CLAY-SIZE FRACTION AND POWDERED WHOLE-ROCK X-RAY ANALYSES OF ALLUVIAL BASIN DEPOSITS IN CENTRAL AND SOUTHERN NEW MEXICO

By Scott K. Anderholm

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CONTENTS

TABLES

CLAY-SIZE FRACTION AND POWDERED WHOLE-ROCK X-RAY ANALYSES OF ALLUVIAL BASIN DEPOSITS IN CENTRAL AND SOUTHERN NEW MEXICO

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ABSTRACT

As part of the study of the geochemistry of the Southwest Alluvial Basins Regional Aquifer-System Analysis, X-ray analyses were performed on powdered whole rock and clay-size fraction of selected samples of alluvial basin deposits. The samples were analyzed to determine the minerals and clay types that may be reacting with ground water and thus affecting water quality.

Nineteen samples from the outcrop of Tertiary and Quaternary alluvial basin deposits in the central and southern Rio Grande rift were collected and analyzed. The analyses of the samples consisted of particle-size analysis (sand, silt, and clay percentages), powdered whole-rock X-ray analysis, clay-size fraction X-ray analysis, and semiquantitative analysis of the relative abundance of different clay-mineral groups present.

The results indicate that calcium smectite and mixed layer illite-smectite are the most abundant clay groups in the samples. Quartz, calcite, plagioclase, and orthoclase are the most common nonclay minerals. The zeolite mineral found in the samples probably is stilbite but could be heulandite.

INTRODUCTION

In 1978, Congress appropriated money to begin a Regional Aquifer-System Analysis (RASA) program. This program was designed to study the hydrology of large areas of the country that are underlain by a regional aquifer system. The Southwest Alluvial Basins (SWAB) regional aquifer study is one of these RASA studies. The SWAB study area consists of basins along the Rio Grande from its headwaters in southern Colorado to Presido, Texas, and several closed basins in southwestern and southern New Mexico. Twenty-two basins were identified in this study area.

The objectives of the study of the geochemistry for the SWAB study were to define the areal water-quality distributions in selected basins and to investigate the chemical reactions or processes that occur in the ground-water system. In conjunction with the study of the geochemistry, X-ray analyses of powdered whole rock and clay-size fraction of selected samples from alluvial basin deposits were performed to examine the minerals and clay types present in the aquifers that may be reacting with ground water.

The purpose of this report is to present the data resulting from the analyses. No interpretations of the data are presented in this report.

Method of Study

Samples from Tertiary and Quaternary alluvial basin deposits in the southern and central Rio Grande rift area were collected from unsaturated outcrops at land surface. In general, the sample sites were chosen to correspond with outcrops of rock units described or mapped in previously published reports or road logs. Sample sites also were chosen so that many of the different ages and rock types of alluvial basin deposits in the southern and central Rio Grande rift were represented. An attempt was made to dig into the outcrop a sufficient distance to get a fresh or unweathered sample. The difference in clay mineralogy or whole-rock mineralogy between unsaturated samples at the surface and saturated aquifer material at depth is unknown. At land surface, there may be leaching of minerals from the rock, concentration or precipitation of minerals in the rock due to evaporation of precipitation, or chemical reactions and thus changes in mineralogy caused by the oxidizing surface environment.

The samples were analyzed by personnel of the New Mexico Bureau of Mines and Mineral Resources. The analyses consisted of particle-size analysis (sand, silt, and clay percentages), powdered whole-rock X-ray analysis, clay-size fraction X-ray analysis, and semiquantitative analysis of the relative abundance of different clay groups present. The Supplemental Information section at the end of this report explains the procedure used in the X-ray examination of the clay-size fraction mineralogy. The semiquantitative relative abundance of different clay groups is based on X-ray peak heights rather than peak areas (George Austin, New Mexico Bureau of Mines and Mineral Resources, written commun., 1981).

Acknowledgments

Charles Kaehler of the U.S. Geological Survey and John Hawley of the New Mexico Bureau of Mines and Mineral Resources collected some of the samples. George Austin of the New Mexico Bureau of Mines and Mineral Resources was helpful throughout discussions concerning the results of the X-ray analysis.

