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HIGHWAY RESEARCH REPORT

MINERALOGICAL ANALYSIS BY X-RAY DIFFRACTION AND

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DEC 29 1972
BERKELEY M&R

October, 1972

STATE OF CALIFORNIA
BUSINESS AND TRANSPORTATION AGENCY
DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS

MATERIALS AND RESEARCH DEPARTMENT
RESEARCH REPORT
CA-HY-MR-2133-1-72-38

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TECHNICAL REPORT STANDARD TITLE PAGE

1. REPORT NO.	2. GOVERNMENT ACCESSION NO.	3. RECIPIENT'S CATALOG NO.
4. TITLE AND SUBTITLE MINERALOGICAL ANALYSIS BY X-RAY DIFFRACTION AND INFRARED SPECTROPHOTOMETRY		5. REPORT DATE October 1972 6. PERFORMING ORGANIZATION CODE Source Code 19203 W.O. 762504-642133
7. AUTHOR(S) Dr. J. L. Post J. Gamble		8. PERFORMING ORGANIZATION REPORT NO. CA-HY-MR 2133-1-72-38
9. PERFORMING ORGANIZATION NAME AND ADDRESS Materials & Research Department California Division of Highways Sacramento, California 95819		10. WORK UNIT NO. 11. CONTRACT OR GRANT NO.
12. SPONSORING AGENCY NAME AND ADDRESS California Division of Highways Sacramento, California 95807		13. TYPE OF REPORT & PERIOD COVERED Final 14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

16. ABSTRACT

Procedures for securing consistent mineral analyses using X-ray diffraction are described, calibration standards are presented, and aids are given for the determinations of feldspar content for mineral aggregates in different stages of weathering. A revised listing of mineral diffraction spacings is assembled for purposes of rapid mineral indentification. An introduction to the use of infrared spectrophotometry in mineralogical investigations is presented.

17. KEY WORDS	18. DISTRIBUTION STATEMENT
Aggregate analysis, calibration clay minerals, correlation, density, feldspar, infrared, mica, minerals, soil aggregate mixtures, weathering, x-ray diffraction.	Unlimited

19. SECURITY CLASSIF. (OF THIS REPORT)	20. SECURITY CLASSIF. (OF THIS PAGE)	21. NO. OF PAGES	22. PRICE
Unclassified	Unclassified	42	

HMRT – 1242 (ORIG. 9/72)

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DIVISION OF HIGHWAYS
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5900 FOLSOM BLVD., SACRAMENTO 95819

October 1972
Final Report
M&R No. 642133

Mr. R. J. Datel
State Highway Engineer

Dear Sir:

Submitted herewith is a research report titled:

MINERALOGICAL ANALYSIS

BY

X-RAY DIFFRACTION

AND

INFRARED SPECTROPHOTOMETRY

Under the direction of.....Raymond A. Forsyth
Supervised by .....M. L. McCauley
Laboratory work by .....Dr. J. L. Post
J. Gamble
Report prepared by .....Dr. J. L. Post
J. Gamble

Very truly yours,

JOHN L. BEATON
Materials and Research Engineer

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REFERENCES: Post, J. L., Department of Civil Engineering, Sacramento State College, Sacramento, California; Gamble, J.; Forsyth, R. A.; McCauley, M. L., "Mineralogical Analysis by X-ray Diffraction and Infrared Spectrophotometry," State of California, Department of Public Works, Division of Highways, Materials and Research Department, Research Report No. 642133, September 1972.

ABSTRACT: Procedures for securing consistent mineral analyses using X-ray diffraction are described, calibration standards are presented, and aids are given for the determination of feldspar content for mineral aggregates in different stages of weathering. A revised listing of mineral diffraction spacings is assembled for purposes of rapid mineral indentification. An introduction to the use of infrared spectrophotometry in mineralogical investigations is presented.

KEYWORDS: Aggregate analysis, calibration, clay minerals, correlation, density, feldspar, infrared, mica, minerals, soil aggregate mixtures, weathering, X-ray diffraction.

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INTRODUCTION

During the summer of 1968 a general procedure was developed for the x-ray identification of aggregate minerals and an approximate method was proposed for the quantitative determination of mineral content of soil and rock samples (X-Ray Diffraction Techniques, Research Report No. M&R 642992, October 1968). Improvements in analytic procedure and the development of auxiliary methods of mineral identification have brought about the need for a supplementary report.

Procedures are given for securing consistent mineral analyses using x-ray diffraction procedures, calibration standards are presented, and aids are given for the determination of feldspar content for mineral aggregates in different stages of weathering. A revised listing of mineral diffraction spacings is assembled for purposes of rapid mineral identification.

An introduction to the use of infrared spectrophotometry in mineralogical investigations is presented.

Summary

The different procedures used for the preparation of powder sample mounts are discussed with respect to consistency of data results which may be achieved. It has been observed that data secured using dense-packed powder samples give the most consistently reproducible reflection intensities. It is recommended that dense-packed powder samples be used for the quantitative determination of mineral content in rock and soil samples. Loose-packed powder samples should continue to be used for mineral identification.

The method of preparation of the powder sample may be varied in a number of ways to improve the consistency of data including grain-size separation of soil samples and finer grind size of the sample powder. The time, cost, and results of these changes in method of sample preparation suggest that the present preparation procedure is adequate. It is recommended that for aggregate minerals showing strong orientation effects, such as feldspars, a minimum of three scans of the calibration peaks be made which are comparable and that the mean value of the reflection intensities be used.

X-ray diffraction data results from any two diffractometers may be correlated by use of mineral standards, especially quartz. The correlation of data using the GE XRD-5 at Materials and Research Laboratory and the Picker XRD at Sacramento State University are given for both slow-speed scans and rapid scan

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rate. Because of the combination of scan rates and beam slits used with the two machines, the intensity calibration curves for feldspar minerals of different weathering stages may be used without alteration.

A complete description of weathering stage and mineral content has previously been prepared by the authors (The Role of Weathering Stages in the Quantitative Determination of Mineral Aggregations) for feldspar, amphibole, and mica minerals. The paper is presently in preparation for publication.

The diffraction intensities of certain minerals vary extremely with weathering and also reconstruction from metamorphism. These minerals, including micas and chlorites, often comprise the predominant portion of 3-phase mineral systems such as slates and phyllites. A method is given for the determination of mineral diffraction intensities and, hence, quantitative mineral determinations.

Diffraction pattern data have been assembled in a number of ways for general and specific uses, but there is no known listing available for the rapid identification of non-clay minerals in soil and rock. Such a listing has been prepared using data secured with different x-ray diffractometers by the authors during recent years.

An auxiliary method of mineral analysis is introduced, that is referred to as infrared spectrophotometry. As discussed, the method has been available for more than 60 years and is widely used in such disciplines as organic chemistry. The method is most useful in identifying different species of clay minerals, poorly crystalline material, and organic materials in soils. Direct quantitative determinations of mineral contents may be made in some cases. Because clays are very amenable to this method of analysis, small clay mineral contents in soils may be identified. An additional advantage to the i.r. method of analysis is that only a very small sample is required, less than 15 milligrams in most cases. A brief, but inclusive, reference list has been included for those interested in the topic.

Sample Preparation

The primary purpose of the investigation of sample preparation methods is to secure consistent data results in the determination of aggregate mineral content of rock and soil samples.

When a powder sample is placed in a sample holder the minerals with perfect cleavage tend to orient in preference to other minerals present in the powder. Along with this orientation some sorting with respect to particle specific gravity and

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grain-size occur. These effects become less pronounced as the mean particle size becomes smaller.

Different packing densities have been tried to develop more consistency in particle orientation, however, only the very loose or very dense powder arrangements can ordinarily be prepared in a consistent manner. The most loosely packed powder that will remain in the G.E. vertical sample holder will show a certain amount of particle orientation. At the same time, the intensity of certain diffraction peaks used for quantitative determinations will be considerably diminished by the more random particle orientation. Thus, the loose-pack powder sample is commonly used for mineral identification but not for quantitative determinations. The moderately dense powder sample shows more particle orientation but is even more difficult to prepare in a consistent manner.

The very densely packed powder sample gives quite consistent reflection intensity results and, thus, has been used to secure data for this report. The data results are readily reproducible with some practice in packing procedures. The reflection intensities secured were found to be not only the result of powder sample density but also a result of the final sample surface preparation. The position of the sample surface in the sample rack then has little effect so long as the x-ray beam strikes entirely on the sample surface.

The orientation of mineral particles in the powder sample may ordinarily vary by as much as a factor of five, however, even platy particles, such as mica, usually show only about a 3-fold variation in degree of orientation. It has been observed that dense packed micas show basal reflection intensities about twice those of loose-packed micas. Micas prepared on oriented slides show basal reflection intensities about 1.5 times those prepared as dense-packed powders.

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CONSISTENT MINERAL ANALYSES

Greater consistency of results may be obtained for measured diffraction peak intensities in a number of ways. However, most of the methods for improvement of consistency are very time-consuming. Two common methods used include physical separation of the sample according to grain-size, and grinding to secure finer particle size.

The clay-size portion of soil samples is often separated and analyzed separately in agricultural studies and also in studies involving sedimentary petrology. One disadvantage is the sample preparation time required. If the sample is to be investigated without pretreatment it is necessary to choose standard grind sizes.

Diffraction peak intensities are usually greatest for particle sizes between about 5μm and 50μm but very widely from one mineral to another. Many clay minerals, and other minerals, cannot be reduced to this size range without some destruction of the crystal structure of the minerals. Grinding media, such as alcohol have been used but are not always satisfactory. Some data are given by Klug and Alexander (1954)[1] and by Tatlock (1966)[2] for diffraction intensities of different minerals at different particle sizes.

Presently, all material is ground to pass a No. 200 sieve because this particle-size powder can be loosely packed in a vertical sample holder without falling out yet the grinding effort is kept to a minimum for the preparation of a uniform powder sample.

More consistent results can be obtained by grinding the material to pass a No. 325 sieve. A more uniform mix is obtained and the effects of preferred orientation are markedly reduced. However, there are certain disadvantages to the use of such finer particle sizes for the investigation of aggregate materials, such as soils.

