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Clay Mineralogical Analysis Using the <0.05-mm Fraction for Forensic Science Investigation— Its Application to Volcanic Ash Soils and Yellow-Brown Forest Soils

ABSTRACT: Clay mineralogical analysis is conducted with a fraction size of less than 0.05 mm in diameter (<0.05-mm fraction), without extraction of the clay fraction (<0.002 mm), using X-ray diffraction (XRD), infrared absorption (IR), and differential thermal (DTA) techniques. Volcanic ash soils and yellow-brown forest soils were used as samples in this experiment. On the basis of IR spectra, clay mineralogical compositions of volcanic ash soil samples could be classified into four groups. XRD was the most effective to discriminate among the yellow-brown forest soil samples and DTA was sensitive for gibbsite. The following procedure was concluded as the most effective one to discriminate small amounts of soil samples. First, IR is used to classify the samples into an allophane-rich type and a siliceous type. To the former type, DTA is applied, and, to the latter, XRD is applied, followed by DTA. Sample was sufficient for this method, including IR, XRD, and DTA, if about 50 mg of the <0.05-mm fraction was obtained.

KEYWORDS: forensic science, soils, spectroscopic analysis, clay minerals, soil discrimination, volcanic ash soils, yellow-brown forest soils

Soil is an important evidence in forensic science. For discrimination of soil samples in criminal investigation, analysis of primary minerals by petrographic microscope [1,2], density gradient distribution [3–5], particle size [6], color reaction [7], soil color comparison [8], pH measurement [9], and enzymatic analysis [10] have been proposed. However, the method currently used in criminalistics laboratories is exclusively restricted to petrological technique, such as optical observation of sand particles. Even though clay minerals are important constituents of soil materials, only slight regard has been paid to them by forensic scientists for discrimination of soil samples.

A fraction size of less than 0.05 mm in diameter (<0.05-mm fraction), the residue after extraction of sand samples (0.05 to 0.2 mm) for petrological examination, has been discarded in Japanese forensic science laboratories. This is a great omission because, for accurate discrimi-

nation of soil samples, clay mineralogical analysis is indispensable. It is, however, very rare to obtain enough of the clay fraction (<0.002 mm), which is usually used for clay mineralogical analysis, because the amount of soil material adhering to suspected shoes, clothing, or automobiles is very small, and often clay minerals have been lost during extraction, transfer, or washing of clay samples. Hence, it is very useful for criminal investigation if a method can be established for the identification of clay minerals in the <0.05-mm fraction remaining after extraction of sand samples without extracting the clay fraction.

In this paper, a procedure has been devised in which the <0.05-mm fraction instead of the clay fraction, extracted by wet-sieving, is used for analysis and examined for clay mineralogical composition by means of X-ray diffraction (XRD), infrared absorption (IR), and differential thermal (DTA) analysis comparing with results obtained by the usual clay mineralogical analysis. The samples used in this experiment are from volcanic ash soils which are azonal soils widely distributed in Japan and yellow-brown forest soils which are zonal soils distributed in southwest Japan.

Materials and Method

Soil Samples

Volcanic ash soil samples were collected from surface (a) and subsurface (b) layers of eight soil profiles. The surface layer is the top one of the upper layers in the soil profile rich in humus appearing dark brown (A horizon) and the subsurface layer is the brown or yellowish-brown one poor in humus under the dark-brown layers. Location and depth of samples are given in Table 1 and Fig. 1.

Yellow-brown forest soil samples were collected from the A_1 and B_2 horizons of five soil profiles. Location, parent material, and depth of samples are given in Table 2 and Fig. 1.

Preparation of <0.05-mm Fraction

An air-dried fine earth (<2 mm) was subjected to decomposition of organic matter with hydrogen peroxide followed by wet-sieving with a 53-μm (270-mesh) sieve. The suspension that passed through the sieve was centrifuged, and after washing successively with water and ethanol, the deposit was air-dried. Decomposition of organic matter may be omitted for the samples which show light color higher than 6 in value on the Munsell system, because carbon content of these samples is less than 3%. Treatment for dispersion of small particles, for instance, addition of a dispersing agent such as calgon or sodium hydroxide or sonic treatment, is not necessary by this method.

IR

IR was carried out on disks (2.0 cm) prepared from a mixture of <0.05-mm fraction (1 mg) and potassium bromide (200 mg) using a Hitachi Model 345 infrared spectrophotometer.

