Special Report

CLAY MINERALOGY OF A GUMBOTIL

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ABSTRACT

Investigation of the clay minerals in a gumbotil associated with the Mahaska soil series in Keokuk County, Iowa, indicates that the principal clay mineral is a dioctahedral calcium montmorillonite. Other clay minerals present in this gumbotil are kaolinite and mica, although they are in much smaller percentages. The investigation also pointed out difficulties involved in analyzing this type of soil by standard X-ray procedures and the complications introduced in the analysis of soil samples with high iron content utilizing standard accepted diffraction techniques.

INTRODUCTION

Gumbotil is a heavy clay soil, usually gray in color and 5 to 20 feet thick, commonly underlying the Wisconsin loess in southern Iowa (1). In better drained locations the color is red-brown and the deposit has been termed "ferreto" (1). Where encountered in foundation excavations, highway cuts, or on eroded slopes it poses unique problems because of its high plasticity and shrinkage and swelling properties. Most gumbotil in this area is believed to be the B horizon of a Yarmouth-Sangamon paleosol formed in Kansas age glacial till. Less extensive gumbotil is associated with Nebraskan and Illinoian age tills.

The clay mineralogy of gumbotil has been a source of concern for engineers for years because of the high swelling and shrinkage properties which these minerals impart to the soil. As a result of these properties, it is probably one of the most difficult soils to work with in the field. Previous analyses have shown the predominance of a calcium-saturated montmorillonite group mineral (1).

X-ray diffraction analysis has been the means principally employed for the analysis of the clay fraction of natural soils. Difficulties inherent in determinations of clay minerals by X-ray methods arise from the fact that clays show wide variation in crystallinity and tend to orient preferentially during preparation for X-ray analysis. Since many of the clay minerals have basal spacings which are similar, various additives have been used to separate the various minerals based on expansive properties due to adsorption. Furthermore, clay minerals with different associated exchangeable cations can be separated and identified on the basis of adsorption.

Greene-Kelley (2) demonstrated in his studies on dehydration of montmorillonites that small interlayer cations such as lithium promote an irreversible decrease in sorption. His studies further showed that trioctahedral montmorillonite re-expands freely upon glycolation after lithium saturation and dehydration, while dioctahedral montmorillonites fail to show this property unless substitution occur in tetrahedral positions.

An investigation of the clay

mineralogy of a gumbotil was conducted. The purpose of the study was to identify the clay minerals present in the soil, and to check the validity of the X-ray analysis techniques in use for classifying dioctahedral and trioctahedral clay minerals in soils.

SOIL PROPERTIES

The sample selected for study was laboratory No. 528-8, samples from a roadcut at NW. 1/4 NW 1/4 Sec. 7 T75NR 10W, Keokuk County, Iowa (1, Fig. 9).

Physical and chemical properties for this soil sample are shown in Table 1. Standard A.S.T.M. Methods D 423-59 and D 424-59 were followed for determination of the Atterberg limits; and particle size distribution was determined by standard sieve and hydrometer analysis procedures.

EXPERIMENTAL PROCEDURES

Preparation of raw gumbotil for analysis: The gumbotil was air-dried and then pulverized with mortar and pestle to pass the standard U.S. No. 100 sieve. This batch of gumbotil was then divided into three portions.

Approximately 50 grams of the gumbotil was left in the original state to be used in analysis of the raw soil. Another 600 grams was converted into Na-clay. Since chemical analysis showed that the original sample had calcium ions associated with the clay fraction, this conversion was considered practical. Earlier diffraction studies had indicated that Ca-clay presented difficulties when attempts were made to obtain a lithium saturated system. To facilitate this later conversion, the clay in the raw soil was converted to a Na-clay by gravity leaching with N NaCl for two days. The sample was washed repeatedly with distilled water to remove the excess chloride ions.

Separation of the 2μ clay fraction: It was felt that the clay fraction of the gumbotil could best be analyzed if the fraction $<2\mu$ was separated from the coarser material. The Na-clay was, therefore, placed in suspension in five-liter beakers and stirred. The 2μ clay was plotted off after a suitable settlement interval (3).

