based on undeferrated sample weight) in addition to 15.2% kaolinite and 22% noncrystalline material. This was confirmed by XRD analyses of oriented mounts (Figure 1) and DSC (Figure 2). Gibbsite was dissolved completely from all samples by NaOH treatments, whereas 6.4 to 21.1% of the samples was dissolved as kaolinite (Table 3). Conversely, none of the standard minerals reacted with the acid ammonium oxalate. Essentially no dissolution of gibbsite and minimal dissolution of kaolinite resulted from ammonium oxalate extractions of soil clay materials (Table 3). The small amounts of kaolinite dissolved may reflect the poorly crystalline nature of the soil clay materials since no dissolution of the well-crystalline Georgia kaolinite was observed (Table 5).

Although the preceding discussion indicates that ammonium oxalate is a more selective extractant for noncrystalline materials than NaOH, some problems remain. From the data in Tables 3 and 4, more soil materials appear to be extracted by NaOH than by ammonium oxalate for all samples except sample 905. Differences between these two extractants range from as low as 7.3% for sample 3 to as high as 19.2% for sample 1. The silica and alumina content of the additional materials extracted by NaOH from soil clays are shown in Table 6. These values were obtained by subtracting the SiO_2 or Al_2O_3 obtained by ammonium oxalate extractions from those obtained by NaOH extractions. The SiO_2:Al_2O_3 mole ratio of sample 1041 indicates that the supervenient material extracted by NaOH from this sample is similar to allophane. Fey and LeRoux (1976) similarly reported greater dissolution of allophane-rich clay fractions by NaOH than by ammonium oxalate. The large difference noted for sample 1, which is derived from alluvial volcanic ash materials, may also be the result of enhanced dissolution of allophane-like materials. Both sample 1041 and sample 1 contain volcanic glass, which is subject to dissolution by NaOH but not by ammonium oxalate (Wada, 1977). This may partially explain the differences in extracted SiO_2, but not those of Al_2O_3. An increased dissolution of the imogolite-rich clay fraction of sample 905 by ammonium oxalate resulted in the negative values noted for this sample in Table 6. The reason for this enhanced dis-

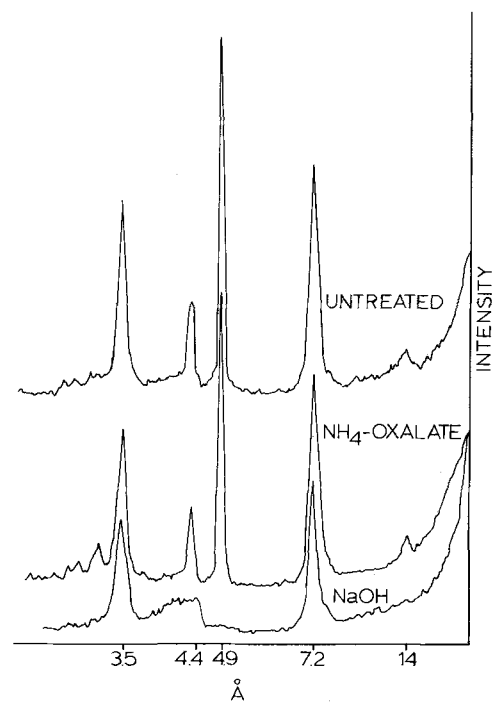


Figure 1. Differential scanning calorimetry endotherms of Pedon 2 (<2 μm) following removal of free Fe and selective dissolution of noncrystalline components with ammonium oxalate and NaOH.

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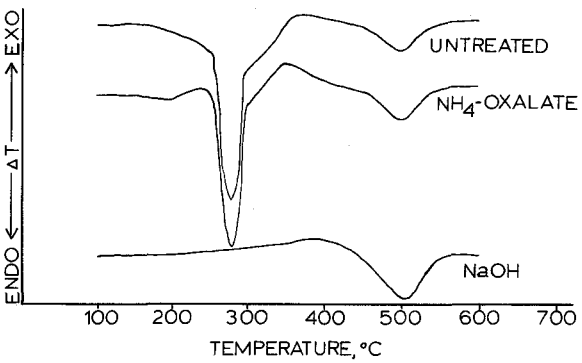


Figure 2. X-ray powder diffractograms of Pedon 2 (<2 μm) following removal of free Fe and selective dissolution of noncrystalline components with ammonium oxalate and NaOH.

solution cannot presently be explained. The additional materials extracted from samples 3 and 4 also have enhanced SiO_2 contents which may be the result of dissolution of 2:1 clay minerals or free SiO_2 from the clay fractions. Quartz peaks were observed on XRD patterns of the clay fraction of these samples (not shown). Extraction of sample 5 with NaOH resulted essentially in supplemental Al_2O_3, most likely the result of dissolution of Al–hydroxy interlayers of vermiculite or smectites that occur in the clay fraction of this soil.

These results support the findings of earlier workers which indicate that NaOH dissolves materials well within the crystalline range, and that ammonium oxalate dissolves materials limited primarily to the noncrystalline range of the soil mineral continuum. Much of the supervenient material extracted by NaOH can be explained, however, further characterization of this material as well as that extracted by ammonium oxalate is needed.

