RELATIONSHIPS OF POTASSIUM FIXATION AND RELEASE TO THE CLAY MINERAL COMPOSITION OF SOME MICHIGAN SOILS

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Samuel L. Cummings
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By

Samuel L. Cummings

AN ABSTRACT

Submitted to the College of Agriculture of Michigan State
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MASTER OF SCIENCE

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Approved M. M. Mortland

Samuel Cummings

ABSTRACT

Some Michigan soils were studied to determine the relationships between potassium fixation and release to the clay mineral composition.

The soils were selected from those of project 413 of the Michigan State Agricultural Experiment Station on the basis of the Michigan potassium soil test (0.13 N HCl extract). They ranged from 36 to 400 pounds of potassium per acre.

Potassium fixation was determined in the laboratory by adding potassium as a KCl solution, drying at 110 degrees centigrade, and extracting potassium with 0.5 N NH_4Cl solution.

Potassium release was determined in the greenhouse by growing wheat plants in a small quantity of soil mixed with quartz sand for 26 days.

The clay minerals were identified from X-ray diffraction patterns.

The nonexchangeable potassium released to the plants was significantly related to the exchangeable potassium, the water soluble potassium, the amount of clay, the amount of illite, and the amount of 2:1 expanding clay minerals. The Michigan soil test and the Woodruff soil test were not significantly related to the nonexchangeable potassium released to the plants.

Potassium fixation was significantly related to the amount of clay and the 2:1 expanding clay minerals.

There is a trend for the soils fixing the higher percentage of applied potassium to contain a larger quantity of 2:1 expanding clay minerals.

In 26 out of 30 cases, the amount of potassium taken up by the plant exceeded the initially exchangeable potassium. Potassium uptake ranged from 69 to 1,950 pounds per acre.

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INTRODUCTION

Potassium is one of the most soluble elements in the form of the simpler chemical compounds, but in the soil-plant system it is characterized by wide variations in solubility and mobility. Soil potassium ranges from the soluble state through a range of decreasing activity to the state of an essential constituent of certain primary minerals from which it is released only by destruction of the crystal structure.

Potassium is found in the soil in the form of primary and secondary minerals. In soils that are strongly weathered, feldspars and micas ordinarily are the most abundant of the potassium-bearing minerals. The most important of these for supplying potassium are orthoclase and microcline feldspars, biotite and muscovite mica, and illite. The feldspars occur almost exclusively in the sand and silt fractions of soils but are found occasionally in the coarse clay. Biotite and muscovite occur mainly in the silt and sand fractions. Illite, the main potassium-bearing mineral of the clay fraction, is micaceous in nature, and is usually of secondary origin. The predominate clay minerals (secondary minerals) include the montmorillonite series, illites or hydrous micas, vermiculite, chlorite, kaolinite, and interstratified minerals in which two or more of the preceding types occur in more or less random arrangement within the same particle. In many soils the entire range of known forms of potassium occurs;

in other soils some forms may be lacking, usually because of the absence of some types of primary or clay minerals.

The order of relative resistance to weathering of these soil minerals is kaolinite, montmorillonite, vermiculite, and interstratified 2:1 layer silicates, muscovite and illite, orthoclase and microcline feldspars, and biotite and chlorite.

The ability of a given soil to supply potassium to crops is a result of many processes which must be studied and evaluated for a proper understanding of potassium availability.

The clay fraction of soils is involved in many physiochemical reactions which are important in the development of soils and in their functioning as a medium for plant growth. The active minerals of clays undergo changes upon contact with water, soil amendments, excretory and secretory products of organisms, or with soluble substances released from weathered minerals and decaying organic matter. Other changes in these minerals occur when water and nutrient elements are removed from them. The many reactions and changes which clays undergo, because of their colloidal nature, have led Kelly (15) to designate the clays as the active fraction of the soil.

The objective of this investigation was to relate potassium release and fixation to the clay mineral composition of some Michigan soils.

REVIEW OF LITERATURE

The release and fixation of potassium in soils has been recognized for many years, but unanswered problems remain which deal with the interaction of the clay minerals.

The proportion of the total potassium held in the water soluble, and exchangeable forms usually is relatively small. The majority of it resides in potassium-bearing primary and clay minerals.

The distinction between the soluble potassium and other forms is arbitrary. Reitemeier (20) has shown that by dilution with water, the soluble potassium will increase, because of hydrolysis of exchangeable potassium. Soluble potassium may also be increased by the replacement of exchangeable potassium by divalent ions or dissolution of potassium-bearing minerals.

Reitemeier (21) stated that the amount of soluble potassium present at any one time, even in fertile soils, is inadequate to meet the major part of the requirements of crops. Apart from the concept of contact feeding by roots on exchangeable cations, as presented by Jenny and Overstreet (12), it is presumed that all potassium entering roots must be in the soluble form. However, the replenishment of soluble potassium may occur rapidly enough directly from the exchangeable form and indirectly from the nonexchangeable form to satisfy plant demands. Although recognizing the importance of this soluble-exchangeable equilibrium, York (27) concluded that the controlling factor in potassium absorption is the available supply in the soil solution..

Usually, the degree of saturation of cation exchange complex of the soil by potassium is small, yet the availability of the exchangeable potassium is relatively high. Jarusov (10) has presented the case of the mobilities of two cations possessing different energies of absorption, and present in varying proportions in the complex. For example, the strength of the bond of one cation with the complex (the one with a higher energy of absorption) decreases as the complex becomes more saturated with this cation, and consequently its mobility increases. For the cation having the lower absorption energy, the mobility changes relatively little as the complex becomes increasingly saturated with this cation. Woodruff (26) using water extracts, expressed activities of potassium and calcium in concentration units (moles per liter) and calculated the energy of exchange, which is a measure of the intensity factor in the delivery of a balanced supply of nutrient cations from the exchange complex of the soil to the growing plant. He found the energies of exchange for the replacement of calcium with potassium to range from -3,500 to -4,000 calories were associated with potassium deficiencies in plants. Energies of exchange from -2,500 to -3,000 calories represented suitable balances between potassium and calcium. Energies of exchange of -2,000 calories or less were associated with escessive amounts of potassium in relation to the amounts of calcium that were present.

Wiklander (24) reported that the ease of release of an ion depends not only on the nature of the ion itself, but also upon the nature of the complementary ions filling the remainder of the exchange positions and the degree to which the replaced ion saturates the exchange complex. Jenny and Ayers (11) found the availability of potassium to plants, especially at lower levels of potassium saturation, was greater when calcium rather than hydrogen, was the complementary ion.

Barber and Marshall (3) have shown that illite tends to hold potassium more tenaciously and calcium less tenaciously, than montmorillonite. Work by Mortland, Lawton, and Uehara (18) indicates a comparatively rapid release to plants of the potassium fixed by montmorillonite and vermiculite and of native potassium of biotite. In this study the release of potassium from illite and muscovite was of much lower magnitude.

There have been numerous reports dealing with potassium fixation in soils in recent years and its relationship to the type of clay minerals present. Seatz and Winters (22) working with soils which developed on material residual from argillaceous limestone, found that fixation tended to be greater in soils that contained the highest proportion of mica and montmorillonite types of clay mineral. Joffe and Levine (14) and Stanford (23) have shown that potassium fixation occurs in the fine clay fraction of 2:1 type clay minerals. In addition, Hoover (9), and Raney and Hoover (19) reported that montmorillonitic soils fix more potassium than kaolinitic soils. Wiklander and Gieseking (25) showed montmorillonitic clays and illitic clays to be more effective in potassium fixation than kaolinitic clays. In studies with illite and vermiculite as single minerals, DeMumbrum and Hoover (7) found that whereas illite did not fix any applied potassium, vermiculite fixed large amounts.

Kunze and Jeffries (16) X-rayed the soil-clays of 15 soils classified as representative of the Gray-Brown Podzolic group which were potassium saturated and magnesium saturated. They found the soil-clays giving a strong 10 angstrom line in the X-ray diffraction pattern when potassium saturated as contrasted to a strong 14 angstrom line when magnesium saturated were relatively high fixers of potassium. Those soil-clays which showed little or no shifting of the larger basal spacings toward 10 angstroms when saturated with potassium were found to be relatively low fixers of potassium.

In 1924 Ames and Simon (1) reported that water soluble potassium determined after heating the soil to 100, 400, or 700 degrees centigrade was greatly increased with temperature. Joffe and Kolodny (13) found the release of potassium from Dover loam to be greatly increased by temperatures up to 600 degrees centigrade. It was pointed out by Bray and DeTurk (6) that heating at 200 degrees centigrade may result in either the release or fixation of potassium depending on the equilibrium conditions at the beginning of the heat treatments.

Attoe (2) was the first to establish that potassium was released to the exchangeable form by soils upon drying and reverted to the nonexchangeable form upon remoistening. If excess potassium was present in the system, fixation occured as a result of drying. Luebs, Stanford, and Scott (17) studied the effect of moisture upon exchangeable potassium and confirmed Attoe's results.

Hanway, Scott, and Stanford (8) have found that a 350 degree centigrade temperature is necessary to fix relative small amounts of potassium in montmorillonite. Even smaller amounts of potassium were fixed at lower temperatures in montmorillonites studied by Barshad (4), who postulated that any fixation was caused by a few highly charged (vermiculite-like) layers present in the montmorillonite as a very minor constituent, presumably as an interstratified material.

EXPERIMENTAL METHODS

Soils

The soils used in the investigation were chosen from project 413 of the Michigan State University Agricultural Experiment Station on the basis of the potassium extracted by the rapid soil (0.13 N HCl) test used in making fertilizer recommendations in Michigan. The values for potassium thus removed ranged from 36 pounds per acre to 400 pounds per acre of potassium as shown in Table 1. This test is purported to remove all water soluble and a high percentage of the exchangeable potassium from the soil.

Laboratory Investigations

Potassium fixation. The potassium fixing capacity was determined by weighing 5 grams of soil into a beaker and added 5 milliliters of 0.005 N KCl plus 50 milliliters of distilled water. Duplicate samples were placed in a 110 degree centigrade oven and dried over night. They were removed from the oven and allowed to stand two hours in contact with 50 milliliters of 0.5 N NH_4Cl. Then they were filtered and washed with 50 milliliters more of the 0.5 N NH_4Cl. Potassium was determined in the filtrate using a Perkin-Elmer Model 52A flame photometer.

Water soluble potassium. Water soluble potassium was determined by weighing 100 grams of soil into a buchner funnel, saturated with distilled water, and allowed to stand over night. Twenty-five milliliters of distilled water were added and the solution removed from the soil by suction. Potassium was determined with a Beckman D. U. flame photometer.

Woodruff soil test. Five grams of soil was placed in a 3 ounce bottle containing 1½ milliliters of distilled water and allowed to stand over night. Then 30 milliliters of 0.01 M $CaCl_2$ was added and gently shaken four times at 15 minute intervals and filtered after one hour. Potassium was determined with the Perkin-Elmer Model 52A flame photometer.

Greenhouse Experiment

To determine the release of soil potassium on cropping, wheat plants were grown in a limited amount of soil. The media for plant growth was prepared by mixing 200 grams of silica sand with 25 grams of soil and placing the mixture in a 16 ounce cottage cheese carton. Four hundred grams of silica sand were placed on top of the mixture of silica sand and soil. There were 2 replications for each soil used. Sixteen wheat seeds were planted and the media moistened with distilled water. Each culture was thinned to 10 plants after emergence. After the cultures were thinned, 15 milligrams of nitrogen was added as a water solution of ammonium nitrate over a period of two weeks. The wheat plants were allowed to grow until it appeared that the rate of growth had markedly decreased. The period was found to be 26 days.

The plants (tops and roots) were ground with a Wiley mill and approximately 0.5 grams weighed into a 250 milliliter beaker. Ten milliliters of concentrated HNO_3 was thoroughly mixed with the sample and digested on a hot plate until all the fiberous material was in solution. Ten milliliters of distilled water and 10 milliliters of $HClO_4$ were added and digestion continued at a moderate temperature. The solution was evaporated to dryness and the residue removed with 0.1 N HCl. This solution was filtered into a 100 milliliter volumetric flask, and potassium was determined using a Beckman D.U. flame photometer. Calcium and magnesium were determined by the Versenate method outlined in USDA Handbook 60.

ANALYTICAL METHODS

Clay Fractionation

The clay was removed by placing 10 grams of soil in a 600 milliliter beaker, and adding 100 milliliters of 6 percent H_2O_2 and a few drops of acetic acid. After standing over night they were heated on a hot plate, followed by 50 milliliters of 30 percent H_2O_2 and again heated. One hundred milliliters of 0.1 N HCl was added and the soil deposited on a number 50 Whatman filter paper. Three additional HCl washes were used, followed by distilled water until free of chlorides. Each sample was transferred to a shaker bottle and titrated with 0.1 N NaOH. Following 24 hours of shaking each sample was transferred to a sedimentation cylinder, filled with distilled water and set in a constant temperature bath. After the suspension temperature was equal to the bath temperature the suspensions were thoroughly mixed and the clay fraction extracted at the appropriate time and depth. The clay suspension was concentrated by drying in an oven at 110 degrees centigrade.

Total Potassium in the Clay Fraction

Total potassium was determined by weighing approximately 0.5 grams of clay into a platinum crucible. The sample was heated and one milliliter of H_2SO_4 (1:5) added. Five milliliters of concentrated HF was added and evaporated to dryness. An additional 5 milliliters of HF was added and evaporated to dryness. The residue was removed by placing the crucible in a beaker containing HNO_3 (1:20) and heating. The crucible was removed and rinsed three times with distilled water. If any of the residue remained, it was removed with a rubber policeman. The resulting solution was evaporated to dryness and taken up with 0.1 N HCl. The solution was filtered into a 100 milliliter volumetric flask. Potassium was determined with a Beckman D.U. flame photometer. Only one sample of each clay was analyzed, since the supply was limited. However, previous work with this method of analysis has shown high precision.

Specific Surface

The specific surface of each clay fraction was determined by using a modified Bower and Gschwend (5) procedure. First 0.3-0.5 grams of air dried clay was weighed into tared weighing bottles. Single samples of each clay fraction were placed in a vacuum dessicator over P_2O_5 and evacuated to a constant weight. Twenty drops of ethylene glycol were added to each sample, distributing it over the entire sample and allowing it to stand over night to get uniform wetting. The clay samples were then evacuated over $CaCl_2$ until they began to assume a dry appearance. They were weighed and evacuated again and reweighed at one hour intervals until the weights remained comparatively constant.

X-ray Diffraction

A small amount of clay suspension was placed in a test tube and a few drops of glycerol added. After standing over night the clay was deposited on a porous plate and washed with three increments of 0.1 N $CaCl_2$ which was 3 percent glycerol by volume. The deposit was allowed to air dry and then placed in a dessicator over $CaCl_2$. The sample was X-rayed as a calcium saturated, glycerol solvated, oriented aggregate using a Norelco diffraction unit with copper radiation and a nickel filter. After the initial X-ray exposure the calcium saturated, glycerol solvated, oriented aggregate was saturated with potassium by leaching with 0.1 N KCl, and the excess KCl washed out with distilled water. The sample was then heated to 110 degrees centigrade and X-rayed. This was followed by a heat treatment to 550 degrees centigrade and a third X-ray exposure.

DISCUSSION OF RESULTS

Potassium Release

The data in Table 1 include several chemical characterizations of potassium from the clays, as well as the order and amount of potassium released to plants during a 26 day period. In general, the clay and loam soils released more potassium than the sandy soils, although there are a few exceptions which would include samples 24, 142, 159, and 55. A good example are soils number 337 and 157. Number 337 with 11.5 percent clay released a little more potassium than did number 157 with 67.5 percent clay. Number 30 with 22.6 percent clay released the largest amount of potassium, while many of the other soils contained a higher percentage of clay. These comparisons are presented graphically in Figure I.

Statistical analysis indicates that the percentage clay mineral composition is a factor regulating the rate of release of potassium to the plant. These analyses are presented in Table 2.

The percent illite in the clay fraction was significantly correlated with the potassium released to the plants. This value for illite is given in Table 2 as total potassium in the clay fractions times the percent clay. The assumption was made that all of the potassium in the clay fraction was in the illite structure. Figure II graphically represents these data.

TABLE 1. SOIL TYPE AND NUMBER, HORIZON, MICHIGAN SOIL TEST, RELEASE OF NONEXCHANGEABLE POTASSIUM, EXCHANGEABLE POTASSIUM, WATER SOLUBLE POTASSIUM, WOODRUFF SOIL TEST, APPLIED POTASSIUM FIXED, PERCENT CLAY, AND SPECIFIC SURFACE OF CLAY

Sample Number	Soil Type	Horizon	Michigan Soil Test (lbs K/acre)
30	Brookston loam	A_{1-2}	276
180	Ontonagon silty clay	A_p	192
158	Paulding clay	C	232
185	Selkirk clay	C	56
108	Conover sandy loam	C	184
192	Selkirk clay loam	C	40
337	Iron River silt	A_p	292
157	Paulding clay	C	156
21	Miami sandy loam	B	104
146	Coldwater sandy clay loam	A_p	245
126	Miami sandy loam	C	64
42	Paulding clay	C	224
24	Brookston sandy loam	A_1	212
142	Mancelona sand	B_1	312
107	Conover loam	B_2	138
18	Miami sandy loam	A_1	72
25	Brookston sandy loam	B_1	120
166	Coldwater sandy loam	B_2	264
27	Brookston sandy loam	B_2	156

Release of Nonexchangeable* K (lbs/acre)	Exchangeable K (lbs/acre)	Water Soluble K (lbs/acre)	Woodruff Soil Test (lbs K/acre)	Applied K Fixed (%)	Percent Clay	Specific Surface of Clay (M^2/gm)
1,481	469	44	366	56	22.6	133
847	383	11	210	30	41.0	94
776	492	3	47	92	70.3	139
521	312	3	68	48	60.7	160
478	148	4	47	74	16.5	167
328	141	1	31	42	32.3	112
327	148	11	227	1	11.5	294
303	607	4	72	84	67.2	125
273	148	3	43	54	19.5	142
235	125	9	135	44	25.0	138
233	62	2	50	56	10.7	104
210	312	4	93	36	41.9	139
197	102	8	92	42	12.2	215
158	109	17	175	-6	2.9	242
157	203	2	35	84	23.6	146
149	86	4	67	40	8.9	190
146	55	2	35	62	11.0	339
119	125	2	47	68	18.8	155
108	125	2	47	74	15.0	147

TABLE 1 (CONTINUED)

Sample Number	Soil Type	Horizon	Michigan Soil Test (lbs K/acre)
141	Mancelona sand	A_p	196
11	Spinks sandy loam	A_1	82
159	Brookston clay loam	A_p	168
8	Granby sand	B_{2g}	36
63	Spinks sand	A	144
52	Fox sand	C_1	90
55	Warsaw loam	B_1	280
71	Warsaw sand	B_1	112
395	Munising loam	C	340
205	Saugatuck sand	A_2	400
349	Mariensco sandy loam	B_p	312

*Potassium in the plant (per acre) minus the exchangeable potassium.

Release of Nonexchangeable * K (lbs/acre)	Exchangeable K (lbs/acre)	Water Soluble K (lbs/acre)	Woodruff Soil Test (lbs K/acre)	Applied K Fixed (%)	Percent Clay	Specific Surface of Clay (M^2/gm)
87	187	22	234	16	3.1	230
86	78	7	67	36	7.5	170
80	352	3	52	72	35.2	184
73	39	4	67	10	2.4	284
72	109	8	131	24	5.2	270
19	70	5	67	20	1.9	333
16	258	9	217	-2	25.6	466
-9	78	7	111	10	4.0	390
-16	125	4	128	36	9.5	173
-33	133	17	161	4	3.4	276
-99	234	10	256	14	4.0	295

TABLE 2

LINEAR CORRELATION COEFFICIENTS

Correlation	Linear Correlation Coefficient
Nonexchangeable K released to plants versus total K in clay times percent clay	.511**
Nonexchangeable K released to plants versus $\Delta I+$ times percent clay	.447*
Nonexchangeable K released to plants versus exchangeable K	.602**
Nonexchangeable K released to plants versus Michigan soil test	.347
Nonexchangeable K released to plants versus Woodruff soil test	.342
Nonexchangeable K released to plants versus water soluble K	.521**
Nonexchangeable K released to plants versus plant $\frac{K}{\sqrt{Ca+Mg}}$	.652**
Nonexchangeable K released to plants versus plant weight	.754**
Nonexchangeable K released to plants versus percent clay	.486**
Applied K fixed versus ΔI^+ times percent clay	.401*
Applied K fixed versus percent vermiculite times percent clay	.359
Applied K fixed versus nonexchangeable K released to plants	.350
Applied K fixed versus specific surface times percent clay	.363*
Michigan soil test versus total K in clay times percent clay	-.104

TABLE 2 (CONTINUED)

Correlation	Linear Correlation Coefficient
Michigan soil test versus plant weight	-.079
Michigan soil test versus plant $\frac{K}{\sqrt{Ca+Mg}}$	.250
Michigan soil test versus the total K taken up by plants	.819**
Water soluble K versus total K times percent clay	-.228
Water soluble K versus specific surface times percent clay	-.228

**1 percent significance.
*5 percent significance.

+ΔI is the change in intensity of the 10 angstrom peak of the X-ray diffraction patterns, going from a calcium saturated, glycerol solvated clay sample to a potassium saturated clay sample heated to 110 degrees centigrade.