Approximately 60 grams of the Na-clay was then subjected to iron removal operations.

Removing free iron oxides from gumbotil: Since converting the 2μ Na-clay to a Li-clay was difficult, it appeared feasible to remove the iron oxide coatings in the clay fraction with sodium hydrosulfite under an acid condition. The method used was so designed that this chemical treatment would not destroy the clay fraction or block the anion exchange sites by adsorption of any organic or inorganic material in the system (4).

Fifty grams of the 2μ clay fraction were mixed in water with an electric stirrer. The clay suspenxion was heated to 80° - $90^{\circ}C$ on a steam bath, and 10 grams of sodium hydrosulfite was added. The mixture was stirred, and heating was continued for fifteen minutes. After the mixture became bluish black, 20 ml. of 1 N HCl was added, and the suspension was centrifuged. A layer of reddish-brown material, perhaps $Fe(OH)_3$, was then skimmed off the surface. This procedure was repeated until no more $Fe(OH)_3$ appeared, and the residue was almost white. The free elemental sulfur was removed by extracting the dried soil with CS_2. The CS_2 was then removed by evaporation over a steam bath and by repeated washing with acetone.

Conversion of Na-clay to Li-clay: Two different kinds of samples of Li-clay were prepared. One set of samples was prepared from the 2μ Na-clay from

Table 1. Properties of soil tested.

Sampling location	Keokuk County, Iowa
Soil series	Mahaska
Horizon	Fossil B
Sampling depth, ft.	6.5-8.5
Textural composition, %	
Gravel (>2.0mm)	0.0
Sand (2.0-0.074mm)	20.1
Silt (0.074-0.005mm)	21.2
Clay (<0.005mm)	58.7
Clay. (<0.002mm)	56.4
Atterberg limits	
Liquid limit, %	68.0
Plastic limit, %	23.0
Plasticity index	45.0
Chemical properties	
pH[a]	7.1
C.E.C.[b] me/100g	39.9
Carbonates,[c] %	1.8
Organic matter,[d] %	.2
Classification	
Textural	Clay
AASHO	A-7-6(20)
Unified	CH

[a]Determined with Bromthymol blue indicator and by glass-electrode measurements.
[b]Ammonium acetate (pH=7) method for soil fraction below 2mm.
[c]Versenate method for total calcium.
[d]Potassium bichromate method.

which the iron oxide coatings had not been removed. The other group of samples were converted from Na-clay to Li-clay by gravity leaching. Samples of clay without the iron coatings removed were leached with 1000 ml of 2 N LiCl for over 48 hours and then washed with distilled water, while the samples with iron removed required only 4 hours of leaching for the same quantity of LiCl to pass through the sample. Chemical analysis showed that the Na-clay samples with iron coatings removed were virtually completely converted to Li-clay in 4 hours; the other samples showed traces of Na-clay remaining even after 4 days of gravity leaching. This suggests that the iron coatings on the clay in this particular soil blocked the entrance of the lithium ions in the attempt at conversion of

the first Na-clay samples.

X-ray methods: All of the samples tested were analyzed on a General Electric XRD-5 diffractometer using CuKα radiation and a Ni filter. A 1° beam slit, 0.2° detector slit, time constant of 2.0 sec., M.R. soller slit, and linear 2000 cps scale were used.

The raw gumbotil was packed into a bakelite sample mount for the X-ray analysis. Diffraction studies were made in the air-dry condition, after the sample had been equilibrated at 50% relative humidity for four days and in a glycolated condition. Diffraction patterns for the air-dry and glycolated specimens are shown in Figure 1. The diffraction patterns suggested a montmorillonitic clay, kaolinite, and mica. The 50% relative humidity sample showed a peak at 15.0 Å , which indicated the two water layers expected with Ca-montmorillonite.