These disadvantages include:

(a) Destruction of some clay mineral structures.

---

[1] Klug, H. P., and L. E. Alexander (1954) X-Ray Diffraction Procedures, J. Wiley and Sons, N.Y., p. 291-297.

[2] Tatlock, D. B. (1966) Rapid Model Analysis of Some Felsic Rocks from Calibrated X-Ray Diffraction Patterns: Geol. Survey Bull. 1209, Wash., D. C.

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(b) Increase of some diffraction peak intensities - decrease of others.

(c) Considerably more time needed for preparation of sample.

(d) Samples containing adsorbed moisture plug sieve screen.

(e) Reduced diffraction intensities for larger books of micas and chlorites while the smaller books are less affected.

(f) Some diffraction line broadening - may obscure other peaks.

(g) Fibrous material very hard to grind and sieve.

(h) Platy materials given curved morphology which makes uniform oriented slides very difficult to prepare.

A consistent quantitative determination of feldspar minerals is most difficult to achieve. Rather than using a finer grind size it is recommended that several slow-speed scans be made across the 002 and 040 reflections of the feldspar series, 27.3° - 28.3°2θ, using a different prepared sample for each scan, until three relatively consistent results are obtained. It has been observed that the more weathered the feldspar the more readily consistent results may be obtained.

Calibration Standards and Machine Correlations

The paper prepared by Post and Gamble concerning the direct quantitative analysis of aggregate minerals (The Role of Weathering Stages in the Quantative Determination of Mineral Aggregates), presented at the A.I.P.E.A. international conference in Madrid, Spain, during the last week of June, 1972, contains a method for determining the weathering stage of feldspar minerals and, hence, calibration curves for the determination of feldspar content in mineral aggregations. Because of the lack of consistency of data results a number of the feldspar samples were further investigated, using both the G.E. XRD-5 unit at the Materials and Research Department and the Picker XRD at Sacramento State University.

To make existing data available for comparison and use, it was necessary to compare the diffraction intensities of the two different diffractometers while the further work with feldspars was being done. The data results, Table 1, show that the two machines give nearly identical data for the feldspar 002 and

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TABLE 1

X-RAY DIFFRACTION INTENSITIES - COMPARISON
FOR FELDSPARS (2000 CPS, TC = 1)

Sample	Weathering Stage	G.E. XRD-5		Picker XRD	
		Trials	I Avg.	Trials	I Avg.
WY. Perthite (Dental Grade)	B	7	352		345
Tucson Fs Soil (Ariz)	E	4	111		110
R.S. Dune Sand (N.M.)	D	4	183		175
Kern Cyn. Fs (Cal.)	C	3	259	4	242
Ladron Mtns. Fs (Sill)(N.M.)	C	2	260		
Whitney Portal Fs (Cal.)	C	2	293		
Orthoclase XLS (C.S.U.S.)	B	3	373		
Weathered XLS (Cal., Dist. 07)	E	7	129	2	168
Folsom Lake Fs (Cal.)	D	1	265	4	220
Sta. Catalina Mtns. (Ariz.)	C	5	249	3	268
Bear Cyn. Fs (Ariz.)	C	5	209	2	234
Pinacate Mtns. (Mexico)	D	3	190		
Georgetown Fs (Cal.)	D	4	173		155

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040 diffraction peak intensities when the data are related at 2000 CPS, TC=1. Thus, Figure 1 from the previously prepared paper may be used without alteration in conjunction with the G.E. XRD-5 data output. The G.E. XRD-5 scan rate is 0.4°2θ/min. with slits: 1°-0.1°, and the Picker XRD scan rate is 0.25°2θ/min. with slits: 1°-1°-0.002 in.

An additional correlation was made for high-speed scans for the two units. The data results are given in Table 2. The G.E. unit gives diffraction peaks of 1.54 times the intensity of the same peaks secured with the Picker unit.

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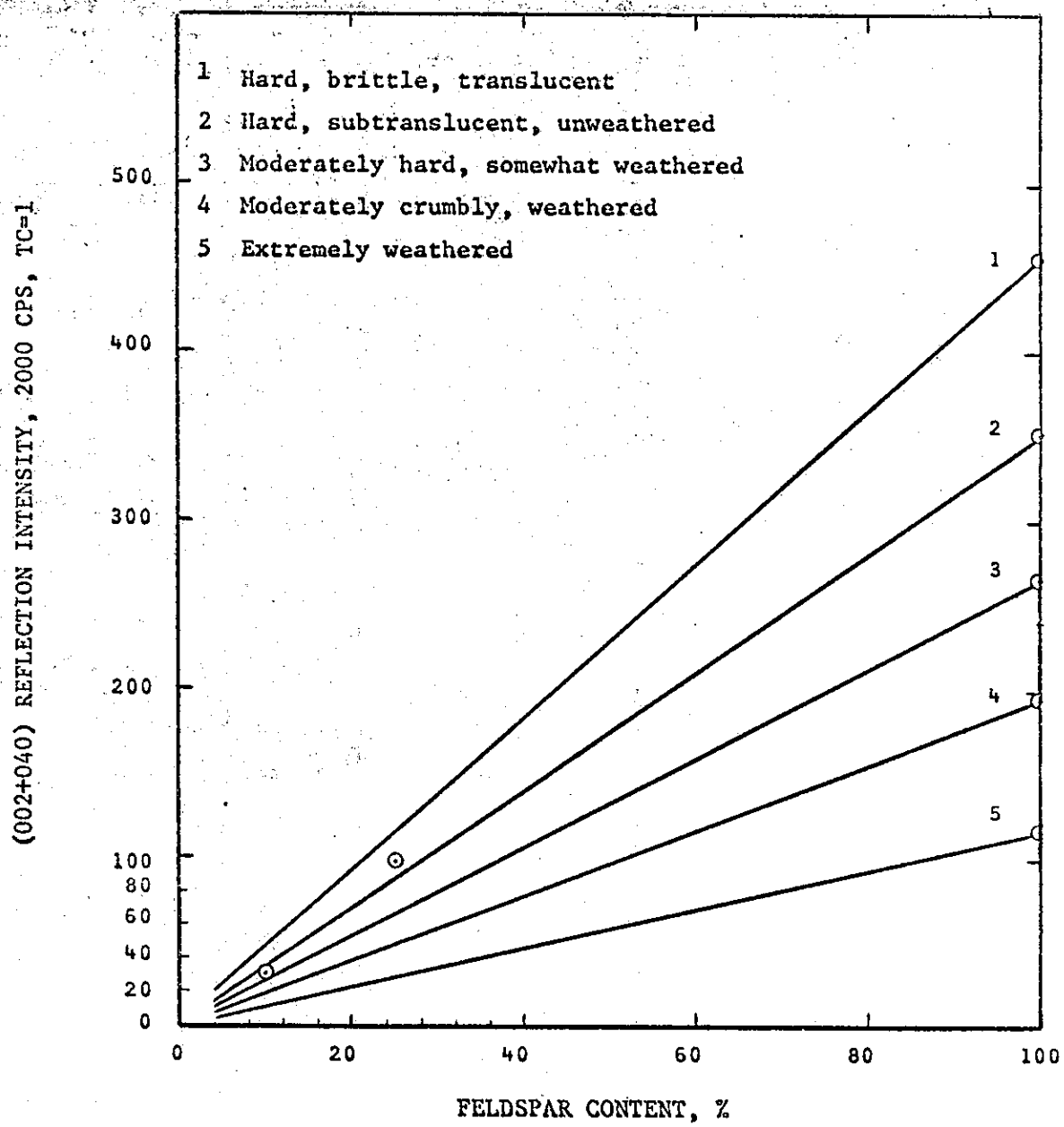

Figure 1 Calibration curves for the determination of feldspar content in a mineral aggregation, with weathering stages indicated by number. The two points on curve 2 show results using artificial mixtures.

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TABLE 2

DATA CORRELATION - X-RAY DIFFRACTION

HIGH-SPEED SCAN

		G.E. XRD-5 Avg. Pk. Ht. (4 scans)	Picker XRD Avg. Pk. Ht. (3 scans)	Ratio
Serpentine	5.0%	13.0	8.5	1.53
Muscovite	5.0	57.0	36.5	1.57
Hornblende	10.0	48.0	32.0	1.50

PERMAQUARTZ

	Avg. Pk. Ht. (3 Pks)	Avg. Pk. Ht. (3 Pks)	Ratio
$\frac{(110)+(102)+(211)}{3}$	69.0	44.8	1.54

G.E. XRD-5

Count Rate: 500 CPS TC = 2

Scan: 2°2θ/min.

Slits: 1° - 0.2°

35KV/32Ma Cu/Ni

ΔE: 2.5 - 32.5

PICKER XRD

Count Rate: 400 CPS TC = 3

Scan: 1°2θ/min.

Slits: 1° - 1° - 0.002"

35KV/16Ma Cu/Ni

Pulse Ht. Sel.: 20.0 - 80.0

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DETERMINATION OF MINERAL COMPOSITION OF 3-PHASE SYSTEMS

Many slates and shales consist essentially of 3-phase systems with only traces of other minerals present. Usually, the primary minerals are quartz, mica, and chlorite. Sometimes the chlorite has altered to an ordered mixed-layer system such as the "swelling chlorites" of the Calavaras formation.

The quartz content of the formations may be determined directly by x-ray diffraction procedures, and simultaneous linear equations may be written to determine the remaining mineral content. It is only necessary to choose two like samples from the same formation which have significantly varied mineral content ratios.

EXAMPLE

Two samples of green slate from Miller Cyn., Ariz.

Dense-Pack

Sample 1	Sample 2
38.1% quartz	28.5% quartz
10Å pk = 83.0 units ht.	10Å pk = 54.0 units ht.
7.1Å = 12.0	7.1Å = 37.0
5Å = 24.0	5Å = 16.0
4.7Å = 5.5	4.7Å = 10.5

Let X = 10Å pk ht. at 100% content - mica
Let Y = 7.1Å pk ht. at 100% content - chlorite

Then $83/X + 12/Y = 0.619$, and $54/X + 37/Y = 0.715$

Calculations:

(a) $83/X + 12/Y = 0.619$ (c) $54/83\ (83/X + 12/Y) = (0.619)\ 54/83$

(b) $54/X + 37/Y = 0.715$ (d) $-(54/X + 7.8/Y) = -(0.403)$

Add eqns b and d, then solve for Y. Y = 93.6 units ht.
Using eqn b, with Y known, solve for X. X = 168.5 units ht.