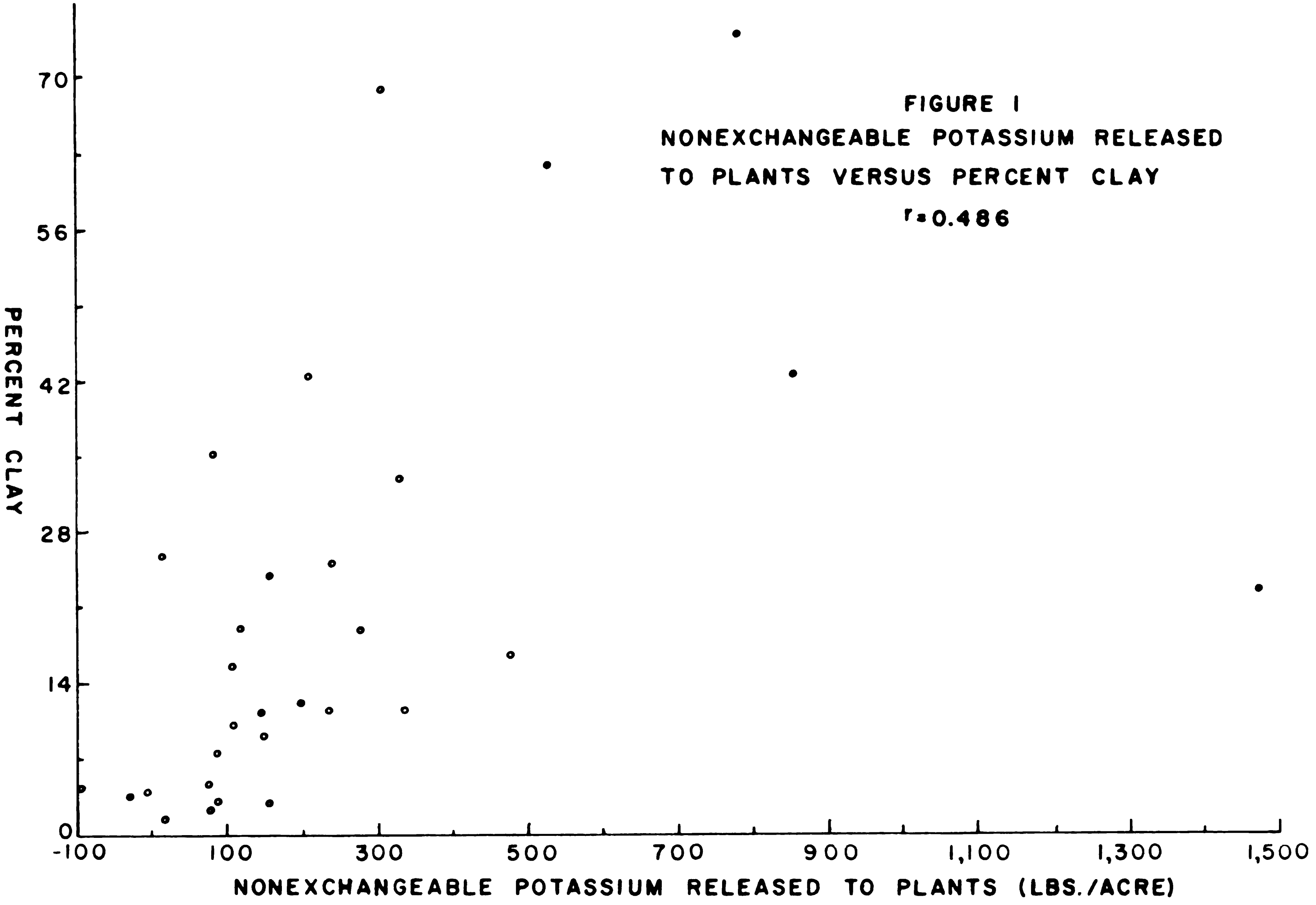


FIGURE I
NONEXCHANGEABLE POTASSIUM RELEASED
TO PLANTS VERSUS PERCENT CLAY
r=0.486

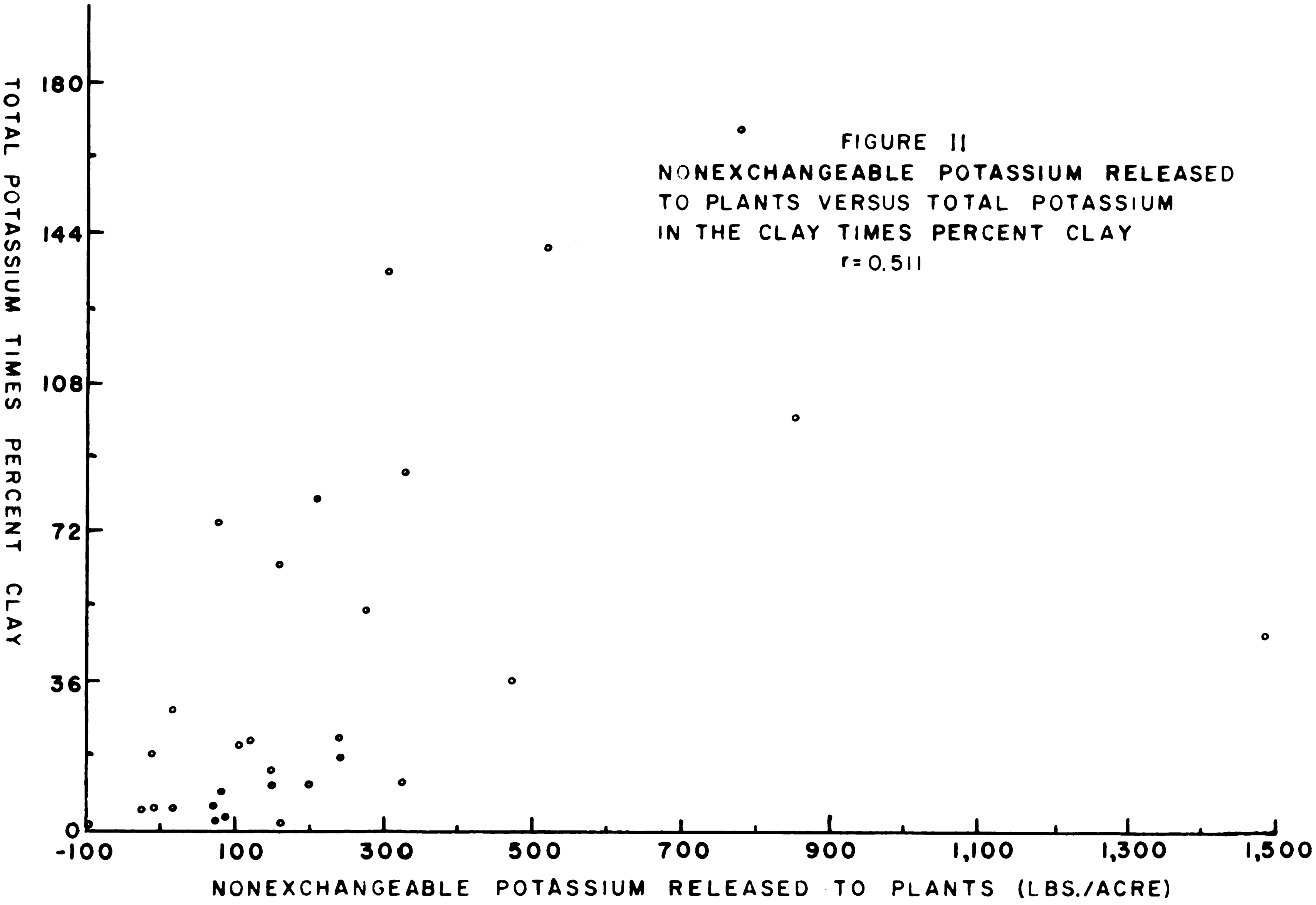
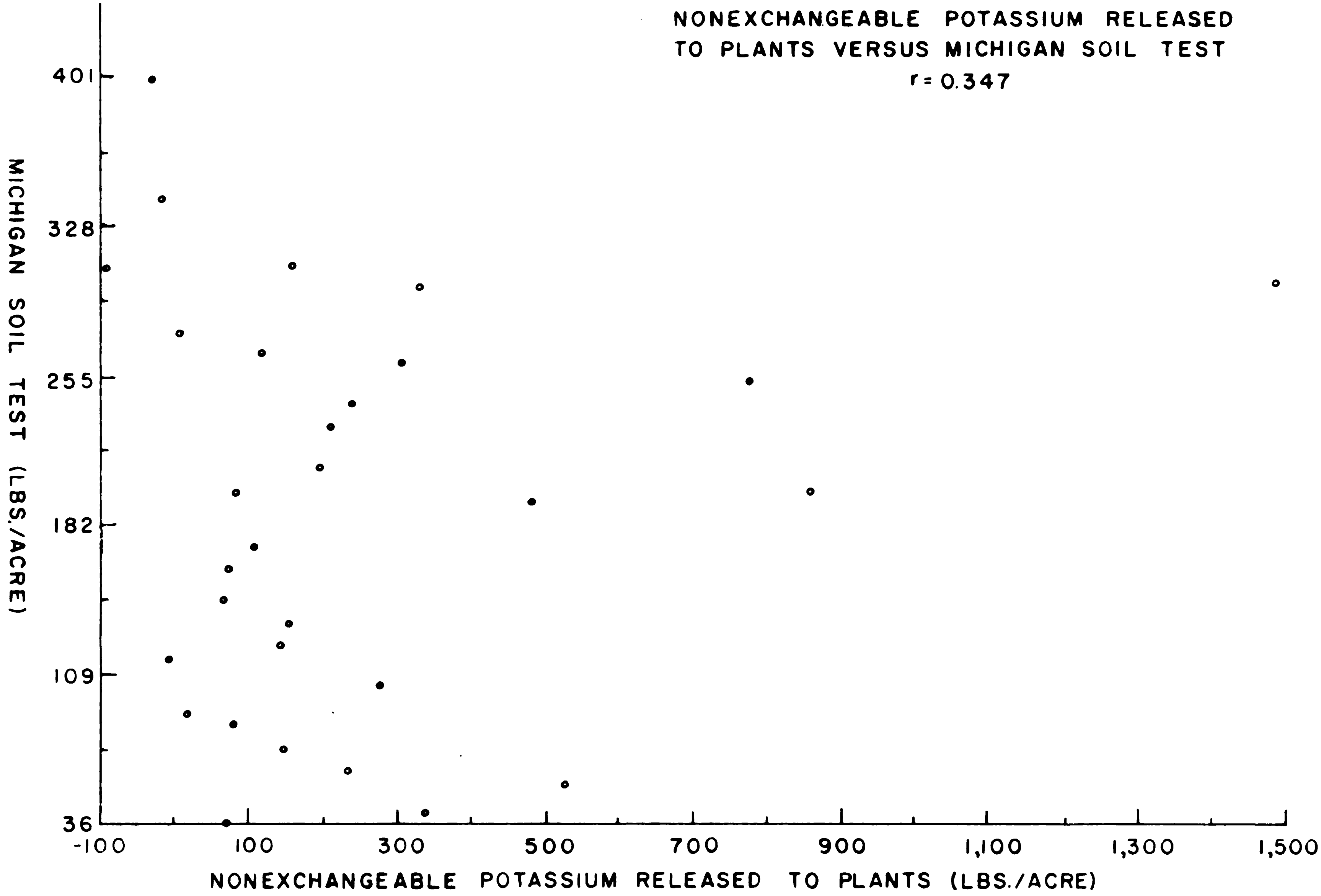


FIGURE 11

NONEXCHANGEABLE POTASSIUM RELEASED TO PLANTS VERSUS TOTAL POTASSIUM IN THE CLAY TIMES PERCENT CLAY

$r = 0.511$

FIGURE III
NONEXCHANGEABLE POTASSIUM RELEASED TO PLANTS VERSUS MICHIGAN SOIL TEST
r = 0.347



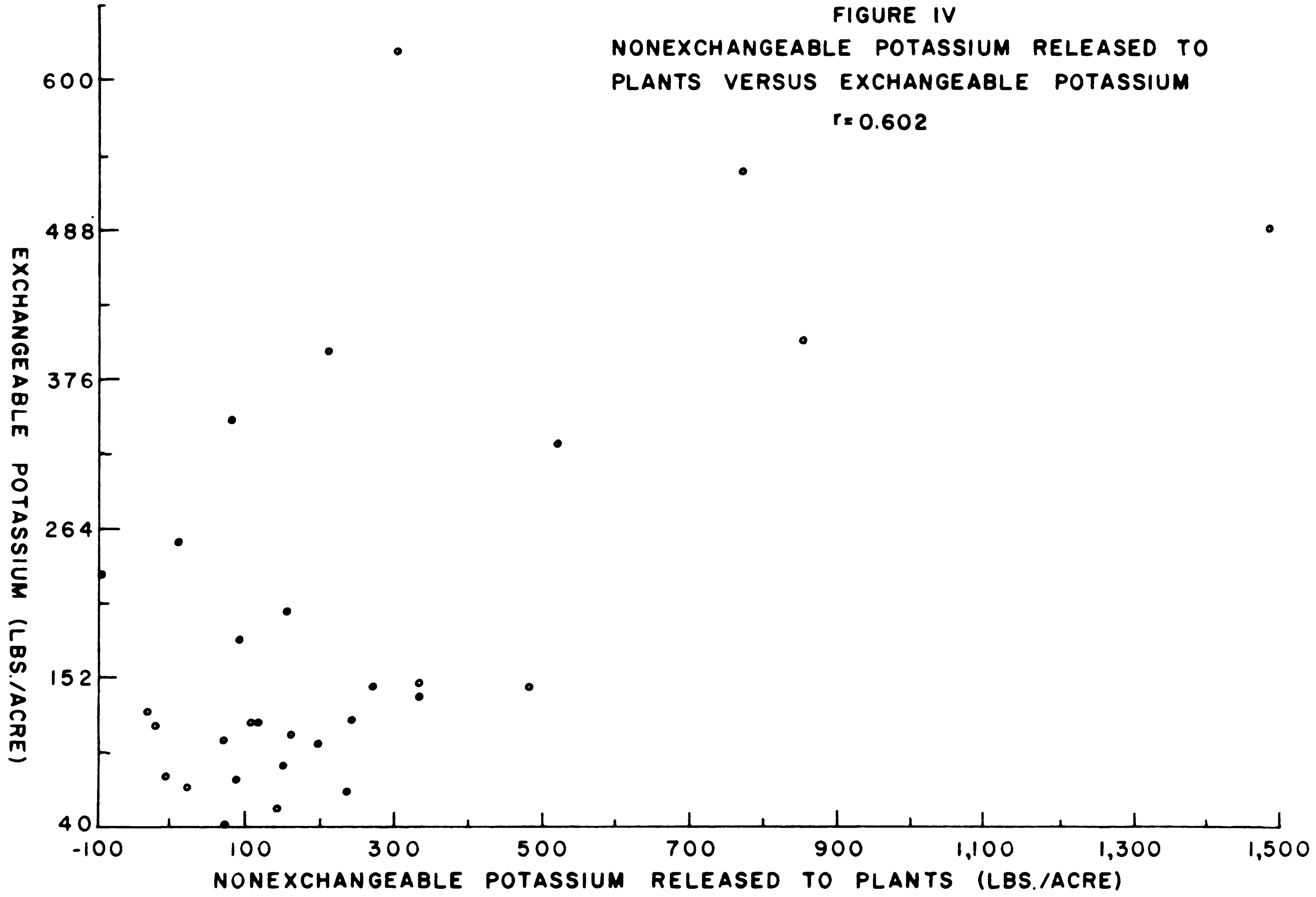


FIGURE IV
NONEXCHANGEABLE POTASSIUM RELEASED TO PLANTS VERSUS EXCHANGEABLE POTASSIUM
r = 0.602

The amounts of 2:1 expanding clay minerals were significantly correlated with the potassium released to the plants. This clay value is given in Table 2 as ΔI times the percent clay. Referring to the X-ray diffraction patterns, ΔI is the change in intensity of the 10 angstrom peak going from a calcium saturated, glycerol solvated clay sample to a clay sample which had been potassium saturated and heated to 110 degrees centigrade. This increase in intensity is a result of the collapsing of the 2:1 expanding clay minerals, namely vermiculite and/or montmorillonite.

The correlation between the Michigan soil test and the release of nonexchangeable potassium to the plants was not significant. Figure III graphically represents this comparison. Many of the soils with rather low Michigan soil test values gave higher amounts of available potassium* than soils testing higher by the Michigan soil test. A good example of this comparison is samples number 30 and 337. Soil number 30 with a Michigan soil test value of 276 pounds of potassium per acre supplied the plants with 1,950 pounds of potassium per acre, while number 337 with a Michigan soil test value of 292 pounds of potassium per acre supplied only 475 pounds of potassium per acre to the plants. The poor correlation is probably due to the inability of the 0.13 N HCl

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*Nonexchangeable potassium plus exchangeable potassium.

to extract proportional amounts of potassium under different situations. The amount and kind of clay minerals and the different degree of saturation of other cations possibly affect the results obtained by this method. It is interesting to note that the nonexchangeable potassium released to plants was significantly correlated with the total potassium in the clay times the percent clay and with the dry plant weight. In contrast the Michigan soil test was not significantly correlated with either of these two factors. It would appear that a greater stress on the potassium equilibrium might be desireable.

Plants are known to grow better where the $K/\sqrt{Ca+Mg}$ ratio in the plant is within a certain range for the particular species. This condition is brought about by the interaction of these elements. The fact that a significant correlation was obtained between this ratio and the release of nonexchangeable potassium to the plants suggests the importance of the contribution of this form of potassium to nutrient balance in the plant.

The Woodruff soil test was also not sighificantly correlated with the nonexchangeable potassium released to plants. This test gave a correlation almost the same as the Michigan soil test, but the amounts of potassium extracted from a given soil were generally quite different.

The quantity of exchangeable potassium in the clays was significantly correlated with the amount of nonexchangeable potassium released to the plants. Figure IV graphically

represents this comparison. The quantity of potassium thus released exceeded the value for exchangeable potassium in 26 of the 30 soils. Of the four soils which didn't fit this situation, three gave the top three Michigan soil test values for this group of soils. With the majority of the potassium taken up by the plants coming directly or indirectly from the exchangeable positions, it would appear that the equilibrium between exchangeable and nonexchangeable potassium is very important in determining the amount of potassium available to the plant. The amount of potassium in the exchangeable and nonexchangeable positions and the type of chemical binding may be controlling factors imposed on this equilibrium.

The water soluble potassium was significantly correlated with the nonexchangeable potassium released to the plants, but the values for the water soluble potassium are very low as compared with the potassium released from the majority of the soils. The quantity of water soluble potassium depends in large part on an equilibrium condition with the exchangeable potassium. This equilibrium in turn depends on the exchangeable-nonexchangeable equilibrium.

Potassium Fixation

The data in Table 1 show that a wide range of fixation of soluble potassium occured within the 30 soils studied. With soils number 55 and 142, Warsaw loam and Mancelona sand respectively, a small amount of potassium was actually released at this level of application.

With the exception of 3 of the soils, all of the clay and loam soils fixed 30 percent or more, while the sandy soils fixed less than 30 percent of the applied potassium. These trends indicate that the amount of clay is a factor in potassium fixation.

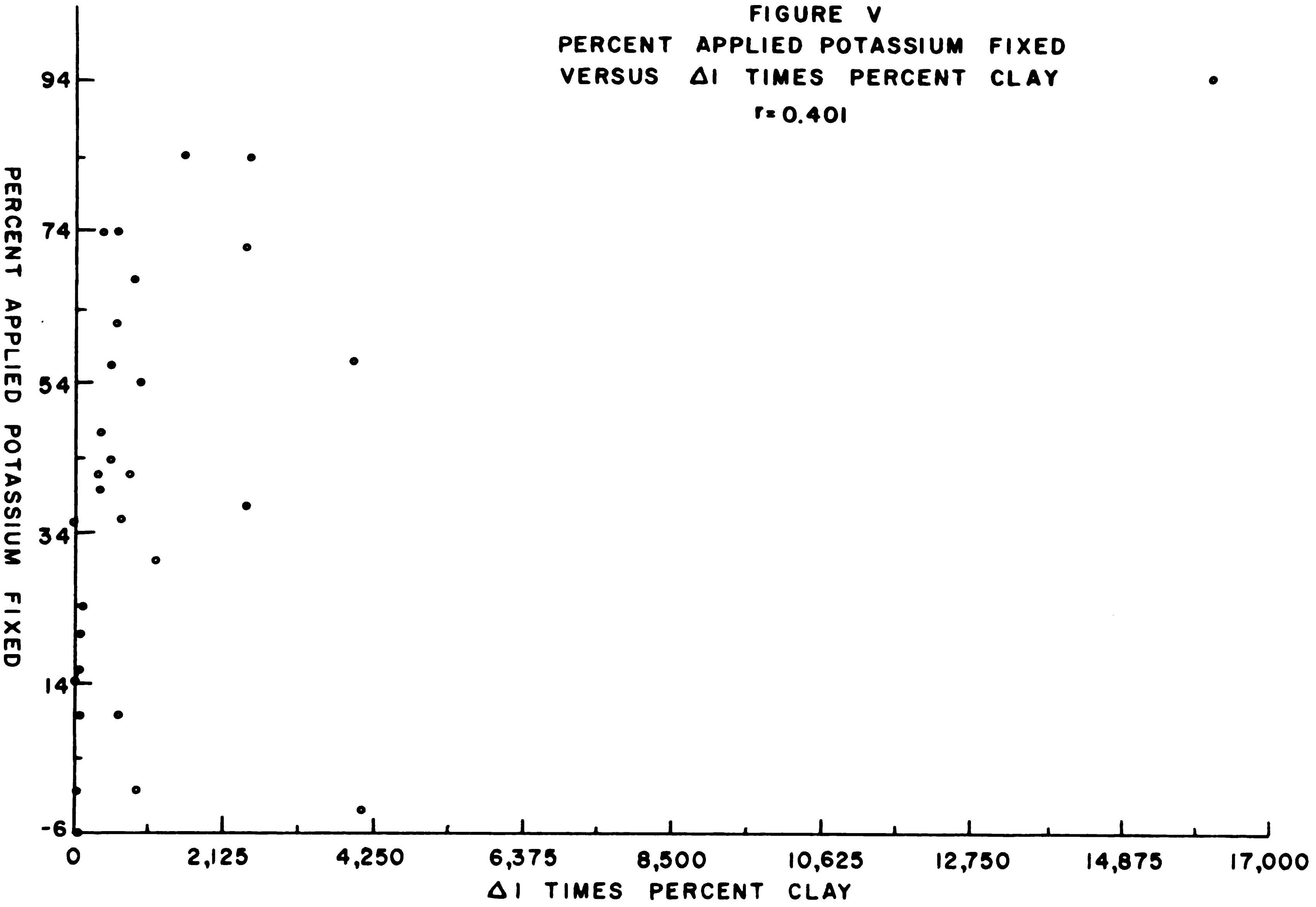
Correlation coefficients given in Table 2 show that the amounts of 2:1 expanding clay minerals were significantly correlated with the potassium fixed, but the percent vermiculite as estimated by the method described in Table 4 was not significantly correlated with the potassium fixed. Figure V graphically represents the percent potassium fixed versus the 2:1 expanding clay minerals. On this basis it could be concluded that montmorillonite is the principle clay mineral responsible for the fixation of potassium. However, this conclusion should be avoided, because the method for the quantitative estimation of the relative amounts of vermiculite and montmorillonite may introduce enough error to make this invalid.