To verify that the 7.19 Å peak was kaolinite rather than a chlorite, which would also give a peak in this range, the sample was heated at 500°C for one hour using a DTA controller. The 7.2 Å peak disappeared, indicating kaolinite rather than chlorite (5). The peak that was tentatively identified as micaceous material at 10 Å remained at 10 Å and showed no shift upon glycolation or increases in moisture. This indicates that non-expanding mica accounted for the 10 Å peak.

The 2μ Na-clay samples were analyzed using the same diffraction geometry. These samples were placed on glass slides for all X-ray work, however. Glass slides were prepared for both the samples with the iron removed and the samples containing iron by placing the clay in suspension in distilled water and then employing a pipette to

place a thin layer of clay on the glass slide. The samples were then allowed to dry on the slides. Heat treatments and glycolation were also performed on samples mounted in this fashion. The glass slide samples showed better basal X-ray diffraction peaks than samples mounted in bakelite (Figures 2 and 3).

Glycolated samples were allowed to cure for periods ranging from 20 minutes to 48 hours, due to the fact that some expansion was noted in samples that had been allowed to cure for longer periods of time following glycolation (Figure 3). Dehydration at 300°C was used to check on the reversible nature of the collapse of the montmorillonite structure. According to studies by Greene-Kelley, reversibility or lack thereof in montmorillonites indicate whether they are dioctahedral or trioctahedral. This analysis was conducted to determine the class of the montmorillonite.

RESULTS

The X-ray analysis of the raw gumbotil indicated that the clay minerals present were kaolinite, mica, and montmorillonite (Figure 1). Thermal tests on the raw soil sample consisting of heating the soil at 500°C for one hour verified the presence of kaolinite. The mica showed a definite non-expanding 10 Å peak in the raw soil, but failed to show any distinct peaks in the 2μ clay fraction (See Figures 1, 2, 3). This suggests that the micaceous material occurs only in the coarse soil fraction. Chemical analysis and X-ray analysis at 50% relative humidity verifies that calcium is the associated cation in the natural montmorillonite. X-ray analysis of the glycolated Li-saturated 2μ clay fraction after thermal treatment at 300°C indicates

Figure 1. Diffraction traces of gumbotil.

Upper: Glycolated natural soil.

Middle: 2μ fraction of Na-clay dried at 104°C.

Lower: Untreated air dry gumbotil.

