NBS TECHNICAL NOTE 977

U. S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Methods For Characterizing Adobe Building Materials

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+ Technical Note

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Abstract

Methods are described for the characterization of those physical properties and mineralogical features of adobe which appear to have the most significant affect on the durability of adobe. These methods include determinations of color, pH, soluble salts, particle size distribution, liquid and plastic limits, and the X-ray "fingerprint" of adobe. In addition, methods are given for the identification of the mineralogy of adobe soils and for the examination of the microfabric of adobe.

Key words: Adobe building materials; adobe soil; color determination; microfabric analysis; mineralogical analysis; particle size distribution; pH; physical properties; plastic and liquid limits; soluble salts.

METHODS FOR CHARACTERIZING ADOBE BUILDING MATERIALS

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1. INTRODUCTION

The need for standard test methods to characterize the important physical properties and mineralogical features of adobe soils and adobe building materials was identified in a review of the technology of preserving historic adobe structures [1][1]. The use of standard test methods should assist the National Park Service in the assimilation and interpretation of data from different sources. Furthermore, standard tests should form the basis for evaluating the effects of preservation materials and methods, and for selecting soils to be used for repairing or replacing deteriorated adobe.

The complete physical and mineralogical characterization of soils is a lengthy process because soils consist of a complex mixture of different mineral and organic substances. However, by only considering the most important properties affecting the performance of a soil used as an adobe building material, the analysis is simplified. This was the approach taken in selecting the test methods described in this report. These methods can form the basis for a standard analysis of the important physical properties and mineralogical features of adobe. Included are test methods which measure those properties of adobes which appear to have the most significant impact on their durability, such as particle size distribution, chemical and phase compositions and the response of adobes to moisture [1]. In addition, methods are provided for the rapid comparison of the properties of repair materials with those of the original adobe.

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[1] Numbers in brackets refer to the literature references listed at the end of this paper.

Most of the tests can be carried out in a modestly-equipped laboratory and the results are easily interpreted. However, the microscopic and X-ray powder diffraction methods should be performed by an analyst experienced in their use. The test procedures given in this report were either taken from existing standard test methods or were developed at the National Bureau of Standards, specifically for the analysis of adobe. In either case, the validity of the procedures was verified by using them to characterize the properties of adobe and soil samples supplied by the National Park Service.

2. SELECTION OF SAMPLES

The usefulness of the information obtained from the analysis of an adobe specimen is largely dependent on the selection of representative specimens. Samples should be carefully chosen according to the objectives of the work and should be representative of the structure at the sampling site. Often the size and number of samples which can be taken from historic sites are limited. A wide range of compositional and microstructural features, which are not visually apparent, may exist within a single adobe specimen. Such variations in the features of an adobe are caused by the heterogeneous nature of soils. In addition, soil could have been taken from several sites during the construction or repair of an adobe structure. Because of these difficulties, either the analyst or personnel experienced in sample selection should actively participate in the selection and removal of specimens.

The size and number of specimens which should be obtained depends on the particular properties which will be studied. For example, the determination of the particle size distribution (Section 3.4) may require a sample which weighs up to 500 grams, whereas only a few grams of material may be needed for mineralogical analysis by X-ray powder diffractometry (Section 3.6). However, the analyst should be aware that the properties of a few small samples may not be representative of those of the "average" adobe in the structure. If carefully carried out, the comparison of analyses of several samples from each of several locations randomly chosen, should provide an indication of the homogeneity of the adobe material in the structure.

The position from which a sample was removed should be recorded and photographically documented at time of removal. In addition, the physical properties of the sample should be recorded including its size and shape, color (using Munsell color charts, Section 3.1), moisture condition, the presence of any large aggregates, and the degree of consolidation (e.g., a hard mass, powdery, etc.). The sample should be transferred to the laboratory in a capped container that maintains its original moisture content.

3. METHODS FOR THE ANALYSIS OF ADOBE

A sequential analysis of adobe building materials, using the methods described in this section, is illustrated in figure 1. This analysis is more complete than that normally necessary. The analysis should be organized to provide the information that is needed in the different aspects of adobe preservation, as indicated in table 1.

The testing procedures for the first five methods are given in detail because these methods can be readily performed using simple laboratory equipment and provide the information most often needed. The x-ray diffractometry and microscopy techniques require more sophisticated equipment and expertise in interpreting the results. Procedures for using them, therefore, is not given in detail. Some results are included under the individual test methods that are based on the characterization of samples taken from structures at Escalante, Fort Bowie and Tumacacori, Arizona. These results are included to indicate the type of information which can be obtained from the individual methods. A brief description of the source of the samples is given in the Appendix.

3.1 Color Determination

The color of an adobe is largely determined by the mineralogy, amount, and particle size of the various constituents. Therefore, color determination is a useful field method for screening soils to be used in the repair of adobe structures. The final selection of the soil to be used for repairs, however, should be made after its composition is more completely characterized by such techniques as X-ray diffraction and particle size distribution analysis.

The color of adobe samples can be easily determined in the field using the methods outlined in this section. The color of a sample should be determined and recorded while the sample is being visually inspected in the field (figure 1).

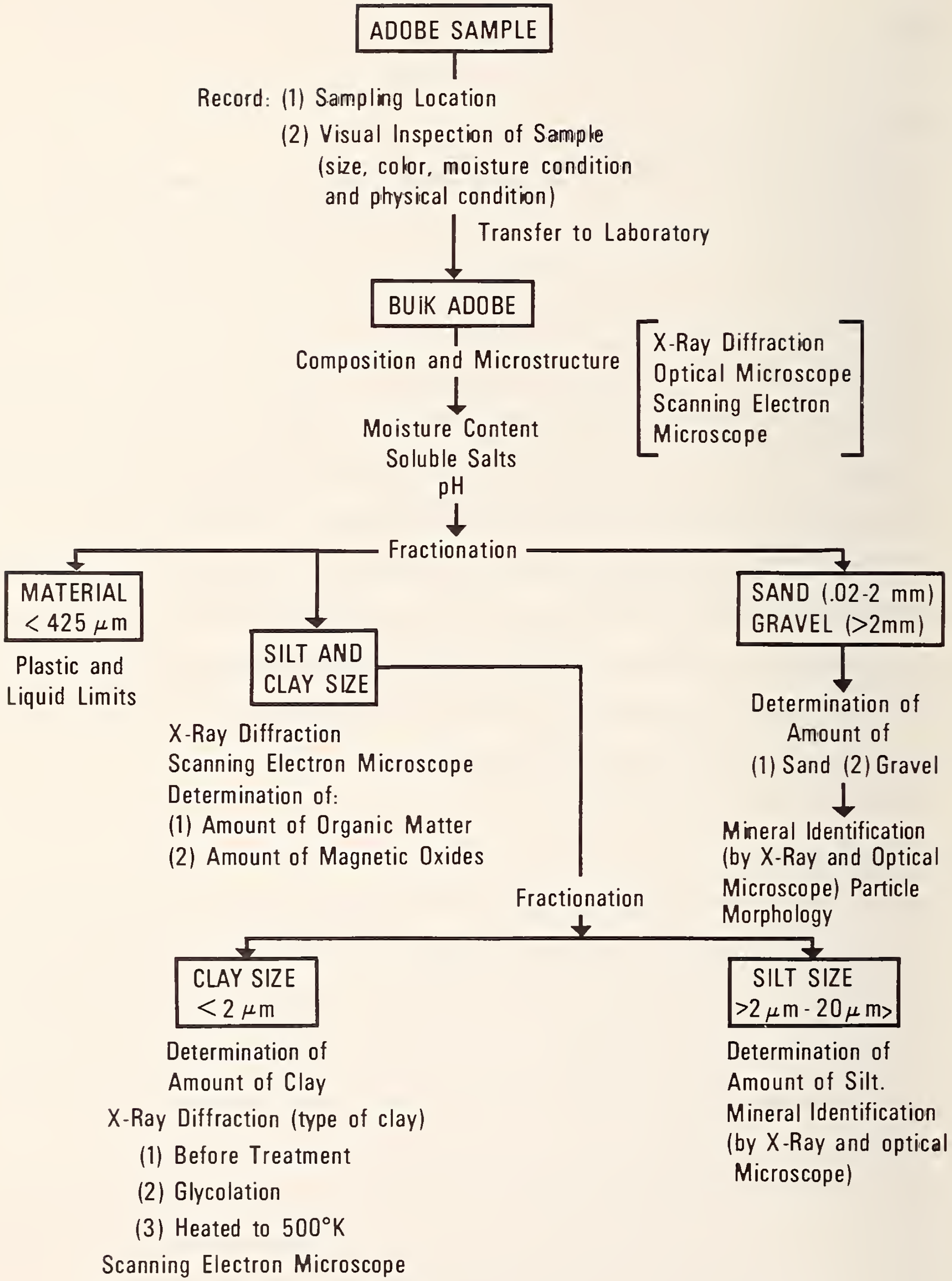

Figure 1. Flow chart for analysis of adobe.