The mica/chlorite peak intensity ratio = 1.80 for equal amounts of each mineral.

Sample 1	Sample 2
83/X = 49% mica	54/X = 32% mica
12/Y = 13% chlorite	37/Y = 40% chlorite

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d(Å) SPACINGS FOR NON-CLAY AGGREGATE MINERALS

(Revised Listing - Aug. 1972)

A number of tabulated listings are available for use in the identification of minerals and chemical compounds with data derived from the x-ray diffraction pattern of an unknown material. The most well known information source is the ASTM Powder Diffraction File. Listings comparable to this file have also been assembled in other countries.

The following additional identification data tables are available:

-----(1951) Reference Clay Minerals, American Petroleum Institute Research Project 49, Columbia Univ., N.Y.

Brindley, G. W. (ed.) (1951) X-Ray Identification and Crystal Structures of Clay Minerals, Mineralogical Society, London.

Warshaw, C. M., and R. Roy (1961) Classification and a Scheme for the Identification of Layer Silicates: Geol. Soc. of Amer. Bull. 72, p. 1455-1492.

Brown, G. (ed.) (1961) The X-Ray Identification and Crystal Structures of Clay Minerals, 2nd Edn., Mineralogical Society, London.

Berry, I. G., and R. M. Thompson (1962) X-Ray Powder Data for Ore Minerals: The Peacock Atlas: Geol. Soc. of Amer., Memoir 85, N.Y.

Borg, I. Y., and D. K. Smith (1969) Calculated X-Ray Powder Patterns for Silicate Minerals: Geol. Soc. of Amer., Memoir 122, Boulder, Colo.

A preliminary listing of diffraction spacings for aggregate minerals was prepared during 1968 at the Materials and Research Department and presented in a Report entitled X-Ray Diffraction Techniques, Research Report No. M&R 642992. Since that time, a large amount of data has become available for correlation.

The data given in the preceding list of six references, and the data given in the ASTM Powder Diffraction File, are for randomly oriented powder samples usually secured with a Straumanis powder camera. The data secured at the Materials and Research Department are derived by scanning a partially oriented powder sample using an x-ray diffractometer. Because of the different procedure used, the relative intensities of the diffraction peaks are varied.

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No complete listing of diffraction patterns for soil and aggregate minerals, using an X-ray diffractometer, has been assembled comparable to the listing for ore minerals by Berry and Thompson.

The available listings of diffraction patterns for clay minerals were considered satisfactory. Thus, only a listing of non-clay aggregate minerals was prepared. A listing of hydrous layered silicates, some of which minerals are also considered clay minerals, was included.

A number of the more common aggregate minerals, such as feldspars, may have more than one hundred diffraction peaks and, thus, the scan range (degrees 2θ) and reflection intensities selected for use must be restricted for practical purposes. The revised listing gives all peaks observed from 3° to 21° 2θ but only the moderate to strong peaks observed from 21° to 63° 2θ.

The relative intensities of the reflections listed, being somewhat variable are simplified as follows:

vs	(very strong)	>>100 chart units	w	(weak)	≈10-30
s	(strong)	≈60-100	f	(faint)	<10
m	(moderate)	≈30-60			

These intensities are derived from rapid scan of the powder sample using a Picker x-ray diffractometer, which gives data comparable to the G.E. XRD-5.

The revised d(Å) spacings for non-clay minerals is given in the Appendix.

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AUXILIARY METHODS OF ANALYSIS

Infrared Spectrophotometry

Infrared spectra have been used for the identification of mineral samples since about 1900 (Lyon, 1962) and since that time infrared analysis of solids, liquids, and gases have become a highly developed procedure.

The methods of sample preparation commonly used are given by Lyon in Chapter VI of the text edited by Rich and Kunze (1964). These methods include the use of alkali halide pellets, deposited films, oriented clay papers and nujol mulls. The preferred method is the use of alkali halide pellets, usually consisting of spectrographic grade KBr.

Two particular advantages to the use of infrared techniques are the short time required and the small amount of sample required, about 10 mg. When quantitative analytic results are not required the method of sample preparation may be simplified so that the time of sample preparation and scanning does not exceed about 15 minutes if the material has been previously ground to size. Ordinarily, the sample which has been prepared for x-ray diffraction, material passing the No. 200 sieve, may be used.

The apparatus required for preparing samples for mineral identification includes: a hydraulic press, two stainless steel press blocks, a dentist's amalgamator (Wig-L-Bug), blotter paper, and a precision balance. The hydraulic press should exceed a unit pressure of 75,000 psi. The blotter paper is cut into 2 in. by 3 in. rectangles with a centered 5/8 in. hole.

To prepare the sample for scanning place about one gram of KBr in the Wig-L-Bug shuttle capsule, followed by about 1.5 mg of sample (about 0.15% of KBr weight). Pulverizer bearings are added and the capsule cover is put on. The shuttle mixer is run for one minute then the contents are removed. The shaped blotter is placed on one press block, the mixed powder is poured on the center hole in the blotter, and the other press block is placed on top. The assemblage is pressed for one minute at pressures in excess of 75,000 psi, at which time the KBr fuses into a transparent disc.

The infrared spectrophotometer is prepared and the pressed blotter paper is put into the specimen holder so that one beam of light shines through the KBr pellet. The pen is positioned on the chart with the aid of the reference attenuator and an absorption spectrum is recorded. (Procedure suggested by Dr. J. Digiorgio, Chemistry Dept., C.S.U.S.)

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Information concerning infrared spectra of minerals is scattered throughout the literature. A brief comprehensive description of infrared analytic procedures and information sources is given by Lyon in the text edited by Rich and Kunze (1964). A bibliography of mineral infrared spectra has also been prepared by Lyon (1962).

Infrared spectra, mainly for non-clay minerals and rock samples, have been assembled by Lyon (1963) and spectra for silicates have been investigated by Saksena (1961). Farmer and Russell have investigated the infrared spectra of layer silicates and Stubican and Roy have investigated the effects of isomorphous substitution on the infrared spectra of layer lattice silicates. The results of many additional investigations have been added to the literature during the last ten years, especially concerning the interaction of organic complexes, such as insecticides, with clay minerals. Copies of infrared spectra for most chemical compounds may be found in the Sadtler Index which is prepared by the Coblentz Society.

The amount of sample required is dependent on the composition of the sample itself. A very small amount of material such as clean kaolinite is required whereas a larger amount of material such as biotite is needed. Such minerals as quartz and feldspar are much less responsive to light transmission and a large amount of sample is required. Sample requirements may vary by as much as a factor of five for different minerals, the hydrous layered silicates generally being most responsive to infrared absorption spectra.

Because of the varied response to infrared spectra evidenced by the minerals which ordinarily comprise soil the clay mineral spectrum will tend to obscure the response of other minerals, except for the carbonates. Generally, the infrared identification technique is most useful in identifying, or verifying, the types of micas and clay minerals present. Identification of other minerals such as feldspars is also possible, as shown in Table 3, however, feldspars are much more amenable to identification by x-ray diffraction procedures.

A typical clay mineral spectrum is shown in Figures 2a and 2b, using a Perkin Elmer 337 grating infrared spectrophotometer. The high range, 4000 Cm^{-1} to 1333 Cm^{-1}, of the spectrum mainly includes the 0-H stretching bonds in the clay crystal structure, but also includes the adsorbed water on the clay particles. The kaolinite sample shown has a unique 0-H absorbance pattern. The low frequency range, 1200 Cm^{-1} to 400 Cm^{-1}, includes the absorbance peaks due to the remaining atomic bonds in the crystal structure, such as the Si-0 stretching absorption

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TABLE 3

INFRARED SPECTRA FOR PLAGIOCLASE FELDSPARS
FREQUENCY (Cm^{-1})

ALBITE	1148m	1094w	1033s—1001s	--	784m	761m	742w	723w	--	647w	607w	586m	531m	473w	458w
OLIGOCLASE	1155m	1090m	1028s— 995s	--	780w	753m—740m		724w	--	634m	--	585m	531m	--	454w
ANDESINE	1152m	1089w	1025m—1002s	930w	774f	756w	745w	718w	--	624w	--	581m	534m	--	460w
Ca-ANDESINE	1135m	1087m	-- 1000s	930f	--	--	736m	--	--	623m	--	576m	535m	--	452w
LABRADORITE	1143m	1094m	-- 992s	945s	774f	--	744m	--	--	620m	--	577m	537m	--	458w
Ca-LABRADORITE	1132m	1084s	-- 980s	940m	--	774f	--	731m	665f	615m	--	573m	534m	--	450f
BYTOWNITE	1135m	1085m	1009s— 985s	937s	--	--	750m	728w	659w	618m	--	565m	534m	--	454w