16.98 Å

7.2 Å   10.0 Å

9.93 Å

7.19 Å

14.7 Å

7.2 Å

13°   11°   9°   7°   5°   2°
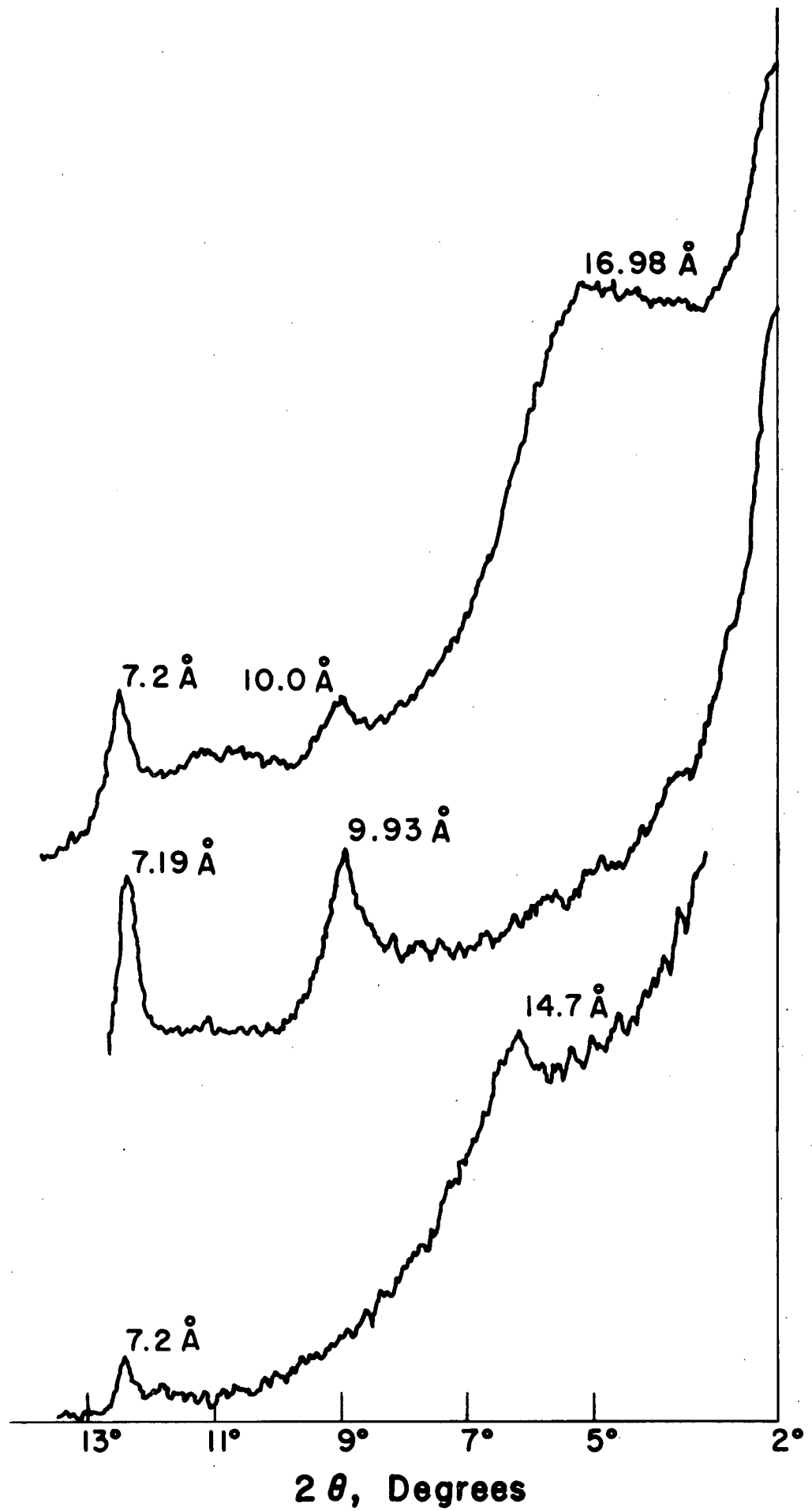
2 θ, Degrees

Figure 2.   Diffraction traces of Li-gumbotil.

    Upper: Fe removed, heated at 300°C for 24 hours, non-glycolated.