Table 1. Selection of Methods Depending on Type of Information Needed

Section Describing Method	Method	Method Applicable[1/]		
		Repair or Replacement of Adobe	Durability Investigations	New Construction
3.1	Color Determination	X		
3.2	Determination of Soluble Salts	X	X	X
3.3	Determination of pH	X (If preservation treatment is applied)	X	X (If adobe is stabilized with chemicals).
3.4	Particle size Distribution	X	X	X
3.5	Liquid and Plastic Limits	X	X	X
3.6	X-Ray Diffractometry			
	Comparison of Adobe (fingerprinting)	X		
	Identification of Adobe mineralogy		X	X (If liquid and plastic limits indicates adobe contains expansive clay).
3.7	Microscopy			
	Optical		X	
	Scanning Electron Microscope		X	

[1/] X indicates method is applicable

3.1.1 Description of Munsell Color Charts

Adobe and soil colors can be readily determined in the field by comparison with a color chart. The collection of charts generally used with soils is the Munsell soil color charts [2]. The soil collection is comprised of seven charts which display 196 different standard color chips systematically arranged according to their Munsell notation. A combination of three variables are used to describe colors. These variables are Hue, Value and Chroma. The Hue notation of a color indicates its relation to red, yellow, green, blue, and purple; the Value notation indicates its lightness; and the Chroma notation indicates its strength (or departure from a neutral of the same lightness). The notation of Hue used for soils is the letter abbreviation of the colors of the rainbow (R for red, YR for yellow-red, and Y for yellow) preceded by a numerical range of 0 to 10. Within each letter range, the Hue becomes more yellow and less red as the numbers increase. Value is given a numerical notation which ranges from 0, for absolute black, to 10, for absolute white. The notation for Chroma is also numerical and ranges from 0 (neutral grays) to 8 for soils, increasing in equal intervals. The complete Munsell designation is given in the sequence Hue, Value/ Chroma.

Descriptive color names can also be obtained by using the Munsell Color Charts and should be reported along with the Munsell notation of color, because each system conveys valuable information. The color names convey a visual description of the color of soil which can be visualized without consulting the color charts, while the Munsell notation gives a more exact description of the color of a soil.

3.1.2 Method of Making Color Determinations

The color names and the appropriate Munsell notation of an adobe or soil are obtained by comparison with the color charts. Apertures between closely matching color chips (figure 2) are placed over the sample thereby permitting direct visual comparison. Normally, the match will not be perfect and the closest match should be selected. The color names and the Munsell notations selected for materials from Escalante, Fort Bowie, and Tumacacori are given in table 2.

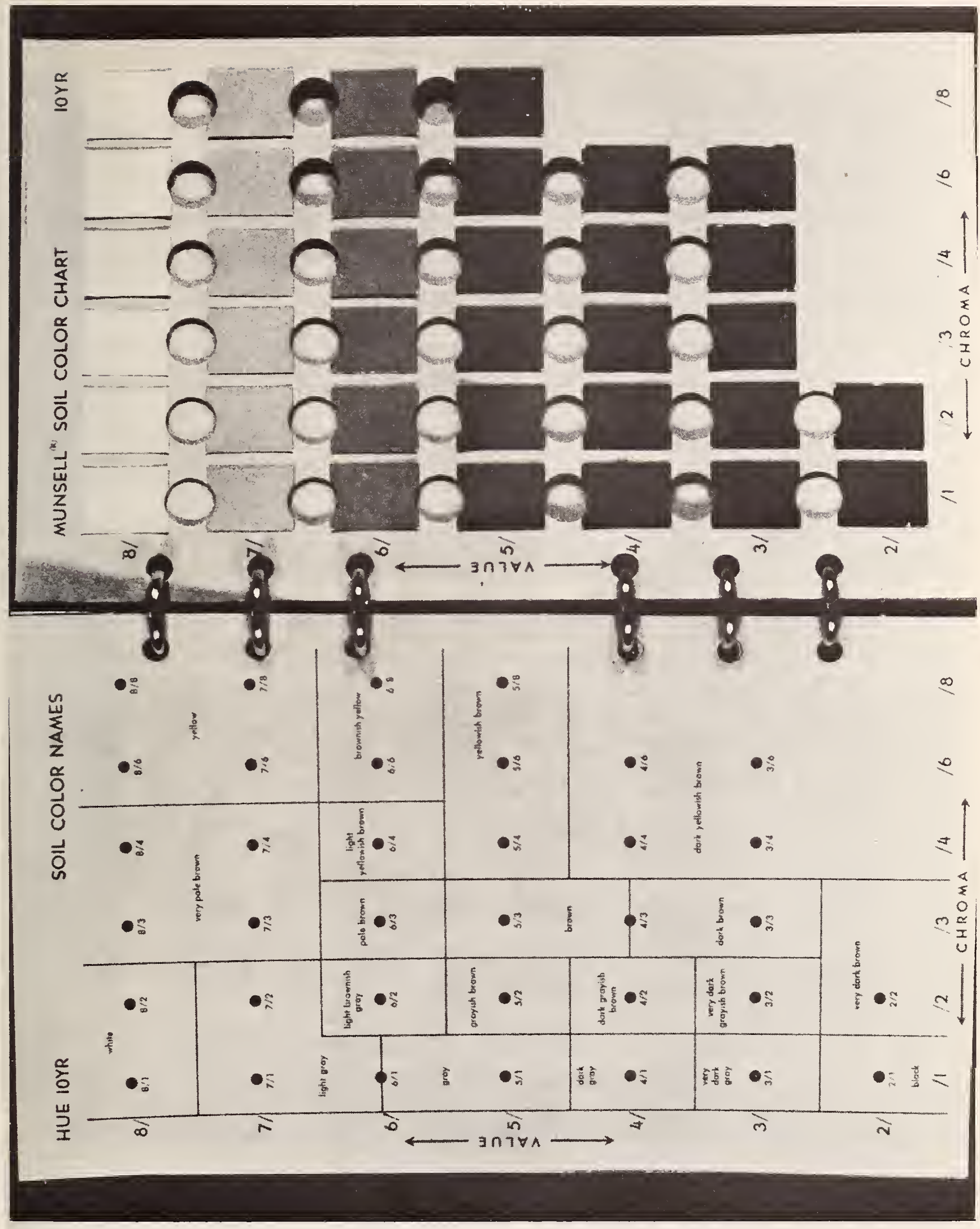

Figure 2. Munsell soil color chart.

Table 2

Color Determination of Adobe and Soil Samples

Sample	Soil Color Name	Munsell Color Notation
Escalante Adobe	Pinkish gray	7.5 YR 7/2
Fort Bowie Adobe	Yellowish brown	10 YR 5/4
Tumacacori Adobe	Dark brown	7.5 YR 4/2
Tumacacori Soil	Dark grayish brown	10 YR 4/2

3.1.3 Factors Affecting Color Matching

An imperfect match may occur when comparing an original adobe with a soil or a freshly prepared adobe of similar composition or even with materials taken from the same approximate site from which the old adobe originated. For example, a perceptible difference was observed in the color notation of the adobe and soil sample from the Tumacacori area (table 2). Some of the causes for such mismatches include the traditional practice of adding straw and small twigs to a soil during the preparation of an adobe, weathering of the adobe, and differences in moisture contents between the adobe and soil. If possible, the soil or new adobe should be compared with an unexposed portion of the original adobe by scraping or rubbing off the exterior weathered adobe layers. It is advisable that the lighting conditions under which a color determination is made should be recorded and, if possible, duplicated when making another determination. For example, if the color of an original adobe is determined in bright sunlight then the color of a freshly prepared adobe should also be determined in bright sunlight.

The color of the adobe of an historic adobe structure may vary throughout the structure. To obtain the best color match between the original and the repair adobe, a series of color determinations should be made around the border of an area to be repaired. Occasionally, an objectional color difference may exist between the original and the repair adobe, and a coloring material used to obtain a closer color match. In such cases, the coloring material should be carefully selected because of the possibility that upon exposure to sunlight its color could change or fade.