s - strong, m - moderate, w - weak, f - faint

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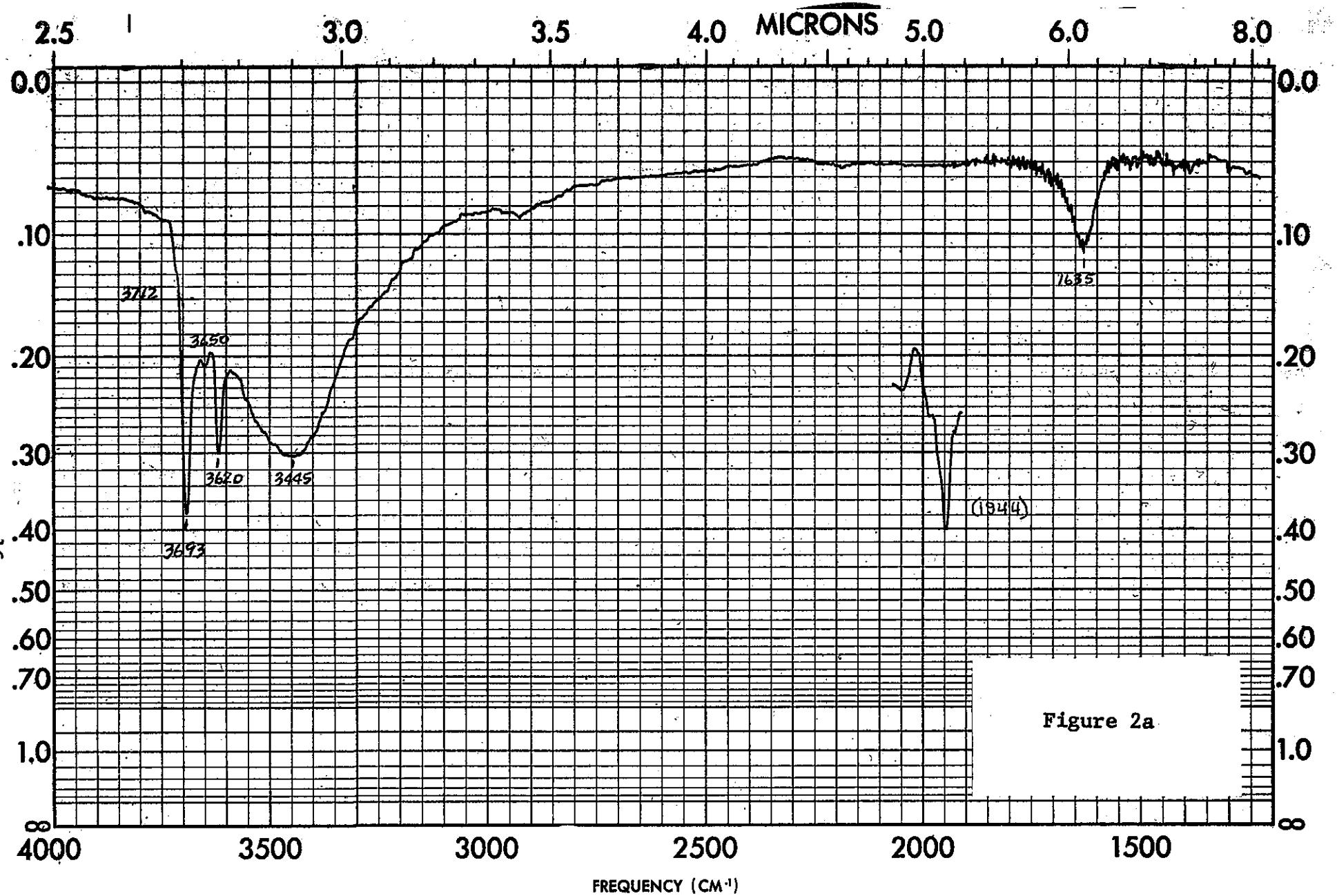

Figure 2a

SAMPLE KAOLINITE	CURVE NO.	SCAN SPEED F	OPERATOR J. L. POST
	CONC. SMALL AMT. OF MAT'L.	SLIT N	DATE 19 JUNE 1972
ORIGIN GEORGIA KAOLIN CO.	CELL PATH	REMARKS	

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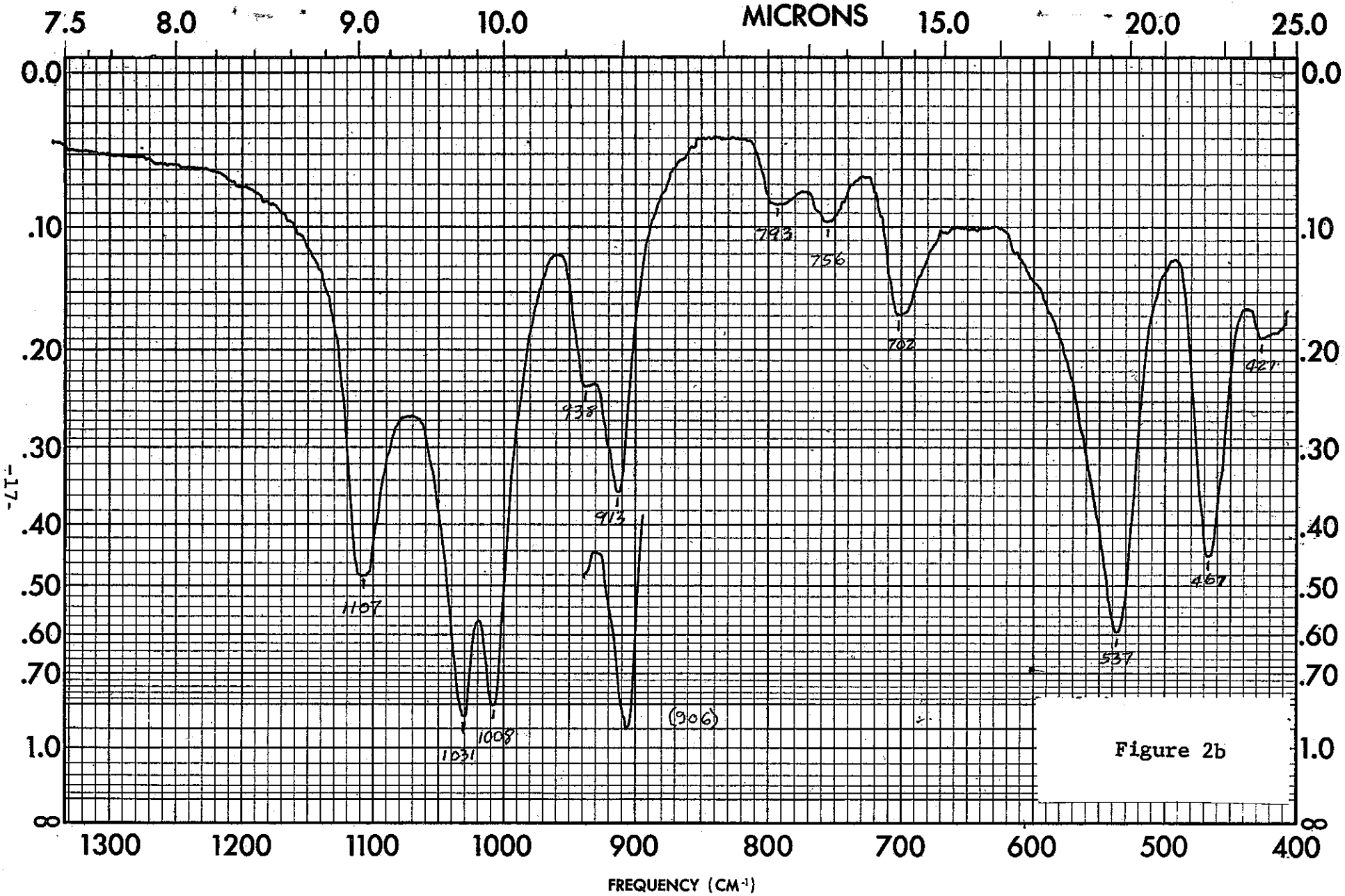

Figure 2b

SAMPLE KAOLINITE B-AXIS DISOR. SANDIA LAB. <0.3μm	CURVE NO.	SCAN SPEED F	OPERATOR J.L. POST
	CONC.	SLIT N	DATE 19 JUNE 1972
ORIGIN GEORGIA KAOLIN CO.	CELL PATH	REMARKS SMALL AMOUNT OF MATERIAL	
SOLVENT KBr PELLET	REFERENCE AIR		

PART NO. 337-1208

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peak at 1004 Cm^{-1}. The peaks which occur at 536 Cm^{-1} and 465 Cm^{-1} are of particular importance in the identification of kaolinite as will be shown.

Infrared absorption data from 600 Cm^{-1} to 400 Cm^{-1} are given in Table 4 for some of the more common clays, serpentine, talc, and pyrophyllite. Not only can the clays be differentiated by this method but solid-state series such as chlorite types can be differentiated as well. Tentative bonding assignments are included for the various absorption frequencies.

Certain parts of the clay mineral infrared spectra are particularly useful for identifying clay types as are shown by the following examples. Illite and kaolinite may be differentiated, as shown in Figure 3, by both absorption peak spacing and relative intensity of peaks. In this case the presence of some chlorite contributes to the illite peak at 460 Cm^{-1} frequency. Vermiculite and montmorillonite may be differentiated, as shown in Figure 4, by the absorption peak spacing and by the absence of a strong $Si\text{-}0\text{-}Al^{VI}$ absorption peak from the vermiculite. Montmorillonite may occur as at least two types of minerals in the smectite suite of clays as shown in Figure 5.

Some montmorillonite minerals, such as examples A and D show only a strong symmetric Si-0 stretching bond at about 1030 Cm^{-1} to 1040 Cm^{-1} whereas others also show a strong doubly degenerated Si-0 stretching bond at about 1095 Cm^{-1}. This is believed to be due to interference from ions emplaced in the mineral structure by isomorphous substitution (Saksena, 1961). The absorption peak at a frequency of about 910 Cm^{-1} as assigned to the 0-H-Al bond in the crystal structure.

Dioctahedral and trioctahedral micas may be readily differentiated by their infrared absorption spectra as shown in Figure 6. Again, the $Si\text{-}0\text{-}Al^{VI}$ absorption peak is absent in the biotite spectra. Most illite spectra closely resemble the muscovite spectra in this frequency range as shown in Figure 2. Other mineral types may be differentiated in the same manner as shown in Figure 7. Antigorite shows a different crystal bonding sequence than chrysotile (Stubican and Roy, 1961). It is sometimes possible to estimate the relative amounts of each present in a mixture of serpentine.

The infrared frequency range 3800 Cm^{-1} to 3100 Cm^{-1} showing the 0-H stretching bonds of hydrous minerals is very useful for differentiating certain clay minerals. Some typical spectra are shown in Figure 8. Other clay minerals, such as degraded biotite may be identified by the lack of any sharp 0-H absorption peaks.

