that, if the presence of anorthoclase is suspected, supplementary thin-section and X-ray studies precede modal analysis of stained slabs.

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HYDRAZINE INTERCALATION—INTERSALATION FOR DIFFERENTIATION OF KAOLIN MINERALS FROM CHLORITES

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It has long been a problem to differentiate the diffraction peak of kaolinite (001) from that of chlorite (002) at 7 Å. Preferential changes in the intensities of these diffraction peaks by heating or acid treatments have been proposed as means of distinction. However, variations in crystallinity and chemical composition, mainly of chlorites, often result in inconclusive differentiation (Brindley, 1961, p. 85 and 262). Resolution of the kaolinite (002) and the chlorite (004) at about 3.5 Å by slow x-ray diffractometer scans (Biscaye, 1964) has merits in simplicity and clarity,

but its application may also be restricted depending on the crystallinity and chemical composition of the chlorite.

Intersalation of kaolin minerals results in shifts of their basal diffraction peaks to larger spacings. Since no similar lattice expansion occurs for chlorites, it provides a definitive criterion for the differentiation of the 7 Å kaolin mineral from the chlorite (Andrew *et al.*, 1960; Wada, 1961). The only difficulty in its application lies in the procedure of grinding, with KCH$_3$COO (KOAC) crystals, which is sometimes detrimental to clays of lower crystallinity such as those in some soils and sediments. Later, Weiss *et al.* (1963) reported an effective intercalation of hydrazine into kaolinites and suggested the use of hydrazine as a "Schlepper" for intersalation. Application of this proposal to clay analyses, however, has not been attempted. The purpose of the present paper is to present procedures for kaolinite-chlorite differentiation based on hydrazine intercalation followed (I) or not followed (II) by intersalation.

I. HYDRAZINE INTERCALATION—INTERSALATION

Take about 50 mg of K saturated clay either as dry powder or sediment from suspension in a 10 ml centrifuge tube. Previous removal of iron oxide extractable in appropriate reagents is preferable. Add 2 ml of hydrazine monohydrate $N_2H_4 \cdot H_2O$, stopper, shake and centrifuge the tube for 5 minutes at 2000 to 3000 rpm. Discard the supernatant, add another 2 ml of hydrazine, stopper, shake, allow to stand for 24 hours at 40 to 50°C and centrifuge again. Add 5 ml of 4 *M* NH$_4$Cl or 8 *M* KOAC to the hydrazine treated clay, stopper, shake and repeat the centrifuge washing twice. Add another 5 ml of the respective salt solution, allow to stand overnight and centrifuge again. Remove the excess salt by washing the NH$_4$Cl treated clay with 8 ml of methyl-isopropyl alcohol mixture (1:1) and the KOAC treated clay with 8 ml of isopropyl alcohol. Repeat a 5 ml isopropyl alcohol wash twice more. Smear a portion of the drained clay on a glass slide and X ray the air-dry sample. Read the peak height on the chart at 7, 10 and 14Å, and calculate the relative intensities 10 Å/7 Å and 14 Å/7 Å for the NH$_4$Cl and KOAC treated samples, respectively. Their concurrent increase in comparison with those for the original K saturated clay, together with an appearance of the 11.6 Å diffraction peak for the KOAC sample heated at 150 to 200°C proves conclusively the presence of the 7 Å kaolin mineral even in the presence of chloritic minerals (Fig. 1).

II. HYDRAZINE INTERCALATION

Dry the suspension containing 10 to 20 mg of K saturated clay, preferably with extractable iron oxide previously removed, on a glass

slide and make an X-ray scan. Carefully transfer $N_2H_4 \cdot H_2O$ with a small pipette in an amount just enough to wet the oriented clay (ca. 0.02–0.05 ml). Allow the slide to stand overnight or longer in a closed petri dish in which free $N_2H_4 \cdot H_2O$ is also placed in a small dish. Remove the excess hydrazine by air-drying and X-ray the slide on which the clay is still moist. Enhancement of the 10 Å peak relative to the 7 Å peak in comparison with the first scan suggests the presence of the 7 Å kaolin mineral. Confirmation is obtained by the reversion which occurs by allowing the slide to stand in air overnight, or by heating to 100–300°C.

The reference kaolin specimens listed in Table 1, except for the dickite,

TABLE 1. LIST OF REFERENCE SPECIMENS

Mineral species	Locality	Particle size
Dickite	Shokozan, Hiroshima	−150 mesh
Dickite-nacrite	San Juanito, Mexico	2–20 μ
Anauxite	Ione, California	−150 mesh
Kaolinite	Keokuk, Iowa	−20 μ
	Mesa Alta, New Mexico	−2, 2–20 μ
	Macon, Georgia	−2, 2–20 μ
	Ibuski, Kagoshima	−150 mesh
Halloysite	Yoake, Oita	−150 mesh
Chlorite	Muramatsu, Nagasaki	−150 mesh
	Besshi, Ehime	−150 mesh
	Kuroshiroishi, Fukushima	2–20 μ
Antigorite	Sasaguri, Fukuoka	−150 mesh
Chrysotile	Kashii, Fukuoka	−150 mesh
Aluminian serpentine	Usagiyama, Fukuoka	−150 mesh

dickite-nacrite and Keokuk kaolinite, gave complete intercalation of hydrazine and intersalation within 24 hours. A longer time, 2 to 3 days, was necessary for the latter coarse and well-crystallized specimens. The intercalation of hydrazine is slowed down further to some extent for these specimens at lower temperatures such as 10°C, but this temperature effect was not appreciable for the other kaolinites and halloysite. Thus, even the procedure II ensures the near completion of hydrazine intercalation for ordinary kaolinites and halloysites. Weiss *et al.* (1963) noted a retardation of hydrazine intercalation for disordered kaolinites in comparison with the well-crystallized one, but in the present study, the halloysite heated at 200°C as well as metahalloysites (Fig. 2; left) and disordered kaolinites (Fig. 2; right) in soils responded quickly to the

treatment. Several chlorite and serpentine specimens listed in Table 1 and those in soils and sediments tested did not expand with the treatment.