XRD

XRD was carried out on oriented magnesium-saturated specimens (Mg-specimen) after air-drying and solvation with glycerol, successively, and on oriented potassium-saturated specimens (K-specimen) after air-drying, heating at 300 and heating at 600°C, successively, using a Rigaku-Denki Geigerflex X-ray diffractometer. Nickel filtered copper $K\alpha$ radiation was used and diffractograms were recorded in the range of 15 to 3° (2θ). The specimens were prepared as follows.

TABLE 1—*Volcanic ash soil samples.*

Sample	Location	Depth, cm (a)[a]	Depth, cm (b)[b]
VA-1	Abuta-gun, Hokkai-do	0–11	37–60
VA-2	Katsuta-gun, Iwate	0–11	45–73
VA-3	Karita-gun, Miyagi	0–9	17–28
VA-4	Agatsuma-gun, Gunma	0–8	67–77
VA-5	Inashiki-gun, Ibaraki	0–20	73–110
VA-6	Maniwa-gun, Okayama	0–7	68–93
VA-7	Shimabara-shi, Nagasaki	0–14	29–63
VA-8	Naoiri-gun, Oita	0–20	110–130

[a]Surface soil.
[b]Subsurface soil.
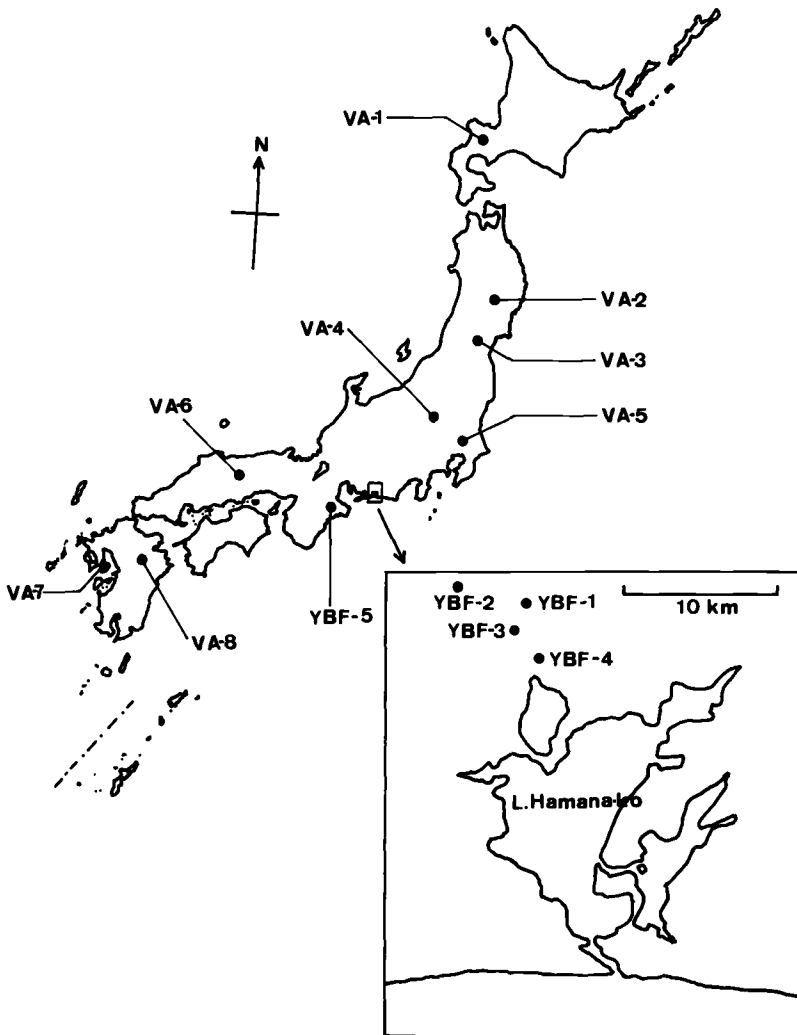
FIG. 1—*Sampling site.*

TABLE 2—*Yellow-brown forest soil samples.*

Sample	Parent Material	Location	Horizon (Depth, cm)
YBF-1	Serpentinite	Inasa-gun, Shizuoka	$A_1(0-9)$, $B_2(28-48)$
YBF-2	Gabbro[a]	Inasa-gun, Shizuoka	$A_{11}A_{12}(0-6)$, $B_2t(18-37)$
YBF-3	Gabbro and diabase	Inasa-gun, Shizuoka	$A_1(0-5)$, $B_2(12-20)$
YBF-4	Chert	Inasa-gun, Shizuoka	$A_1(0-10)$, $B_2(18-40)$
YBF-5	Granite	Taki-gun, Mie	$A_{11}(1-7)$, $B_2(36-56)$

[a]Slightly serpentinized.