There was no significant correlation between the amount of potassium fixed and the quantity of potassium released from the soil. However, there was a trend for some of the soils which fixed large amounts of potassium to release large quantities of this ion. This relationship supports the correlation of the amount of 2:1 expanding clay minerals with the potassium released on cropping. Therefore

FIGURE V

PERCENT APPLIED POTASSIUM FIXED
VERSUS ΔI TIMES PERCENT CLAY

r = 0.401



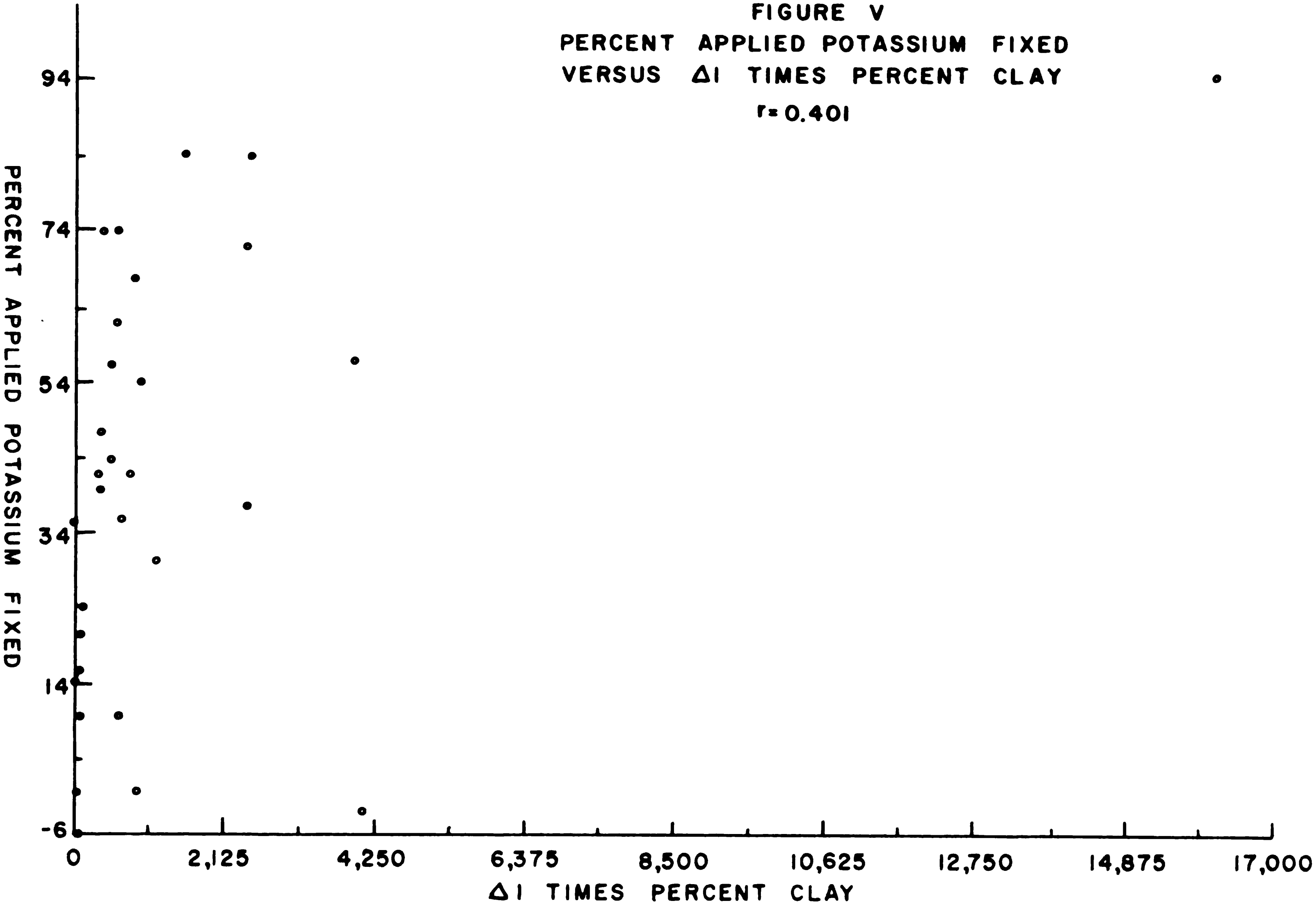


FIGURE V
PERCENT APPLIED POTASSIUM FIXED
VERSUS ΔI TIMES PERCENT CLAY
r = 0.401

it would appear that the 2:1 expanding clay minerals partially control the release of potassium as well as being the principle clay minerals associated with the fixation of potassium.

Upon comparing potassium fixation with the X-ray diffraction patterns (appendix), there is a trend for the higher potassium-fixing clays to have a more intense 10 angstrom peak when they are potassium saturated and heated to 550 degrees centigrade.

Clay Minerals

In Table 3 the discrete and interstratified clay minerals of each soil clay fraction are listed. These data were obtained from the X-ray patterns given in the appendix.

Discrete illite and quartz were identified in all of the soil clay fractions. Illite was identified by the presence of a 10 angstrom peak with the calcium saturated, glycerol solvated clay sample. Quartz was identified by the occurence of a 4.26 angstrom peak.

Discrete kaolinite was identified in all of the soil clay fractions except soil number 205. The criteria for the presence of kaolinite was the disappearance of the 7 and 3.5 angstrom peaks when the clay sample was potassium saturated and heated to 550 degrees centigrade.

Vermiculite was found randomly interstratified in all but one of the soil clays as a vermiculite-chlorite interstratification or a vermiculite-chlorite-montmorillonite interstratification. Discrete vermiculite was noted in only 12 of the soil clays. Identification of this soil clay mineral was accomplished by noting the disappearance of or a decrease in the 14 angstrom peak on comparing a calcium saturated, glycerol solvated clay sample with a potassium saturated clay heated to 110 degrees centigrade. The randomly interstratified vermiculite-chlorite clay minerals were identified by first observing, for example, a 14 angstrom peak when the clay sample was calcium saturated, glycerol solvated and then observing if a broad peak occurred between 10 and 14 angstroms after potassium saturating and heating the clay sample. If a broad series of peaks occurred between 10 and 14 angstroms, vermiculite and chlorite were said to be randomly interstratified. Relative amounts of chlorite or vermiculite may be estimated by the position of the broad peak relative to the 10 and 14 angstrom position.

Discrete chlorite was found in 20 of the soil clays and appeared in randomly interstratified systems in all samples except one. The clay mineral, chlorite was identified by the persistence of a 14 angstrom peak after potassium saturation and heating to 110 degrees centigrade. X-ray data indicate discrete chlorite present in 72 percent of the loamy soil clays, in 60 percent of the clayey soil clays, and in 57 percent of the sandy soil clays. Forty-four percent of the soils classed as loamy soils bordered on the

TABLE 3

CLAY MINERALS

Sample Number	Soil Type	Discrete	Interstratified
158	Paulding clay	I K Q	V-C
42	Paulding clay	I K Q	V-C
52	Fox sand	I K Q	V-C-M
30	Brookston loam	I K Q	V-M-C
8	Granby sand	I K Q C	C-V
18	Miami sandy loam	I K Q C	V-C
63	Spinks sand	I K Q C	V-C-M
55	Warsaw loam	I K Q C	V-C-M
166	Coldwater sandy loam	I K Q M	V-C
159	Brookston clay loam	I K Q M	V-C
25	Brookston sandy loam	I K Q M	V-C
71	Warsaw sand	I K Q M	V-C
349	Mariensco sandy loam	I K Q M C	C-V
27	Brookston sandy loam	I K Q M C	V-C
21	Miami sandy loam	I K Q V C	V-C
180	Ontonagon silty clay	I K Q V C	V-C
157	Paulding clay	I K Q V C	V-c

TABLE 3 (CONTINUED)

Sample Number	Soil Type	Discrete	Interstratified
337	Iron River silt	I K Q V C	V-C-M
185	Selkirk clay	I K Q V C	C-M-V
107	Conover loam	I K Q V C	V-C-M
146	Coldwater sandy clay loam	I K Q V C M	V-C
192	Selkirk clay loam	I K Q V C M	V-C
11	Spinks sandy loam	I K Q V C M	V-C
108	Conover sandy loam	I K Q V C M	V-C
395	Munising loam	I K Q V C M	V-C
126	Miami sandy loam	I K Q	V-M
24	Brookston sandy loam	I K Q C M	C-M
205	Saugatuck sand	I Q C F	V-C
142	Mancelona sand	I K Q F C	C-V
141	Mancelona sand	I K Q F C	C-V

Legend: I=Illite
K=Kaolinite
Q=Quartz
C=Chlorite
M=Montmorillonite
V=Vermiculite
F=Feldspars

sandy side, this grouping suggests that a higher percentage of the sandy soils contained discrete chlorite.

Discrete montmorillonite was noted in 12 of the soil clays and as a randomly interstratified clay mineral in 10 other samples. It was found that fifty-five percent of the loamy soil clays and 14 percent of the sandy soil clays contained discrete montmorillonite. It is interesting that none of the clayey soil clays contained discrete montmorillonite. Criteria for the presence of discrete montmorillonite was the occurence of a 17.7 angstrom peak when the soil clays were calcium saturated, glycerol solvated.

Feldspars were identified by the persistance of a 3.25 angstrom peak in three of the clay fractions separated from soils which were of a sandy texture.

Several of the soils have the same type of discrete and interstratified clay minerals but the amounts of each type and degree of interstratification varies with each soil.

Table 3 shows 14 different assortments of the clay minerals. Nine of the assortments are represented by two to four soils and five are represented by only one soil. The variability and complexity of these clay mineral systems makes a comparison of the clay mineral composition with the release of nonexchangeable potassium and fixation of potassium quite difficult.

Soils number 158 and 42 containing the same type of clay minerals and the same type of interstratification had very similar X-ray diffraction patterns, with 158 having the more intense peaks. Soil number 42 with 41.9 percent clay released only 27 percent as much nonexchangeable potassium as did soil number 158 with 70.3 percent clay. These data suggest that the amount of the different clay minerals is a factor helping to regulate the release of nonexchangeable potassium. More potassium was fixed by soil number 158 than by soil number 42, a condition which was again possibly related to the amounts of the different kinds of clay minerals. The same general pattern of fixation and release that was evident in these two soils, was also found for soils number 52 and 30, 8 and 18, and 349 and 27. It is apparent that the soil with the more intense X-ray diffraction pattern peaks containing the highest percentage of clay, released the most nonexchangeable potassium and fixed the most applied potassium. In these particular soils the total amount of clay may have been more important in controlling fixation and release of potassium from the soil clays than the particular type of clay mineral.

The remainder of the soils do not show this same conformity and consequently no adequate comparison of these soil clay minerals with the release of nonexchangeable potassium and fixation of added potassium can be made using the X-ray diffraction patterns. By comparing the potassium released and fixation data with the amount of clay in each soil of these different assortments, it appears that the amount of certain type of clay minerals, probably 2:1 expanding minerals, are more important than the total amount of clay.

GENERAL DISCUSSION

The complexity of the clay minerals found in the soils and the present methods of making quantitative estimation of clay minerals does not allow an adequate comparison of the clay mineral composition with the release of nonexchangeable potassium and potassium fixation. Although rather general statements can be made concerning clay mineral composition and the release of nonexchangeable and fixed potassium, it is difficult to state exactly what the difference is between two particular soils. A good example of this can be seen in a comparison of data of soils number 180 and 55. Soil number 180 with a specific surface of 94 square meters per gram, with 41 percent clay, of which 50 percent was illite, contained vermiculite and no montmorillonite. This soil clay released 847 pounds of nonexchangeable potassium per acre, while soil number 55 with a specific surface of 466 square meters per gram, with 25.6 percent clay of which 25 percent was illite, contained vermiculite and montmorillonite. Release of nonexchangeable potassium from this soil clay amounted to only 16 pounds per acre. These values for potassium release seem to be correct assuming all the potassium is in the illite clay mineral. The picture of potassium fixation becomes somewhat more complicated, since soil number 180 fixed 30 percent of the applied potassium while soil number 55 released potassium at this particular rate of application. It would appear that soil number 55 should fix the most potassium due to the presence of montmorillonite and vermiculite. In addition this soil clay had a specific surface approximately 4.5 times larger than soil number 180. This high specific surface indicates that approximately 50 percent of the clay minerals are probably present as 2:1 expanding clay minerals. As previously mentioned such clays are considered to be the main fixers of potassium. The fact that the clay from soil number 55 has interstratified vermiculite and montmorillonite and vermiculite with no discrete vermiculite or montmorillonite, while clay from soil number 180 contains discrete vermiculite as well as interstratified vermiculite may have an effect on the fixing properties of the two clay systems. Actually there is no experimental evidence to verify this conclusion.

The correlation between the relative amounts of 2:1 expanding clay minerals times the percent clay versus the nonexchangeable potassium released to the plants is very interesting, as one would expect an inverse rather than a direct relationship. The positive correlation may indicate that some of the initially nonexchangeable potassium may occur in the expanded 2:1 minerals and that it may be an oversimplification to allocate all nonexchangeable potassium to 10 angstrom layers.

The release of nonexchangeable potassium to the plants was positively correlated with the amount of illite clay mineral, the exchangeable potassium, and the water soluble potassium, but none of these give a very good estimation of the amount of available potassium to the plants.

It is concluded that a better understanding of the water soluble-exchangeable-nonexchangeable equilibrium of each soil is very important in interpreting the release of potassium to the plants.

SUMMARY

1. The following factors are related to the release of nonexchangeable potassium to the plants:
    - A. The amount of clay in the soil.
    - B. The types of clay minerals.
        - a. Illite mineral.
        - b. 2:1 expanding minerals.
    - C. The exchangeable potassium.
    - D. The water soluble potassium.
2. The following factors affect the fixation of potassium:
    - A. The amount of clay in the soil.
    - B. The 2:1 expanding clay minerals.
3. There is a trend for the higher fixers of potassium to have a more intense 10 angstrom X-ray diffraction peak when potassium saturated and heated to 550 degrees centigrade.
4. In most of the cases, the amount of potassium taken up by the plants exceeded the exchangeable potassium.
5. The Michigan soil test and the Woodruff soil test did not give significant correlations with the release of nonexchangeable potassion to plants.

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APPENDIX

TABLE 4

QUANTITATIVE ESTIMATION OF THE CLAY MINERALS*

Sample Number	I	K	C	M	V	Q
205	x	xx	xx	0	xxxx	x
395	xxx	xx	xx	x	x	x
349	x	xx	xx	xx	xx	x
142	x	xx	xx	0	xxxx	x
337	xx	xx	xx	xx	xx	x
55	xx	xx	x	xx	xx	x
30	xxx	xx	xx	x	x	x
166	xx	xx	xxx	x	x	x
157	xxx	xx	xx	0	xx	x
146	xxx	xx	xx	x	xx	x
158	xxx	xx	x	0	xx	x
42	xxx	xx	xx	0	xx	x
24	xx	xx	xxx	xx	0	x
141	x	xx	xx	0	xxx	x
180	xxx	xx	xx	0	x	x
108	xxx	xx	xx	x	x	x
159	xxx	xx	xx	x	x	x
27	xx	xx	xxx	x	x	x
63	xx	xx	xx	xx	xx	x
107	xxxx	xx	x	x	x	x
25	xx	xx	x	xx	xx	x

TABLE 4 (CONTINUED)

Sample Number	I	K	C	M	V	Q
71	xx	xx	x	xx	x	x
21	xxxx	xx	x	0	xx	x
52	xxx	xx	x	xx	x	x
11	xx	xx	xx	x	xx	x
18	xx	xx	xx	0	xxx	x
126	xxx	xx	xx	x	x	x
185	xxxx	xx	x	x	x	x
192	xxxx	xx	x	x	x	x
8	xxx	xx	x	0	xxx	x

Legend: x=0-10 percent.
xx=10-30 "
xxx=30-50 "
xxxx=50-70 "

I=Illite
K=Kaolinite
C=Chlorite
M=Montmorillonite
V=Vermiculite
Q=Quartz

*The quantitative estimation of the clay minerals was determined by using the specific surface values of the pure clay minerals and the total potassium in the <2 μ clay and by solving simultaneous equations.

TABLE 5

ΔI, PLANT WEIGHT, TOTAL POTASSIUM, POTASSIUM IN PLANTS, AND PLANT $K/\sqrt{Ca+Mg}$

Sample Number	Plant Weight (mgms)	ΔI (%)	Total K in <2 μ clay (%)	K in Plants (%)	Plant $K/\sqrt{Ca+Mg}$
30	955	197	1.92	1.52	5.37
180	961	27	2.32	1.28	1.73
158	805	230	2.39	1.97	7.44
185	840	6	3.23	1.24	1.45
108	1,045	23	2.12	0.75	1.22
192	783	8	2.68	0.75	2.21
337	826	75	1.23	0.72	1.53
157	707	23	2.02	1.60	5.65
21	726	48	2.82	0.58	1.93
146	729	45	1.78	0.62	0.89
126	699	37	1.95	0.53	0.79
42	686	57	1.85	0.95	1.22
24	734	63	0.97	0.51	0.74
142	671	28	0.78	0.50	1.07
107	749	100	2.72	0.60	1.46
18	699	30	1.20	0.42	0.50
25	718	39	1.47	0.35	0.76
166	726	42	1.18	0.42	0.79
27	677	35	1.43	0.43	1.04
141	717	12	0.90	0.48	2.15
11	707	0	1.41	0.29	0.58

TABLE 5 (CONTINUED)

Sample Number	Plant Weight (mgms)	ΔI (%)	Total K in <2 μ clay (%)	K in Plants (%)	Plant $K/\sqrt{Ca+Mg}$
159	683	67	2.08	0.79	1.72
8	467	27	1.71	0.30	0.66
63	597	14	1.19	0.38	1.10
52	697	32	1.96	0.16	0.34
55	701	159	1.18	0.49	1.27
71	575	118	1.32	0.15	0.34
395	506	64	1.94	0.27	1.35
205	502	0	1.09	0.25	1.25
349	601	0	0.46	0.28	1.06

X-RAY DIFFRACTION PATTERNS

Sample Number	Soil Type	Horizon	Page
30	Brookston loam	A_{1-2}	51
180	Ontonagon silty clay	A_p	52
158	Paulding clay	C	53
185	Selkirk clay	C	54
108	Conover sandy loam	C	55
192	Selkirk clay loam	C	56
337	**Iron River silt**	A_p	57
157	**Paulding clay**	C	58
21	**Miami sandy loam**	B	59
146	**Coldwater sandy clay loam**	A_p	60
126	Miami sandy loam	C	61
42	Paulding clay	C	62
24	Brookston sandy loam	A_1	63
142	Mancelona sand	B_1	64
107	Conover loam	B_2	65
18	Miami sandy loam	A_1	66
25	Brookston sandy loam	B_1	67
166	Coldwater sandy loam	B_2	68
27	Brookston sandy loam	B_2	69
141	Mancelona sand	A_p	70
11	Spinks sandy loam	A_1	71
159	Brookston clay loam	A_p	72
8	Granby sand	B_{2g}	73

X-RAY DIFFRACTION PATTERNS
(CONTINUED)

Sample Number	Soil Type	Horizon	Page
63	Spinks sand	A	74
52	Fox sand	C_1	75
55	Warsaw loam	B_1	76
71	Warsaw sand	B_1	77
395	Munising loam	C	78
205	Saugatuck sand	A_2	79
349	Mariensco sandy loam	B_p	80

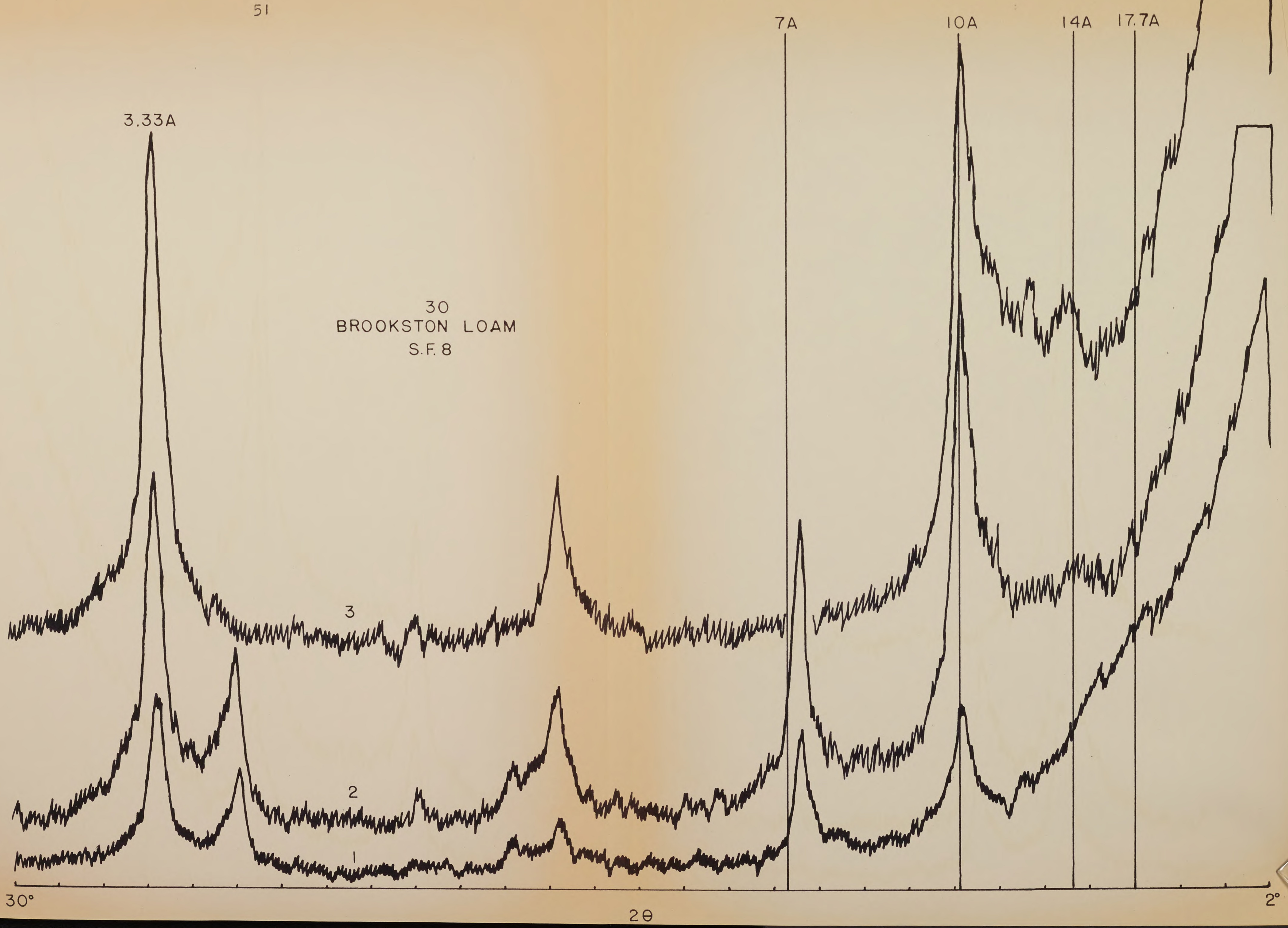
1 = Calcium saturated, glycerol solvated.

2 = Potassium saturated, heated to 110 degrees centigrade.

3 = Potassium saturated, heated to 550 degrees centigrade.

S.F. = Scale factor.