CONCLUSIONS

1. The determination of the noncrystalline material content by loss in sample weight following SDA, washing with $(NH_4)_2CO_3$, and heating agrees well with chemical analysis. Time-consuming chemical analyses are thus avoided, and no assumptions need be made concerning sample homogeneity or water content of the noncrystalline component.
2. If the noncrystalline material content of samples is to be related to soil systems, it is recommended that whole samples be used for analyses, rather than only the <2-μm fractions.
3. The use of acid ammonium oxalate as an extractant is recommended over NaOH because of its greater selectivity for noncrystalline components and because no correction is necessary for the dissolution of gibbsite and kaolinite.

Table 6. Difference in selective dissolution analyses of soil clays with NaOH and ammonium oxalate.[1]

Sample	SiO_2 (wt. %)	Al_2O_3 (wt. %)	SiO_2/Al_2O_3 (molar)
1041	8.1	11.1	1.2
905	(−1.5)	(−3.8)	(−0.7)
1	12.1	12.3	1.7
2	5.7	0.0	—
3	1.1	1.1	1.7
4	4.6	3.2	2.5
5	(−0.4)	7.9	—

[1] Values listed in percent = $SDA_{NaOH} - SDA_{ammonium\ oxalate}$.

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Резюме—Была разработана процедура для оценки содержания некристаллического материала почв, переотложенного из самых разнообразных материнских источников. Процедура основана на потере веса после применения анализа селективного растворения (АСР) и промывания $(NH_4)_2CO_3$. После экстрагирования с помощью 0,2 N щавелевокислого аммония или кипящего 0,5 N раствора NaOH образцы промывались 1 N $(NH_4)_2CO_3$, чтобы удалить излишки растворителей и предотвратить разрушение образцов. Количество некристаллического материала, выделенного из образца экстрагирующим раствором, определялось взвешиванием выщелоченных продуктов после их высушивания до постоянного веса при 110°C. Результаты хорошо согласуются с результатами, полученными с помощью химических анализов продукта растворения и использования соответствующей воды. Предложенный метод потери веса требует меньше времени, чем химический метод, и не требуется никаких предположений об однородности образцов или насыщенности водой некристаллических материалов.

Экстракции всей почвы и рассеянных глинистых частиц указывают, что определения некристаллического материала на глинистых частицах приводят к недооценке содержания некристаллического материала для всей почвы от 0 до 34%. Кислота щавелевокислого аммония оказалась гораздо более селективным экстрагентом для некристаллических материалов, чем NaOH. [N. R.]

Resümee—Es wurde eine Untersuchungsmethode entwickelt, die auf dem Gewichtsverlust beruht, der nach selektiver Auflösung und Waschen mit $(NH_4)_2CO_3$ auftritt. Mit dieser Methode wird der Gehalt an nichtkristallinem Material in Böden bestimmt, das von sehr unterschiedlichem Ausgangsmaterial stammt. Nach der Extraktion mit 0,2 N Ammoniumoxalatlösung oder dem Kochen mit 0,5 N NaOH wurden die Proben mit 1 N $(NH_4)_2CO_3$ gewaschen, um überschüssiges Lösungsmittel zu entfernen und die Dispersion der Probe zu verhindern. Die Menge des aus der Probe durch Extraktion entfernten nichtkristallinen Materials wurde durch Wäugen der bei 110°C konstant getrockneten Auslaugungsprodukte bestimmt. Das Ergebnis kommt nahe an das Ergebnis heran, das bei der chemischen Analyse des Lösungsproduktes unter Berücksichtigung der entsprechenden Wassermenge erhalten wurde. Die vorgeschlagene Gewichtsverlust-Methode erfordert weniger Zeit als die chemische Methode. Außerdem müssen keine Annahmen bezüglich der Probenhomogenität oder des Wassergehaltes des nichtkristallinen Materials gemacht werden.

Die Extraktion des Gesamtbodens und dispergierter Tonfraktionen deutet darauf hin, daß die Bestimmung des nichtkristallinen Materials in den Tonfraktionen den Gehalt an nichtkristallinem Material in den Gesamtböden um bis zu 34% zu klein erscheinen läßt. Es zeigt sich, daß saures Ammoniumoxalat ein viel selektiveres Extraktionsmittel für nichtkristallines Material ist als NaOH. [U. W.]

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Résumé—Une procédure basée sur la perte de poids après une analyse de dissolution sélective (SDA) et un lavage au $(NH_4)_2CO_3$ a été développée pour estimer le contenu de matériel non-cristallin de sols dérivés de mantériaux parents très différents. Après extraction à l'ammonium oxalate 0,2 N ou par des solutions de 0,5 N NaOH bouillantes, les échantillons ont été lavés avec du 1 N $(NH_4)_2CO_3$ pour enlever les agents de dissolution en excès et pour prévenir la dispersion de l'échantillon. La quantité de matériel non-cristallin retiré de l'échantillon par la solution extrayante a été estimée en pesant les produits lessivés, sechés à 110°C à un poids constant. Les résultats sont très proches de ceux obtenus par analyses chimiques du produit de dissolution et l'attribution de l'eau appropriée. La méthode de perte de poids proposée prend moins de temps que la méthode chimique, et on ne dot rien assumer concernant l'homogénéité de l'échantillon ou le contenu en eau du matériel non-cristallin.

Des extractions de fractions de sol entier et d'argile dispersée ont indiqué que les déterminations de matériel non-cristallin sur les fractions d'argile sous-estimaient le contenu de matériel non-cristallin pour les sols entiers de 0 à 34%. L'acide d'ammonium oxalate s'est montré être un extrayant beaucoup plus sélectif pour les matériaux non-cristallins que NaOH. [D. J.]

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