3.2 Determination of Soluble Salts

The presence of water-soluble salts in adobe may have a significant impact on the durability of an adobe structure. The build-up of soluble salts, which occurs as the result of the migration of groundwater into an adobe structure, and their subsequent crystallization may result in surface deterioration [1]. The soluble salt content of a sample may also be an important consideration in the determination of the particle size distribution. Because the clay content of a sample is determined by the procedures given in section 3.4, as the difference between the total sample weight and the combined weights of the gravel, sand, silt and organic fractions, the soluble salt fraction is calculated as clay. Accordingly, a high soluble salt fraction can cause significant error in this determination of clay content. For example, the soluble salt content in one sample of adobe from Tumacacori was found to be in excess of 4 percent of the sample weight. Therefore, corrections should be made for the amounts of soluble salts when the clay content is calculated. The determination of soluble salt content is straight-forward and can be completed within 72 hours using the following procedure.

A representative adobe sample is dried for 24 hours at 105 °C and weighed to the nearest 0.01 gram. In most cases a sample weight of about 200 g is adequate to yield a sufficient amount of soluble salt for subsequent analysis, if desired. The sample is then placed in a 600 ml borosilicate glass filter funnel (Buchner type with a fritted filter disc) attached to a 1000 ml filter flask. About 200 ml of distilled water are poured over the sample which is then allowed to slake for a minimum of three hours. The sample is stirred from time to time to aid in dispersion. Then, the water containing the dissolved salts is collected in the filter flask by evacuating the flask with an aspirator. When water stops dripping from the tail of the filter funnel, the aspirator is disconnected. Additional 200 mls of distilled water is then poured over the sample, the slurry is stirred and the aspiration procedure is repeated. This process is repeated four times altogether to remove the soluble salts.

After the sample has been washed four times, the soluble salt solution which has been collected in the filter flask is transferred to a glass beaker which has been weighed to the nearest 0.01 g. The free water is evaporated by warming the beaker on the hot plate, however, the water should not be allowed to boil. Once the water has evaporated, the beaker is placed in an oven at 105 °C for 24 hours to remove any residual moisture from the salts and then reweighed to the nearest 0.01 g. The weight difference is the weight of the soluble salts and this can be calculated as a percentage of the original sample weight. The elemental composition of the soluble salts can be obtained by a variety of techniques including X-ray powder diffraction, flame photometry, X-ray fluorescence or by atomic absorption [3].

3.3 Determination of pH

The behavior of clay particles when wet is largely controlled by the exchangeable cations of the clay and the pH of the clay-water system. For example, a low pH promotes flocculation of the clay particles from suspension, while a high pH can lead to the formation of a stable suspension or dispersion of clay particles [4]. Preservation treatments such as electroosmosis, electrochemical hardening, and preservatives which can produce either basic or acid solutions, may drastically change the pH of an adobe. In the cases of a dimensionally unstable and deteriorating adobe, such a change in pH could improve its performance. However, a change in pH could have an adverse effect on a stable adobe. In addition, pH measurements can be used to determine if repair materials are compatible with the original adobe. It is suggested that the pH of a repair adobe should be within ± 2 pH units of the original adobe. A pH difference of 2 units indicates a difference of a factor of 100 in the concentration of hydrogen ions. Therefore pH measurements provide a rapid method for monitoring the effect of preservation techniques on the properties of the adobe.

The pH of the bulk adobe samples should be measured, i.e., the unfractionated material (figure 1). Portable pH meters with internally contained batteries are available so that field measurements are possible.

3.3.1 Method of Measuring pH

The pH of a soil or adobe can be readily measured in an hour or two using an inexpensive commercially available pH meter. Usually a saturated calomel electrode is used as the reference electrode and a glass electrode as the indicating electrode.

The calibration of the pH meter and electrodes should be checked at least daily using standard buffer solutions. Usually standard buffer solutions and calibration instructions are included when a pH meter is purchased.

Several procedures have been developed for preparing a soil prior to making pH measurements [5] and the following procedure was found to be satisfactory and is recommended for the analysis of adobe.

(1) A sample of 20 grams of the adobe or soil which passed through a No. 40 sieve is mixed with 40 ml of distilled water in a 50 ml beaker.

(2) The mixture is stirred well using a glass rod and then allowed to stand for one hour.

(3) After one hour the electrodes are placed in the mixture and its pH is measured.

3.3.2 Results of pH Measurements

The pH's of samples from Escalante, Fort Bowie and Tumacacori, measured by the methods described herein, are in the range of 8 to 9 units (table 3), indicating that these are slightly basic materials, i.e., the concentration of available hydroxide ions is greater than that of the available hydrogen ions.

Table 3. Results of pH Determinations

Sample	pH	
	Test Conditions[1/]	
	1	2
Fort Bowie Adobe	8.72	8.18
Escalante Adobe	8.19	7.61
Tumacacori		
Adobe	8.12	7.58
Soil	8.25	7.77
Soil-Sand[2/]	8.39	7.97

[1/] Testing condition No. 1 (recommended method); 40 ml of distilled water mixed with 20 gm of sample mixture, allowed to equilibrate for an hour then pH measured. Condition No. 2; 30 ml of distilled water mixed with 20 gm of sample mixture allowed to equilibrate for 24 hours before pH was measured.

[2/] One part soil was mixed with one part Ottawa grade sand.

In another set of measurements the testing conditions were changed: 30 ml of distilled water were mixed with 20 gm of the samples, and the mixture was allowed to stand for 24 hours. The measured pH's were lower than those measured by the recommended procedure. This indicates the necessity of adhering to a standard procedure when characterizing adobes and soils.

3.4 Determination of Particle Size Distributions

The particle size distribution of an adobe is one of the main factors controlling its durability [1, 6-9]. A good adobe will consist of between 60 and 80 percent sand, 20 to 40 percent silt and clay, with little or no gravel present. The clay and, to a lesser extent, the silt portion of an adobe act as the binder forming a matrix in which the sand particles are embedded. A large proportion of clay could cause excessive swelling in the adobe through absorption of moisture by the clay and excessive shrinkage when the clay dries. Clearly, knowledge of the particle size distribution of an historic adobe can provide valuable insights to some of the problems which will be encountered in its preservation. Additionally, in repairing deteriorated adobe, the repair material should have a particle size distribution either close to that of the original adobe, or a distribution which is in the range found for durable adobes. Therefore, the particle size distribution is one of the most important properties of an adobe which should be characterized.

As indicated in the flow chart for the analysis of adobe (figure 1), fractionation of the adobe into different particle size fractions is necessary to obtain the particle size distribution and also to identify the mineralogy of the various fractions. The procedures for fractionating adobe and for obtaining the particle size distribution are given in this section.

3.4.1 Method for Determining Particle Size Distribution

Several methods are available for the determination of the particle size distribution in soils. These include ASTM D1140, "Amount of Material in Soils Finer than the No. 200 Sieve," [10] and ASTM D422, "Particle Size Analysis of Soils" [11]. The present method is a modification of these ASTM standard methods. The modifications simplify the analytical procedure while permitting retention of the various size fractions for further analysis.

The method described herein can be used to separate the gravel, sand, silt and organic fractions which are found in adobe. These size fractions are defined as follows:

gravel:	particle diameters larger than 2 mm
sand:	particle diameters between 20 microns (0.02 mm) and 2 mm
silt:	particle diameters between 2 and 20 microns
clay:	particle diameters less than 2 microns.

Separation of size fractions within the sand-gravel size ranges relies on sieving, while separation of the clay and silt fractions from the coarser fractions, as well as from each other, relies on particle sedimentation.

3.4.1.1 Sample for Analysis

The adobe sample selected for analysis should, if possible, be in the form of a well consolidated agglomeration. Powdery material, which may collect at the bases of severely eroded areas, is generally not representative of the structure. Similarly, consolidated material which has been eroded by rain or flowing water should not be considered representative. In the former case, a sample would probably be deficient in coarse particles while in the latter it may be deficient in the finer clay and silt fractions.

The mass of the sample to be analyzed is largely determined by the size of the largest particles present. For example, ASTM D422 recommends the following sample sizes:

Nominal Diameter of the Largest Particles		Approximate Minimum Sample Mass
mm	inches	grams
9.5	(3/8)	500
19.0	(3/4)	1000
25.4	(1)	2000
38.1	(1-1/2)	3000

However, from the standpoint of characterizing the particle size distribution in the size range below about 9.5 mm, sample masses in this fraction need only range from 65 grams for clay-silt soils to 115 grams for sandy soils. If the adobe to be analyzed is removed from an historically significant structure, it is generally desirable to limit the mass of the collected sample. Based on the analyses of adobe from three historical structures (Tumacacori, Fort Bowie, and Escalante), a sample mass between 150 and 300 grams was found to be adequate.