A = Angstroms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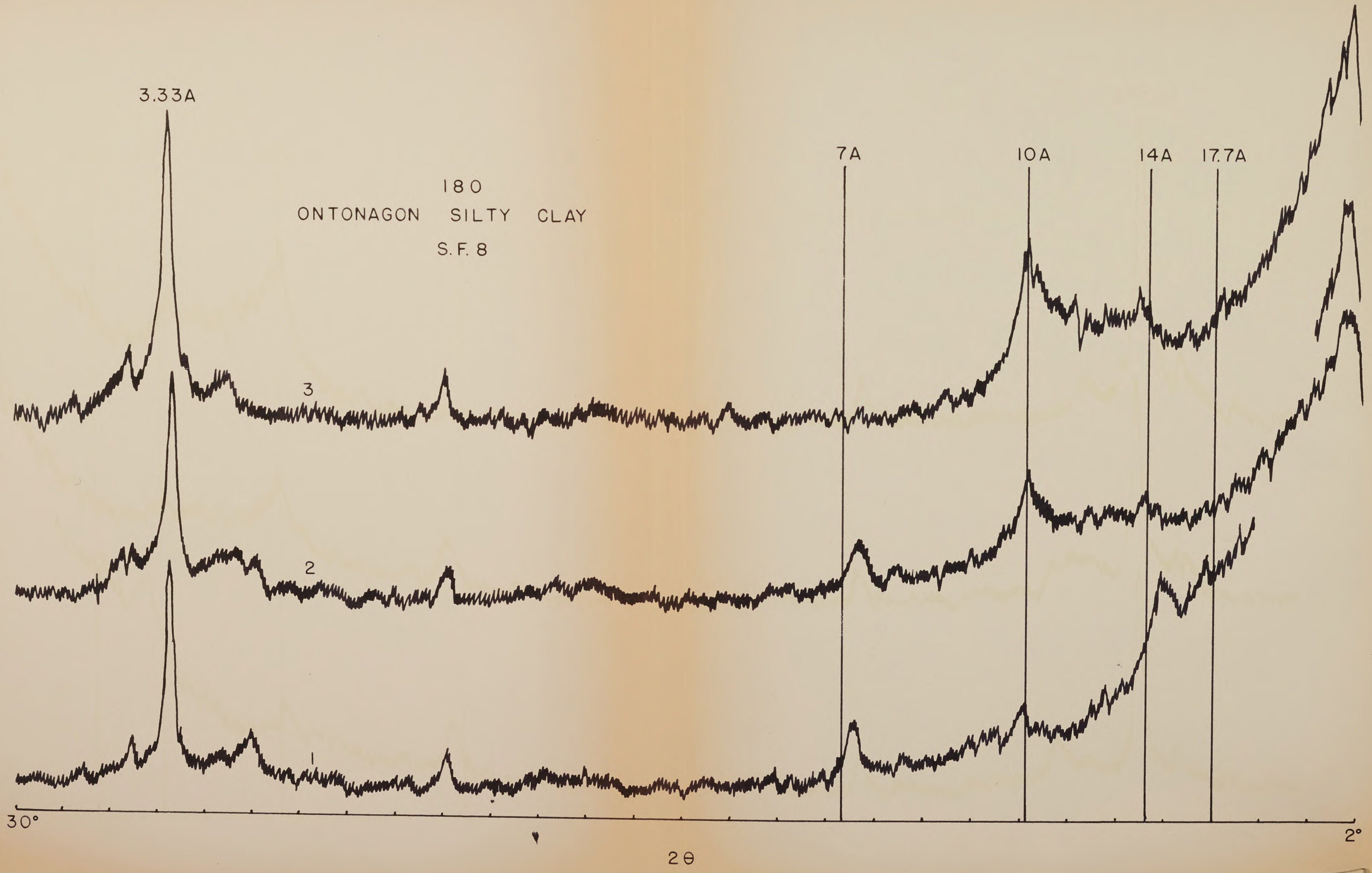
10A
14A
17.7A
30
BROOKSTON LOAM
S.F. 8
3
2
1
30°
2°
2θ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S.F. 8
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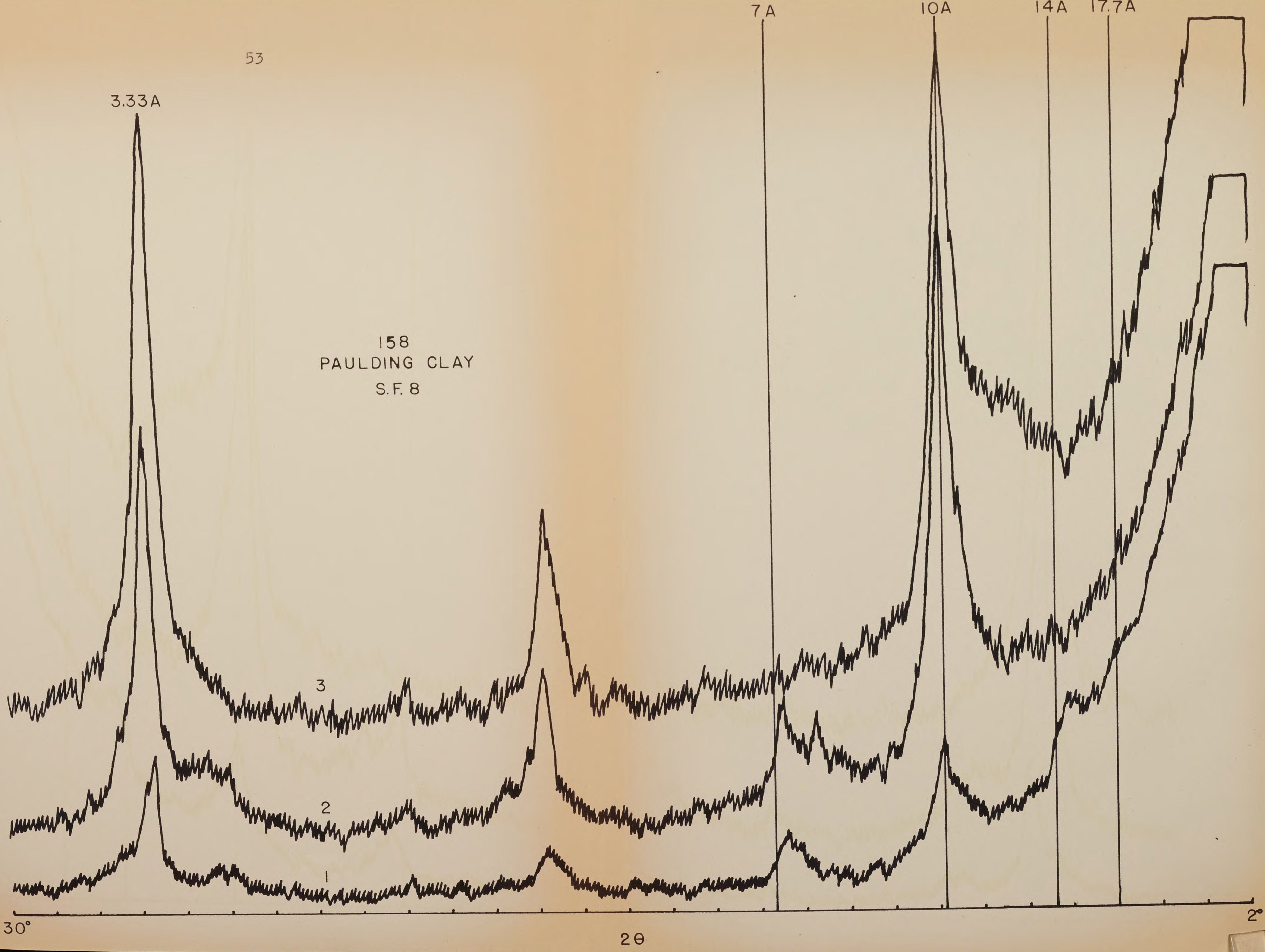
10A
14A
17.7A
3
2
1
30°
2°
2θ



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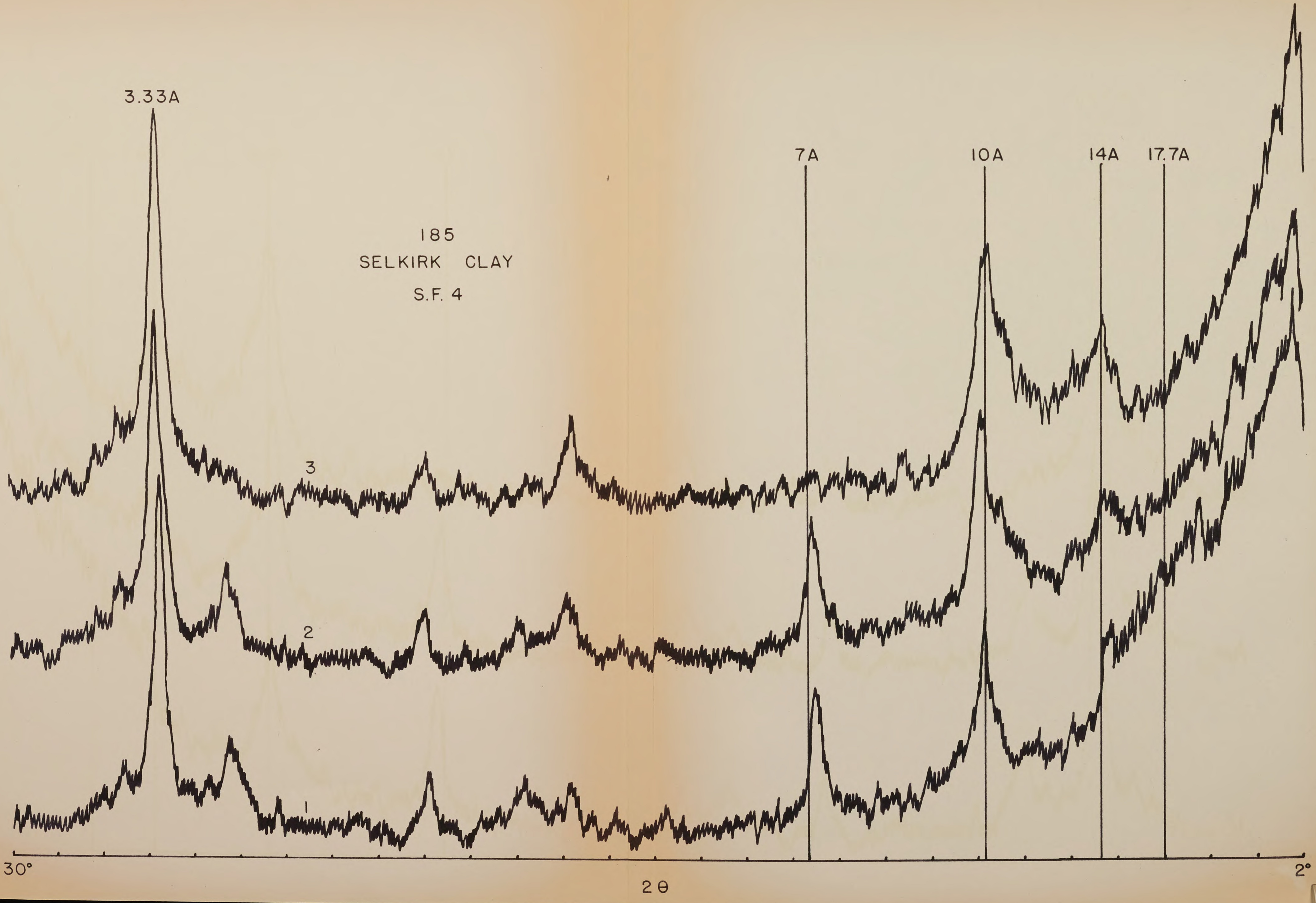
PAULDING CLAY
S.F. 8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14A
17.7A
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SELKIRK CLAY
S.F. 4
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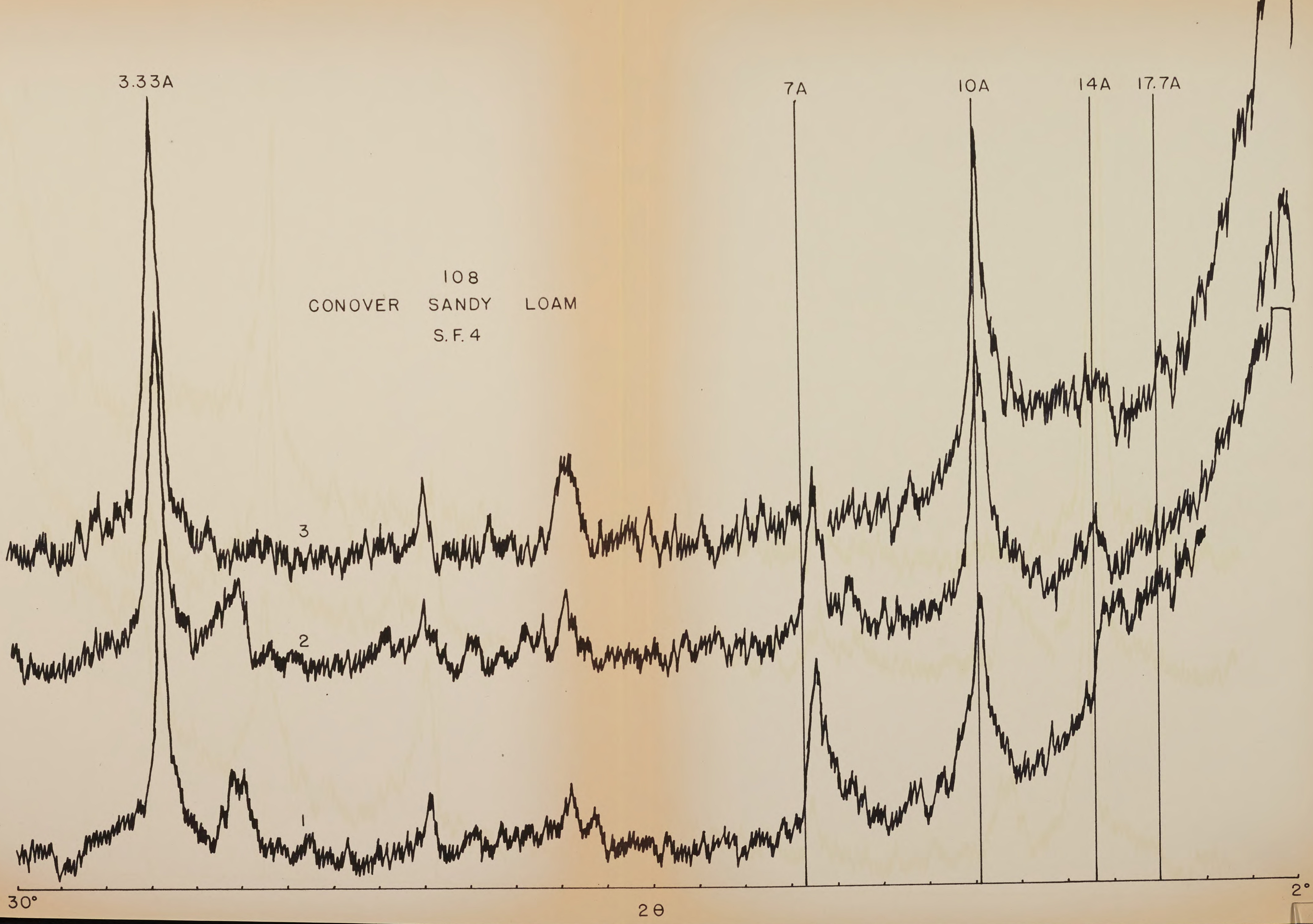
2
1
30°
2°
2θ



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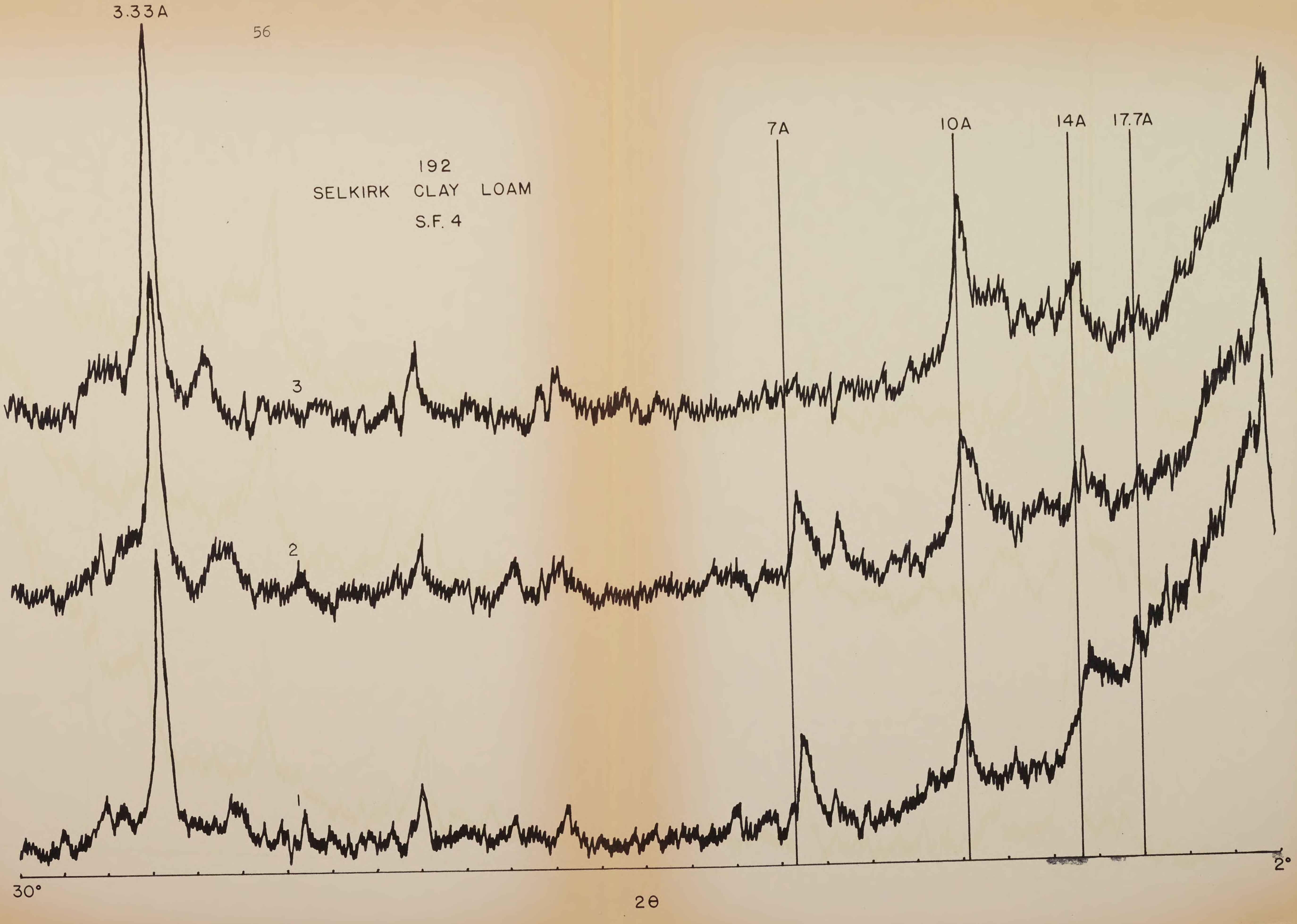
CONOVER SANDY LOAM
S.F. 4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SELKIRK CLAY LOAM
S.F. 4
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17.7A
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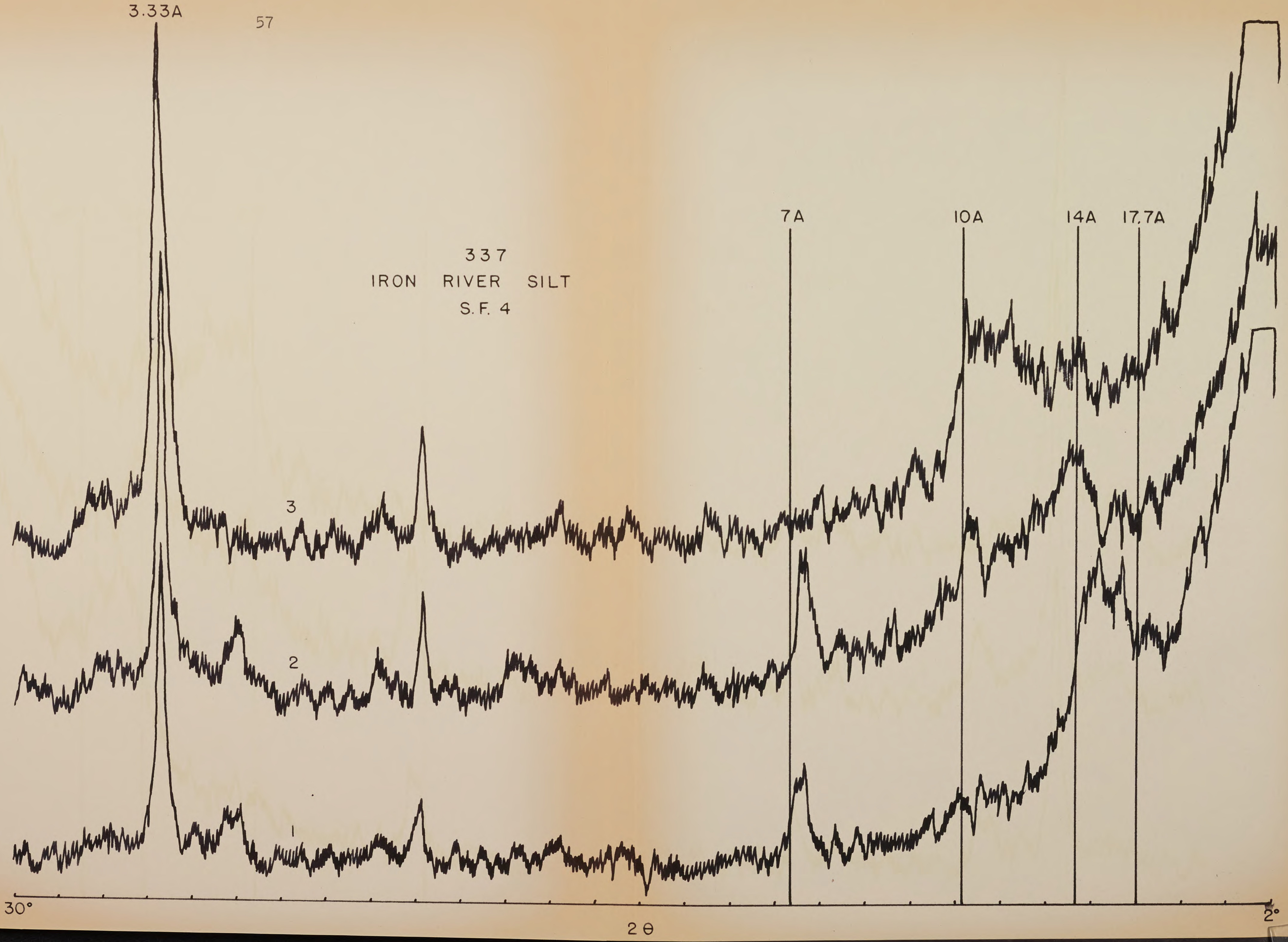
2
1
30°
2θ
2°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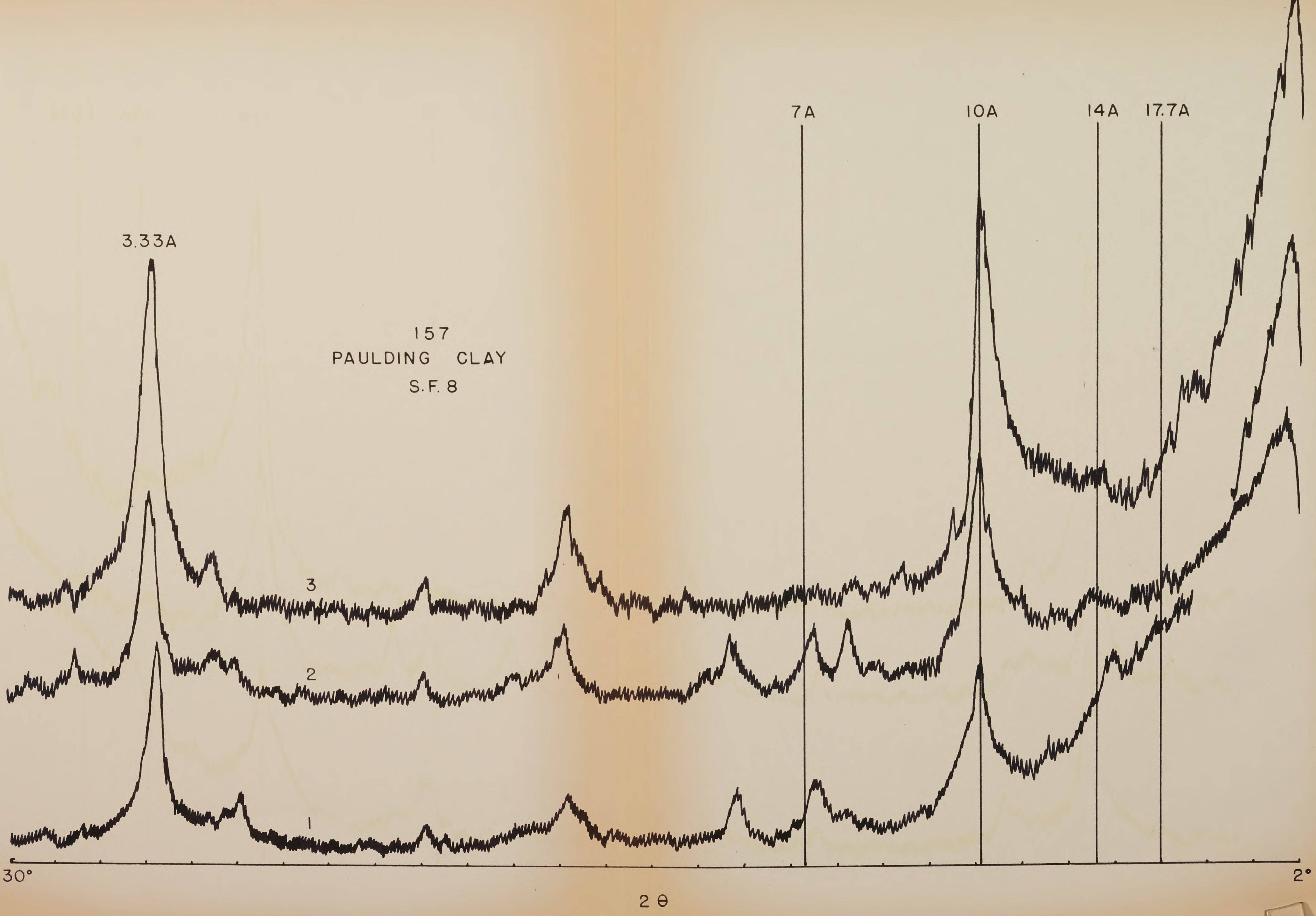
17.7A
3
2
1
30°
2°
2 θ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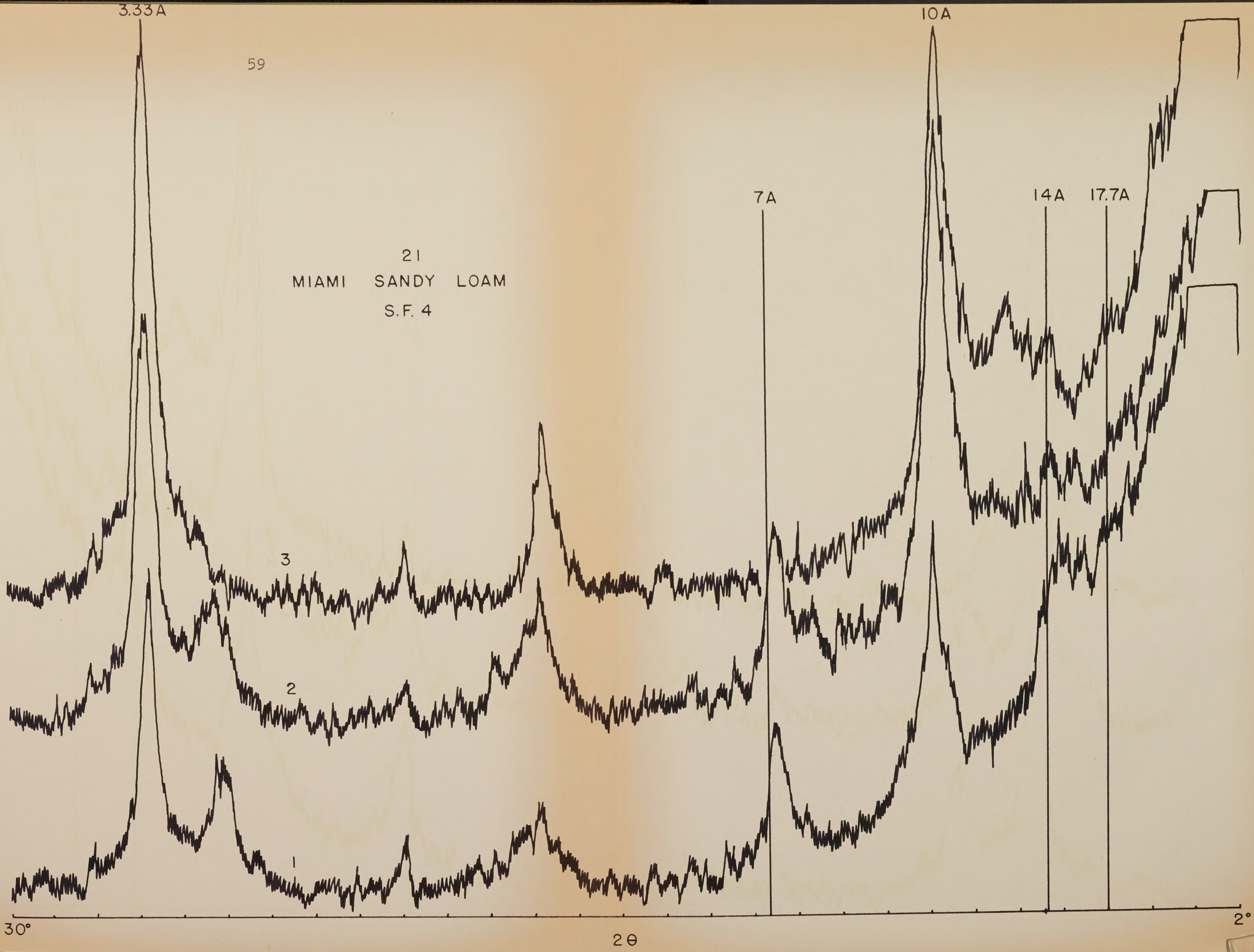
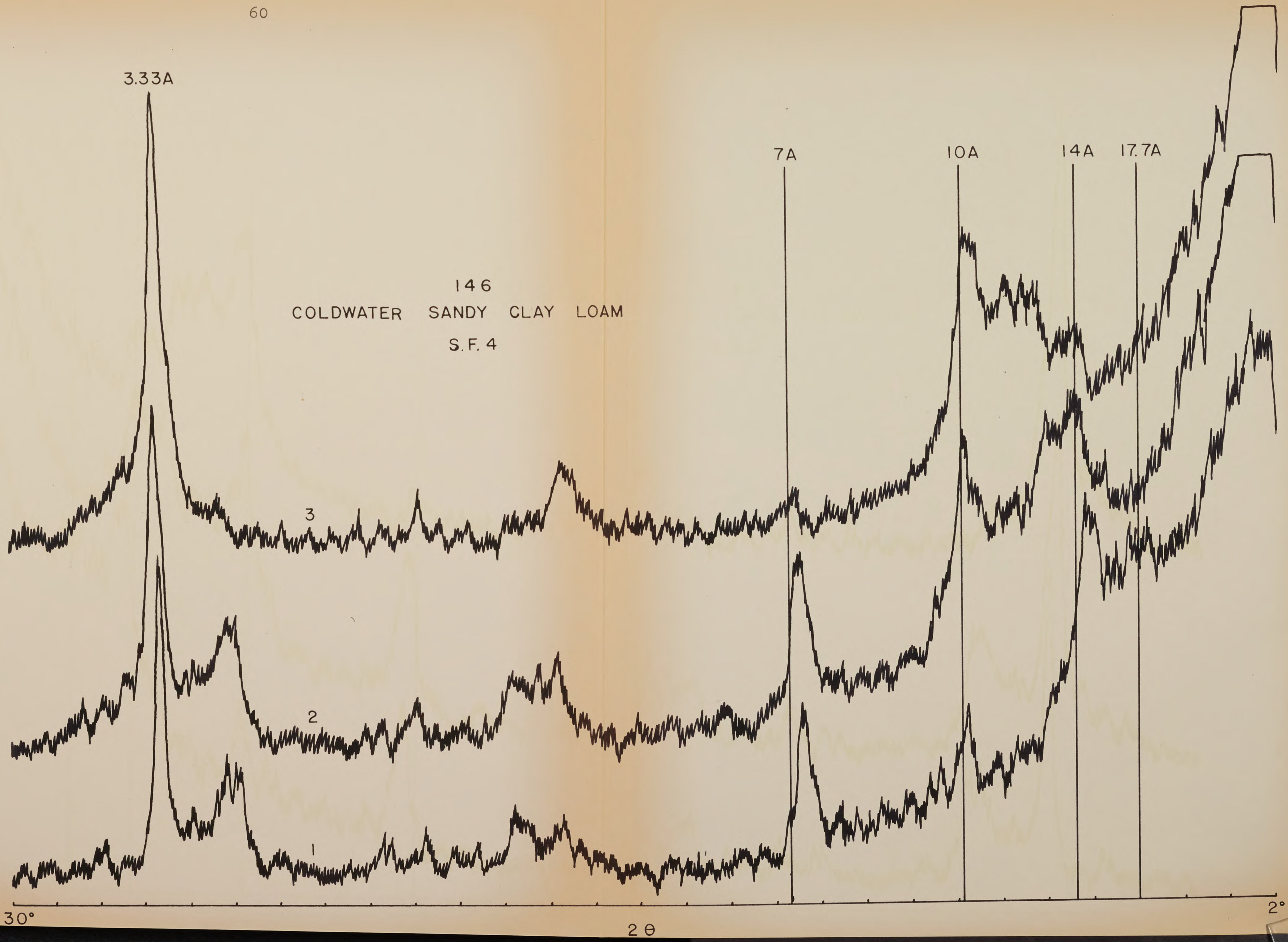
2
1
30°
2°
2 θ