DATA

Nineteen samples were collected for analysis. In general, the samples are listed from youngest to oldest. The description of each sample includes the geologic formation or member from which the sample was collected, the U.S. Geological Survey topographical quadrangle map used for sample location, the latitude (lat.) and longitude (long.) of the sample location, a brief description of sample location, and the name(s) of the sample collector. The results of the particle-size analysis are given next. The clay-size fraction data give the parts-in-ten abundance of the clay group first and then list the nonclay minerals in the clay-size fraction of the sample in order of abundance. The powdered whole-rock X-ray data are given last, and the minerals are listed in order of abundance in the sample. A condensed tabular form of the data is presented in table 1.

SAMPLE 1

Edith Formation of Lambert (1968), Bernalillo 7½-minute quadrangle. Lat. 34°16'17", long. 106°33'22". Sample collected from middle unit of 2B of Lambert's (1968) Bernalillo measured section. Measured section on U.S. Interstate 25 about 4 miles north of the Bernalillo-Sandoval County line. Sample collected by Scott Anderholm.
Particle Size: clay--5.2 percent, silt--8.0 percent, sand or larger--86.8 percent.
Clay-Size Fraction Mineralogy: clay minerals: 4/10 kaolinite, 3/10 mixed layer illite-smectite, 2/10 illite, 1/10 calcium smectite; nonclay minerals: calcite, quartz, plagioclase, and orthoclase.
Whole-Rock Mineralogy: quartz, plagioclase, orthoclase, calcite, gypsum, and unknown.

SAMPLE 2

Camp Rice Formation of Strain (1966) equivalent, piedmont facies, Caballo 7½-minute quadrangle. Lat. 32°50'12", long. 107°18'49". Sample collected from road cut along U.S. Interstate 25 north of Animas Arroyo bridge, 0.2 mile south of milepost 65. Sample collected by Charles Kaehler and John Hawley.
Particle Size: clay--11.7 percent, silt--14.6 percent, sand or larger--73.7 percent.
Clay-Size Fraction Mineralogy: clay minerals: 3/10 mixed layer illite-smectite, 3/10 kaolinite, 2/10 illite, 2/10 calcium smectite; nonclay minerals: orthoclase, plagioclase, quartz, gypsum (?), calcite, and zeolite.
Whole-Rock Mineralogy: quartz, orthoclase, calcite, plagioclase, and illite.

SAMPLE 3

Upper Buff Formation of Lambert (1968), La Mesita Negra SE 7½-minute quandrangle. Lat. 35°05'14", long. 106°52'28". Sample from unit 6 of Lambert (1968) El Rincon measured section. Sample collected from sandy unit near base of outcrop shown on plate 7-A (Lambert, 1968, p. 307). Sample collected by Scott Anderholm.
Particle Size: clay--3.8 percent, silt--1.6 percent, sand or larger--94.6 percent.
Clay-Size Fraction Mineralogy: clay minerals: 6/10 calcium smectite, 2/10 mixed layer illite-smectite, 1/10 kaolinite, 1/10 illite; nonclay minerals: plagioclase, calcite, and quartz.
Whole-Rock Mineralogy: quartz, plagioclase, orthoclase, calcite, dolomite, and smectite.

SAMPLE 4

Upper Buff Formation of Lambert (1968), La Mesita Negra SE 7½-minute quandrangle. Lat. 35°05'12", long. 106°52'30". Sample collected from middle of unit 2 of Lambert (1968) El Rincon measured section. Sample collected by Scott Anderholm.
Particle Size: clay--33.2 percent, silt--52.2 percent, sand or larger--14.6 percent.
Clay-Size Fraction Mineralogy: clay minerals: 8/10 calcium smectite, 1/10 kaolinite, 1/10 illite, trace of mixed layer illite-smectite; nonclay minerals: calcite, quartz, and feldspars.
Whole-Rock Mineralogy: quartz, calcite, plagioclase, orthoclase, smectite, illite, dolomite, and hornblende (?).

SAMPLE 5

Fluvial facies in upper Santa Fe Group, Williamsburg 7½-minute quadrangle. Lat. 33°01'13", long. 107°18'53". Sample collected from thick, maroon clay band in fluvial sand sequence in road cut along U.S. Interstate 25, 1.2 miles north of rest area. Sample collected by Charles Kaehler and John Hawley.
Particle Size: clay--48.9 percent, silt--48.2 percent, sand or larger--2.9 percent.
Clay-Size Fraction Mineralogy: clay minerals: 4/10 mixed layer illite-smectite, 3/10 calcium smectite, 2/10 illite, 1/10 kaolinite; nonclay minerals: quartz, feldspar, and calcite.
Whole-Rock Mineralogy: quartz, calcite, illite, plagioclase, and smectite.