3.4.1.2 Dispersion of Sample and Separation of Organic Material and Gravel

Once a suitable sample has been selected, it is crushed lightly using a mortar and a rubber covered pestle, the sample is weighed to the nearest 0.01 gram and dried at 105 °C for about 24 hours. Then it is again weighed to the nearest 0.01 gram. The moisture content of the sample is then calculated and expressed in percent based on the dry weight. After weighing, the sample is transferred to a 600 ml high-form beaker, covered with about 200 ml of a freshly prepared sodium hexametaphosphate solution, stirred until the soil is completely wet, and allowed to stand overnight. The sodium hexametaphosphate solution is prepared by adding 40 grams of sodium hexametaphosphate crystals to one liter of distilled or deionized water. Distilled or deionized water should be used exclusively throughout this procedure. The sodium hexametaphosphate is used to disperse or separate the clay particles. To ensure complete dispersion of the adobe it is usually necessary to agitate the mixture of adobe and dispersion solution by mechanical means. However, prior to this step it was found advisable to hand stir the mixture using a stirring rod and then filter it through a number 8 or number 10 sieve, collecting the filtrate. This removes the larger particles which are retained on the sieve as well as most of the twigs and larger pieces of organic material which may be present. Removal of the larger particles facilitates mechanical stirring. The organic material retained on the sieve is removed with a tweezers from the gravel particles after the fraction has been dried at 105 °C. The weight of the gravel is measured and recorded or further fractioned using coarse sieves if desired. The organic fraction should be retained for identification purposes.

If a significant amount of organic material is washed through the sieve (no. 8 or 10), the majority of it may be retrieved when the contents of the pan are transferred back into the 600 ml beaker by adding about 100 ml of water, stirring and allowing the sediment to settle. The organic fraction, which is less dense than water, floats on the surface, and can be removed with a tweezers or a tea strainer. This organic fraction may then be dried and combined with the other organic fraction, weighed, and retained for further analysis. Usually, most of the organic material is removed by this method. The unremoved portion is usually sufficiently small so it does not introduce significant errors into the particle size distribution analysis.

Prior to further separation of the sand, silt and clay size fractions it is necessary to completely disperse the particles. While ASTM D422 recommends the use of a high speed blender, the use of a magnetic stirrer was found to be adequate. Dispersion was accomplished by stirring the mixture at high speed for 15 minutes using a 5 cm long Teflon coated stirring bar. The only disadvantage to this dispersion technique is that any magnetic fraction of the sample will adhere to the stirring bar. However, this magnetic material is easily rinsed from the bar with a wash bottle. Then the amount of magnetic material is estimated by drying the material at 105 °C and weighing it.

3.4.1.3 Partial Separation of Silt-Clay Fraction

Partial separation of the silt-clay fraction may be accomplished by adding sufficient water to cover the sample with a 8-10 cm high column of water, dispersing the sample by magnetic stirring, allowing the solid material to settle for a prescribed period and then siphoning the clay-silt fraction which remains in suspension into a large beaker. These fractions should be retained for further separation. If this procedure is carried out 3 or 4 times prior to sieving, a substantial amount of the silt-clay fraction can be separated, thus minimizing clogging of the finer sieves.

The appropriate settling time can be determined using the nomograph given in figure 3, in which the settling time in cm per second is plotted against the particle diameter in microns for various temperatures. For example, diameters of 20 microns or less, require a settling time of 30 s per cm if the solution is at a temperature 18 °C. Thus if the column of water is 10 cm high, the settling period should be 300 s or 5 minutes. This allows the particles having diameters larger than 20 microns to settle out of the solutions. However, because the particles with diameters smaller than 20 microns are initially relatively uniformly distributed throughout the solution, those near the bottom of the column of water will settle out before 300 seconds and will not be removed by siphoning. As a consequence it is necessary to carry out this procedure a number of times, until little change in the color of the solution is observed.

3.4.1.4 Sieve Fractionation and Determination of Coarse Particles

Once a portion of the silt-clay fraction has been separated from the larger particles, the contents of the beaker are poured into a set of sieves for size fractionation of the coarser material. The sieve sizes used are as follows:

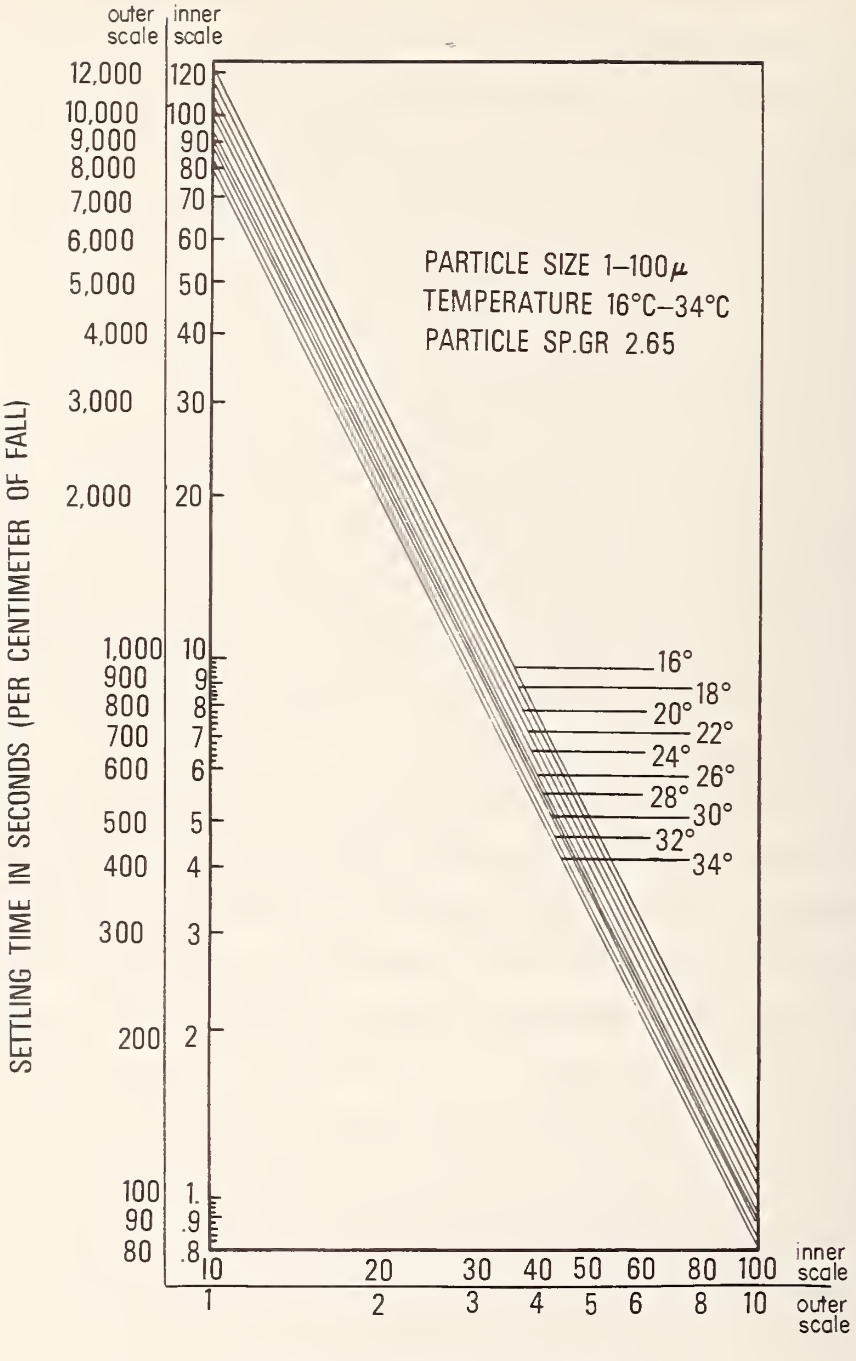

Figure 3. Particle settling time. (Nomograph was developed by Tanner and Jackson [12].)

Sieve No. [13]	Size of Opening in Sieve Screen
No. 16	118 mm
No. 30	600 μm
No. 50	300 μm
No. 100	150 μm
No. 200	75 μm
No. 270	53 μm
No. 400	38 μm

A catch pan is placed beneath the No. 400 sieve. The material retained on the No. 16 sieve is washed with water to remove any fine particles which are in turn washed on to the No. 30 sieve. The No. 16 sieve is then removed from the stack of sieves and placed in an oven at 105 °C for about an hour. The particles are then transferred to a weighing dish and weighed. This procedure is repeated in turn until the contents retained on each sieve in the series have been thoroughly rinsed, dried, and weighed. The material in the catch pan, which consists of the size fraction below 38 μm, is then transferred to a beaker. This material is then stirred and fractionated by the previously described settling time technique to separate the minus 20 μm silt-clay fraction from the plus 20 μm minus 38 μm fraction. This minus 20 μm fraction is then dried and the silt-clay fraction combined with that obtained previously (section 3.4.1.3).