Mg-Specimen—The <0.05-mm fraction (15 mg) was weighed into a 10-mL polyethylene centrifuge tube and treated by small-scale Mehra and Jackson's method [11] for removal of free iron oxides, that is, 4 mL of $0.3M$ sodium citrate and 0.5 mL of $1M$ sodium bicarbonate were added, the tube warmed to 80°C in water bath, then 0.1 g of sodium hydrosulfite was added and allowed to stand for 15 min, occasionally stirring. After centrifugation, the deposit was treated with 8 mL of $1N$ sodium acetate-acetic acid buffer (pH 5.0), 8 mL of $1N$ magnesium acetate (pH 7.0), and 8 mL of water, successively. Finally, the deposit was suspended in 0.5 mL of water and then the suspension was decanted on a glass slide and spread to 2.5 × 3.0 cm and allowed to stand to dryness at room temperature.

K-Specimen—Using 15 mg of <0.05-mm fraction, the K-specimen was prepared in the same manner as that of the Mg-specimen, but $1N$ potassium acetate (pH 7.0) and a quartz slide were used instead of $1N$ magnesium acetate and a glass slide, respectively.

DTA

DTA was carried out on 20 mg of <0.05-mm fraction with a Shiku-Riko Ulvac Model TGD-3000 differential thermal microbalance.

Results

Effect of Grinding on Clay Minerals

About 0.5 g of <0.05-mm fraction of YBF-5(B_2) was ground manually for 0 to 60 min with an agate mortar before the instrumental analyses. It had been expected that grinding of the <0.05-mm fraction was necessary to prepare a homogeneous specimen for measurement, but contrary to the expectation, grinding was harmful, as shown in Fig. 2 (0 to 20 min). Diffraction peaks of clay minerals at 1.4 and 0.7 nm in the <0.05-mm fraction were progressively weakened with grinding time and finally disappeared. While a similar tendency was observed in the clay fraction, a decrease in intensity was less marked than in the <0.05-mm fraction. The results indicate that the destruction of the crystal structure of clay minerals by grinding is enhanced by coexisting hard primary minerals such as quartz and feldspar, which are more abundant in the <0.05-mm fraction than in the clay fraction. Therefore, grinding of the <0.05-mm fraction must be carried out only for mashing clods as mildly as possible with an agate mortar, for the minimum amount of time.

IR

Volcanic Ash Soils—Although IR spectra of the <0.05-mm fraction showed somewhat siliceous patterns compared with those of the clay fraction, they could be classified into four
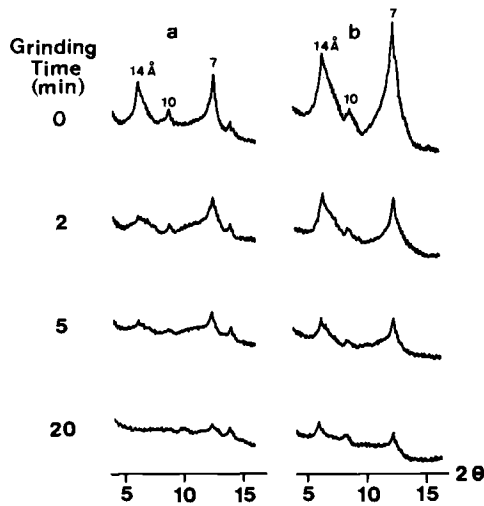
FIG. 2—*Effect of grinding on diffractogram.* (a) *<0.05-mm fraction of YBF-5(B$_2$) and* (b) *clay fraction of YBF-5(B$_2$).*

groups (Type 1 to Type 4) on the basis of categories simplified from Kobo and Oba's classification system [12], which had been proposed for characterization of clay fraction in volcanic ash soils.

Type 1 is dominated by allophane. Its spectrum shows strong broad absorption bands at 960 to 990 cm^{-1} and 570 to 540 cm^{-1}, the former band shifts toward 990 from 960 cm^{-1} and the latter shifts toward 540 from 570 cm^{-1} with decrease of allophane content, respectively. Spectra of comparatively siliceous samples show weak absorption bands at 1100 and 480 cm^{-}1 because of the Si-O bond.

Type 2 is dominated by crystalline clay minerals. In its spectrum, absorption bands caused by the Si-O bond appear more clearly than in Type 1, and absorption bands caused by Si-OH appear at 920 and 3620 cm^{-1}, which are characteristic of kaolin minerals.

Type 3 is dominated by amorphous silica. Its spectrum shows two strong broad absorption bands at 1090 and 480 cm^{-1} and a medium absorption band at 900 cm^{-1} caused by amorphous silica.