    Lower: Same sample glycolated and cured for 1 hour.
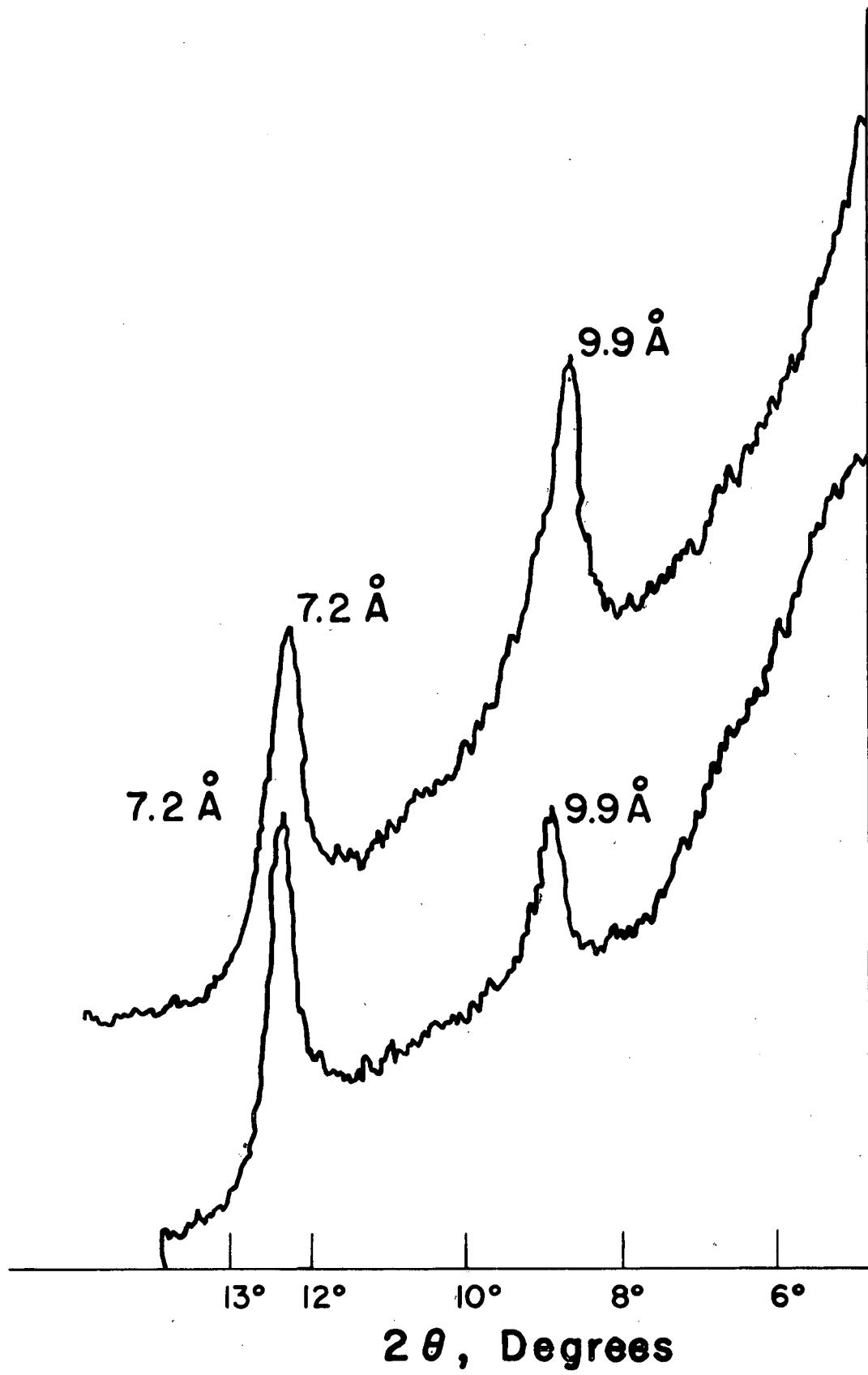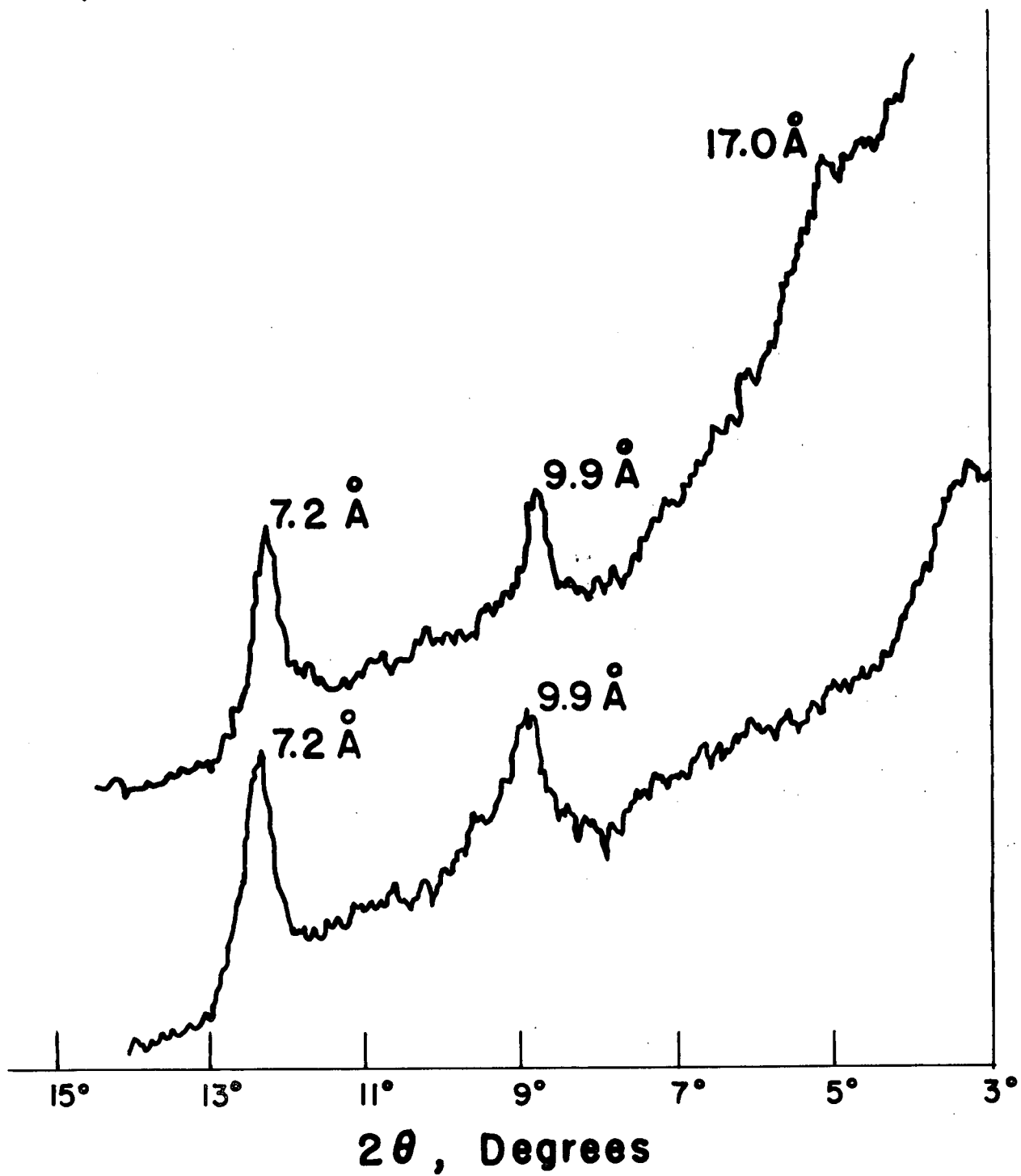
Figure 3. Diffraction traces of Li-gumbotil.