After the various sieve fractions have been dried they are weighed and the weight of each size fraction recorded. Care should be taken to ensure that minimal material remains entrapped in the mesh of each sieve. The weight of the clay fraction is calculated as the difference between the total sample weight and the combined weights of the silt, sand, gravel and organic fractions. As a consequence, particles that remain entrapped in the sieves are not weighed as members of the various sieve size fractions and will be calculated as part of the clay fraction. Most of the particles entrapped in the finer sieves may be removed by brushing the inverted sieve with a stiff brush. Particles entrapped in coarser sieves may be removed with a pin or tweezers.

3.4.1.5 Separation of Silt from Clay

Prior to separating the silt from the clay fraction, the remaining solution (from 3.4.1.3) was usually left undisturbed for 2 to 3 days thereby allowing the fine clay particles to settle. However, lengthy settling is necessary only if the analyst desires to retain the clay fraction for additional analysis. After several days of settling, most of the solution may be siphoned off and discarded with minimal loss of the clay fraction. This reduces the volume of the solution, which may be as much as 4 liters, to a few hundred milliliters, and also reduces the concentration of soluble salts (initially present in the adobe) and the concentration of the sodium hexametaphosphate dispersant. It was found convenient to allow the solution to settle sufficiently well so that after siphoning off the clear portion, the remaining mixture could be transferred to a 600 ml high-form beaker.

Separation of the silt from clay can be carried out by the same procedure as was described for separating the silt-clay fraction from the coarser particles. In this case, however, because the particle diameters are smaller, longer settling times are required. For example, the settling rate for a 2 micron clay particle in a solution at 18 °C is 3000 seconds per cm (figure 3). Thus, if the column of water above the sediment is 10 cm high, the settling time is 8 hours 20 minutes. As a consequence, several days are required to adequately separate the silt and clay fractions. When this separation is complete, the silt is dried at 105 °C and weighed. The clay fraction is allowed to sit undisturbed until the solution becomes clear. The solution is then siphoned off and the clay sediment is dried at 60 °C and retained for further analysis, such as identification of the clay mineralogy by X-ray diffraction methods. The clay fraction should be dried at 60 °C rather than 105 °C to avoid the possibility of irreversible dehydration of the clay minerals.

3.4.2 Useful Information from Particle Size Determinations

Two examples of useful information obtained from the determination of the particle size distribution are provided by an analysis of adobes from Fort Bowie and Tumacacori. The first example involves the adobe from Fort Bowie (table 4). This adobe contains about 25 percent, by weight, of particles larger than 2.4 mm (gravel-sized particles) and about 60 percent of particles between 2.4 mm and 300 μm (coarse sand). As a consequence, when the adobe walls become damp and the cohesiveness of the silt-clay binder is reduced, the gravel and coarse sand particles tend to settle downward disrupting the matrix. This matrix can be readily eroded by rain and accumulated run-off water, which probably accounts for the observed rapid deterioration of the walls at Fort Bowie.

As a second example, a variety of adobe samples from the church at Tumacacori were analyzed along with a soil sample obtained from the vicinity of the church. The range of particle size distributions of the adobe are compared with the distribution of the soil in table 4. The distribution for the adobe samples are in the range characteristic of durable adobe [1]. However, the soil is rich in silt and clay. This soil is probably not dimensionally stable, i.e., it may expand when wet and shrink upon drying to a larger extent than the adobes. The addition of 150 parts of sand, on a weight basis, to 100 parts of the soil will increase the proportion of sand to about 70 percent and reduce the silt and clay content to about 30 percent. This will produce an adobe material with a particle size distribution in the approximate range of the adobe from Tumacacori.

Table 4. Measured Particle Size Distribution

Type of Particle	Amount (percent on weight basis)		
	Adobe from Fort Bowie	Adobe from Tumacacori [1]	Soil from Tumacacori Area
Gravel	25	5-8	2
Sand	59	65-80	24
Silt	9	8-12	26
Clay	7	8-12	46

[1] Range based on analyses of 3 different adobe samples from the Tumacacori church.

3.5 Liquid and Plastic Limits

The rheological behavior of a soil changes with a change in water content [14,15]. At high water contents, soils are suspensions, with the flow properties of liquids. As the water content decreases, the soil becomes consecutively pastelike, sticky, then plastic, and finally at a low water content the soil has the properties of a solid. The physical state of a soil at a given water content is termed its consistency, which is a measure of the resistance of a soil to flow. Different soils usually have different consistencies at the same water contents; therefore, the determination of consistency provides information about both the constituents and the rheological properties of a soil or adobe.

The specific consistencies that are used for soil classification purposes are the liquid and plastic limits.[2] The liquid limit (LL) is the water content, in percent by

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[2] Sometimes called the Arterberg limits, in recognition of his early work on soil consistency.

weight, at which a soil ceases to behave as a liquid and begins to exhibit the behavior of a plastic. The plastic limit (PL) is the water content, in percent by weight, below which the soil is not plastic and crumbles when worked. The plasticity index of a soil is also sometimes given in reports, and is defined as the liquid limit minus the plastic limit. The plasticity index largely depends on the amount of clay present; the strength of a soil increases as the plasticity index increases. However, the tendency of a clay to expand when wet and shrink when dried also increases as the plasticity index increases.

3.5.1 Determination of Liquid and Plastic Limits

Determination of the liquid and plastic limits requires only an hour or two of actual work and the final results are obtained within 24 hours. One pound (454 grams) of bulk adobe will normally be sufficient for the determinations. The determinations are made using fractionated material (figure 1).

The liquid limit of a soil or adobe can be determined by following the methods given in ASTM D423 [16]. The determination essentially consists of:

(1) Separating and collecting the portion of the sample passing a No. 40 (425 μm) sieve. About 100 grams of the fine material is required.

(2) Mixing this fine material with distilled water, if available.

(3) Placing this mixture in the cup of a mechanical device which jars the sample in a reproducible way (figure 4).

(4) A groove of about 1/2 inch (1.27 cm) is made in the moist sample with a standard grooving tool (figure 4).

(5) Then the cup is dropped repeatedly from a standard height (0.3937 in (exactly 1 cm)) until the sample flows together in the groove. The liquid limit is defined as the water content at which 25 of the standardized blows are required to close the groove over a distance of about 1/2 in (1.27 cm). Because it would be time consuming to add exactly the correct amount of water, several additions of water are made, proceeding from the dryer to the wetter condition of the soil. At each addition of water, the number of blows required to close the groove is recorded. The purpose of this procedure is to obtain samples of such consistency that the number of blows will be above and below 25. The range of data should be between 10 to 40 blows. A best-fit straight line is