Type 4 contains gibbsite. Its spectrum shows sharp absorption bands at 3535 and 3450 cm^{-1} due to gibbsite. This type is used in combination with the others for the samples whose spectra show the characteristics of the other types, for example, Type 4-2.

Typical IR spectra of each type and the results of classification of clay mineralogical composition in the <0.05-mm fraction of the volcanic ash soil samples were given in Fig. 3.

Yellow-Brown Forest Soils—All spectra of the yellow-brown forest soil samples showed characteristics of Type 2 mentioned above. Among them, YBF-5(A$_{11}$) and (B$_2$) showed those of Type 4-2, and YBF-1(A$_1$) and (B$_2$) showed no absorption band caused by kaolin minerals at the OH stretching region; however, other absorption bands agreed with those of Type 2. Absorption bands caused by kaolin minerals in the OH stretching region in the yellow-brown forest soil samples were clear and sharp doublet (3700 and 3620 cm^{-1}) with the exception of YBF-1(A$_1$) and (B$_2$), whereas Type 2 of the volcanic ash soil samples showed only a single small absorption band at 3620 cm^{-1}. This fact indicates that kaolin minerals in yellow-brown forest soils have higher crystallinity than those in volcanic ash soils. In the spectra of YBF-4(A$_1$) and (B$_2$), which were from the soil developed from chert, a strong doublet absorption band of quartz at about 800 cm^{-1} were observed.
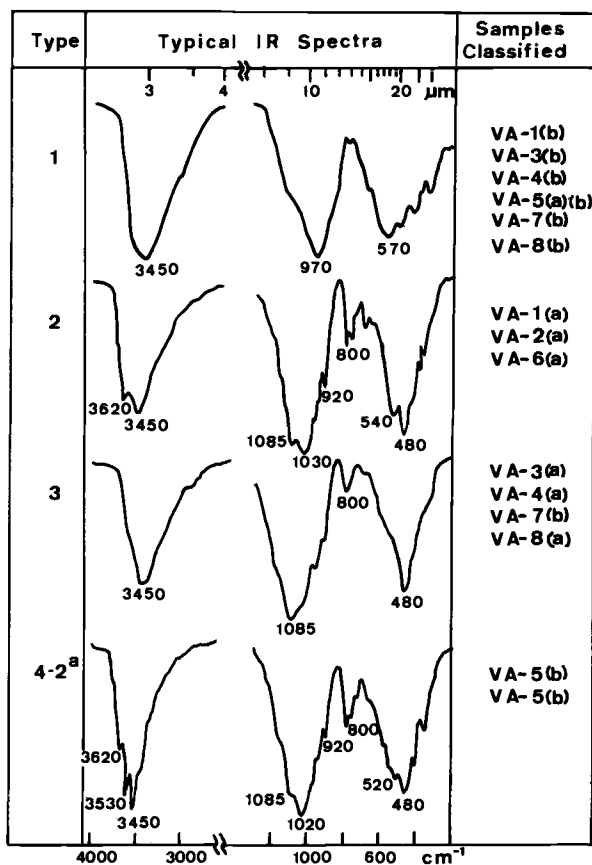
FIG. 3—*Typical IR spectra of volcanic ash soils and classification of the samples. [a]The spectrum shows the characteristics of Types 4 and 2 together.*

XRD

Clay minerals in soils were identified according to the variation of diffraction peaks (001) given in the Appendix.

Volcanic Ash Soils—Crystalline clay minerals were not always major clay minerals in the volcanic ash soil samples. Diffraction peaks of clay minerals were observed chiefly in the siliceous samples whose IR spectra showed Type 2 to Type 3. Especially VA-7(b), which belonged to Type 1, showed a diffraction peak at 1.4 nm. The XRD patterns of volcanic ash soil samples and the results of identification were given in Fig. 4 and the results in Table 3, respectively.

Yellow-Brown Forest Soils—As diffraction peaks of crystalline clay minerals were observed in all samples, XRD was an effective technique to discriminate the samples from yellow-brown forest soils. The XRD patterns of yellow-brown forest soil samples were given in Fig. 5 and the results were given in Table 4.