The hydrazine-kaolin complex gives its strong (001) diffraction peak at 10.4 Å (Weiss *et al.*, 1963). One must be sure that the complex collapses in air when the excess hydrazine has been removed by drying. The
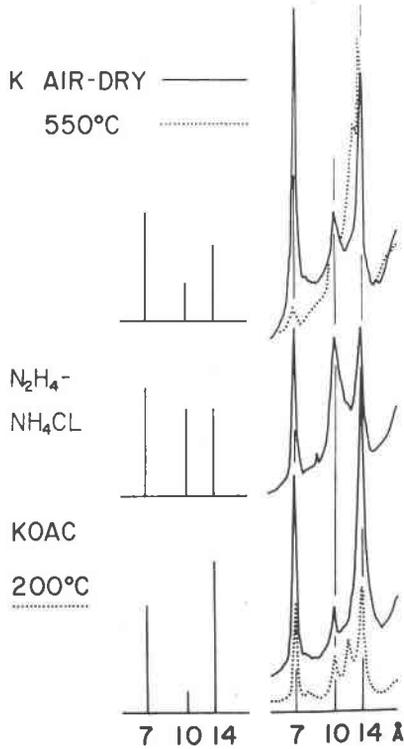

Fig. 1. X-ray diffraction patterns for the $-2\,\mu$ fraction from the 6th horizon of a paddy soil at Hinai, Akita showing differentiation of the 7 Å kaolin mineral in the presence of chlorite and illite. The line diagram indicates changes in the relative intensities of the diffraction peaks at 7, 10 and 14 Å in the hydrazine intercalation—intersalation procedure.

reaction, however, is not always reversible as claimed by Weiss *et al.* (1963). The incomplete collapse in spacing, and a considerable broadening and decrease in the intensity of the 7 Å diffraction peak suggest partial fixation of hydrazine. The complete reversion often requires heating at 200 to 300°C.

Washing the salt-kaolin complexes with water and air-drying resulted

in the following variable spacings (Å), where the values in parentheses give spacings of minor diffraction peaks.

	NH_4Cl complex	KOAC complex
Dickite	10 (7)	7
Dickite-nacrite	10 (7) & 8.35	7 & 8.35
Kaolinite I	10 (7)	7
Kaolinite II	7 (10)	7
Halloysite	10	10

The 8.35 and 10 Å diffraction peaks obtained for the nacrite and halloysite were ascribed to the formation of their water complexes, whereas the
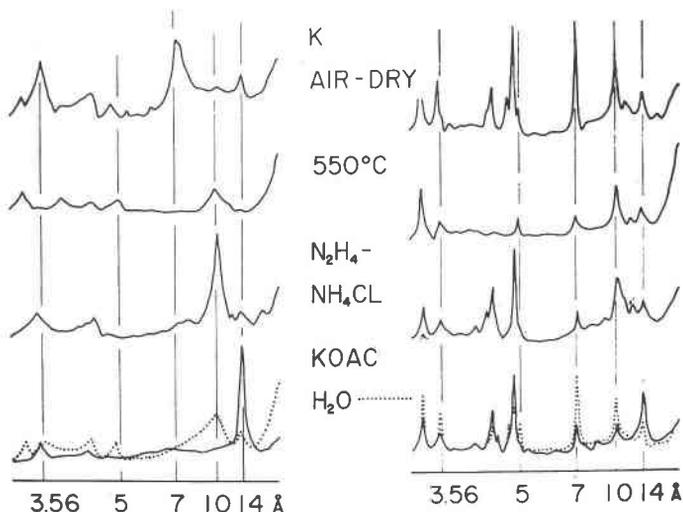

FIG. 2. X-ray diffraction patterns for the $-2\,\mu$ fractions of the two soils showing the effect of hydrazine intercalation—intersalation on the 7 Å kaolin minerals. Left; from the AB horizon of a Red-Yellow Podzolic soil developed on a basaltic rock at Oura, Saga (Higashi and Aomine, 1958). Right; from the 3rd layer of a volcanic ash deposit at Kuroishibaru, Kumamoto (Wada and Aomine, 1966).

10 Å diffraction peak for the dickite and kaolinite to the partial NH_4Cl fixation between the kaolin layers (Wada, 1965a; 1965b). The result for kaolinite I was obtained for the Keokuk specimen; the other reference kaolinites together with the anauxite gave values listed for kaolinite II. The Keokuk specimen was proved to be well-crystallized kaolinite on the basis of X-ray diffraction, but yielded a "dickite-like" DTA curve with an asymmetric endothermic peak at 690–695°C (Keller *et al.*, 1966). The correlations between the mineral species and intercalation phenomena may give a guide for the species differentiation in a multi-component

mixture, where the identification based on the powder pattern is often difficult.

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HIGH PRESSURE EPOXY IMPREGNATION OF POROUS MATERIALS FOR THIN-SECTION AND MICROPROBE ANALYSIS[1]

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The need for preparing thin sections and microprobe samples from porous and friable rock specimens such as altered pyroclastics and manganese nodules led to devising a successful method of high pressure impregnation with a thermally-resistant epoxy resin. The method described below provides thorough penetration and rigid cementation of materials which could not be treated satisfactorily by conventional means. In brief, the process consists of encapsulating small specimens in epoxy-filled rubber bags after vacuum treatment to remove trapped gases, followed by compression in a cylindrical die at 12,000 lb/in² to drive in