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TABLE 4

ASSIGNMENT (TENTATIVE) OF INFRARED ABSORPTION PEAKS FOR HYDROUS LAYERED SILICATES. (FREQUENCY IN Cm^{-1})

	(MgO)?	Si-O-Al^{VI}	Si-O-Fe^{+3}	Si-O	Si-O-Mg^{VI}	Si-O
Kaolinite		533s		463m		425w
Meta-Halloysite		537s		464m		425w
Illite		518m		463s	432w (Chlorite)	
Montmorillonite		513-521m		457m		
Nontronite	580f		486m			418m
Muscovite		529m		470m		
Phlogopite		510f	490f		457s	
Biotite	558f	520f	486f		448s	
Ripidolite	546m		495f	452m	424s	
Sheridanite	545-552w			456w	434s	
Clinochlore	550f	530f		450w	431s	
Penninite	555f	520m		452w	436s	
Antigorite	563m		490f		434s	
Chrysotile	555f			470w	425s	
Talc	532m			460w	448s	425w
Pyrophyllite	573w	535m		477m		412w
Vermiculite		520f			435s	

NOTE: 400-900 Cm^{-1} - mainly Si-O bending, some metal-O stretching.

BONDING ASSIGNMENTS FROM:
Stubican, J., and R. Roy "Isomorphous Substitution and Infra-red Spectra of the Layer Lattice Silicates," Amer. Min., 46, 1961, p 32-51.

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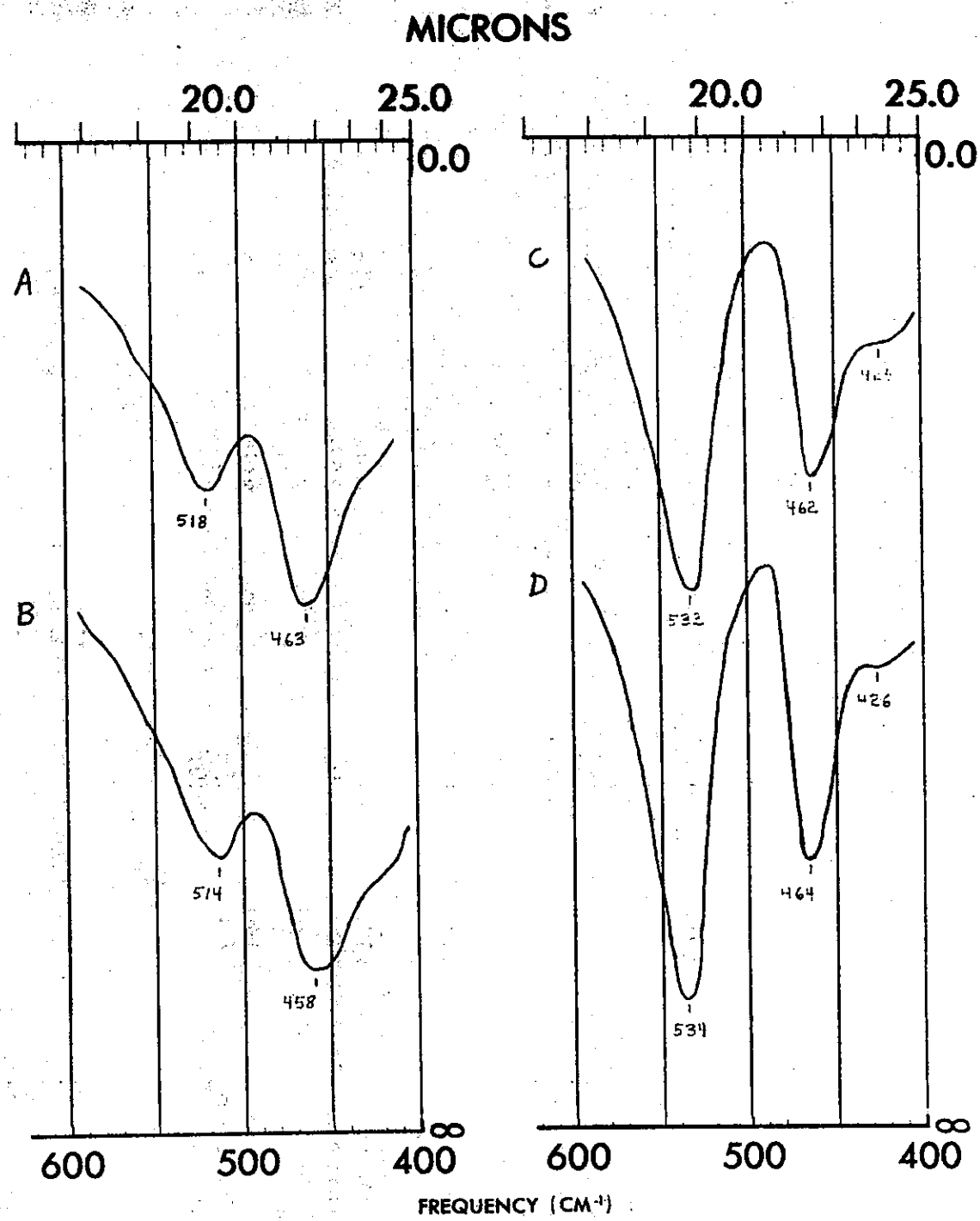

Fig. 3 Infrared spectra for illite and kaolinite in the frequency range 600 to 400 cm^{-1}. Curve A - illite, Kl 10.50, Verdal, Norway; Curve B - illite, Kl 11.20, Verdal, Norway; Curve C - kaolinite, Edgar, Fla.; Curve D - kaolinite, API No. 9, N.M.

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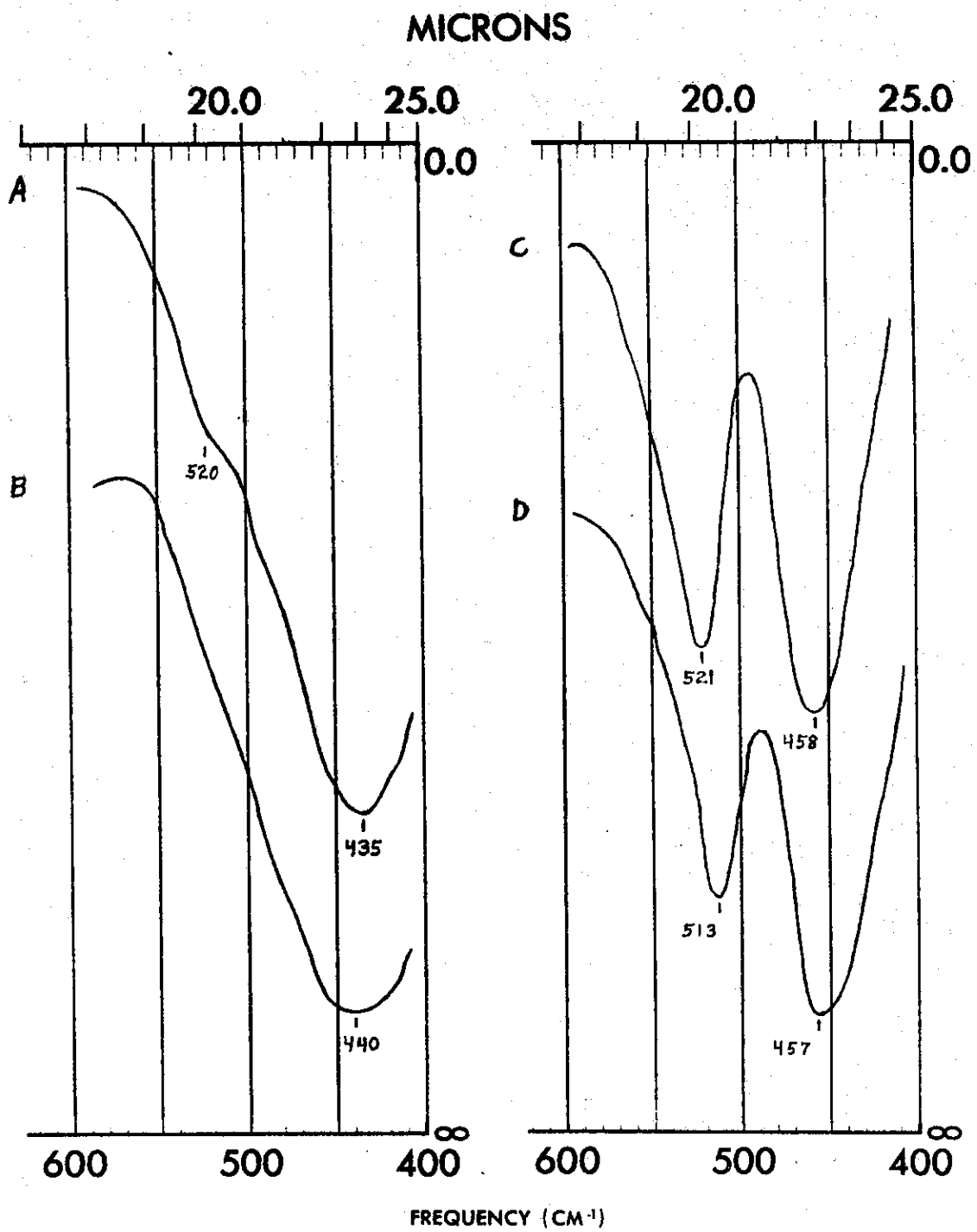

Fig. 4 Infrared spectra for vermiculite and montmorillonite in the frequency range 600 to 400 cm^{-1}. Curve A - Ca — vermiculite, Amer. Cyn., Calif.; Curve B - vermiculite, Gold Bluff, Nev.; Curve C - Na — montmorillonite, Colony, Wy.; Curve D - montmorillonite, Grant Co., N.M.