The clay fraction of the yellow-brown forest soil samples were subjected to XRD to compare the results of this method with those of usual clay mineralogical analysis. The results (Table 5) showed that diffraction peaks of clay minerals were slightly weakened by using the <0.05-mm fraction instead of the clay fraction. There was, however, no error in identification of clay minerals; they were identified clearly by the use of the <0.05-mm fraction.
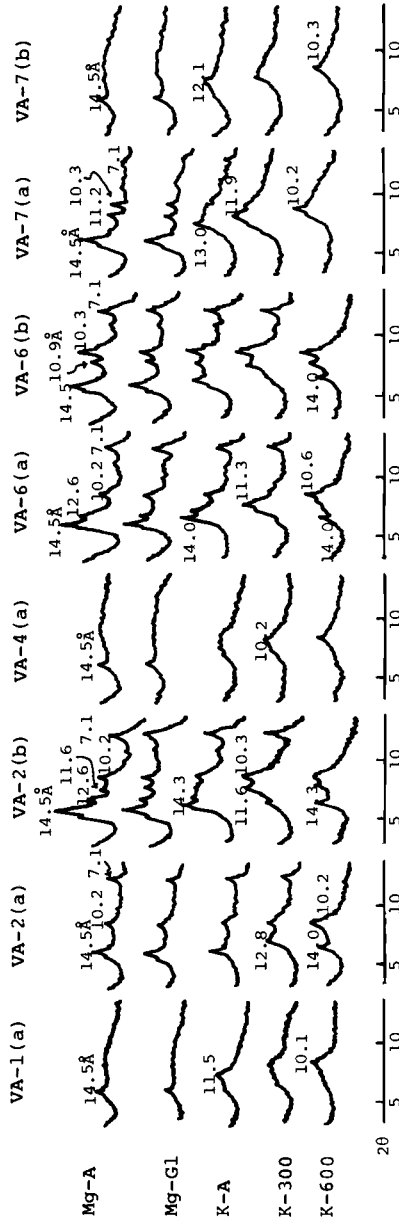
FIG. 4—XRD patterns of the <0.05-mm fractions of volcanic ash soil samples. Abbreviations: Mg, Mg-saturated specimen; K, K-saturated specimen; A, air-dried; Gl, glycerol-solvated; 300, heated at 300°C; and 600, heated at 600°C.

TABLE 3—*Clay mineralogical composition in the <0.05-mm fraction of the volcanic ash soil samples by XRD.*[a]

Sample	Layer	Ch	Al	Mc	Ch/Mc	Al/Mc	Kn
VA-1	(a)		±				
VA-2	(a)	+	+	±			+
	(b)	+	+ +	+	±		+ +
VA-4	(a)		±				
VA-6	(a)	+	+ + +	+		±	+
	(b)	+	+ +	+ +	±		+ +
VA-7	(a)		+	±	±		
	(b)		±				

[a] Abbreviations—minerals: Ch, chlorite; Al, aluminum interlayer clay minerals; Mc, mica; X/Y, interstratified clay mineral of X and Y; Kn, kaolin minerals (without halloysite). Abundance: + + +, abundant; + +, medium, +, little; ±, very little.
The volcanic ash soil samples that are not listed here showed no diffraction peak of clay minerals.
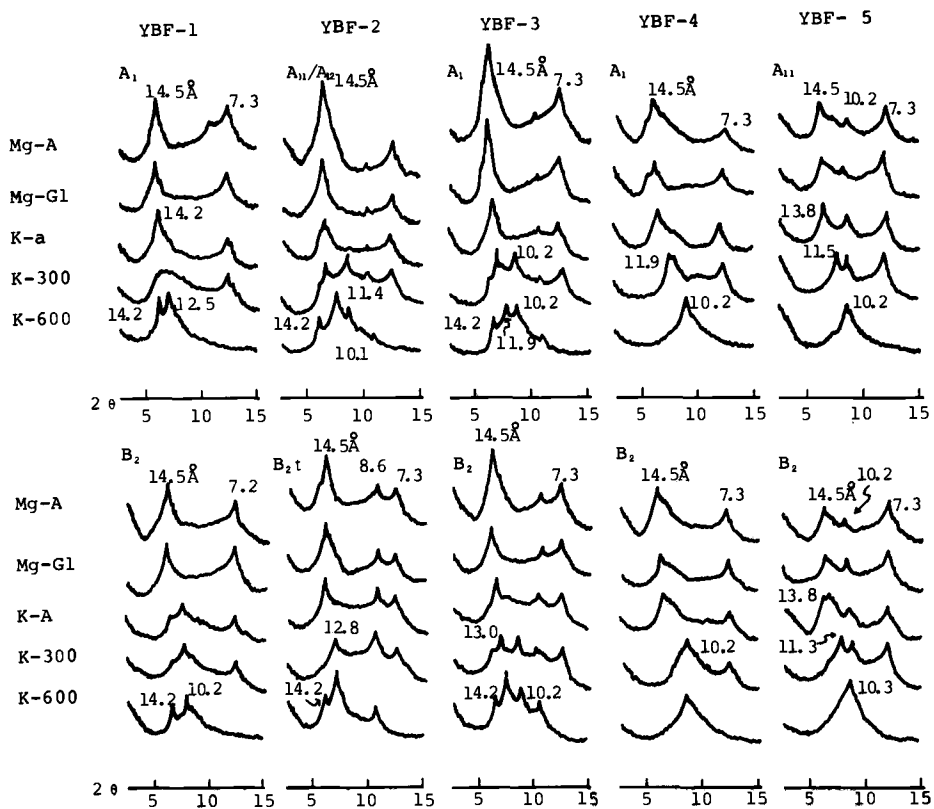