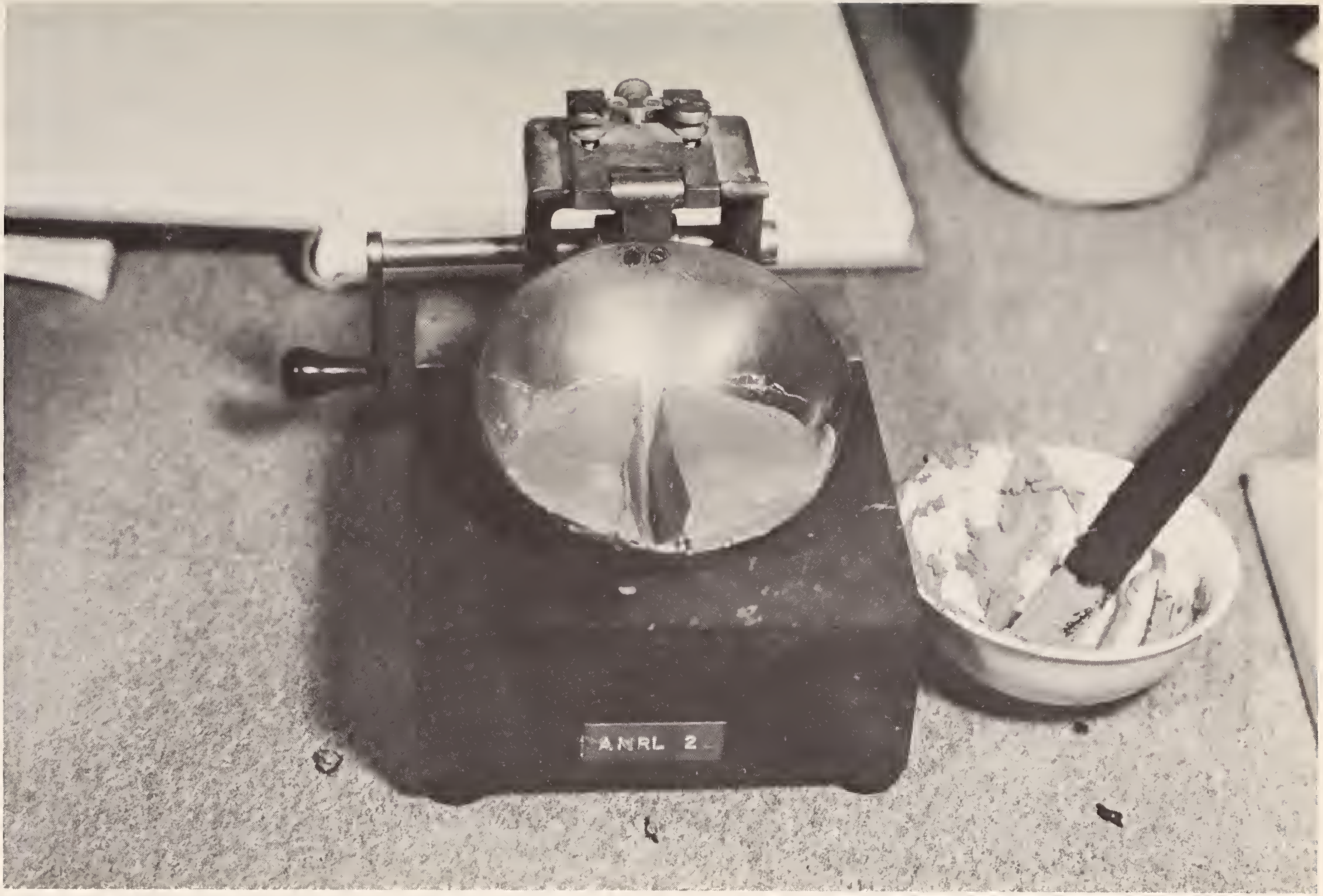

Figure 4. Mechanical device used for measuring the liquid limit of soils

drawn through the data and the water contents at 25 blows is estimated (fig. 5). The water contents are measured weighing the moist specimen then drying it at 110 °C until at constant weight (overnight drying is usually sufficient).

The plastic limit can be determined by the methods given in ASTM D424 [17]. In this method, a wet sample is dried by being rolled back and forth over a ground glass plate or a piece of absorbent paper until the sample crumbles at a diameter close to 1/8 in (3.2 mm) (fig. 6). The rolling promotes the drying of the sample. The sample is weighed and then oven dried at 110 °C until at constant weight. The water content of the sample is defined as its plastic limit.

Both the liquid and plastic limits should be reported in percent, as calculated by the formula:

$$\text{percent moisture content} = \frac{\text{weight of moisture}}{\text{dry weight of soil}} \times 100.$$

Practice is required to obtain reproducible results in measuring both the liquid and plastic limits. With practice, standard deviations should be within 10 percent of the average values [18-19]. Therefore, it is recommended that triplicate determinations be made and the average and standard deviation of the data reported.

3.5.2 Information from Liquid and Plastic Limit Determinations

The liquid and plastic limits determined as described herein, can be useful in the process of characterizing the properties of particles smaller than 425 μm. However, it is the clay size particles i.e., those smaller than 2 μm which have the greatest effect on the magnitude of these limits. The limits for some clays are listed in table 5. Clearly, a clay which is predominantly montmorillonite can be distinguished from other clays such as kaolinite, illite, chlorite on the basis of its liquid and plastic limits. The liquid and plastic limits of some samples from Escalante, Fort Bowie and Tumacacori are given in table 6. Based on these data it can be concluded that little, if any, montmorillonite is present in these samples. Note that the adobe sample from Fort Bowie has a small plastic index, which occurs with soils having small amounts of fine particles. Furthermore, the

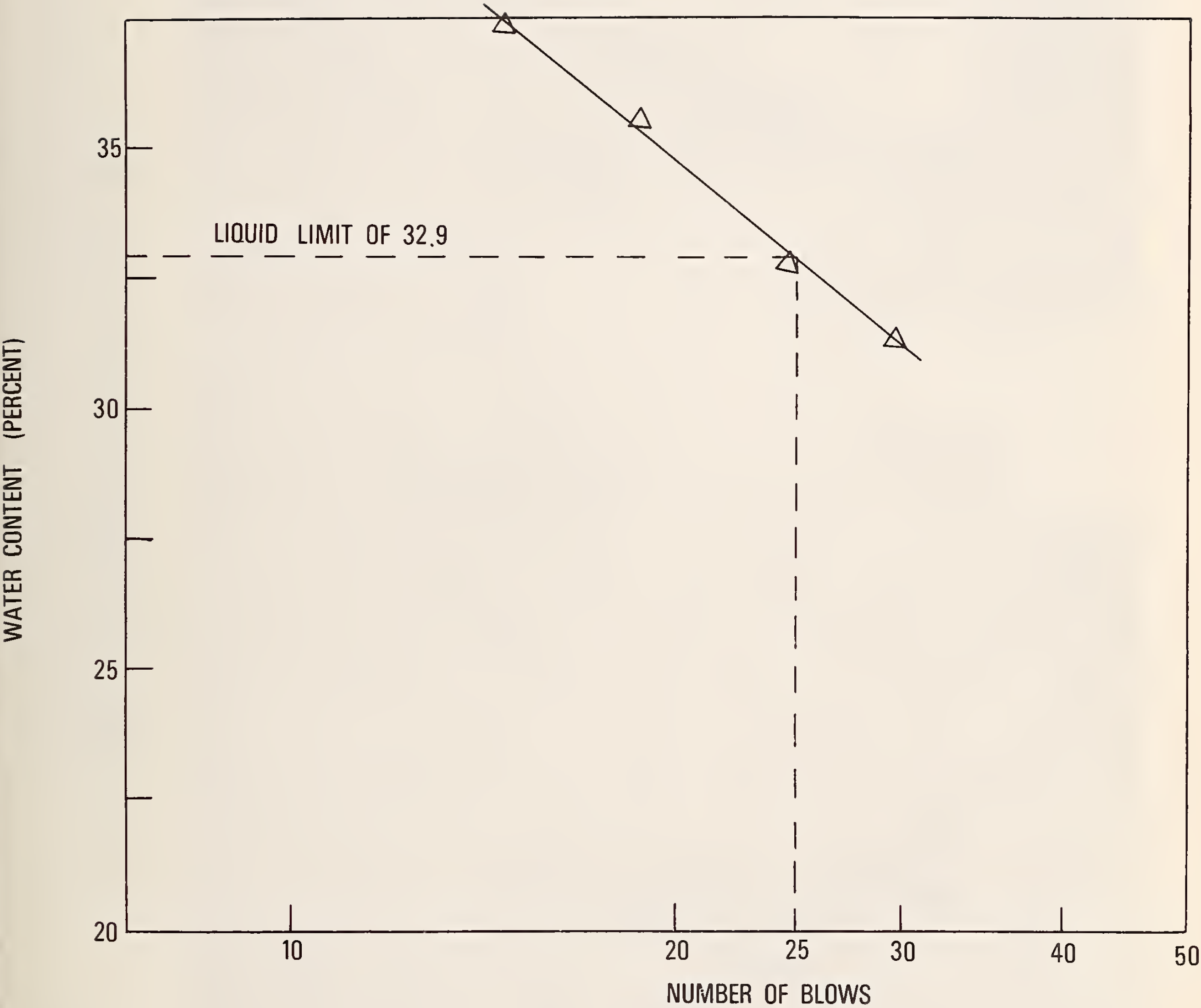

Figure 5. Plot of a liquid limit determination. The water content at 25 blows is estimated by constructing a straight line between data points denoted by the symbol Δ.