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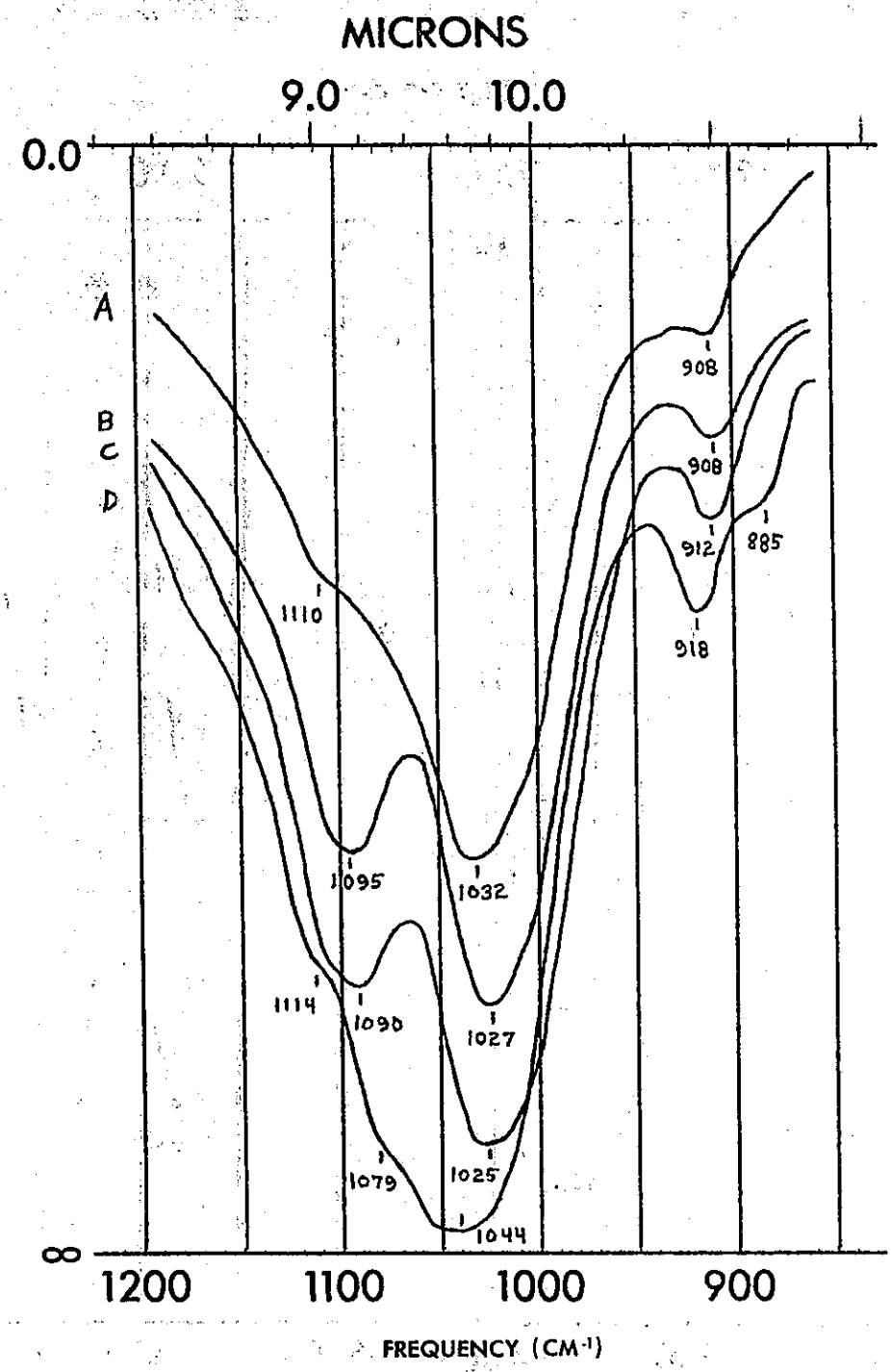

Fig. 5 Infrared spectra in the frequency range 1200 Cm^{-1} to 850 Cm^{-1} for montmorillonite samples. Curve A - Pena Blanca, Ariz.; Curve B - API 30a, Santa Rita, N.M.; Curve C - Ray Pit, Ariz.; Curve D - Bed A, Colony, Wy.

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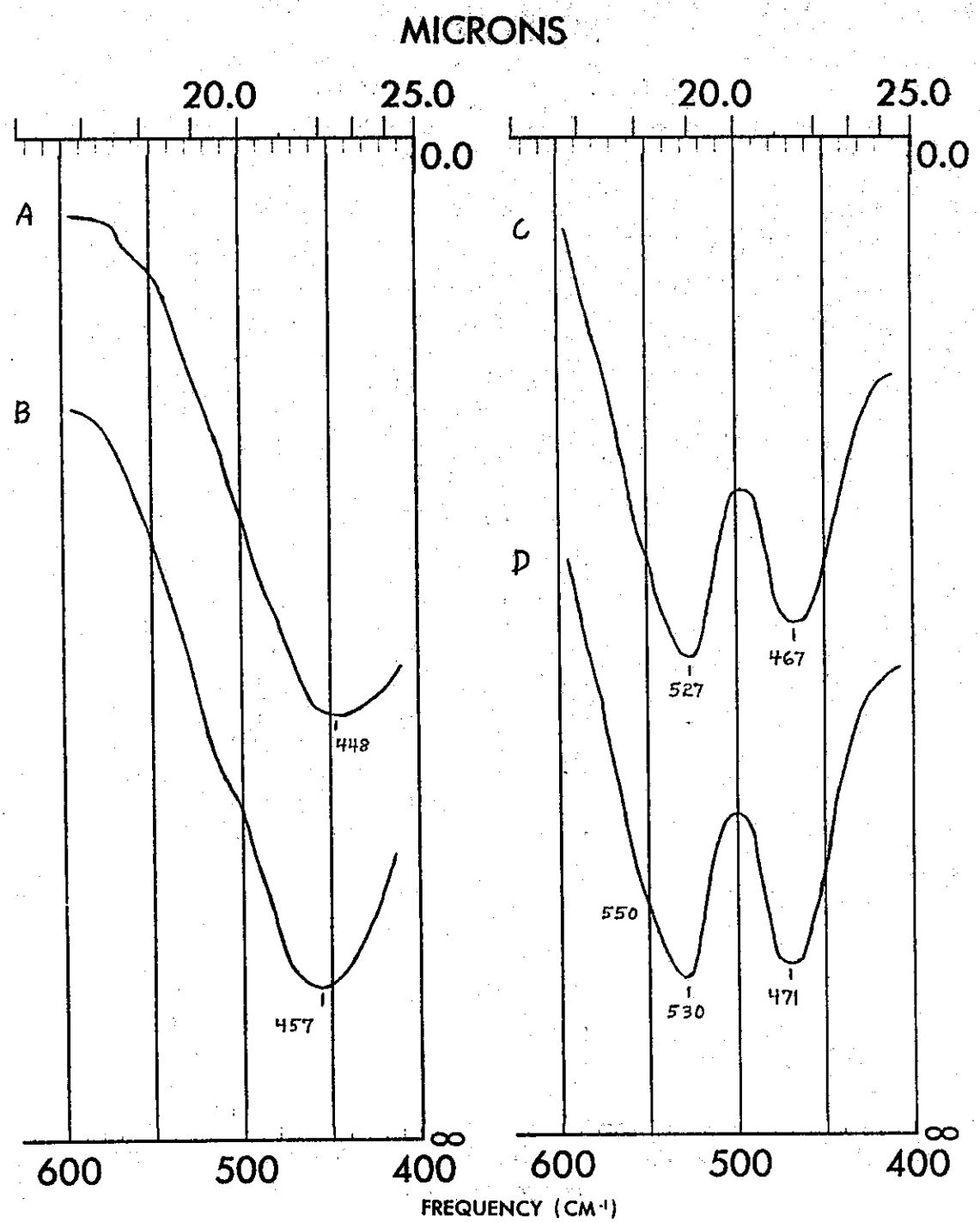

Fig. 6 Infrared spectra for trioctahedral mica and dioctahedral mica in the frequency range 600 Cm^{-1} to 400 Cm^{-1}. Curve A - biotite, Sabino Cyn., Tucson, Ariz.; Curve B - phlogopite, CSUS Geol. Dept., Calif.; Curve C - muscovite, Summerhaven, Ariz.; Curve D - muscovite, Pala, Calif.

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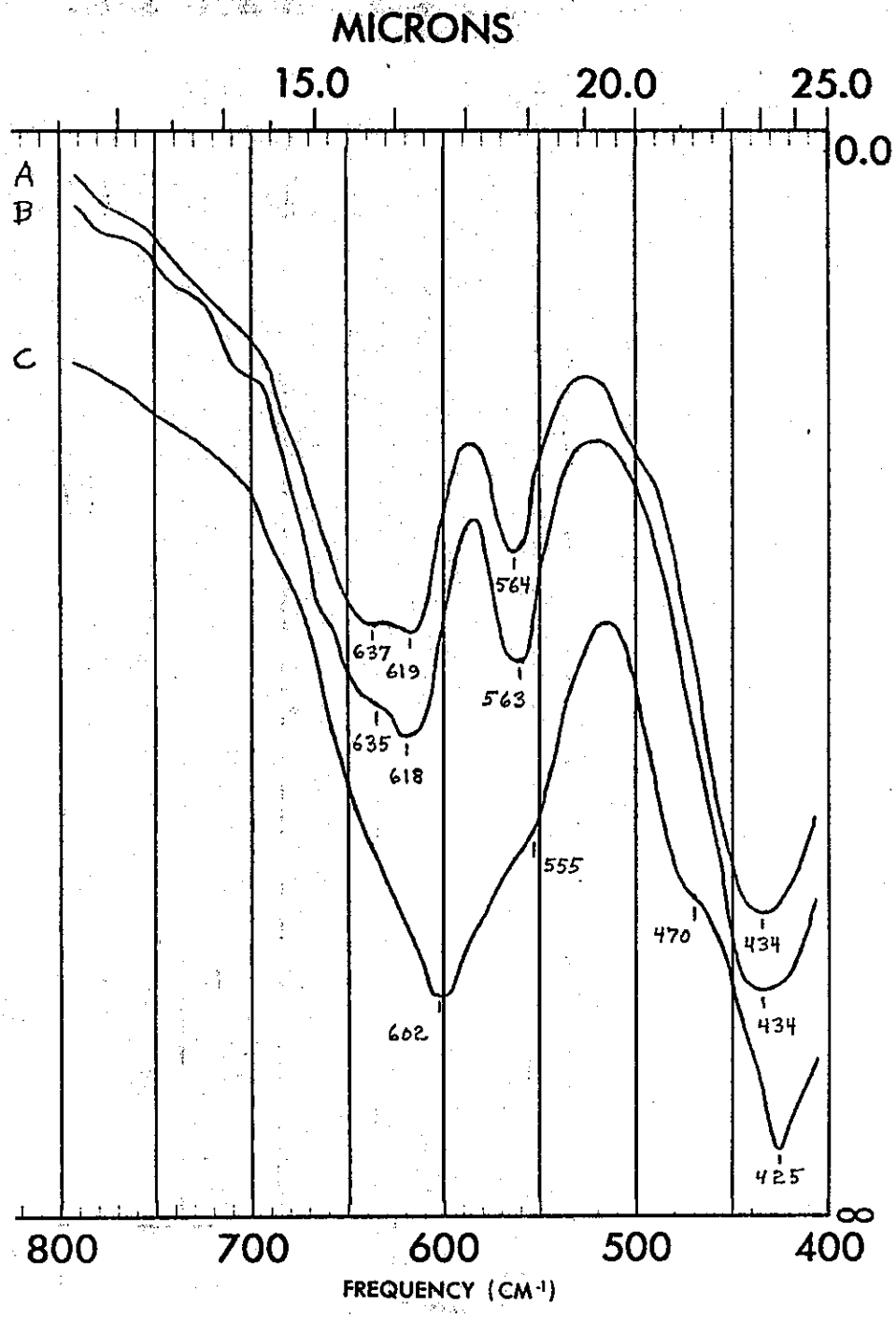