FIG. 5—*XRD patterns of the <0.05-mm fractions of yellow-brown forest soil samples. Abbreviations: Mg, Mg-saturated specimen; K, K-saturated specimen; A, air-dried; Gl, glycerol-solvated; 300, heated at 300°C; and 600, heated at 600°C.*

TABLE 4—*Clay mineralogical composition in the* <0.05-*mm fraction of the yellow-brown forest soil samples by XRD.*[a]

Sample	Horizon	Ch	Vr	Al	Mc	Ch/Al	Ch/Mc	Al/Mc	Kn
YBF-1	A_1	++				+			+
	B_2	++				+			+
YBF-2	A_{11}/A_{12}	+				+			+
	B_2t	+				++			+
YBF-3	A_1	+		±		+			+
	B_2	+		+		+			+
YBF-4	A_1			+				±	++
	B_2			+				±	++
YBF-5	A_{11}		+	+	±			±	++
	B_2			+	±			±	++

[a] Abbreviations—minerals: Ch, chlorite; Vr, vermiculite; Al, aluminum interlayer clay minerals; Mc, mica, X/Y, interstratified clay mineral of X and Y; Kn, kaolin minerals (without halloysite). Abundance: +++, abundant; ++, medium; +, little; ±, very little.

TABLE 5—*Clay mineralogical composition in the clay fraction of the yellow-brown forest soil samples by XRD.*[a]

Sample	Horizon	Ch	Vr	Al	Mc	Ch/Al	Al/Mc	Kn
YBF-1	A_1	+++				++		±
	B_2	++				+++		±
YBF-2	A_{11}/A_{12}	++				+++		++
	B_2t	++				+++		++
YBF-3	A_1	++		±		++		++
	B_2	++		+		++		++
YBF-4	A_1			+++	+		++	++
	B_2			+++	+		++	++
YBF-5	A_{11}		++	++	+		++	++
	B_2		±	++	+		++	++

[a] Abbreviations—minerals: Ch, chlorite; Vr, vermiculite; Al, aluminum interlayer clay minerals; Mc, mica; X/Y, interstratified clay mineral of X and Y; Kn, kaolin minerals (without halloysite). Abundance: +++, abundant; ++, medium, +, little; ±, very little.

DTA

Clay minerals were identified according to DTA curves as shown in Table 6.

Volcanic Ash Soils—On the basis of peak height at 100°C, large amounts of allophane were estimated in the samples of Type 1. On the other hand, allophane was absent or in small amounts in the samples of Type 3. In the other samples, content of allopane was presumed to be intermediate between the above two groups.

A large amount of gibbsite was estimated in the samples of Type 4-2. Nevertheless, absorption bands due to gibbsite had not been observed in IR spectra; a small amount of gibbsite was identified in VA-2(a), VA-3(a), VA-5(a) and (b), VA-7(b), and VA-8(b) by DTA, which is more sensitive for gibbsite than IR.

An exothermic peak at about 300°C was observed in VA-1(a), VA-4(a), VA-6(a), VA-7(a),

TABLE 6—*Identification of clay minerals according to DTA curves.*

Clay Mineral	Endothermic Peak, °C	Exhothermic Peak, °C
Allophane	100[a]	920[b]
Gibbsite	270	
Amorphous iron oxide[c]		300
Kaolin minerals	500	900[b]

[a]Broad peak.
[b]This peak does not always appear.
[c]Or organic matter that had not been decomposed completely by peroxidation.

and VA-8(a). It may be due to amorphous iron oxide, but there is doubt that it is due to organic matter that had not been decomposed completely by peroxidation, because it was observed exclusively in the samples from surface soil.