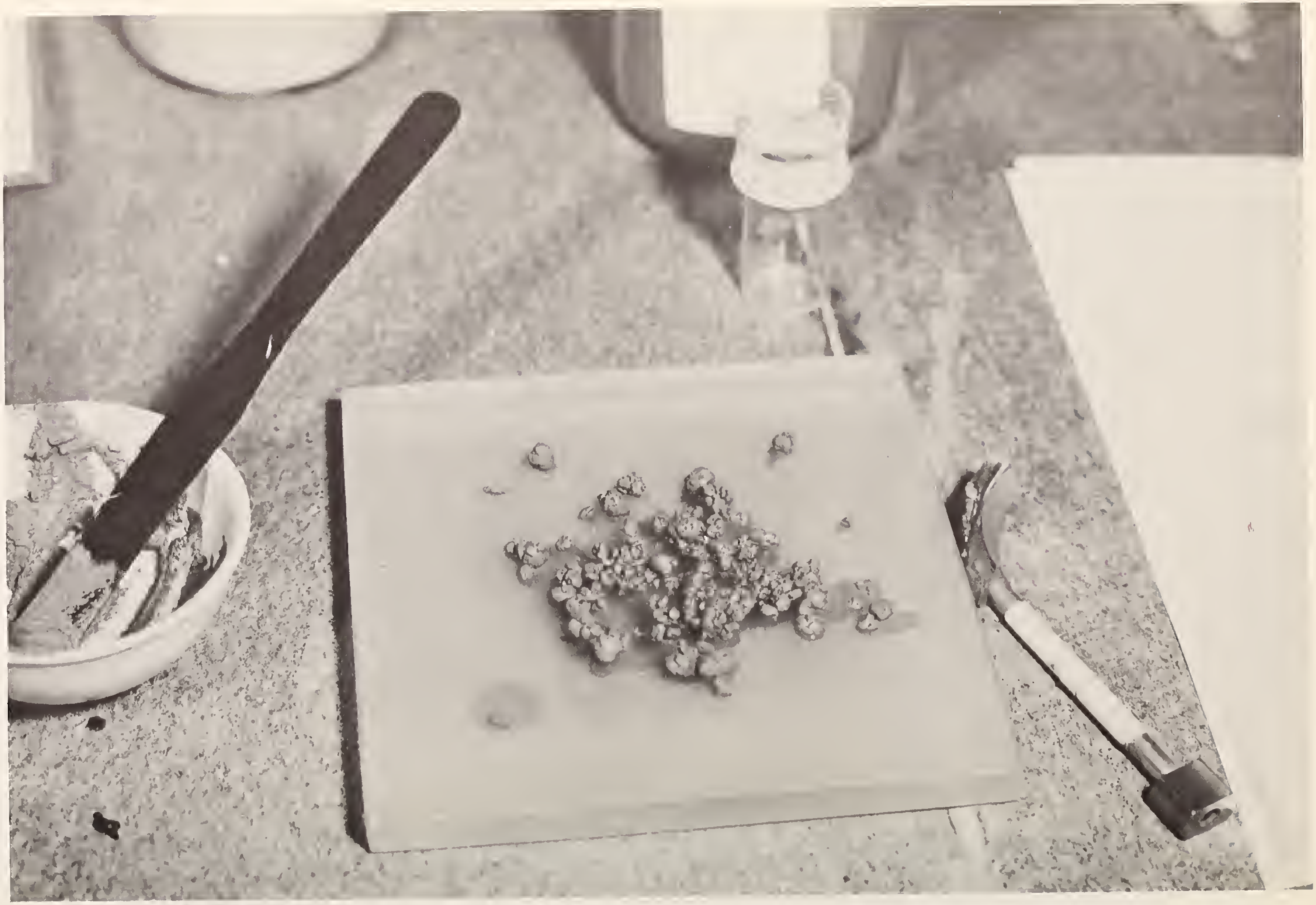

Figure 6. Determination of plastic limit. The plastic limit is defined as the water content of a soil sample which crumbles upon rolling at a diameter close to 1/8 in (3.2 mm).

Table 5. Compilation of Liquid and Plastic Limits for Clays [14,20]

Clay	Liquid Limit (percent)	Plastic Limit (percent)
Kaolinite-Na	52	26
Kaolinite-K	73	36
Illite-Na	61	34
Illite-Ca	90	40
Montmorillonite-Na	700	97
Montmorillonite-Ca	177	63
Attapulgite	171	145
Halloysite	94	65
Allophane (undried)	231	136
Allophane (dried)	85	78
Chlorite	44-47	36-40

Table 6. Measured Liquid and Plastic Limits of Adobe and Soil Samples [1/]

Sample	Liquid Limit (LL) (percent)	Plastic Limit (PL) (percent)	Plasticity Index (PI) [2/] (percent)
Escalante			
Adobe	32.9	21.8	11.1
Fort Bowie			
Adobe	20.2	14.6	5.6
Tumacacori [3/]			
Adobe T-011	23.5	16.0	7.5
Adobe T-012	24.8	21.6	3.2
Adobe T58	42.5	31.7	10.8
Soil	36.5	20.7	15.8

[1/] For particles less than 425 μm.

[2/] PI = LL - PL.

[3/] Adobe specimens taken from different parts of the church at Tumacacori.

small plastic limit for this adobe indicates that it has poor cohesion and poor resistance to cracking. Liquid and plastic limits are significantly affected by several factors [4,9] including the presence of organic matter (straw, manure, etc.), type and amount of soluble salts, and by the specific exchangeable cation of the clay (table 5). Therefore, while the liquid and plastic limits give useful information that can be used to supplement the mineral identification of clays obtained from X-ray diffraction and optical microscopy techniques, they do not provide definitive mineralogical identification.[3/]

Important information concerning the dimensional response of a clay to moisture is provided by the value of the plasticity index [4,20]. Higher plasticity indices are accompanied by larger expansions upon wetting and larger shrinkages upon drying. For example, a clay with a plasticity index of 50 percent could have a total expansion, when saturated with water, of between 33 and 40 percent, while a clay with a plastic limit of 10 percent may expand by only 4 to 10 percent (table 7). Therefore, usually the closer that adobes match in their plastic indices the more compatible are their dimensional responses to moisture. The soil from Tumacacori has a plastic index of 15.8 percent (table 6), which is probably sufficiently close to the plasticity indices of the adobe samples from Tumacacori to permit its use, provided sand is added (see section 3.4.2). Of course, as close a match in plasticity indices as possible would be desirable.

Another use of the liquid limit determination is to obtain an estimate of the amount of mix water required to make good adobe brick, and adobe test specimens. Usually the amount of required water will be less than the water contents at the liquid limit as the adobe will contain particles larger than 425 µm. Particles larger than 425 µm absorb much less water than clay-size particles and the reduction in mixing water was found to be somewhat proportional to the percentage of material larger than 425 µm.

[3/] Except in the case of allophane clay minerals which are identified on the basis of the effects of drying on their plastic limits and plastic indices. However, it is doubtful that allophane clays will be found in abundance in the arid southwestern region of the United States.

Table 7. Relationship between Total Expansion and Plasticity of Soils [20]

Plasticity Index of Soil[1/] (percent)	Total Expansion (percent)
10	4.5 - 10.0
20	13.5 - 18.7
30	21.4 - 28.0
40	28.0 - 35.0
50	33.0 - 40.0

[1/] For particles smaller than 2 µm.

3.6 Determination of Minerals in Adobe by X-ray Diffractometry

The mineral composition and particle size distribution of adobes and soils largely controls their mechanical properties and durabilities. For example, an adobe containing a montmorillonite type of clay would swell excessively when wet and shrink upon drying, which would impair its durability. Therefore, such a material should not be used to replace or repair deteriorated adobe. If possible, the material used to replace or repair the adobe of historic structures should have the same mineral composition as the original adobe.

3.6.1 Method of Obtaining and Interpreting Powder Diffraction Patterns.

X-ray powder diffractometry is a rapid method for identifying the mineralogy of the constituents of adobe materials and soils. It is also useful in ascertaining if a soil selected for repair purposes has the same mineralogical features as the original adobe. Because the diffraction patterns reflect the types and amounts of the various minerals present, they serve to characterize or "fingerprint" a particular adobe. Therefore, by comparing the pattern of the original adobe with that of the repair material the analyst can ascertain if minor or gross differences exist, without identifying the mineralogical composition of the adobe.

The type of the X-ray powder analysis selected will depend on the information desired. Usually the "fingerprint" will be sufficient for the analysis of adobes. In some cases, a more detailed analysis in which the mineralogical composition is determined may be desired. However, identification of the clay mineralogy can be difficult, even for an experienced analyst. The procedure for making either a fingerprint or detailed analysis are discussed to acquaint the preserver with these techniques and with the type of information they provide.

It is assumed that the reader is familiar with the use of standard X-ray equipment. The diffractometric powder technique is recommended. Detailed discussions of theory and of the equipment and its operation are given by Klug and Alexander [21]. Cu radiation is commonly used.