        Upper: Fe removed, heated at 300°C for 24 hours, glycolated and

              cured for 48 hours.

        Lower: Same sample immediately after glycolation.

17.0 Å

9.9 Å

7.2 Å

9.9 Å

7.2 Å

15°   13°   11°   9°   7°   5°   3°

2θ , Degrees

that the montmorillonite is irreversably collapsed after heating to 300°C for a period of one hour or longer (Figure 2). According to Greene-Kelley (2), this irreversibility indicates that the mineral is dioctahedral montmorillonite.

One oddity noted in the X-ray analysis of the glycolated collapsed montmorillonite was a tendency for expansion to occur with long periods of equilibration. This tendency was more pronounced if equilibration of 24 hours or longer was permitted. The expanded peak occurred in the 17 Å range, which would indicate an expanding montmorillonite (Figure 2). The diffraction pattern, however, still indicated a sharp peak at 9.9 Å for a collapsed montmorillonite structure. Since this expansion tendency was pronounced for the Li-clay samples with no iron removed, it is felt that a trace of sodium remaining in the sample, too small to be detected by qualitative analysis, is responsible.

The difficulty of conversion of the Na-clay with iron coatings present and the high background on the diffraction charts (Fig. 3), leads to the suggestion that removal of iron coatings from the natural soil clays is advisable before attempting to convert to a Li-clay.

CONCLUSIONS

1. The predominant clay minerals in the gumbotil analyzed are in approximate order of abundance, dioctahedral montmorillonite, kaolinite, and mica. Mica is mainly limited to the coarser fractions.

2. It is advisable to remove iron oxide coatings from natural soil clays before attempting lithium saturation for differentiating between diocta-

hedral and trioctahedral montmorillonites.

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