Yellow-Brown Forest Soils—A large endothermic peak of gibbsite was observed in YBF-5(B_2). A broad doublet exothermic peak at about 300°C was observed in YBF-5(A_{11}). This doublet was probably formed by the overlap of the endothermic peak of gibbsite and the exothermic peak of iron oxide or organic matter resistance to peroxidation. An endothermic peak caused by kaolin minerals was observed in all samples.

Discussion

The samples from volcanic ash soils are classified into four groups (Type 1 to Type 4) on the basis of IR spectra. For discrimination of the samples of Type 1, DTA is useful and XRD is not so useful. Among seven soil samples of Type 1, VA-8(b) is characterized by a very large amount of allophane, VA-5(b) is characterized by a large amount of allophane with gibbsite, and VA-5(a) and VA-7(b) are characterized by gibbsite according to DTA. XRD can discriminate only VA-7(b) from the other Type 1 samples. Remaining Type 1 samples, VA-1(b), VA-3(b), and VA-4(b), cannot be discriminated from each other by the technique used here. For discrimination of these soil samples which contain only amorphous clay minerals such as allophane, clay mineralogical analysis brings less information than the other techniques, such as optical observation of sand particles.

For discrimination of the volcanic ash soil samples of Type 2 to Type 4, XRD is effective because of the presence of crystalline clay minerals with the exception of two samples of Type 3, VA-3(a) and VA-8(a). They are, however, discriminated from each other by the presence of a small amount of gibbsite in VA-3(a) based on DTA.

XRD is the most effective among the techniques examined here to discriminate the yellow-brown forest soil samples, as these soils are dominated by crystalline clay minerals. While IR is less effective for discrimination of these soil samples, it is useful to distinguish soil samples to which XRD is applicable from the others by the use of only 1 mg of <0.05-mm fraction.

From the results mentioned above it is concluded that the following procedure is the most effective to discriminate between small amounts of soil samples in crime investigation (Fig. 6). At the beginning IR is applied to the <0.05-mm fraction which remains after the extraction of sand particles for petrological examination. To samples classified as Type 1, DTA is applied. The amount of the <0.05-mm fraction necessary for the above analyses is 21 mg. Then XRD is applied if the <0.05-mm fraction remaining after the above analyses is more than 30 mg. To samples classified as Type 2 to Type 4 by IR, XRD is applied. The amount of the <0.05-mm fraction necessary for the above analyses is 31 mg. Finally, DTA is applied, if the <0.05-mm fraction remaining after the above analysis is more than 20 mg.

The amount of the <0.05-mm fraction required for the whole procedure of this method is about 50 mg.
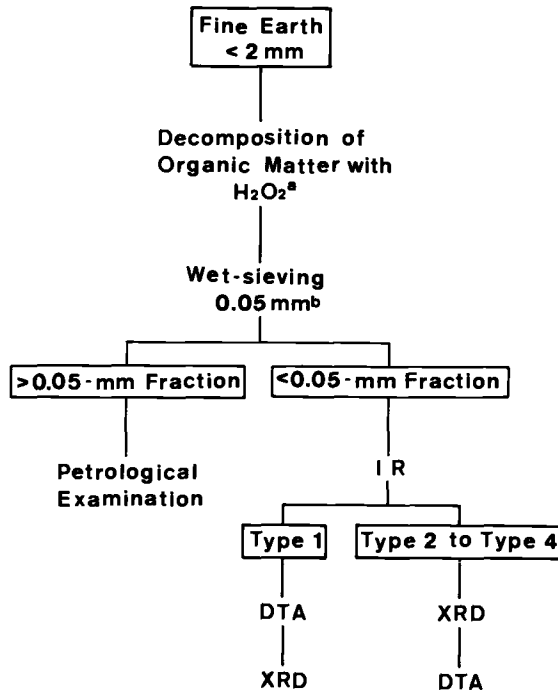
FIG. 6—*Procedure of clay mineralogical analysis for discrimination of small amount of soil samples.* *[a] Peroxidation can be omitted for samples which show light color higher than 6 in the value of Munsell system. [b] 53-μm (270-mesh) sieve.*

APPENDIX

XRD is performed on oriented clay specimens after the following treatments, and clay minerals are identified according to the variation of the diffraction peak of basal spacing (001).

1. Air-drying of magnesium-saturated clay specimen: a suspension of clay, which has been treated with 1*N* magnesium acetate (pH 7.0) previously, is spread on a glass slide and air-dried (mentioned previously as Mg-specimen).

2. Solvation of Mg-specimen with glycerol: On the above specimen (1) after measurement of XRD, 5% glycerol (in ethanol) is sprayed, and the specimen is allowed to stand for 1 h to evaporate ethanol.