SAMPLE 6

Sierra Ladrones Formation, alluvial-fan facies, Black Hill 7½-minute quadrangle. Lat. 33°32'52", long. 107°12'30". Sample collected from buried soil horizon in road cut along U.S. Interstate 25 near Nogal Canyon. Sample collected by Charles Kaehler and John Hawley.
Particle Size: clay--44.2 percent, silt--33.8 percent, sand or larger--21.8 percent.
Clay-Size Fraction Mineralogy: clay minerals: 5/10 mixed layer illite-smectite, 2/10 kaolinite, 2/10 illite, 1/10 calcium smectite; nonclay minerals: feldspar (?) and calcite (?).
Whole-Rock Mineralogy: quartz, plagioclase, orthoclase, illite, and smectite.

SAMPLE 7

Rincon Valley Formation of Seager, Hawley, and Clemons (1971), piedmont facies, Sierra Alta 7½-minute quadrangle. Lat. 32°35'02", long. 107°00'25". Sample collected from road cut at highway level, south of Hayner Ranch on old highway 85 at Rio Grande overlook. Sample collected by Charles Kaehler and John Hawley.
Particle Size: clay--13.2 percent, silt--12.0 percent, sand or larger--74.8 percent.
Clay-Size Fraction Mineralogy: clay minerals: 5/10 mixed layer illite-smectite, 3/10 calcium smectite, 1/10 illite, 1/10 kaolinite; nonclay minerals: quartz, illite, calcite, and zeolite.
Whole-Rock Mineralogy quartz, orthoclase, calcite, plagioclase, illite, zeolite, and smectite.

SAMPLE 8

Rincon Valley Formation of Seager, Hawley, and Clemons (1971), playa facies, Rincon 7½-minute quadrangle. Lat 32°40'36", long 107°04'42". Sample collected from road cut along U.S. Interstate 25, 0.1 mile west of milepost 37. Sample collected by Charles Kaehler and John Hawley.
Particle Size: clay--49.1 percent, silt--37.8 percent, sand or larger--13.1 percent.
Clay-Size Fraction Mineralogy: clay minerals: 5/10 mixed layer illite-smectite, 2/10 kaolinite, 2/10 illite, 1/10 calcium smectite; nonclay minerals: quartz, calcite, feldspar.
Whole-Rock Mineralogy: quartz, albite, calcite, gypsum, smectite, and illite.

SAMPLE 9

Middle Red Member of Santa Fe Formation of Bryan and McCann (1937), Bernalillo 7½-minute quadrangle. Lat. 35°16'13", long. 106°33'23". Sample collected from sand unit approximately 6 feet below top of Middle Red Formation of Lambert (1968) in an arroyo approximately 200 feet south of Lambert's (1968) Bernalillo measured section. Sample collected by Scott Anderholm.
Particle Size: clay--5.4 percent, silt--6.4 percent, sand or larger--88.2 percent.
Clay-Size Fraction Mineralogy: clay minerals: 4/10 mixed layer illite-smectite, 3/10 calcium smectite, 2/10 kaolinite, 1/10 illite; nonclay minerals: feldspar, quartz, and calcite.
Whole-Rock Mineralogy: quartz, calcite, plagioclase, orthoclase, gypsum, illite, smectite, and zeolite.

SAMPLE 10

Middle Red Member (?) of Santa Fe Formation of Bryan and McCann (1937), Bernalillo 7½-minute quandrangle. Lat. 35°18'58", long. 106°31'52". Sample collected from sand unit 3 feet below base of 4-foot red clay unit at road cut on the northeastern side of the Cuba-Bernalillo-Placitas exit on U.S. Interstate 25. Sample collected by Scott Anderholm.
Particle Size: clay--5.2 percent, silt--21.5 percent, sand or larger--73.3 percent.
Clay-Size Fraction Mineralogy: clay minerals: 4/10 mixed layer illite-smectite, 3/10 calcium smectite, 2/10 kaolinite, 1/10 illite; nonclay minerals: calcite, quartz, and feldspar.
Whole-Rock Mineralogy: calcite, quartz, feldspar, smectite, unknown, and zeolite.