Fig. 7 Infrared spectra for varieties of serpentine in the frequency range 800 Cm^{-1} to 400 Cm^{-1}. Curve A - fibrous antigorite, Flagstaff Hill area, Calif.; Curve B - antigorite, Santa Catalina Mtns., Ariz.; Curve C - chrysotile, Napa Co., Calif.

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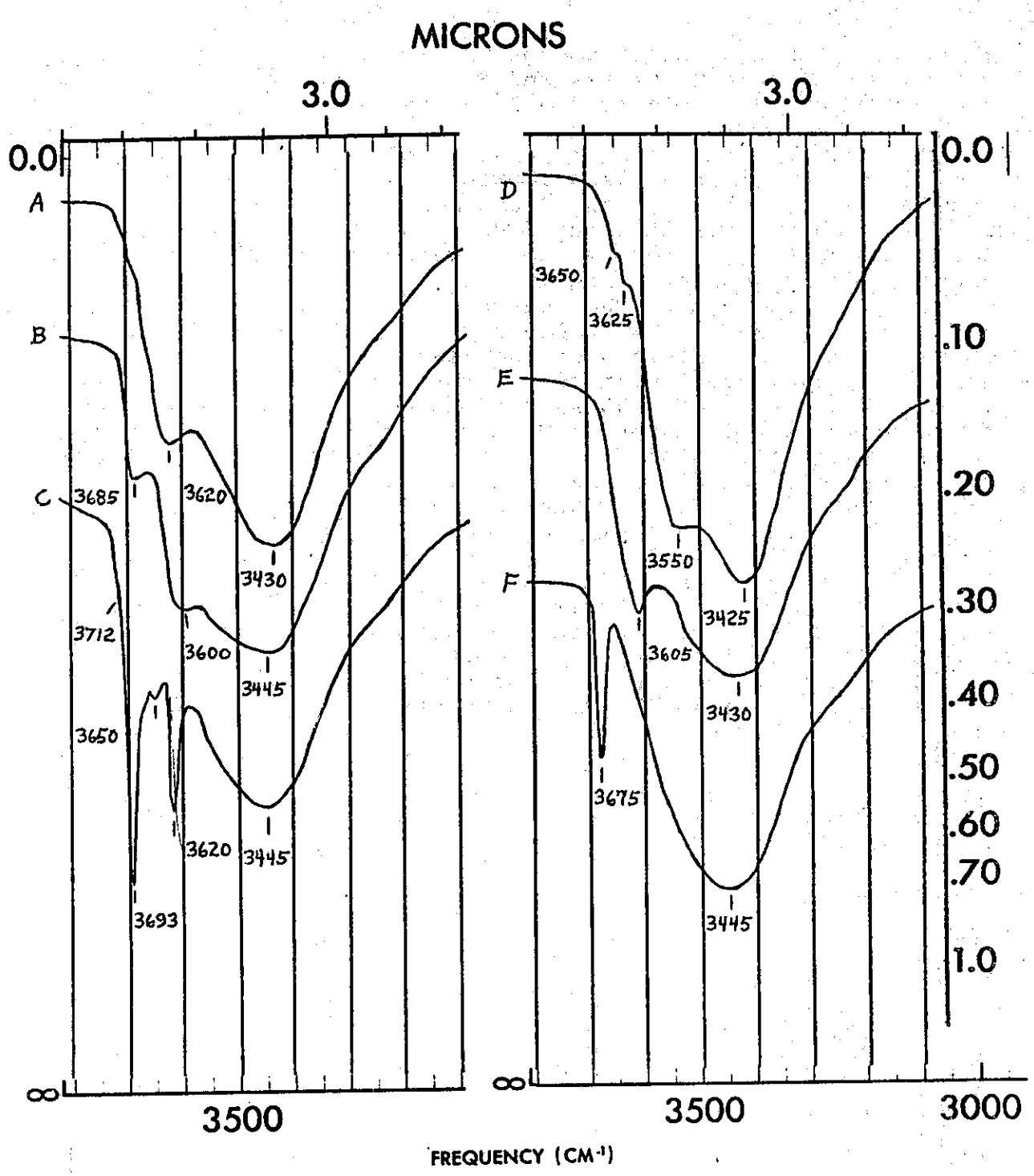

Fig. 8 Infrared spectra in the frequency range 3800 cm^{-1} to 3100 cm^{-1} showing the O-H stretching bonds of six typical hydrous layered silicates. Curve A - Na montmorillonite, Colony, Wy.; Curve B - halloysite (endellite), A.P.I. No. 13; Curve C - kaolinite, Georgia Kaolin Co.; Curve D - ripidolite, Flagstaff Hill, Ca.; Curve E - muscovite, Pala, Ca.; Curve F - talc, Van Horn, Texas.

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A preliminary list of infrared spectra for aggregate minerals now available at the Materials and Research Department, frequency range 4000 Cm^{-1} to 400 Cm^{-1}, is given in Table 5.

There has been considerable discussion about the particle size necessary for securing a clean reproducible spectrum. Stephens and Tuddenham (1971) and Lyon (Rich and Kunze, eds, 1964) suggest that the average particle size of material used be less than 2 μm. Such particle sizes are not necessary for mineral identification and, indeed, grinding may cause partial destruction of some crystal structures. The reason for control of the predominant particle size is that there may be light transmission interference in the sample when the mean particle size approximates the wave length of the transmitted light. Material pulverized to pass a No. 200 sieve does not appear to be affected very markedly in this manner. More coarse clay particles may show increased effects of orientation. For some investigations particle orientation is deliberately effected. In fact, some of the early i.r. investigations were made with thin plates of cleaved material, for example, mica.

Infrared investigations are also useful in the investigation of very poorly crystalline materials such as the sodium silicate glasses where x-ray diffraction procedures are of little help. The infrared analytical approach to the identification and quantitative analysis of hydrous layered silicates can be very useful in supplementing the more conventional x-ray analytic methods.

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TABLE 5

Infrared Spectra of Aggregate Minerals Now Available at M&R

Frequency Range 4000 Cm^{-1} to 400 Cm^{-1}

Mineral	Source
Calcite	Santa Catalina Mtns., Ariz.
Dolomite	E/O Lone Pine, Calif.
Halloysite	Eureka, Utah, API 13
Kaolinite	Georgia Kaolin Co.
Na-Montmorillonite	Bed A, Colony, Wy.
Montmorillonite	Almeria, Spain
Muscovite	Pala, Calif.
Nontronite	Manito, Wash., API 33b
Palygorskite	San Bernardino Co., Calif.
Pyrophyllite	Graves Mtn., Ga.
Quartz	20-30 Ottawa Sand
Saponite	Clay Camp, near Lathrop Wells, Nev.
Sepiolite	Two Crows, Nev. (Near Henderson)
Talc	25 mi. E/O Garberville, Calif.
Tremolite (fibrous)	In case at M&R; Dunsmuir, Calif.
Vermiculite #1	S/O I-80, Amer. Cyn., Calif.
Calcareous Duripan	Tucson, Ariz.
Slate (tan)	Hwy 193, So. Fk. Amer. Riv., Calif.
Slate (brown layer)	Hwy 193, So. Fk. Amer. Riv., Calif.
Slate (black)	Hwy 193, So. Fk. Amer. Riv., Calif.
Halloysite soil	Hwy 88, W/O Fiddletown Rd., Calif.

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APPENDIX

d(Å) Spacings for Non-Clay Aggregate Minerals

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AGGREGATE MINERALS (3°-21°2θ)

	5°	10°	15°	21°
Alunite			5.77w 5.71w 4.96w	
Amblygonite		6.16w	4.78w 4.65s	
Analcite			5.60m 4.84w	
Andalusite			5.52vs 4.51 vs	
Axinite		6.30f		
Barite				
Basic $Fe_2(SO_4)_3 \cdot 18H_2O$	18.40vs	9.18vs	6.125vs	5.56w 5.39f 4.72f 4.58f 4.49f
Brucite				4.76vs
Chabazite		9.11vs	6.37f	4.95f
Clinoptilolite		8.97m	7.91w 6.75f	5.24f 5.11f 4.35f
Cordierite			8.51vs	4.90w 4.66w
Cryolite				4.52m 4.42m
Epidote			8.04w	5.02w 4.78f 4.60f
Fluorapatite			8.13f	5.24f
Forsterite				5.12m 4.31f
Gibbsite				4.84s
Goethite				4.98f
Gypsum			7.60vs	4.28vs
Hydromagnesite		9.19vs	6.42s	5.81vs 4.60w 4.48w
Idocrase		10.98f		5.90f 4.72w
Jarosite			5.94w	5.75w 5.09w
Kyanite			6.70w	5.90f 4.43f 4.30f
Leonardite		9.42vs	6.82vs 6.51f 6.17f	5.02f 4.70f 4.49w 4.30f
Ludwigite			7.43f 6.19w	5.12vs
Magnetite				4.83f

vs - very strong; s - strong; m - moderate; w - weak; f - faint.