3. Air-drying of potassium-saturated clay specimen: Suspension of clay, which has been treated with 1*N* potassium acetate (pH 7.0) previously, is spread on a quartz slide and air-dried (mentioned previously as K-specimen).

4. Heating of K-specimen at 300°C: The above specimen (3), after measurement of XRD, is heated at 300°C for 1 h in an electric furnace.

5. Heating of K-specimen at 600°C: The above specimen (4), after measurement of XRD, is heated at 600°C for 1 h in an electric furnace.

Chlorite

Chlorite has 1.4-nm basal spacing and its spacing is never varied by any treatment, so the diffraction peak is always observed at 1.4 nm in the specimens after Treatments 1 through 5. While the peak is weakened by heating at 300°C, it becomes stronger by heating at 600°C.

Smectite

Smectite (montmorillonite) is characterized by swelling when wetted and shrinking when drying. In an air-dried Mg-specimen, smectite shows 1.4-nm basal spacing. The spacing swelled to 1.8 nm by solvation with glycerol, then collapsed to 1.2 to 1.3 nm (sometimes to 1.0 nm) showing a broad diffraction peak by potassium saturation, and entirely collapsed to 1.0 nm by heating at 300°C.

Vermiculite

Vermiculite is characterized by collapsing when treated with potassium ion. The basal spacing shows 1.4 nm in an air-dried Mg-specimen; it is not swelled by solvation with glycerol. By the treatment of potassium saturation, the spacing is collapsed to 1.0 nm and, furthermore, not varied by heating.

Mica

Mica as a clay mineral (illite) has the same crystalline structure as that of rock-forming mica minerals (primary minerals). The basal spacing of mica is 1.0 nm, and it is never varied by any treatments.

Kaolinite and Metahalloysite

Kaolinite and metahalloysite show 0.7 nm basal spacing by Treatments 1 through 4, and they are destroyed by heating at 600°C. The diffraction peak at 0.7 nm disappeared after heating at 600°C.

Halloysite

Halloysite is a water-solvated type of metahalloysite, characterized by collapsing when heated, losing water in its interlayer region. Halloysite shows a 1.0 nm basal spacing in the air-dried Mg-specimen, and it is not swelled by solvation with glycerol and not collapsed by potassium saturation. The spacing is collapsed to 0.7 nm by heating at 300°C, and destroyed by heating at 600°C.

Aluminum Interlayer Clay Minerals

Aluminum interlayer clay minerals are smectite or vermiculite, which have aluminum in their interlayer region (chloritized smectite or vermiculite). The basal spacing in an air-dried Mg-specimen is 1.4 nm; it is not varied by solvation with glycerol and potassium saturation. The spacing collapsed progressively by heating toward 1.0 nm; the diffraction peak shifted to 1.2 to 1.3 nm by heating at 300°C and completely reached 1.0 nm by heating at 600°C. To distinguish chloritized smectite and chloritized vermiculite, Tamura's treatment [13] is applied, that is, by the treatment, chloritized smectite shows swelling to about 1.8 from 1.4 nm, whereas chloritized vermiculite does not show any change.

Chlorite/Mica

Chlorite/mica is a randomly interstratified clay mineral composed of a chlorite layer and mica layer. Weaver [14] reported that interstratified clay mineral appears as if it had a basal spacing of average value of constituent minerals resulting from the simultaneous scattering by each type of layer. Chlorite/mica shows a diffraction peak between both basal spacing chlorite (1.4 nm) and mica (1.0 nm), which is never varied by any treatment.

Chlorite/Aluminum Interlayer Clay Mineral

This interlayer clay mineral shows a diffraction peak at 1.4 nm in an air-dried Mg-specimen; it is not varied by solvation with glycerol and potassium saturation. The diffraction peak shifted toward 1.0 nm by heating, but never reached 1.0 nm.

Chlorite/Vermiculite

Chlorite/vermiculite exhibits similar characteristics to those of vermiculite, but collapsing by potassium saturation is incomplete. While the diffraction peak shifts toward 1.0 nm from 1.4 nm, it never reaches 1.0 nm.

Chlorite/Smectite

Chlorite/smectite exhibits similar characteristics to those of smectite, but swelling and collapsing are incomplete and the diffraction peak never reached 1.8 and 1.0 nm by solvation with glycerol and potassium saturation, respectively.

Smectite/Mica, Vermiculite/Mica, and Aluminum Interlayer Clay Mineral/Mica

These show a diffraction peak between 1.4 and 1.0 nm in an air-dried Mg-specimen, and exhibit similar characteristics to those of smectite, vermiculite, or aluminum interlayer clay mineral, respectively.

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