SAMPLE 11

Middle Red Member of Santa Fe Formation Bryan and McCann (1937), Mesa Aparejo 15-minute quadrangle. Lat. 34°43'08", long. 107°02'15". Sample collected from north side of small ridge that separates two major arroyos in Gabaldon badlands. Sample collected by Scott Anderholm.
Particle Size: clay--19.5 percent, silt--10.2 percent, sand or larger--70.3 percent.
Clay-Size Fraction Mineralogy: clay minerals: 5/10 mixed layer illite-smectite, 2/10 kaolinite, 2/10 calcium smectite, 1/10 illite; nonclay minerals: quartz, feldspars, calcite, and zeolite.
Whole-Rock Mineralogy: quartz, calcite, plagioclase, orthoclase, gypsum, and illite.

SAMPLE 12

Middle Red Member of Santa Fe Formation of Bryan and McCann (1937), Mesa Aparejo 15-minute quadrangle. Lat. 34°43', long. 107°02'25". Sample collected from outcrop along the northern margin of the Gabaldon badlands. Sample collected by Scott Anderholm.
Particle Size: clay--37.2 percent, silt--21.5 percent, sand or larger--41.2 percent.
Clay-Size Fraction Mineralogy: clay minerals: 4/10 mixed layer illite-smectite, 3/10 kaolinite, 2/10 calcium smectite, 1/10 illite; nonclay minerals: quartz, feldspar, and calcite.
Whole-Rock Mineralogy: quartz, orthoclase, plagioclase, calcite, and smectite.

SAMPLE 13

Post-Popotosa Formation, San Acacia 7½-minute quadrangle. Lat. 34°17'54", long. 106°55'23". Sample collected from sand outcrop under the powerline approximately 50 feet south of road. Sample collected by Scott Anderholm.
Particle Size: clay--14.7 percent, silt--10.3 percent, sand or larger--75.0 percent.
Clay-Size Fraction Mineralogy: clay minerals: 4/10 mixed layer illite-smectite, 3/10 kaolinite, 2/10 calcium smectite, 1/10 illite; nonclay minerals: quartz, calcite, and feldspar.
Whole-Rock Mineralogy: quartz, orthoclase, plagioclase (?), calcite, and illite.

SAMPLE 14

Popotosa Formation, playa facies, San Acacia 7½-minute quadrangle. Lat. 34°17'25", long. 106°59'38". Sample taken from claystone outcrop below resistant sandstone south of Cañada de la Tortola. Sample collected by Scott Anderholm.
Particle Size: clay--42.39 percent, silt--38.1 percent, sand or larger--19.6 percent.
Clay-Size Fraction Mineralogy: clay minerals: 5/10 calcium smectite, 2/10 kaolinite, 2/10 illite, and 1/10 mixed layer illite-smectite.
Whole-Rock Mineralogy: quartz, calcite, gypsum, illite, and feldspar.

SAMPLE 15

Popotosa Formation, alluvial-fan facies, Riley 15-minute quadrangle. Lat 34°17'28", long. 107°00'23". Sample taken from oxidized sandstone at the end of the road in the arroyo. Sample collected by Scott Anderholm.
Particle Size: clay--9.2 percent, silt--3.6 percent, sand or larger--87.2 percent.
Clay-Size Fraction Mineralogy: clay minerals: 5/10 mixed layer illite-smectite, 4/10 calcium smectite, 1/10 illite, trace of kaolinite; nonclay minerals: quartz, orthoclase, plagioclase, and zeolite.
Whole-Rock Mineralogy: plagioclase, orthoclase, quartz, calcite, zeolite, and smectite.

SAMPLE 16

Popotosa Formation, Riley 15-minute quadrangle. Lat. 34°17'15", long. 107°16'42". Sample collected approximately 150 feet east of fault that separates andesite and Popotosa Formation. Sample collected by Scott Anderholm.
Particle Size: clay--8.5 percent, silt--12.2 percent, sand or larger--79.3 percent.
Clay-Size Fraction Mineralogy: clay minerals: 8/10 calcium smectite, 1/10 illite, 1/10 mixed layer illite-smectite; nonclay minerals: quartz, feldspar, calcite, and zeolite.
Whole-Rock Mineralogy: calcite, plagioclase, quartz, gypsum, and zeolite.