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AGGREGATE MINERALS (3°-21°2θ)

	5°	10°	15°	21°
Natrojarosite		5.94w	5.56m 5.05w	
Quartz			4.255vs	
Sillimanite			5.35m 4.56f	
Sphene			4.91w	
Spinel			4.68m	
Staurolite		8.22f 7.08w		
Tourmaline		6.34-6.41m	4.97w 4.61w 4.22-4.25m	
Ulexite	12.34vs	8.00m 7.76s 6.00s	5.81w 5.68w 5.38w 5.18f 4.60w 4.34w	

AMPHIBOLES AND PYROXENES (3°-21°2θ)

	5°(17.66Å)	10°(8.84Å)	15°(5.90Å)	21°
Glaucophane	8.93f	8.25vs	4.84f 4.64f 4.45w	
Tremolite	9.02w	8.41vs	5.06w 4.87f 4.73m 4.50w 4.20w	
Actinolite	9.02w	8.40vs	5.09f 4.88f 4.75w 4.51w 4.21m	
Hornblende	9.05f	8.41-8.44vs	4.91f 4.77f 4.51f 4.21f	
Richterite	9.06f	8.48-8.51vs	4.79f	
Enstatite		6.32f	4.56f 4.41f	
Bronzite		6.34w	4.43f	
Hypersthene			4.57f	
Diopside		4.46m		
Augite		6.45w 4.68f 4.46w		
Hedenbergite		6.58w 4.77w 4.55f		
Omphacite		6.34w 4.39m		
Spodumene		6.10s 4.44w 4.36w		

HYDROUS LAYERED SILICATES (3°-21°2θ)

	5°(17.66Å)	10°(8.84Å)	15°(5.90Å)	21°
Muscovite	9.96vs		4.98s	
Biotite	10.06vs		5.02f	
Phlogopite	10.05vs		5.02w	
Lepidolite	9.96vs		4.98vs	
Phengite	9.91vs		4.94s	
Vermiculite	15.1-14.4vs	7.56-7.20f	5.03-4.80f 4.58w	
Hydrobiotite	10.11vs		5.05f 4.60f	
Antigorite		7.26 vs	4.61f	
Chrysotile		7.32s	4.54w	
Talc	9.30vs		4.66vs 4.57w	
Pyrophyllite	9.18vs		4.59vs	
Penninite	14.31vs	7.16vs	4.77vs 4.61f	
Clinochlore	14.19vs	7.09vs	4.73vs 4.60f	
Sheridanite	14.16vs	7.08vs	4.72vs 4.61f	
Ripidolite	14.14vs	7.07vs	4.71vs 4.64f	

FELDSPARS

Albite	6.43f 6.38s
Oligoclase	6.44f 6.38w 6.37s
Andesine	6.45f 6.41w 6.36m 6.28f
Labradorite	6.47f 6.42w 6.36w
Bytownite	6.58f 6.54w
Microcline	6.73f 6.53w 6.49m 6.47s
Interm. Microcline	6.67f 6.59f
Orthoclase	6.61f 6.49w 6.48s
Sanidine	6.58f 6.51m 6.44w
Anorthoclase	6.50w 6.44w

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AGGREGATE MINERALS (21°-63°2θ)

	21°(4.23Å)	30°(2.976Å)	45°(2.013Å)	63°
Alunite	3.48m 2.99vs	2.88s 2.29vs	1.90m 1.50m	
Amblygonite	3.32m 3.26m 3.23m 3.19m 3.16vs	2.96vs 2.50m	1.58m	
Analcite	3.43s	2.93m		
Andalusite	3.92m 3.52s 3.50s	2.77vs 2.48m 2.46m 2.27m 2.25m 2.17s		
Aragonite	3.40s 3.27m	2.70m 2.48m 2.37m	1.98m	
Axinite	3.46m 3.15vs	2.80m 2.19m		
Barite	3.57s 3.43vs 3.30m 3.09m	2.72m 2.11m 2.10m		
Brucite		2.363s		
Calcite	3.03vs	2.28m 2.09m	1.91m 1.87m	
Cassiterite	3.35vs	2.64s 2.37m	1.76s 1.68s 1.50s	
Chabazite	3.02vs		1.82s	
Chromite		2.70m 2.50vs		
Clinoptilolite	3.97m			
Cordierite	4.09s 3.37s 3.15m 3.13s 3.04s 3.02m			
Cristobalite	4.04s			
Cryolite	4.52m 4.42m 3.89vs	2.75s 2.33m 2.32m	1.94vs	
Dolomite		2.89vs 2.19m	1.80m 1.78m	
Epidote	4.01s 3.40m 3.06m	2.90m 2.68m 2.60m 2.04m	1.64m	
Fluorapatite	3.43m	2.81s 2.77m 2.71m	1.94m 1.84m	
Fluorspar	3.15 vs		1.93s 1.65m	
Forsterite	3.89m 3.00m	2.77m 2.52m 2.46m	1.75m 1.62m	
Goethite	4.18m	2.69m 2.45m		
Grossular	2.98m	2.67vs 2.43m	1.72m 1.59m	
Gypsum	3.81vs 3.07s	2.87m	1.90m	
Halite		2.82vs	1.99m	
Hematite	3.69m	2.70vs 2.52s	1.84m 1.70m	
Hydromagnesite	4.19m 3.32m	2.90vs 2.69m 2.30m 2.16m		

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AGGREGATE MINERALS (21°-63°2θ)

	21°(4.23Å)	30°(2.976Å)	45°(2.013Å) 63°
Idocrase		2.75s 2.59m 2.04m	
Ilmenite		2.74s	
Jarosite	3.11m 3.08s	2.87m 2.29m	
Kyanite	3.74m 3.33vs 3.18m 3.06m	2.69m 2.51m	1.96vs 1.93m
Leonhardite	4.14vs 3.50m 3.26m 3.02m		
Ludwigite		2.56vs 2.17m	2.00m
Magnesite		2.74vs 2.50m	2.10vs 1.94m 1.70s
Magnetite		2.53s	1.48m
Marcasite		2.71m 2.69m	
Natrojarosite	3.11m 3.05m	2.77m	
Psilomelane	3.47m	2.41m	
Pyrite		2.71vs 2.42m	1.63m
Quartz	3.343vs	2.455m 2.279m 2.122m	1.817s 1.541m
Siderite		2.79vs	1.74m 1.73m
Sillimanite	3.41vs 3.36vs	2.68s 2.54s 2.21m 2.11m	1.52m
Smithsonite	3.56vs	2.75vs 2.33s 2.11vs	1.95m 1.71vs
Spinel		2.87m 2.45s 2.02m	1.56m
Staurolite	3.00m	2.68m 2.54m 2.39m 2.36m	1.97m
Tourmaline	3.98m 3.46s	2.95s 2.58m 2.04m	1.92m 1.65m
Tridymite	4.30m 4.27m 4.10s		
Ulexite	4.16m 3.09m	2.82m 2.67m	

AMPHIBOLES AND PYROXENES (21°-63°2θ)

	21° (4.23Å)	30° (2.976Å)	45° (2.013Å)	63°
Glaucophane	3.06vs	2.74m 2.69m		
Tremolite	3.27m 3.12vs	2.97m 2.80m 2.70m		
Actinolite	3.28m 3.12vs	2.80m 2.71m		
Hornblende	3.13vs	2.71m		
Richterite	3.14vs	2.72m		
Enstatite	3.17vs 3.16s	2.87s		
Bronzite	3.17vs	2.87m		
Hypersthene	3.18s	2.88m 2.54m 2.51m		
Diopside	3.23vs 2.99m	2.95s 2.56m 2.52m 2.15m	1.75m 1.62m	
Augite	3.22m 2.99s	2.95m 2.52s 2.15m		
Hedenbergite	3.01m			
Omphacite	4.39m 3.18m 2.97s	2.90m		
Spodumene	4.20vs	2.92s 2.79vs	1.86s	

FELDSPARS (21°-63°2θ)

Albite	4.03m	3.77m	3.67m	3.22m	3.195vs		2.93m	
Oligoclase	4.03m	3.77s	3.65m	3.49m	3.21m	3.185vs	2.93m	
Andesine	4.03m	3.75s	3.63m	3.36m	3.205vs	3.180vs	2.93m	
Labradorite	4.04m	3.76m	3.74m	3.37m	3.208vs	3.181vs	2.93m	
Bytownite			3.25m	3.209s	3.178vs	3.131s		
Microcline	3.83m	3.59m	3.48m	3.37m	3.29m	3.245vs	2.95m 2.16s	
Interm. Microcline	3.74m	3.47m	3.35m	3.31m	3.235vs		2.99m 2.16m	
Orthoclase	4.22m	3.76m	3.46m	3.31s	3.240vs		2.98m	1.80m
Sanidine	4.19m	3.93m	3.77m	3.30m	3.230vs		2.99m	1.80m
Anorthoclase	3.24s	3.22vs	3.20vs					

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HYDROUS LAYERED SILICATES (21°-63° 2θ)

	21° (4.23Å)	30° (2.976Å)	45° (2.013Å)	63°
Muscovite	3.33vs		1.99m	
Biotite	3.35vs		2.02m	
Phlogopite	3.35vs	2.62m 2.51m	2.01m	
Lepidolite	3.32vs		1.99s	
Phengite	3.30vs		1.98m	
Vermiculite	3.02-2.88m			
Hydrobiotite	3.37s			
Antigorite	3.61s	2.53m		
Chrysotile	3.65m			
Talc	3.11vs		1.87m	
Pyrophyllite	3.32m 3.06vs	2.29m	1.83s	
Penninite	3.58vs	2.86vs		
Clinochlore	3.55vs	2.84vs		
Sheridanite	3.54vs	2.83vs		
Ripidolite	3.54vs	2.83vs		

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