21
MIAMI SANDY LOAM
S.F. 4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17.7A
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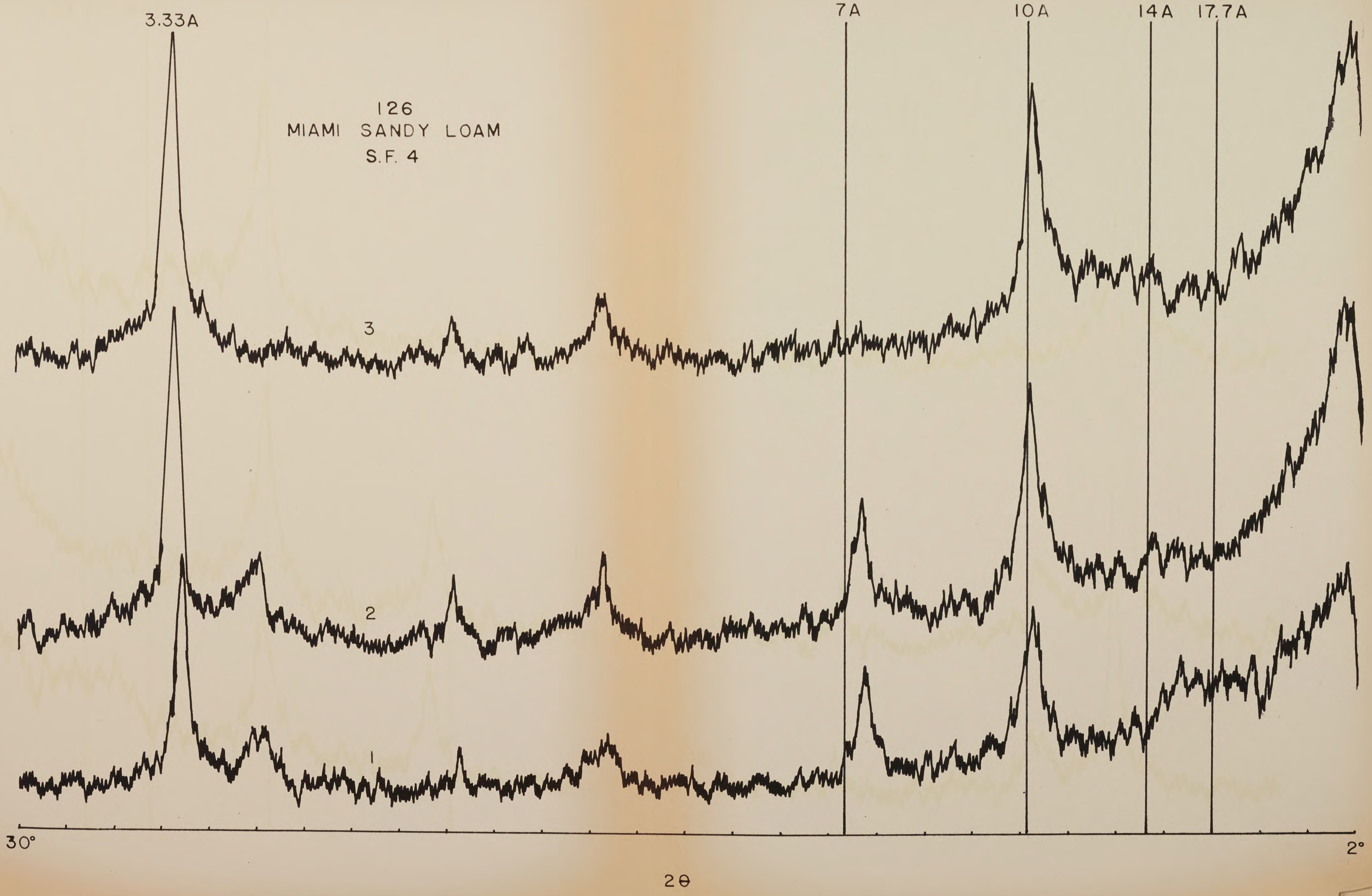
1
30°
2°
2 θ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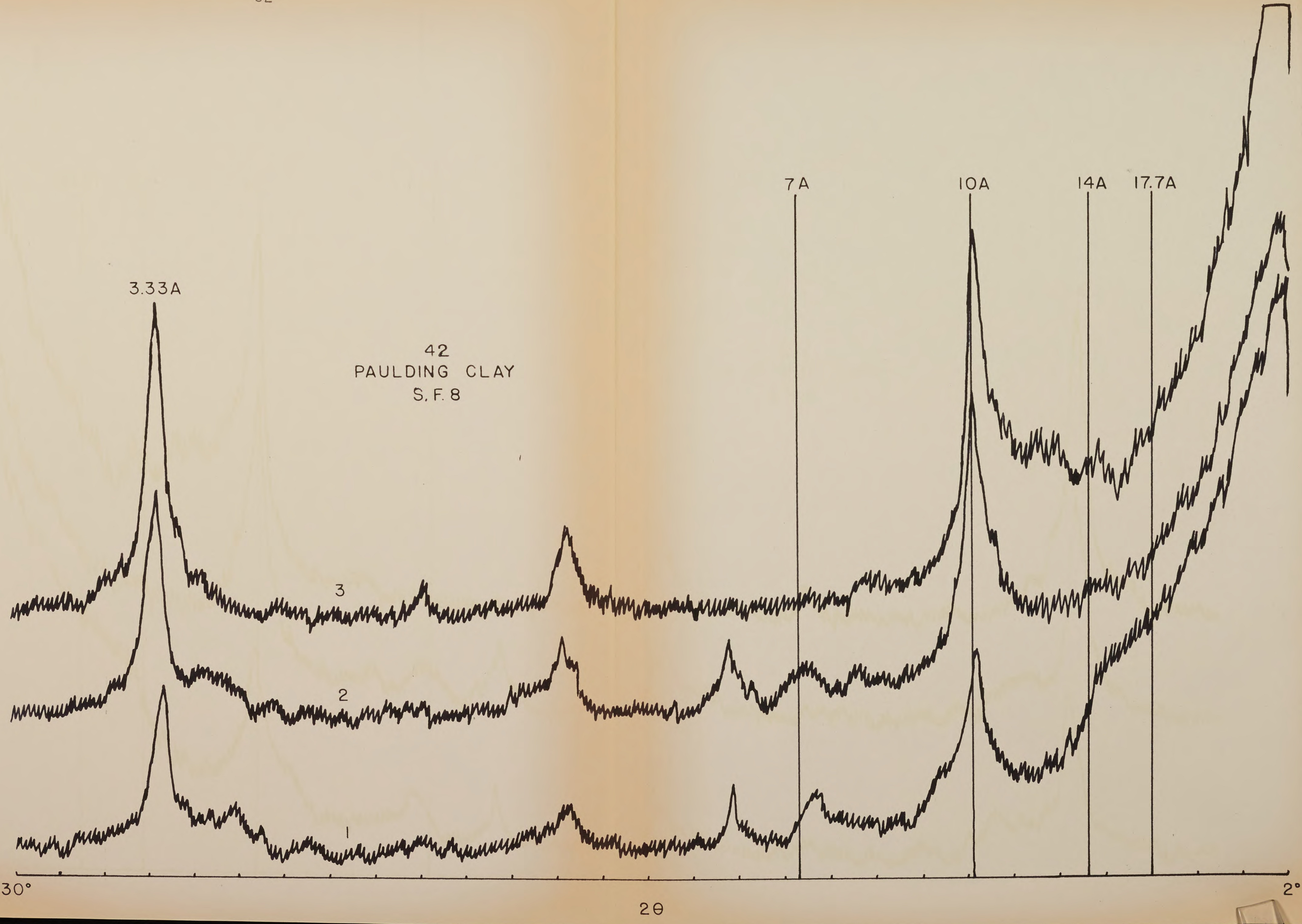
14A
17.7A
3
2
1
30°
2°
2θ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S.F.8
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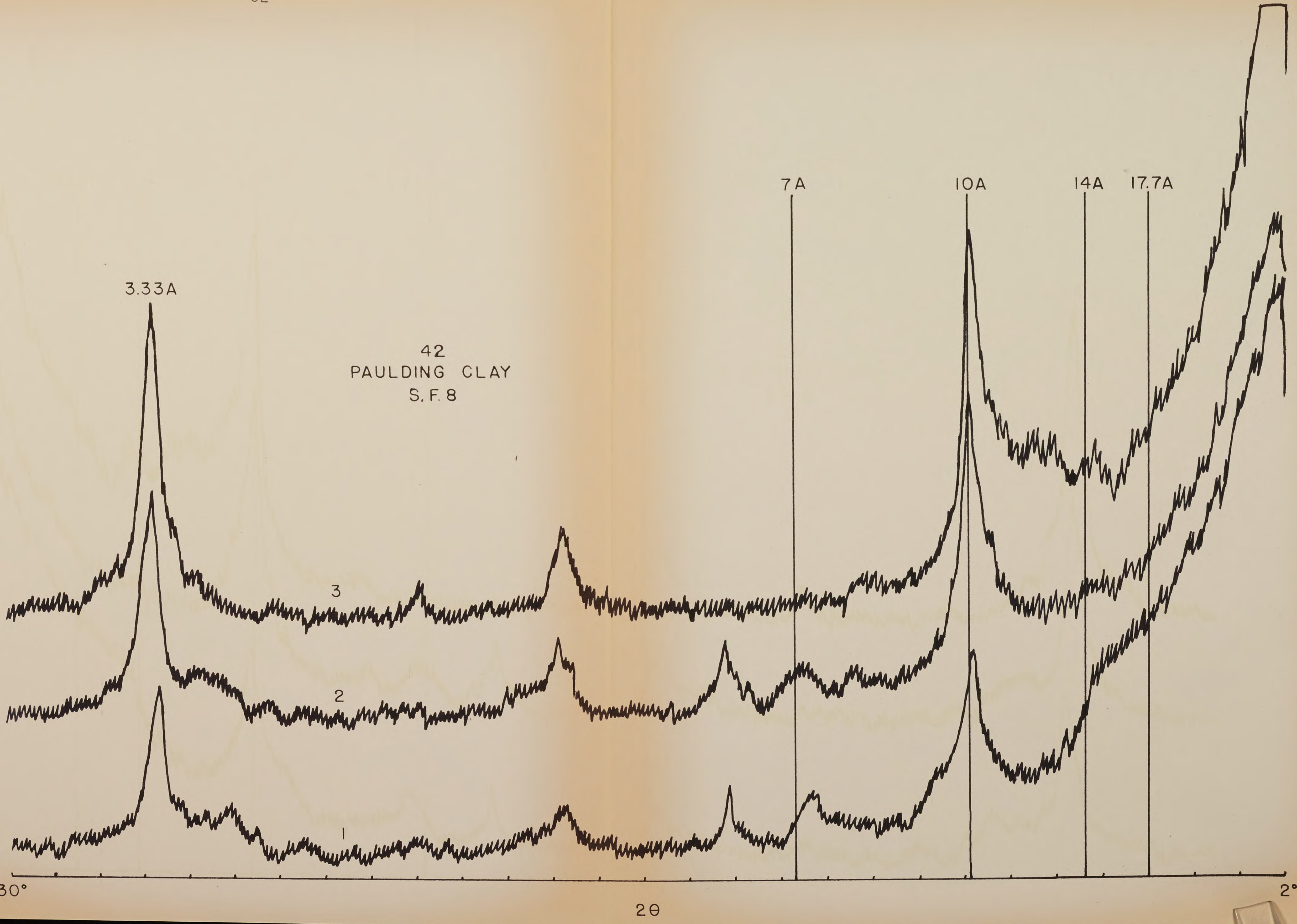
10A
14A
17.7A
3
2
1
30°
2°
2θ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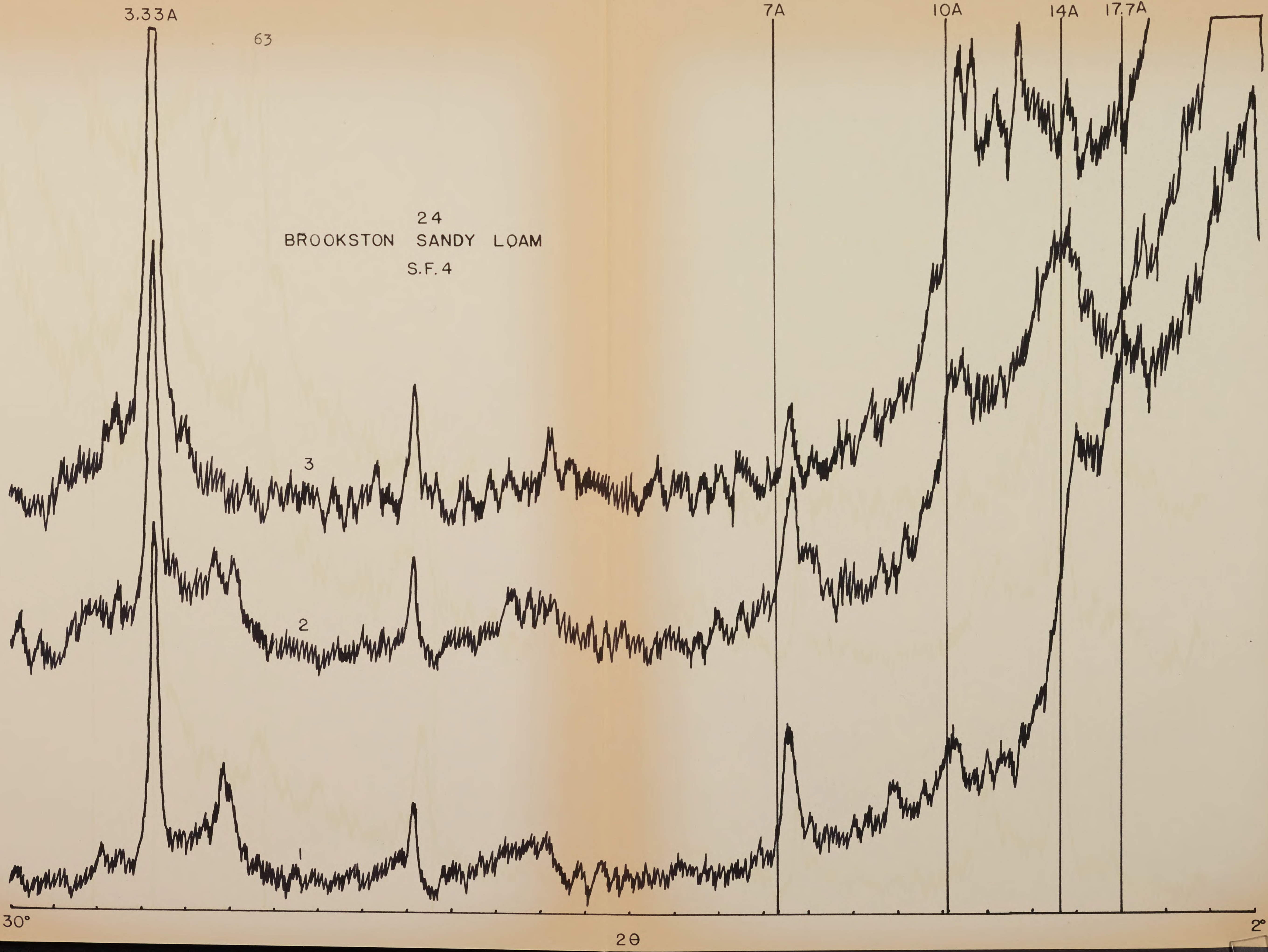
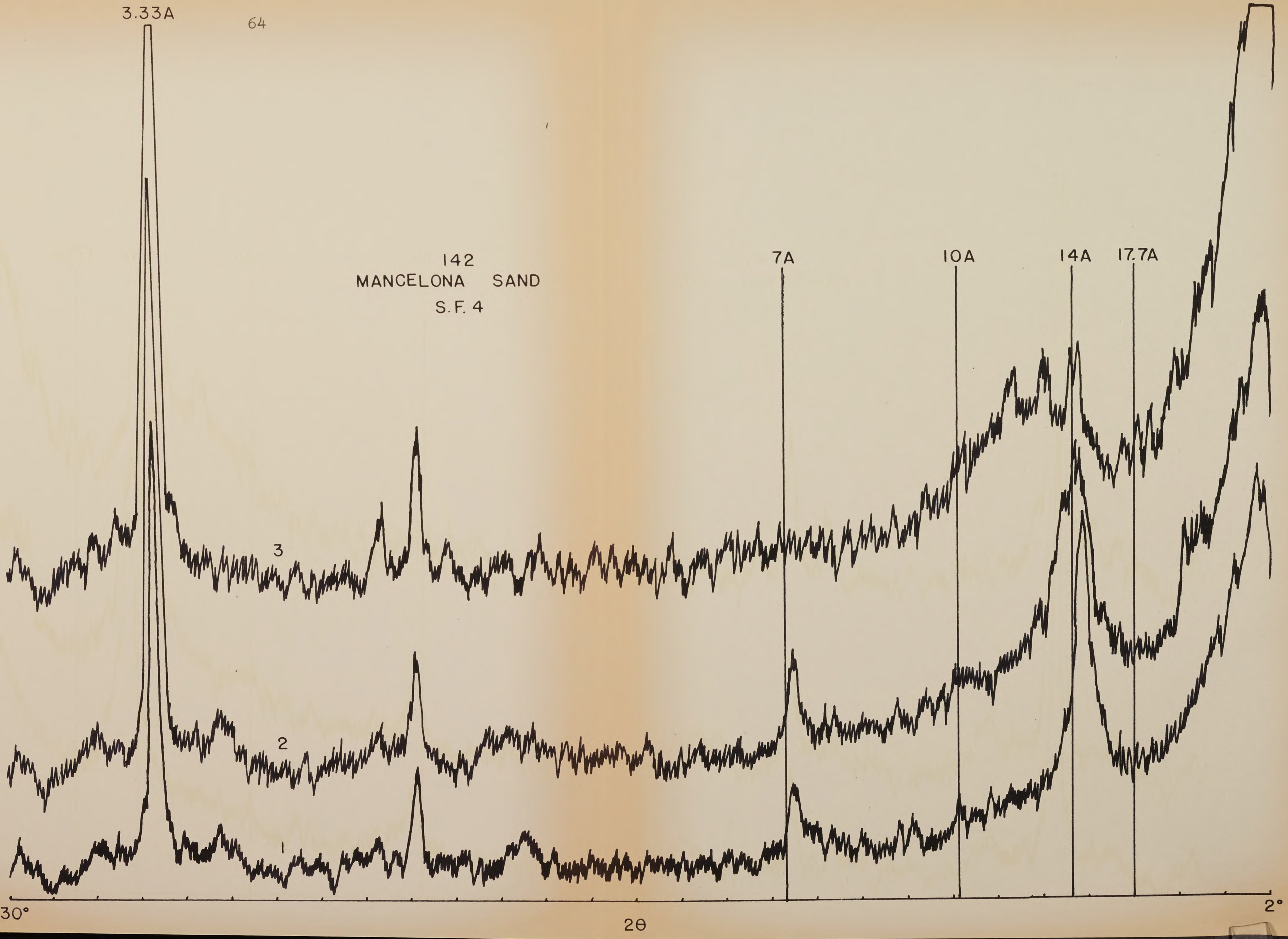
42
PAULDING CLAY
S.F.8
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ

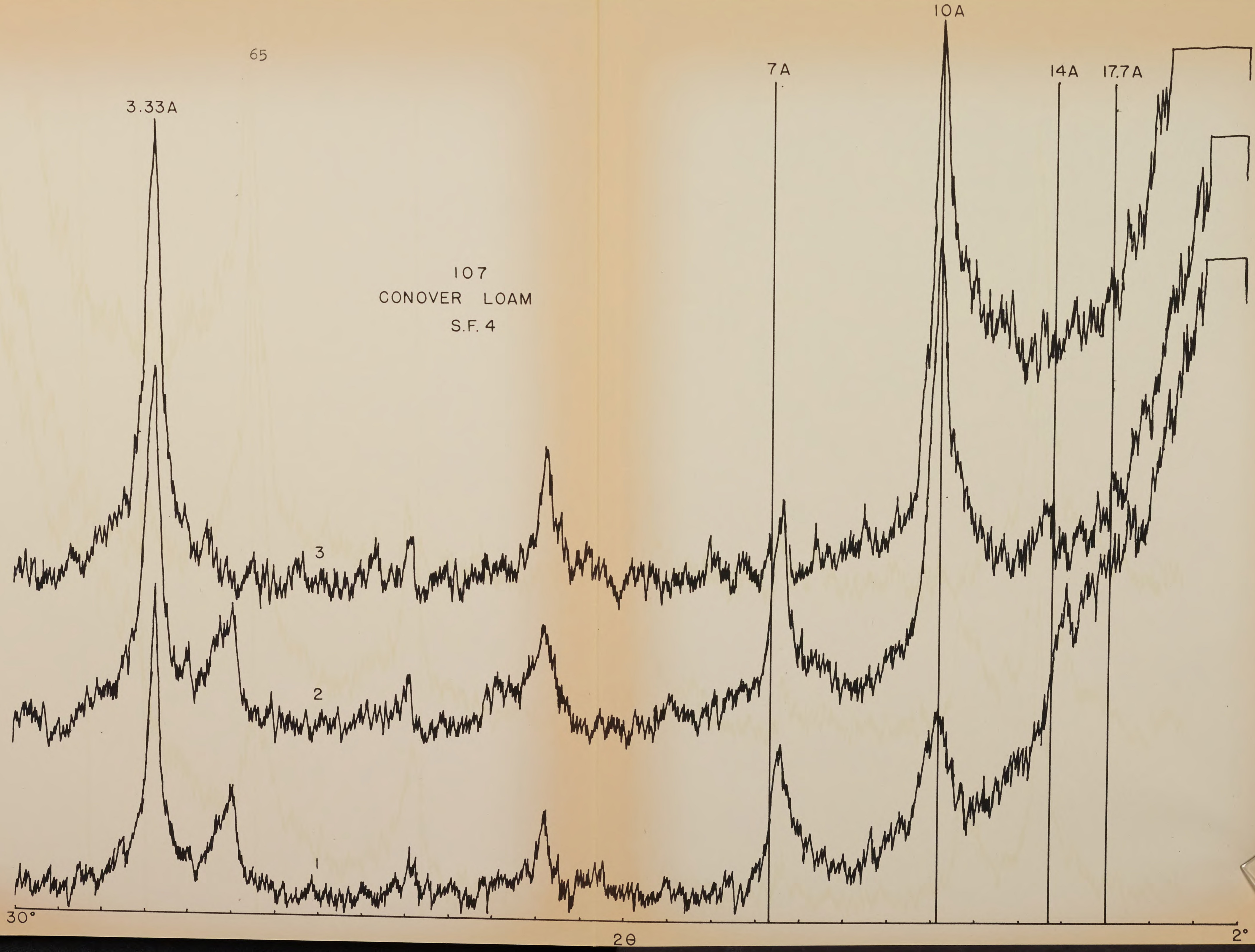
24

BROOKSTON SANDY LOAM

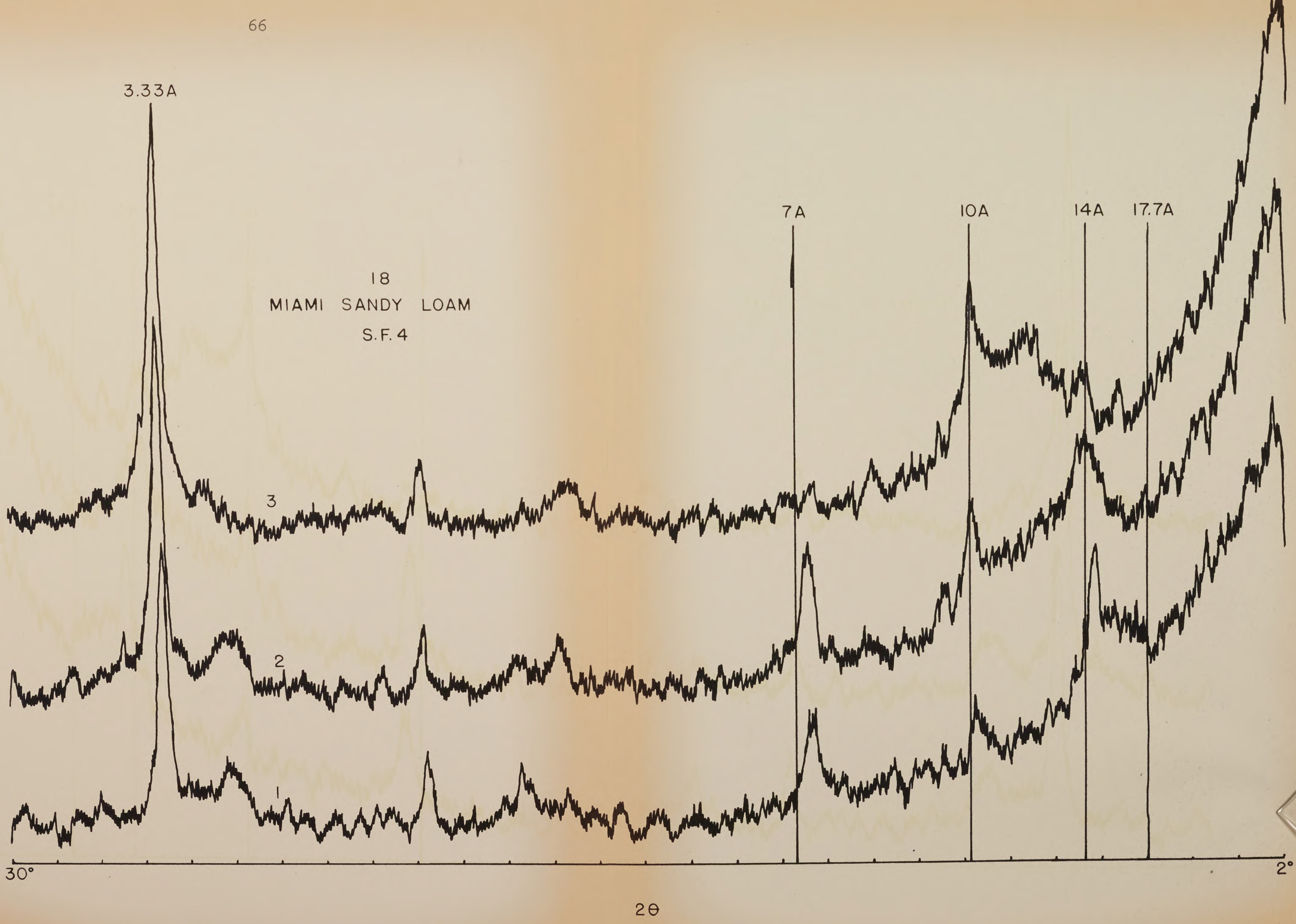
S.F. 4



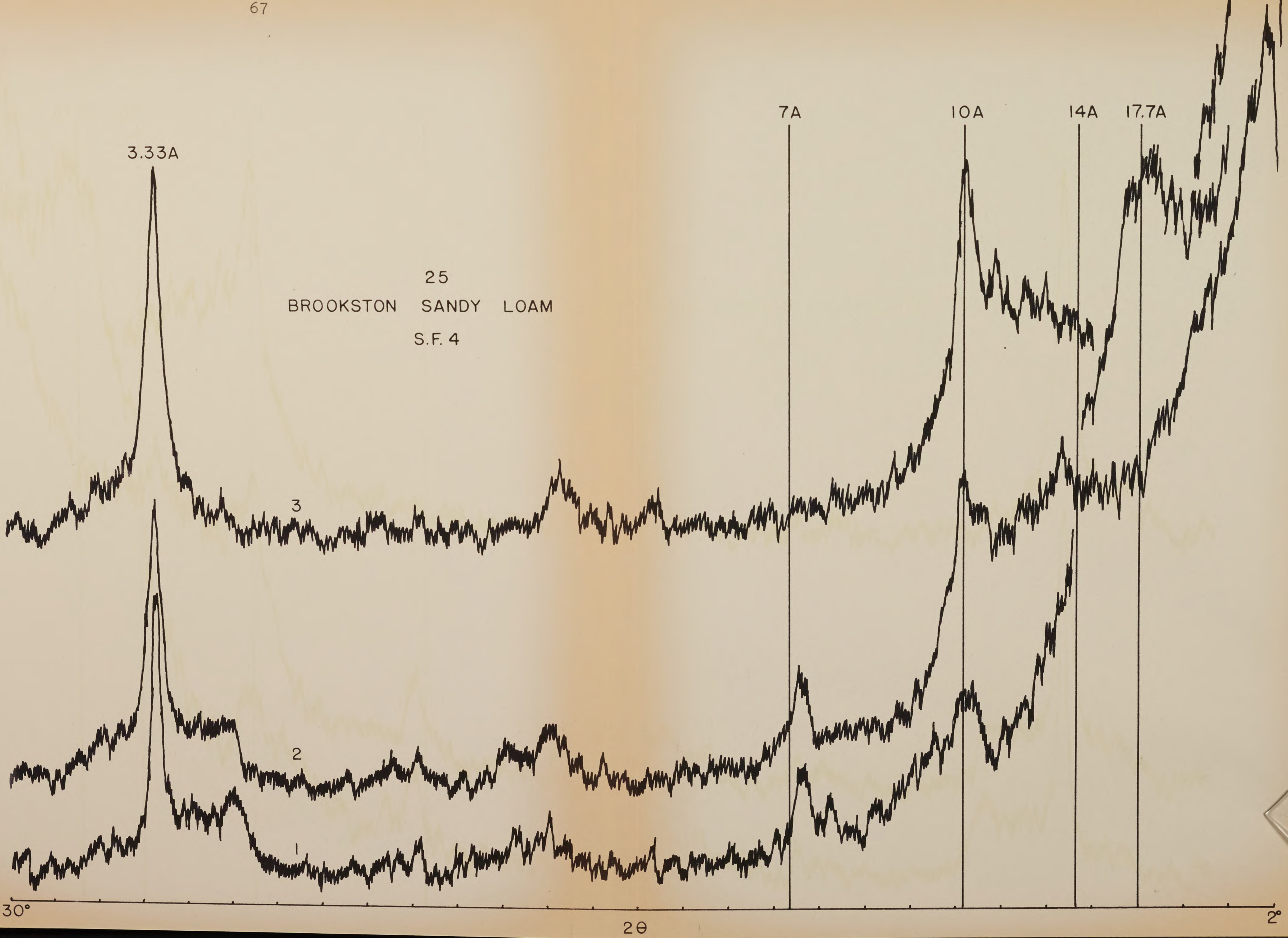

3.33A
142
MANCELONA SAND
S.F. 4
7A
10A
14A
17.7A
3
2
1
30°
2θ
2°

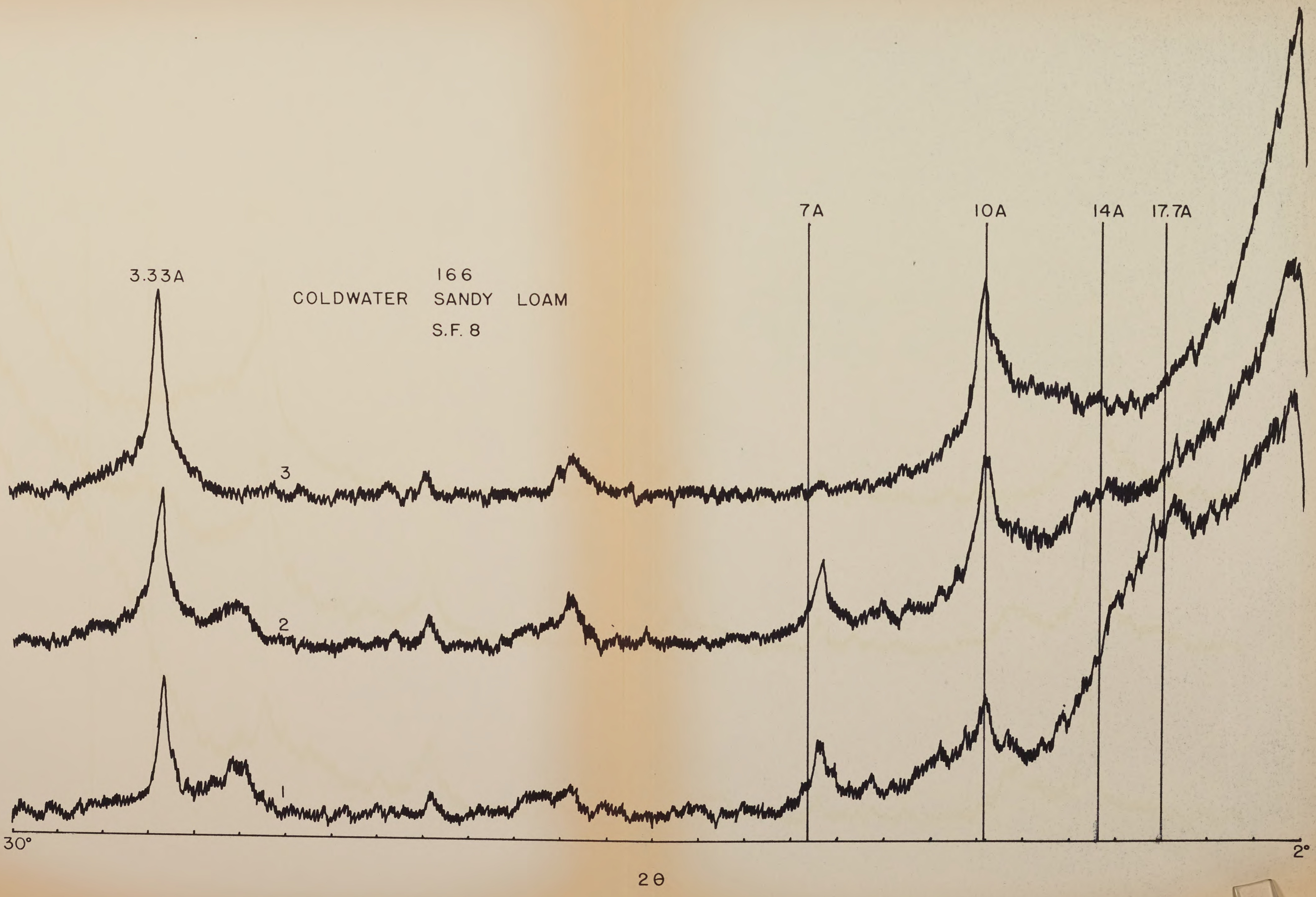


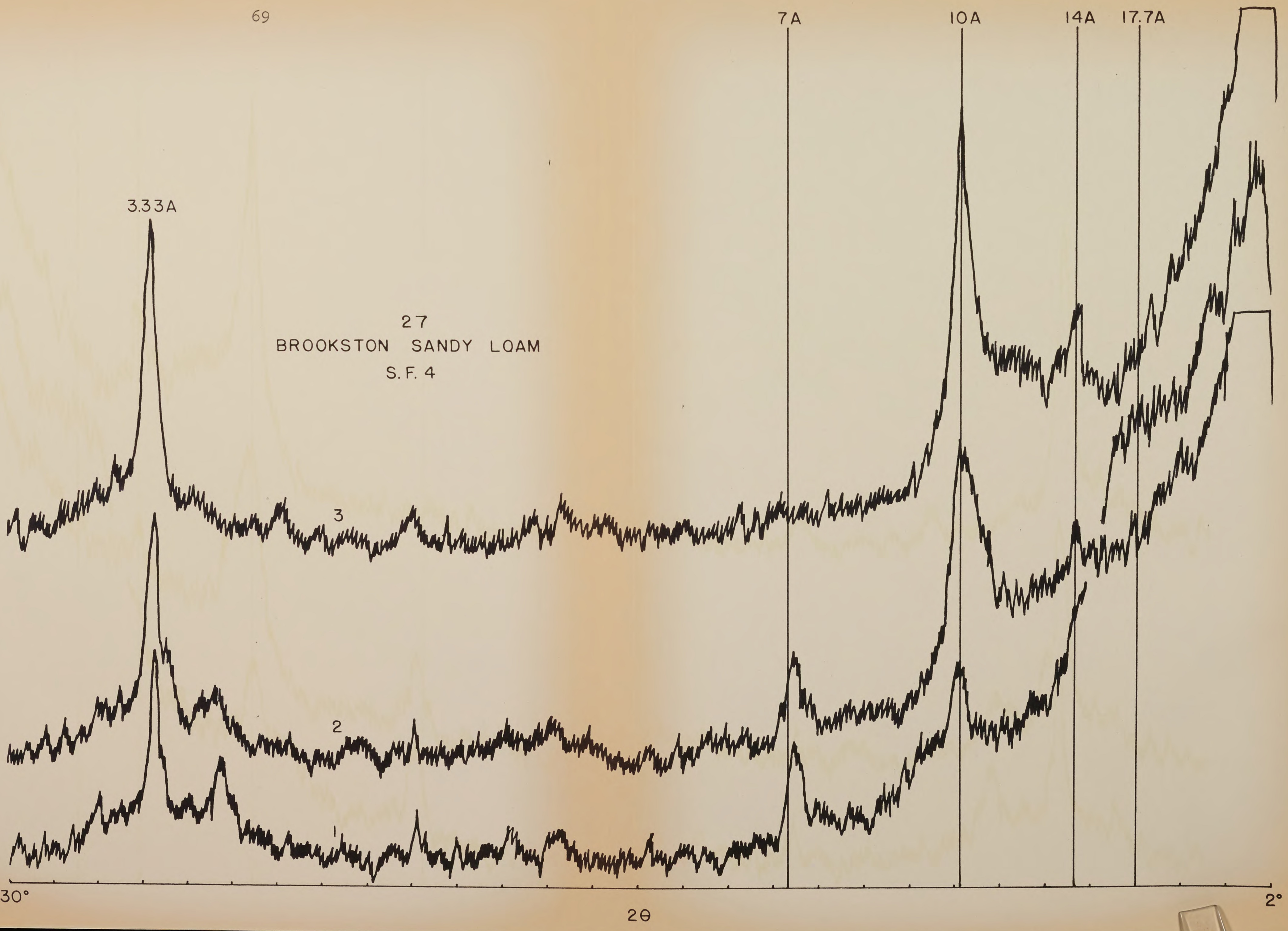
107
CONOVER LOAM
S.F. 4

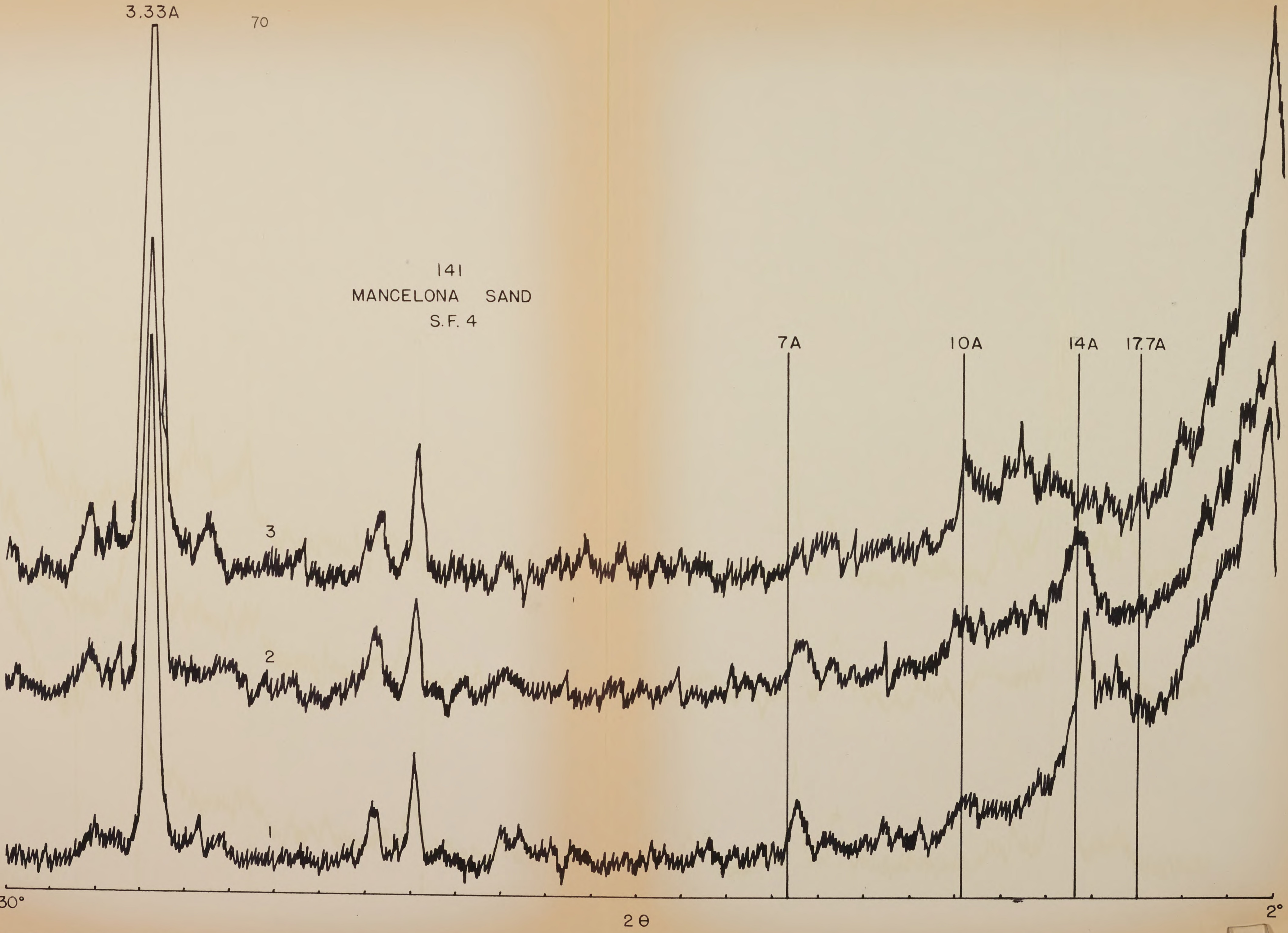


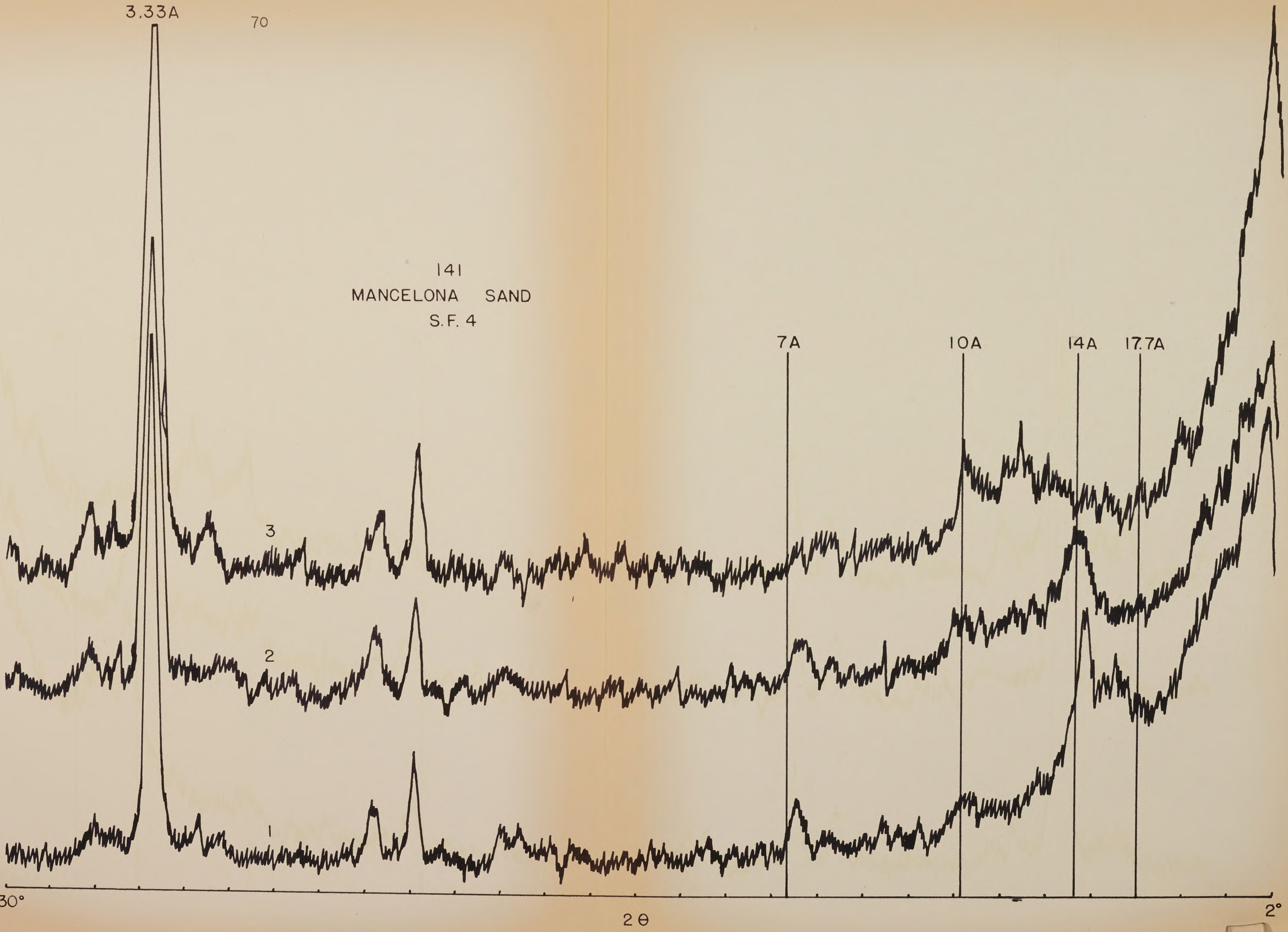
18
MIAMI SANDY LOAM
S.F. 4

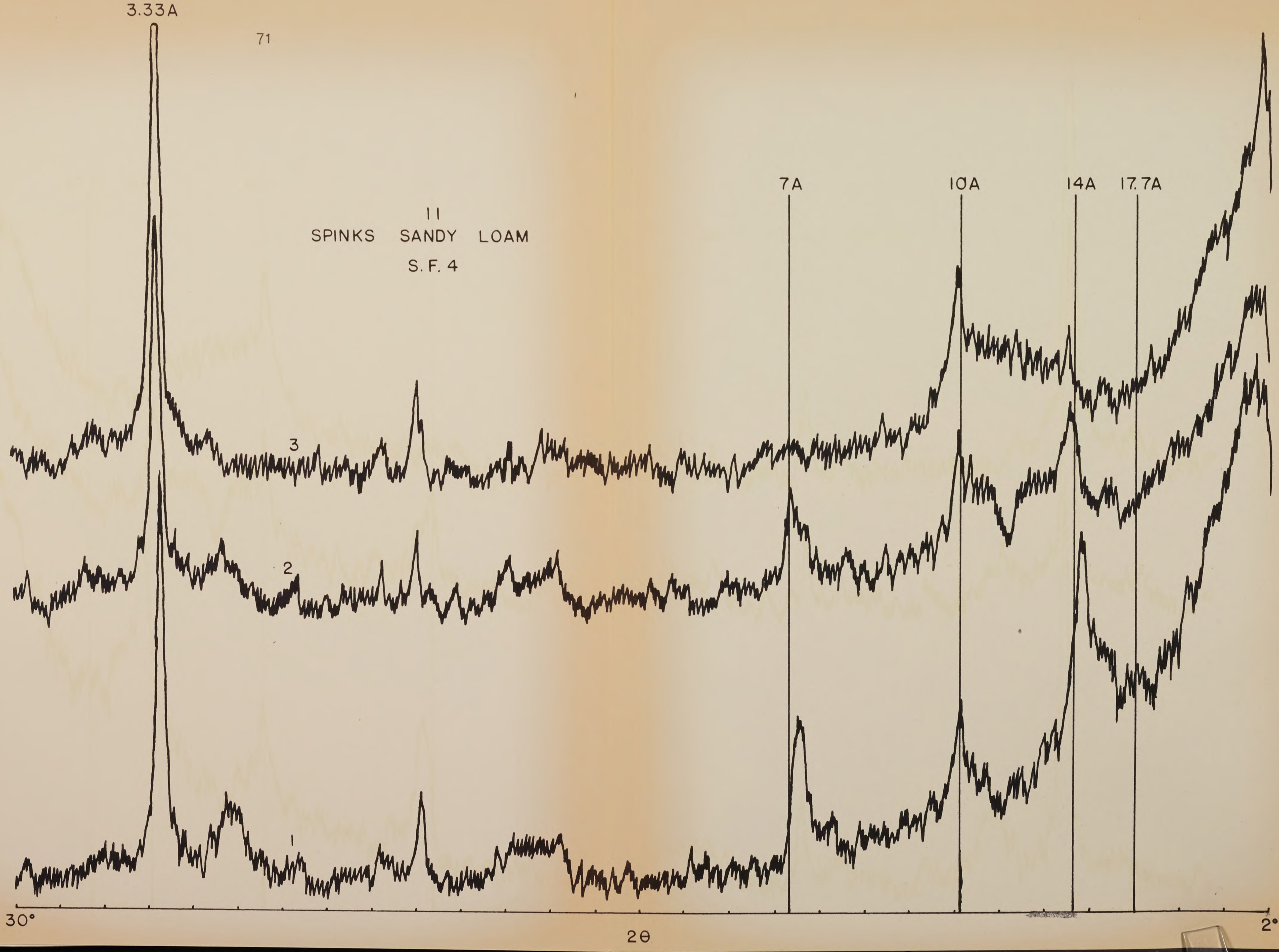

25
BROOKSTON SANDY LOAM
S.F. 4
3.33A
7A
10A
14A
17.7A
3
2
1
30°
2θ
2°


3.33A
166
COLDWATER SANDY LOAM
S.F. 8
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ

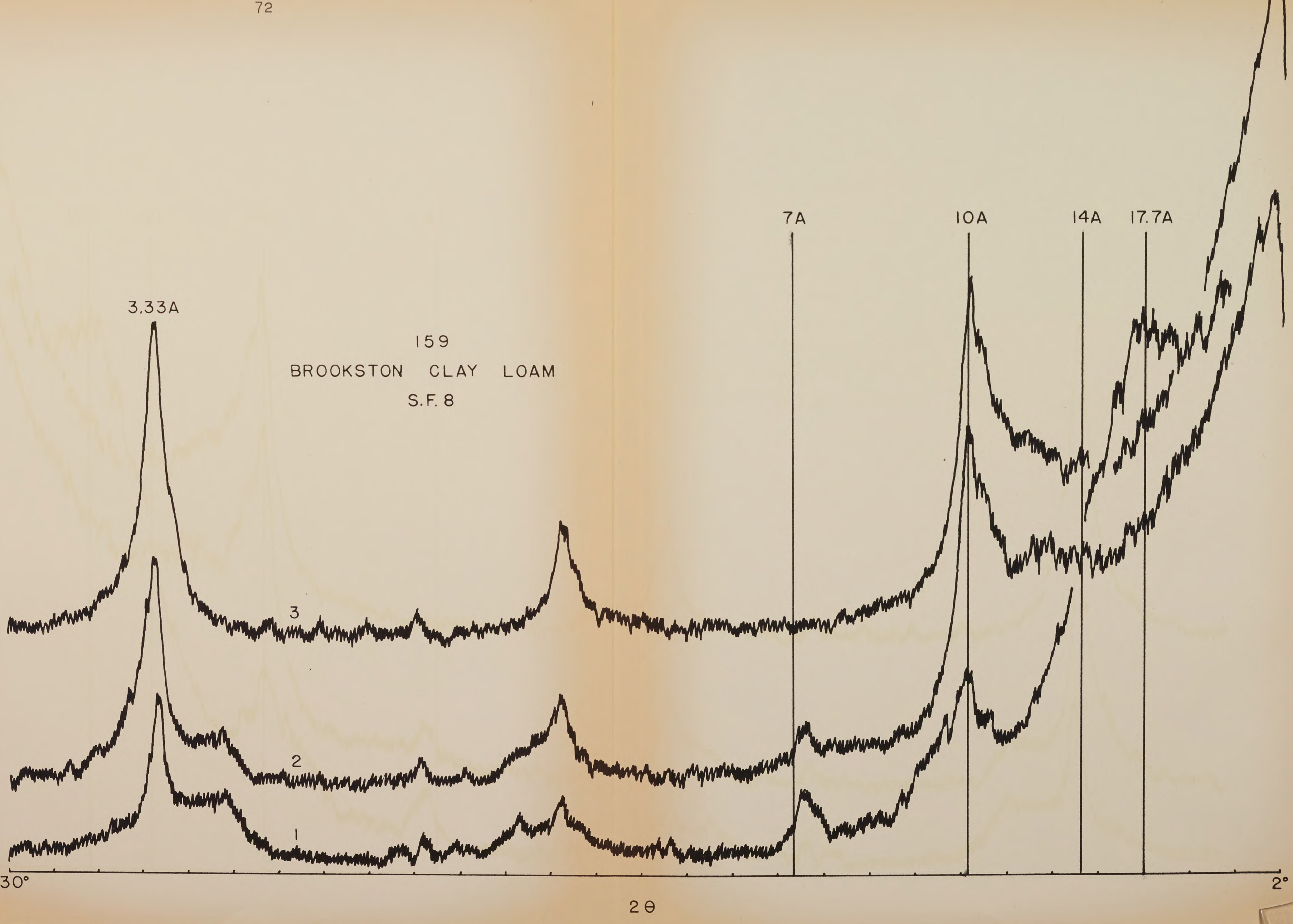

27
BROOKSTON SANDY LOAM
S.F. 4
3.33A
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ

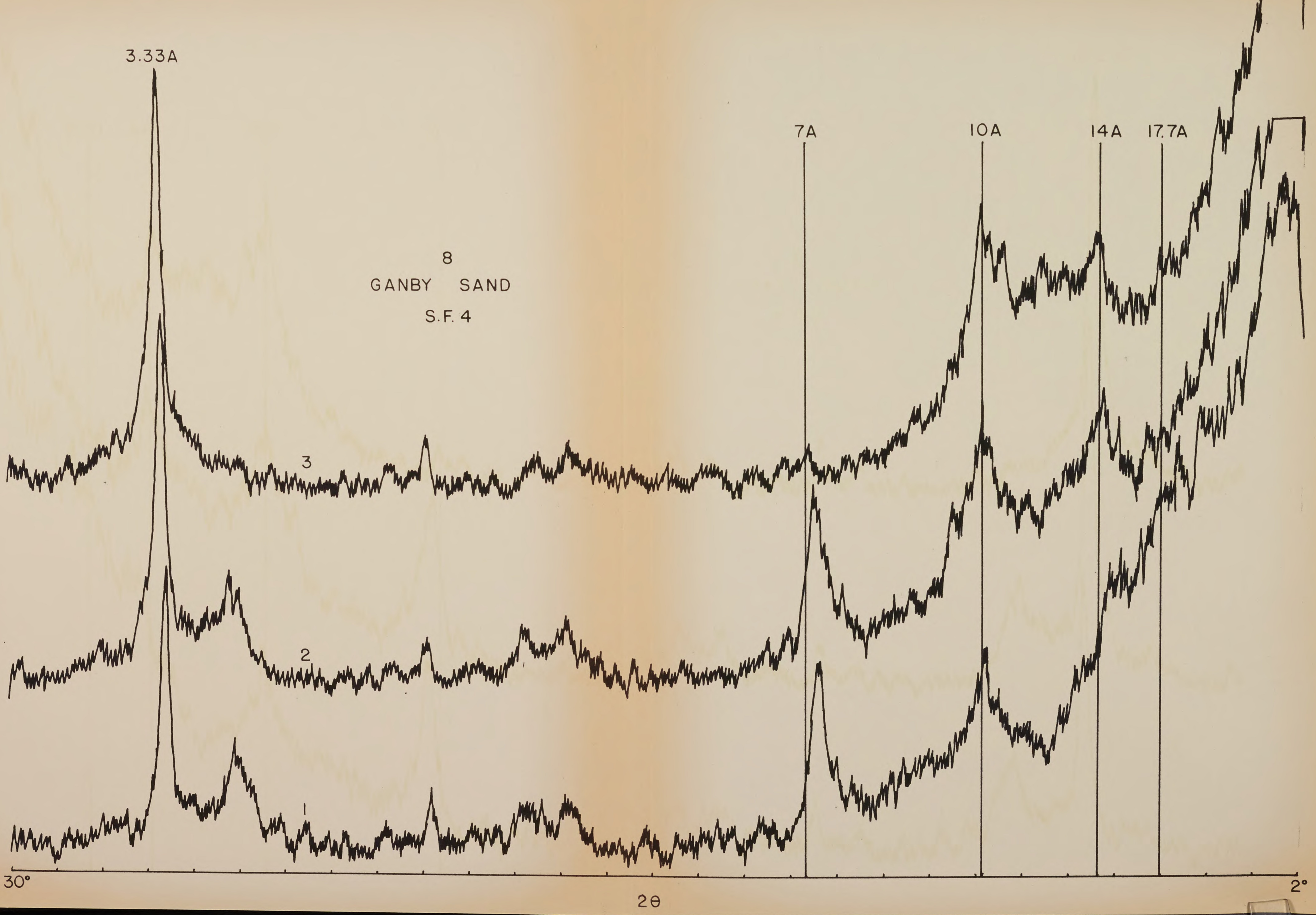

3.33A
141
MANCELONA SAND
S.F. 4
7A
10A
14A
17.7A
3
2
1
30°
2θ
2°


3.33A
141
MANCELONA SAND
S.F. 4
7A
10A
14A
17.7A
3
2
1
30°
2θ
2°

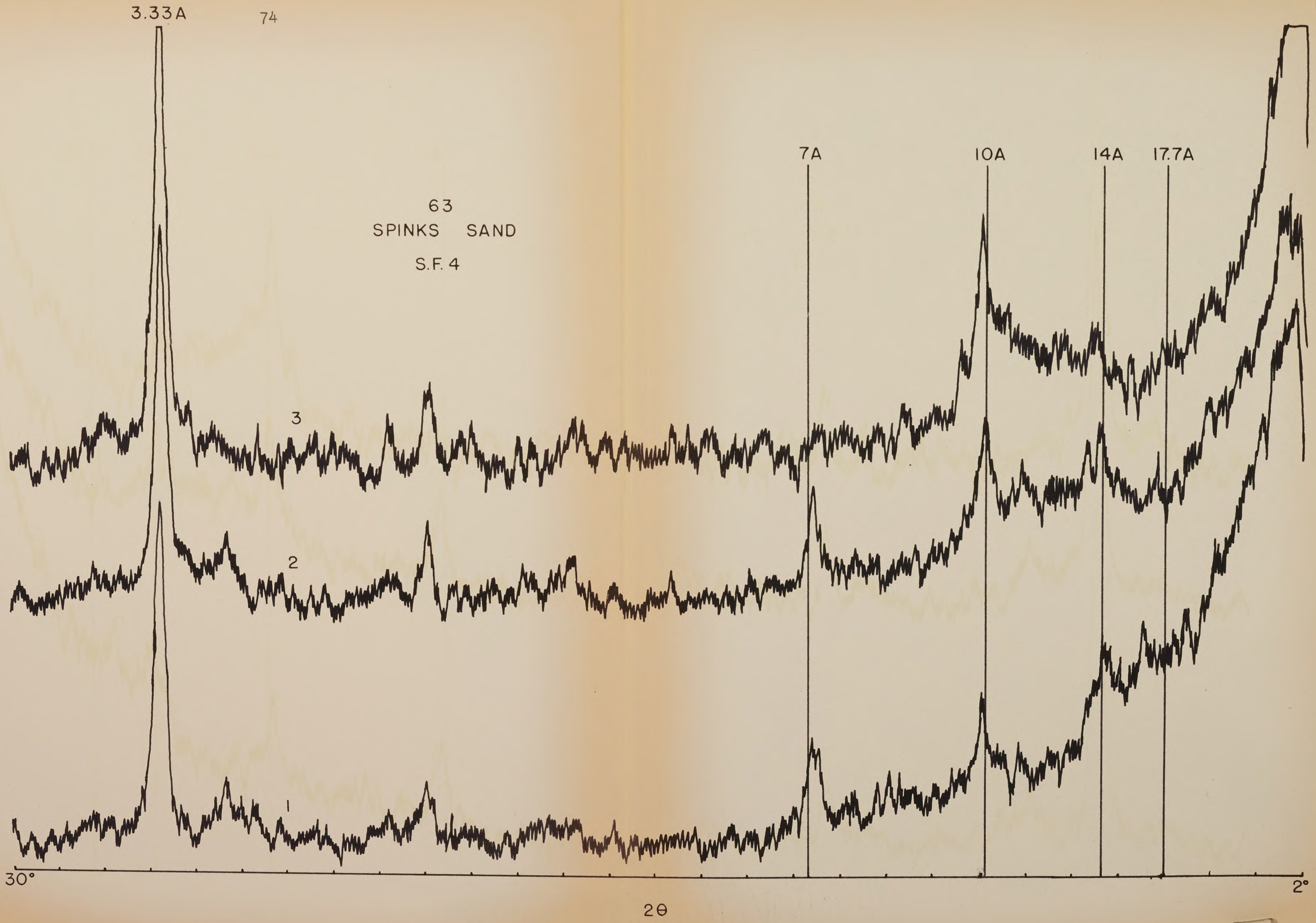


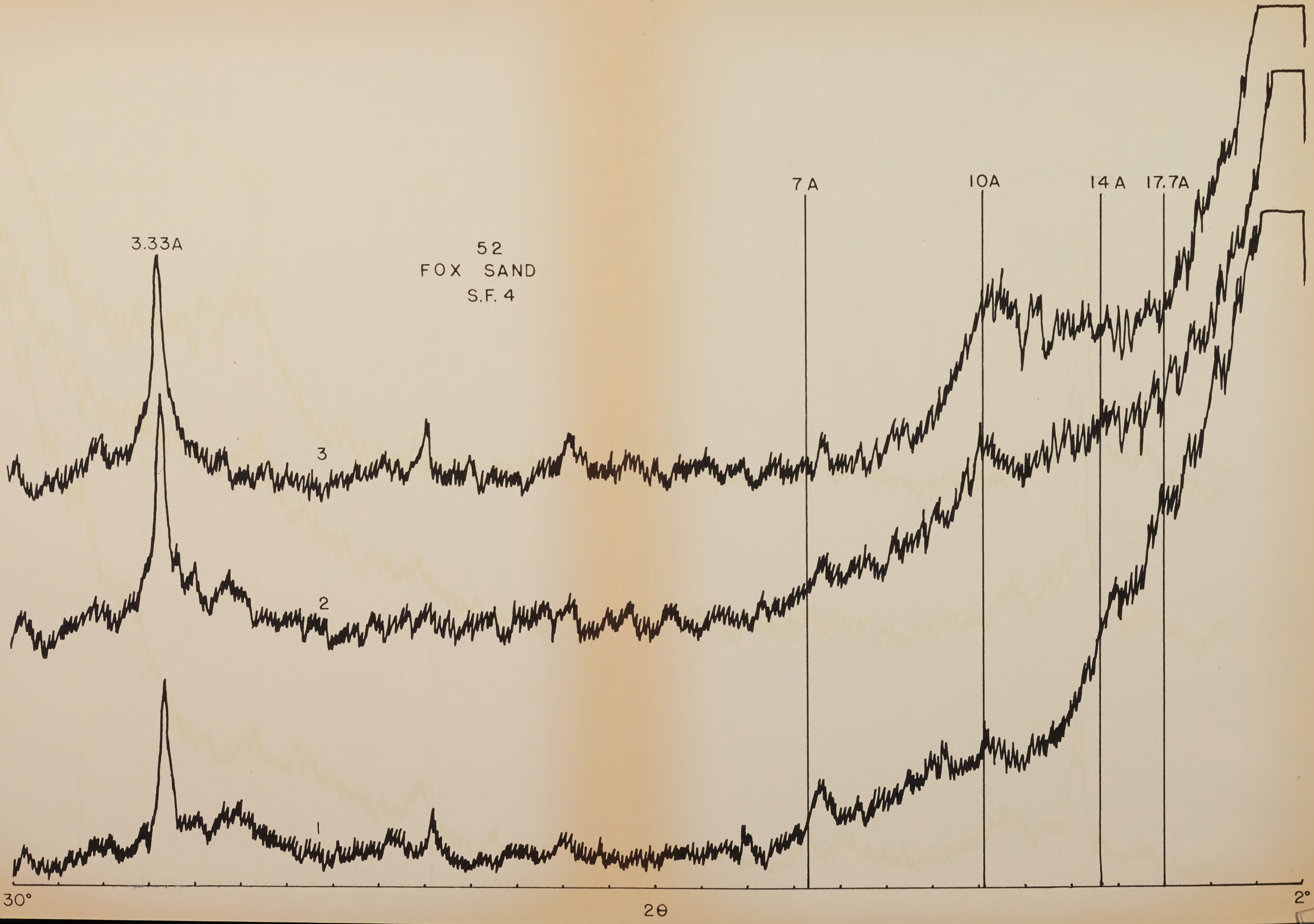
11
SPINKS SANDY LOAM
S. F. 4

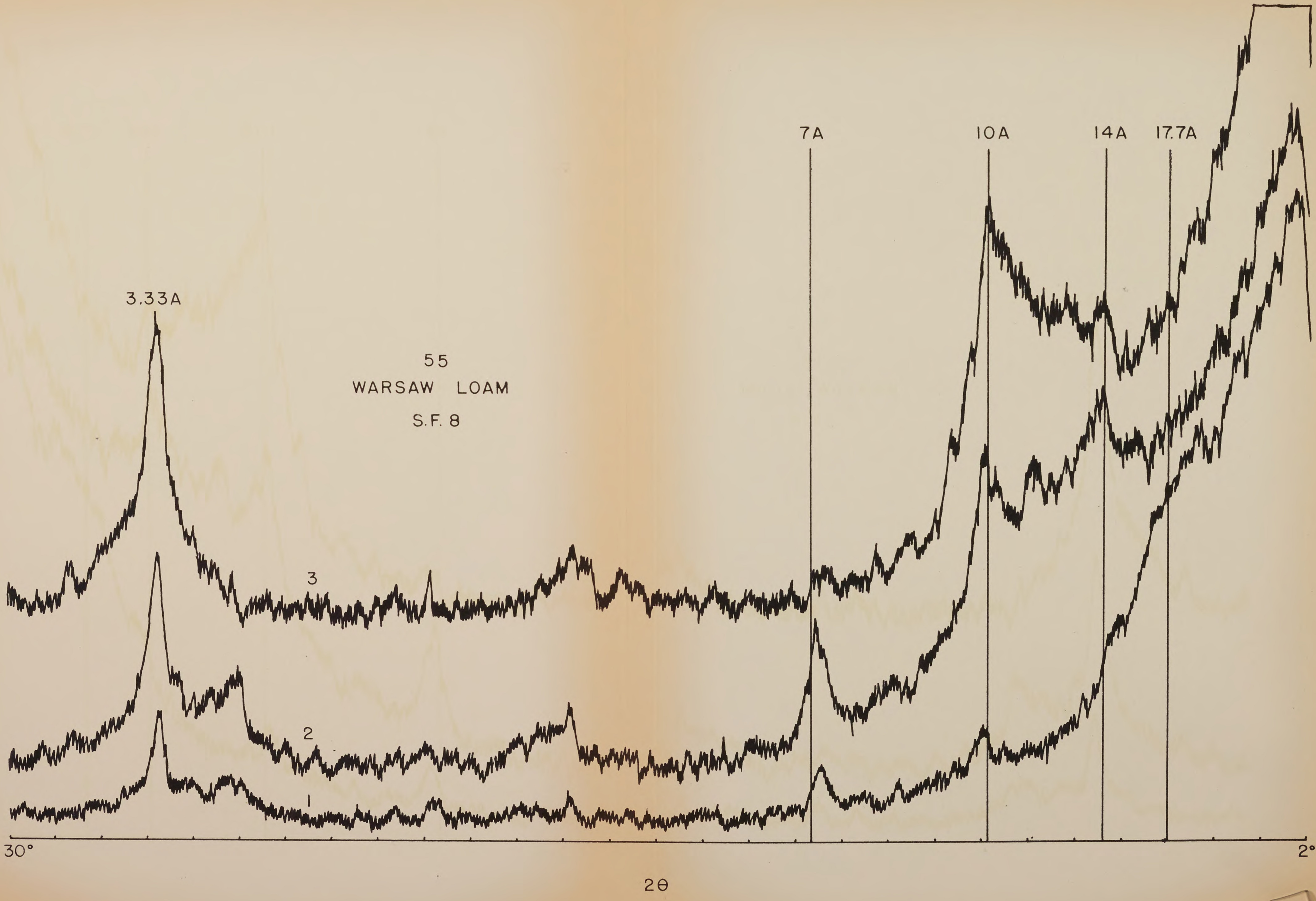

159
BROOKSTON CLAY LOAM
S.F. 8
3.33A
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ

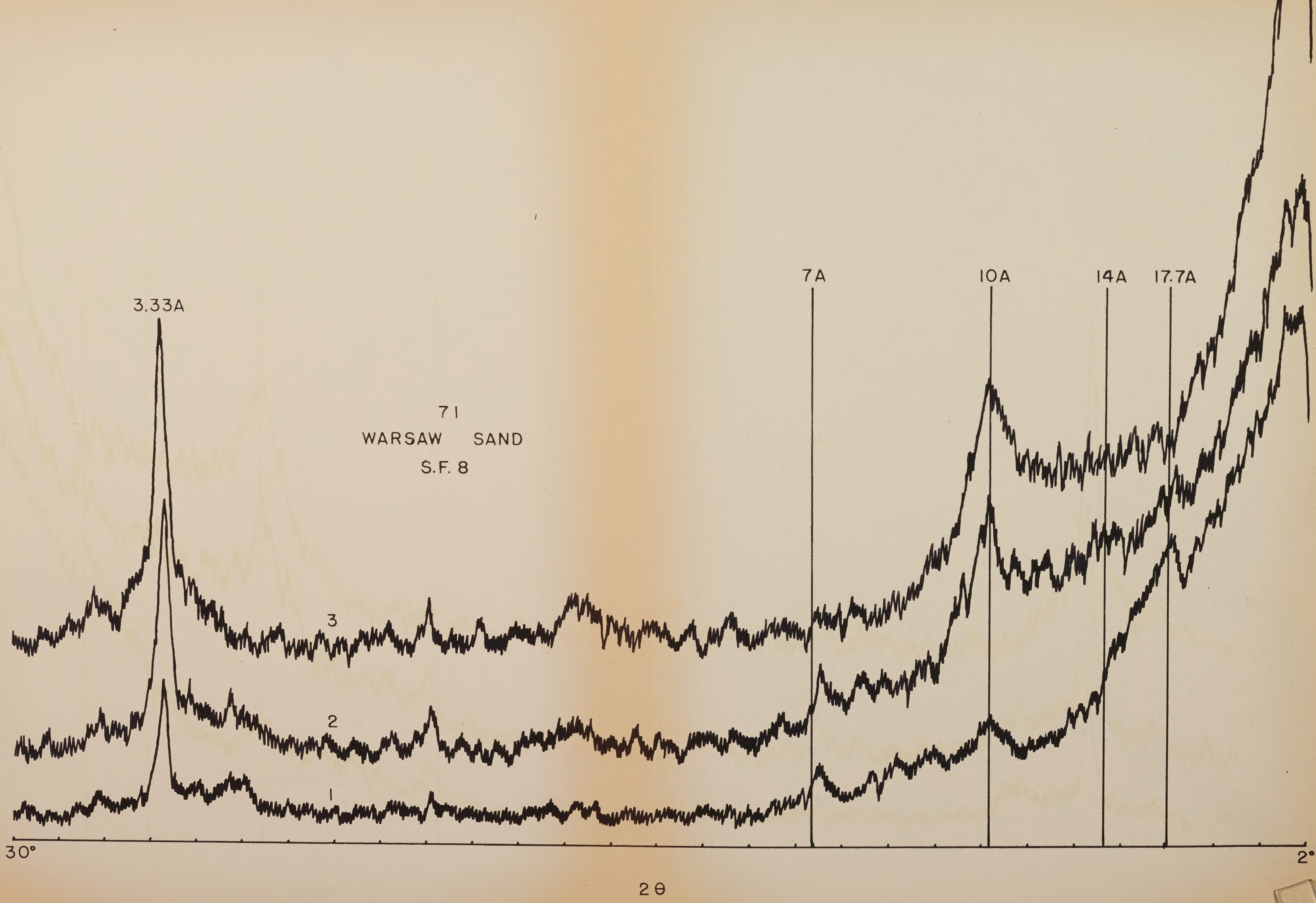


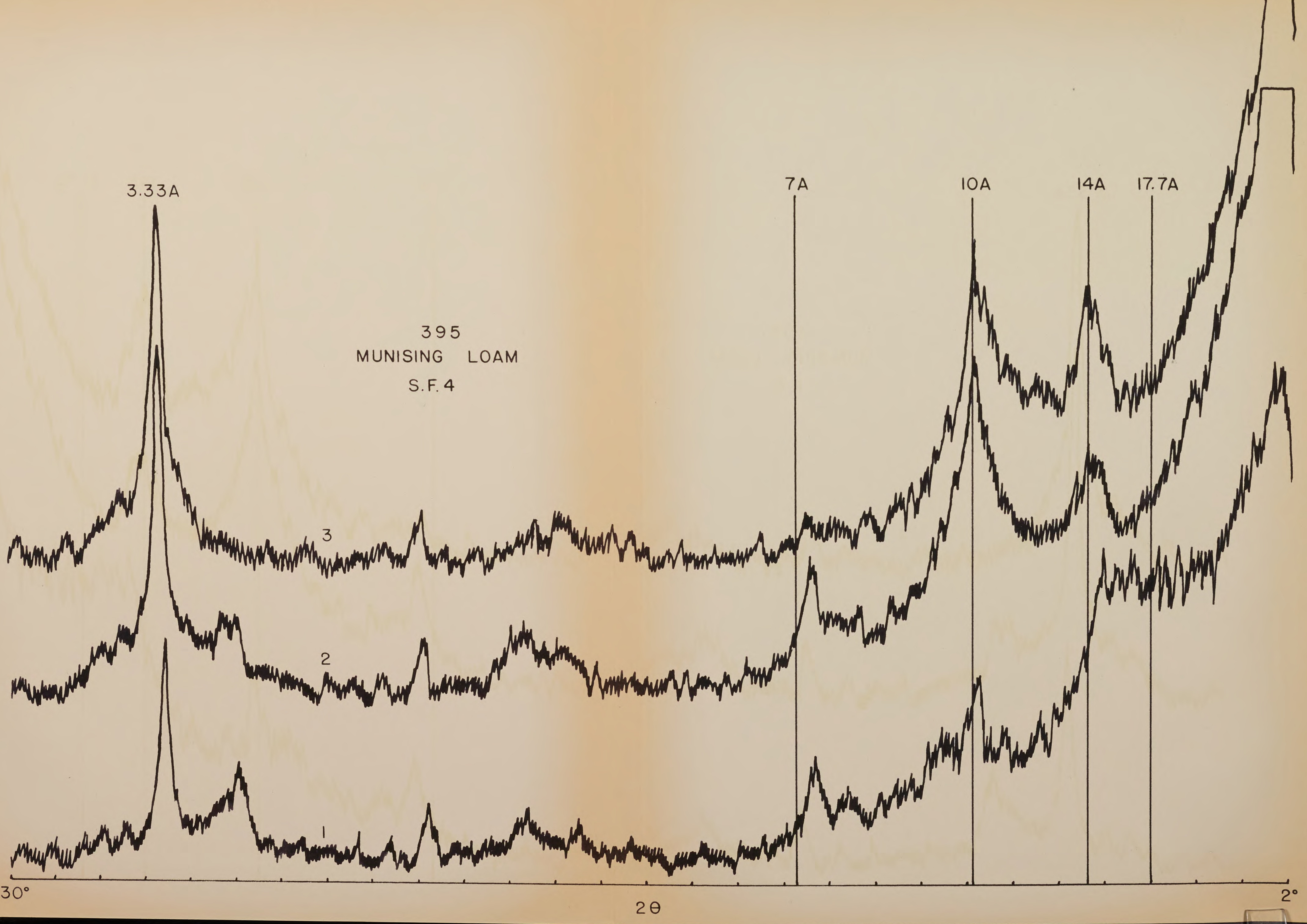