SAMPLE 17

Cochiti Formation, Loma Machete 7½-minute quadrangle. Lat. 35°21'53", long. 106°37'47". Sample taken from first sandstone above the basal gravel of the formation in outcrop along State Highway 44, 0.3 mile west of milepost 6. Sample collected by Scott Anderholm.
Particle Size: clay--4.3 percent, silt--5.2 percent, sand or larger--90.5 percent.
Clay-Size Fraction Mineralogy: clay minerals: 9/10 calcium smectite, trace of kaolinite, trace of illite; nonclay minerals: plagioclase, orthoclase, calcite, and zeolite.
Whole-Rock Mineralogy: quartz, calcite, plagioclase, orthoclase, smectite, and zeolite.

SAMPLE 18

Zia Sand of Galusha (1966), Sky Village NE 7½-minute quadrangle. Lat 35°28'12", long. 106°49'24". Sample collected from pink sandstone approximately 5 feet above the base of the outcrop in large north-facing cliff. Sample collected by Scott Anderholm.
Particle Size: clay--7.5 percent, silt--40.5 percent, sand or larger--52.0 percent.
Clay-Size Fraction Mineralogy: clay minerals: 9/10 calcium smectite, 1/10 mixed layer illite-smectite, trace of illite; nonclay minerals: quartz, feldspar, calcite, and zeolite.
Whole-Rock Mineralogy: quartz, smectite, plagioclase, orthoclase, and gypsum.

SAMPLE 19

Love Ranch Formation, Las Cruces 15-minute quadrangle. Lat. 32°20'51", long 106°52'30". Sample collected near Apache Canyon Dam in the southern Robledo Mountains. Sample collected by Charles Kaehler and John Hawley.
Particle Size: clay--9.8 percent, silt--17.6 percent, sand or larger--72.6 percent.
Clay-Size Fraction Mineralogy: clay minerals: 6/10 calcium smectite, 2/10 mixed layer illite-smectite, 1/10 kaolinite, 1/10 illite; nonclay minerals: calcite, quartz, and zeolite.
Whole-Rock Mineralogy: quartz, gypsum, plagioclase, dolomite, and smectite.

RESULTS

Calcium smectite and mixed layer illite-smectite are the most common clay groups found in the samples (table 1). Quartz, calcite, plagioclase, and orthoclase are the most common nonclay minerals found in the samples (table 1).

The zeolite mineral probably is stilbite but could be heulandite (George Austin, New Mexico Bureau of Mines and Mineral Resources, written commun., 1981). Brenner-Tourtelot and Machette (1979, p. 13) identified the zeolite minerals clinoptilolite and mordenite as the most common zeolite minerals in samples from the Popotosa Formation near San Acacia. They (1979, p. 13) also found the zeolite minerals analcime and chabazite in their samples.

Table 1. ***Results of clay-size fraction and powdered whole-rock X-ray analyses***

[Numbers for clay groups are parts-in-ten abundance. Nonclay minerals and powdered whole rock are expressed in order of relative abundance. x_1 is most abundant.]

		Clay-size fraction							
		Clay group				Nonclay			
Sample number	Formation	Calcium smectite	Mixed layer illite-smectite	Kaolinite	Illite	Plagio-clase	Zeolite	Calcite	Ortho clase
1.	Edith Formation of Lambert (1968)	1	3	4	2	x_3		x_1	x_4
2.	Camp Rice Formation of Strain (1966) equivalent	2	3	3	2	x_2	x_6	x_5	x_1
3.	Upper Buff Formation of Lambert (1968)	6	2	1	1	x_1		x_2	
4.	do	8	tr	1	1			x_1	
5.	Fluvial facies in upper Santa Fe Group	3	4	1	2			x_3	
6.	Sierra Ladrones Formation	1	5	2	2			x_2	
7.	Rincon Valley Formation of Seager, Hawley and Clemons (1971), piedmont facies	3	5	1	1		x_3	x_2	
8.	Rincon Valley Formation of Seager, Hawley and Clemons (1971), playa facies	1	5	2	2			x_2	
9.	Middle Red Member of Santa Fe Formation of Bryan and McCann (1937)	3	4	2	1			x_3	
10.	do	3	4	2	1			x_1	
11.	do	2	5	2	1		x_4	x_3	
12.	do	2	4	3	1			x_3	
13.	Post-Popotosa Formation	2	4	3	1			x_2	
14.	Popotosa Formation, playa facies	5	1	2	2				
15.	Popotosa Formation, alluvial-fan facies	4	5	tr	1	x_3	x_4		x_2
16.	Popotosa Formation	8	1		1		x_4	x_3	
17.	Cochiti Formation	9		tr	tr	x_1	x_4	x_3	x_2
18.	Zia Sand	9	1		tr		x_4	x_3	
19.	Love Ranch Formation	6	2	1	1		x_3	x_1	

			Powdered whole rock									
minerals												
Gypsum	Quartz	Feldspar (undiff-erentiated)	Quartz	Cal-cite	Feldspar (undiff-erentiated	Ortho-clase	Plagio-clase	Gypsum	Dolo-mite	Smectite	Illite	Zeo-lite
	x_2		x_1	x_4		x_3	x_2	x_5				
x_4	x_3		x_1	x_3		x_2	x_4				x_5	
	x_3		x_1	x_4		x_3	x_2		x_5	x_6		
	x_2	x_3	x_1	x_2		x_4	x_3		x_7	x_5	x_6	
	x_1	x_2	x_1	x_2			x_4			x_5	x_3	
		x_1	x_1			x_3	x_2			x_5	x_4	
	x_1		x_1	x_3		x_2	x_4			x_7	x_5	x_6
	x_1	x_3	x_1	x_3	x_2			x_4		x_5	x_6	
	x_2	x_1	x_1	x_2		x_4	x_3	x_5		x_7	x_6	x_8
	x_2	x_3	x_2	x_1	x_3					x_4		x_5
	x_1	x_2	x_1	x_2		x_4	x_3	x_5			x_6	
	x_1	x_2	x_1	x_4		x_2	x_3			x_5		
	x_1	x_3	x_1	x_4		x_2	x_3				x_5	
			x_1	x_2	x_5			x_3			x_4	
	x_1		x_3	x_4		x_2	x_1			x_6		x_5
	x_1	x_2	x_3	x_1			x_2	x_4				x_5
			x_1	x_2		x_4	x_3			x_5		x_6
	x_1	x_2	x_1			x_4	x_3	x_5		x_2		
	x_2		x_1				x_3	x_2	x_4	x_5		

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SUPPLEMENTAL INFORMATION

Procedures used in the X-ray examination of clays (George Austin, New Mexico Bureau of Mines and Mineral Resources, written commun., 1981):

Procedure for preparation of oriented clay-mineral aggregates

1. Place small sample (20-25 grams) in a 100 milliliter beaker with distilled or deionized water. Mix and wait 5 minutes. Remix, wait 15 seconds, and pour suspension into another 100 milliliter beaker.

2a. If clay flocculates or settles out, pour off clear water, add more water and remix. If clay does not disperse, repeat this step several times.

2b. If clay still flocculates, wet grind a sample in distilled or deionized water in a mortar until slurry is developed. Place slurry in 100 milliliter beaker and fill with distilled or deionized water.

2c. If clay still settles, centrifuge, wash it with distilled water and centrifuge again.

2d. If clay still does not disperse, add a few drops of concentrated ammonium hydroxide (NH_4OH) and remix. If clay flocculates, repeat step 2a.

2e. If clay still flocculates, add a few drops of a dilute solution (50 grams per liter) of sodium hexametaphosphate (Calgon). Remix. If clay does not disperse, repeat step 2a.

3. Once the clay is in a dispersed state, allow the beaker and its contents to remain undisturbed for 10 minutes. At the end of the 10-minute period, touch eye-dropper to the surface of the suspension and draw off enough material to cover a glass slide completely. This decanted fraction is less-than-2-micrometer equivalent size diameter. It is generally desirable to prepare two or three slides of each sample at this time. Slides should be allowed to air dry.

4. Use petrographic glass slides or flat, porous ceramic plates having a high melting point. The latter generally will not warp at temperatures over 550 to 600 degrees Celsius (°C). If desired, exchange of the saturated cations may also be accomplished with clay minerals mounted on porous plates.

This procedure generally yields a strongly oriented sample with the c axis perpendicular to the slide's surface, thus giving intense basal X-ray reflections. Rarely the clay slurry will flocculate on the slide surface during drying and preferred orientation of the clays will be absent. If it does, remake slide; this time washing clay several times with distilled or deionized water before placing the suspension on the slide.

5. Run slide of oriented clay on diffractometer at 2° 2θ per minute from 2° 2θ to 38° 2θ with monochromatic or Ni-filtered Cu radiation. Subsequent runs will vary depending on the mineralogy and nature of the information needed.