8
GANBY SAND
S.F. 4

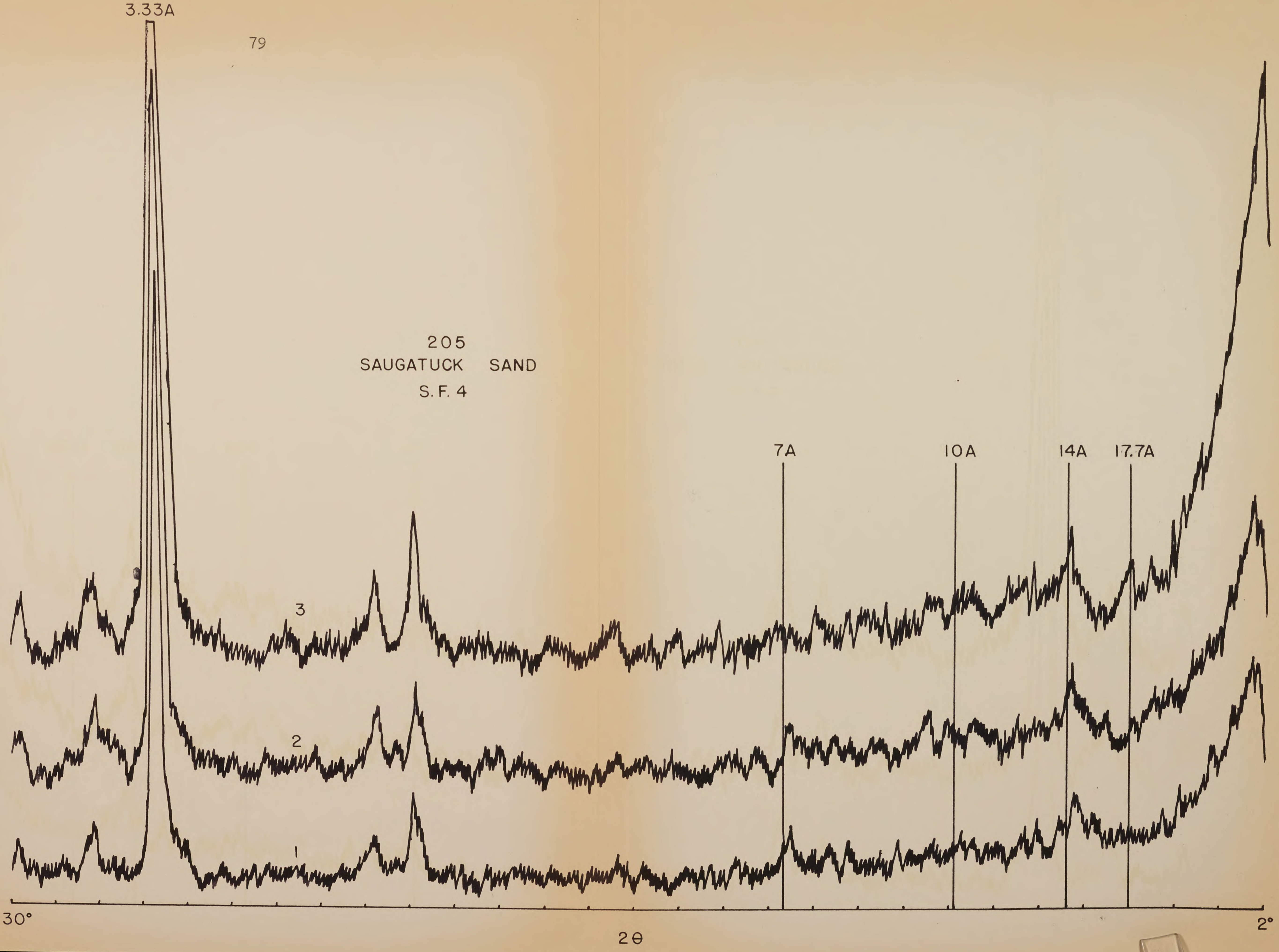

3.33A
63
SPINKS SAND
S.F. 4
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ

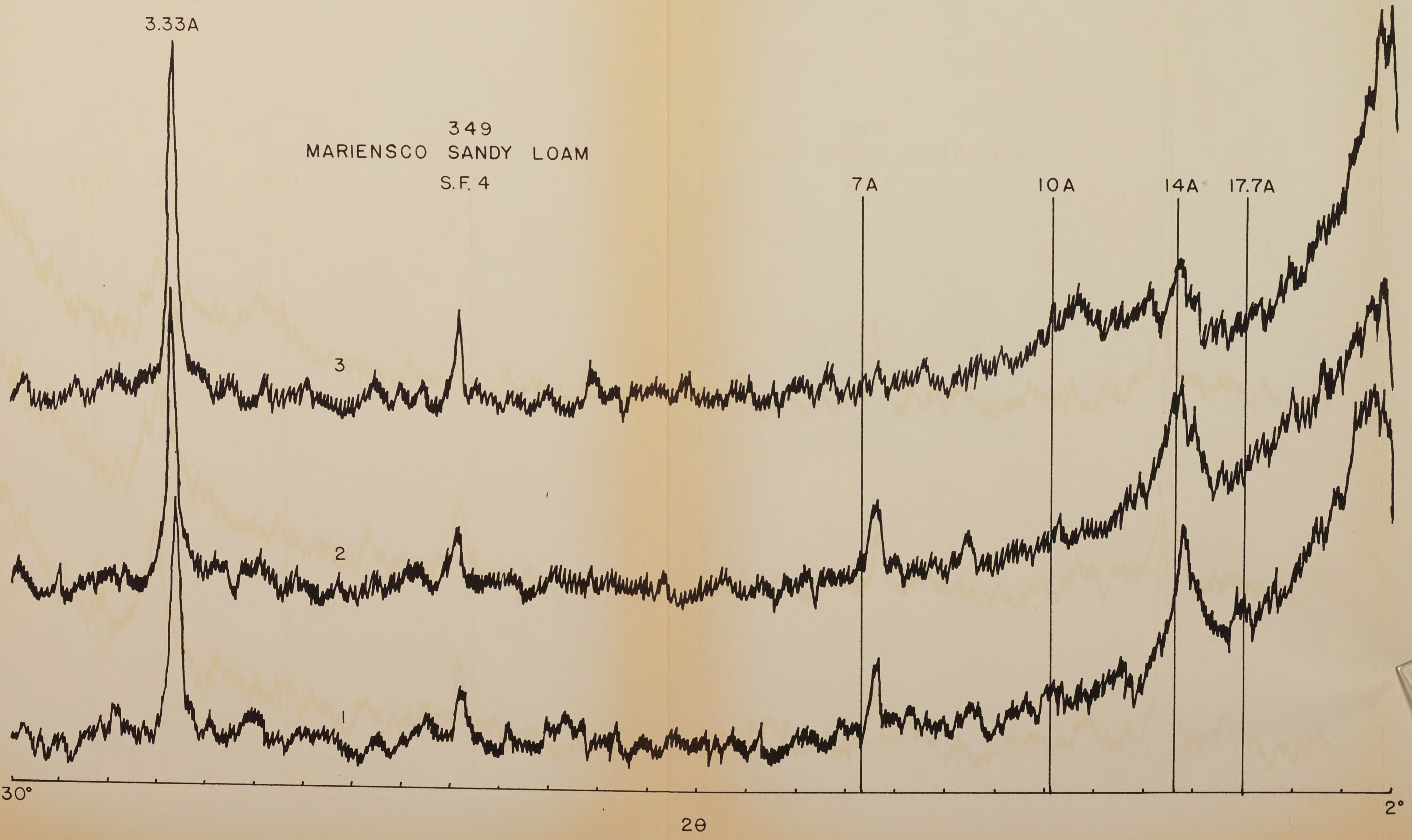

3.33A
52
FOX SAND
S.F. 4
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ


3.33A
55
WARSAW LOAM
S.F. 8
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ


3.33A
7A
10A
14A
17.7A
71
WARSAW SAND
S.F. 8
3
2
1
30°
2°
2θ


3.33A
7A
10A
14A
17.7A
395
MUNISING LOAM
S.F.4
3
2
1
30°
2θ
2°


3.33A
205
SAUGATUCK SAND
S. F. 4
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ


3.33A
349
MARIENSCO SANDY LOAM
S.F. 4
7A
10A
14A
17.7A
3
2
1
30°
2°
2θ