The approximate basal spacings for the various clay mineral groups are as follows:

Basal spacings in A and 2θ Cu Kα

Clay mineral group	001		002		003		004	
	A	2θ	A	2θ	A	2θ	A	2θ
Chlorite	14.1 to 14.2	6.2 to 6.3	7.05 to 7.1	12.5 to 12.6	4.7	18.9	3.52 to 3.54	25.3 to 25.1
Kaolinite	7.16	12.4	3.57	24.9	2.38	37.8	1.78	51.3
Mica (illite)	9.98	8.8	4.96	17.9	3.32	26.8	2.49	36.1
Ca montmorillonite*	15.4	5.7	7.7	11.5	5.1	17.4	3.8	23.4
Na montmorillonite*	12.4	7.1	6.2	14.3	4.1	21.7	3.1	28.8
Ca mont. plus glycol Na mont. plus glycol	17.0	5.2	8.5	10.4	5.7	15.6	4.2	21.2
Vermiculite	14.1 to 14.3	6.2	w		w		w	

w - weak if present at all * general positions on broad peaks

The mineral assemblage encountered is likely to be a mixture of clay minerals some of which may have regular or random mixed-layer structures: illite-smectite, illite-chlorite, etc. In identifying mixed-layer clay minerals, consideration of the individual components should guide the treatment of the sample.

Many times when kaolinite and chlorite are both present in a sample, distinction of the two may be made by the separation of the 002 kaolinite peak from the 004 chlorite peak at 24.9° and 25.1° 2θ respectively. A slower scanning speed of 0.4° 2θ per minute from 24° 2θ to 26° 2θ will be helpful in distinguishing these basal reflections.

In further identification of the various clay minerals, the following treatments are helpful:

a. Treatment of the sample with ethylene glycol.

b. Heat treatment of the sample.

c. X-ray of sample under controlled humidity.

d. Saturating the sample with various cations.

Ethylene glycol treatment

Oriented aggregate is placed in closed container above ethylene glycol overnight. Smectite will expand and absorb the glycol between the layers, thus changing the basal spacing (see chart on previous page). Ethylene glycol is also absorbed by smectite layers within mixed-layer clay minerals, thus aiding in their identification. The container may be heated to speed up the process, making glycolation of the sample possible in 10 to 30 minutes.

Heat treatment

The original or second oriented slide may be heat treated to help differentiate the various clay minerals. However, response of a mineral to heat treatment varies with composition and crystallinity, and with heating time and temperature. If minerals response to heat treatment appears anomalous, heat another slide in stages (375°C, 450°C, etc.) and examine by X-ray diffraction after each stage.

Heating Example:
14A peak present. Heat to 250°C to 300°C for 30 to 60 minutes. If peak shifts to approximately 10A, vermiculite is present. If peak does not shift, but increases in intensity after 500°C for 60 minutes, Fe-rich chlorite is present; Mg-rich chlorite indicated by increase in intensity of 14A peak at 600°C after 60 minutes. 10A peak present along with poorly defined reflections between 10 and 17A. Heat to 375°C for 30 minutes, ill-defined reflections collapse to 10A, illite-smectite mixed layers present.

Controlled humidity

Air pumped through a drying tower and directed on sample during X-ray diffraction will cause the 001 peak of Na^+ montmorillonite to shift to about 10A whereas Ca^{++} montmorillonites 001 peak will shift to approximately 14.0A. Air pumped through various saturated salt solutions and directed over the sample during X-ray diffraction is also helpful in determining exchangeable cations on montmorillonite.

Saturating sample with various cations

It is particularly useful in the study of soil clays in which X-ray diffraction maxima are diffuse to saturate the clay with various cations, e.g. Mg^{++}, Na^{+}., K^{+}, etc., before using other techniques for identification. Samples can be mixed with 1 normal solution of the desired salt ($MgCl_2$, KCl, etc.) and then washed until they disperse.

Example:

Mg^{++} saturate sample. X-ray shows 14A peak. If montmorillonite is present, glycol will shift peak to 17A. If 14A peak is not affected by glycol, heat slide to 250°C to 300°C for 30 to 60 minutes. If 14A peak persists, heat sample to 600°C and if peak becomes more intense, mineral is chlorite.

This is a very general outline for clay-mineral sample preparation and treatment. Tailoring of these procedures to suit an individual's needs probably will be necessary. It should be kept in mind that there are many other procedures and treatments not mentioned here that can aid substantially in clay-mineral identification.