In practice, a subsample of adobe is crushed and ground using a mortar and pestle. Although an average particle size of less than 10 μm is preferred for intensity measurements, it may not be desirable to grind the material to that extent. Excessive grinding may destroy the structure of layer silicates such as the micas and most of the clay minerals. In order to avoid orientation of the crystallites and hence to obtain diffraction intensities more proportional to the amount of a mineral present, a special aluminum specimen holder is used. Figure 7 shows the manner in which the specimen is prepared by gently shaking the powder specimen so that the powder falls into the cavity formed by the holder and a glass slide. This technique is used by the Joint Committee on Powder Diffraction Standards (JCPDS) Associates at the National Bureau of Standards and has proven very effective.

The diffraction angles (2) of the diffractometer traces may be converted to _d_ spacings in Ångstrom units by reference to tables such as those of Fang and Bloss [22]. The experimentally obtained _d_ spacings are then compared with _d_ spacings of known compounds until identification is achieved. The Powder Diffraction File of the JCPDS is a standard reference for powder diffraction analysis. A laboratory doing X-ray analysis should have the powder diffraction file and appropriate search manuals.

3.6.2 Bulk Analysis

X-ray powder diffractometry provides the most rapid method for the bulk characterization of adobe materials. For example, the bulk X-ray analysis of several adobe specimens from Tumacacori showed that all contain quartz, feldspar (both alkaline and calcic), biotite mica, muscovite mica, amphibole, gypsum, rutile, hematite, illite clay, kaolinite clay and calcite. In general, estimates of amounts of minerals present in a complex mixture on the basis of diffraction intensities are semiquantitative at best because of differences in mass absorption coefficients, degree of crystallinity, lack of random particle orientation, variation in sample weights, degree of hydration and other variables. Quantitative analysis is sometimes

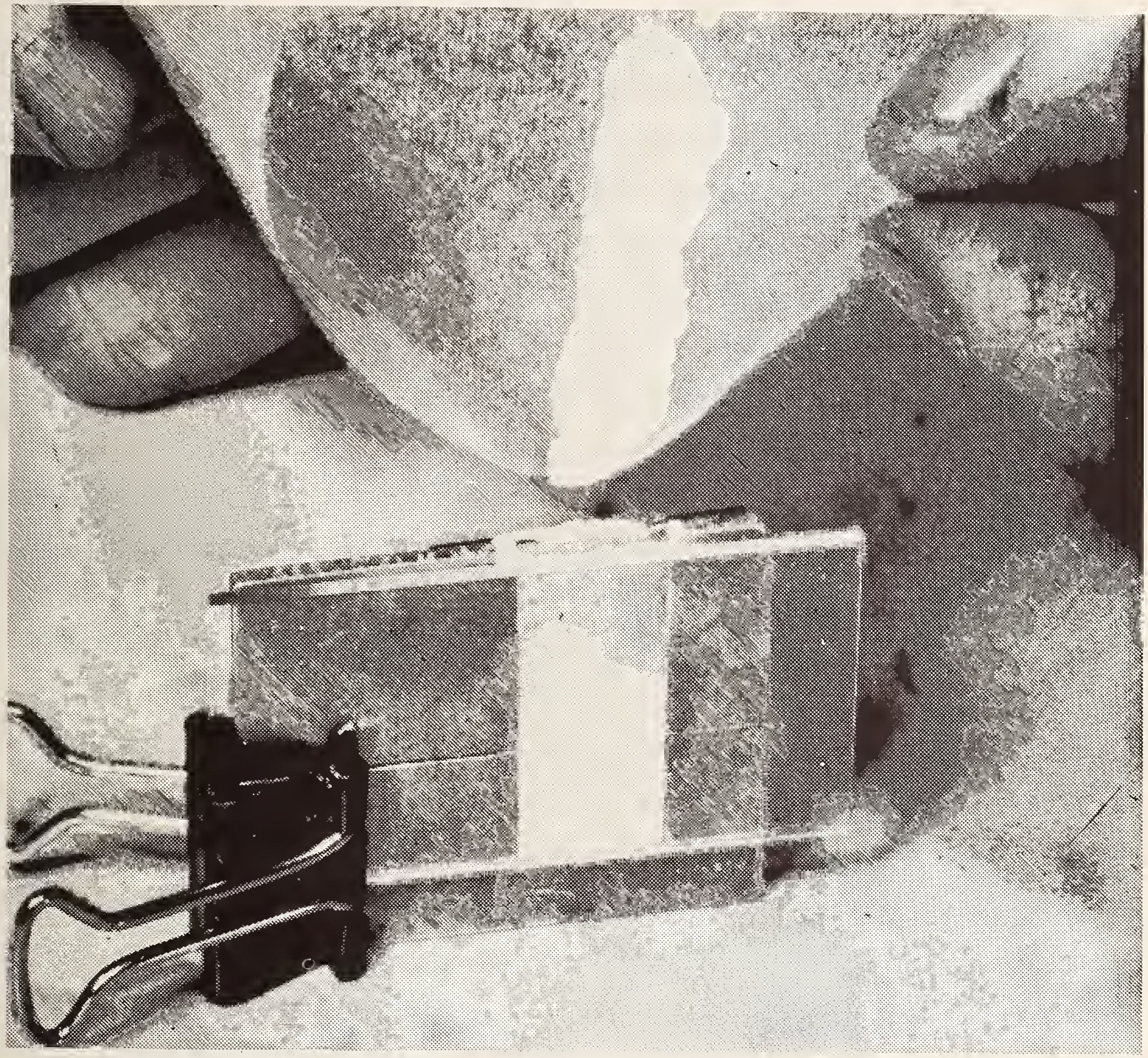

Figure 7. Aluminum holder for preparing powder samples for X-ray analysis.

possible, however, and a detailed discussion of the subject is given in reference 21.

References 4 and 23 are particularly good sources of information on the properties and identification of minerals in soils.

3.6.3 Identification of Clay Minerals

With exception of the clays, the minerals listed above are mainly primary minerals formed during the cooling of a magma. Their resistance to chemical weathering is roughly inversely proportional to the sequence in which they were formed, or the reverse of Bowen's reaction series [4]. The clay minerals are one product of chemical weathering. In the discussion which follows, the term "clay" will refer to the less than 2 μm size fraction of the adobe and will therefore include both the clay minerals and clay-sized minerals. Before analysis, the clay fraction (figure 1 and section 3.4) must first be separated from particles larger than 2 μm by sedimentation.

Precise identification of clay minerals is a specialized and sometimes complex X-ray powder diffraction problem. Therefore, it is recommended that standard texts be consulted when attempting identification of clay minerals. A useful discussion of experimental methods is given by Brindley in [24]. This book is also an excellent source of X-ray data on the clay minerals and many of the common soil minerals, as is the text by Grim [25].

Brindley [24] points out that on the order of a dozen X-ray diffractograms may be required for the definitive identification of a particular clay sample, including patterns of oriented, unoriented, glycolated and heated samples. Oriented sample mounts are prepared by evaporation of a clay-water suspension on a Vycor glass slide [26], or by deposition by centrifugation on a porous ceramic plate [27]. Basal spacings are obtained from oriented specimens and are most useful in clay identification. The specimen should be scanned from 60 to 2 degrees at a rate of 0.5 degrees per minute. A slower scanning rate may be found desirable when a definitive identification is needed. Diffractograms of both oriented and unoriented specimens should be recorded after specimens have been air dried and then placed in a desiccator for 24 hours. Ethylene glycol can enter between the layers of certain types of clays, causing a controlled separation of the layers, which can be diagnostic. The specimens are subjected to glycolation by heating them in a desiccator containing a beaker of ethylene glycol. The desiccator should be placed in an oven at 60 °C and held for five hours. If the clay has been deposited on porous tile by centrifugation, the glycol may be applied directly to the sample surface. A 25 percent glycol-water solution is frequently used. After treatment, the specimen is air dried and the X-ray diffraction pattern obtained.

As discussed by Brindley [24] and by Carroll [26] a variety of techniques are often required for identification of the clay minerals. In addition to glycolation, a heat treatment of the clays may be necessary. Two very useful tables of data on clay minerals have been compiled by Carroll. The first (table 8) permits a preliminary identification of most clay minerals on the basis of their X-ray diffraction data. The second (table 9) gives characteristic reactions and structural changes of clay minerals when heated in a muffle furnace. By these methods, the predominant clay minerals of adobe materials may be identified. For example, the diagnostic method for montmorillonite clay is to record the basal d spacings (001), which are between 14 to 15 Å for the montmorillonite group. If the clay does belong to the montmorillonite group, this spacing will increase to about 17 Å upon treatment with glycol and collapse to 9 Å when the sample is heated at 300 °C. It is difficult to identify small amounts of montmorillionate because x-ray diffraction lines of this clay group are weak.

3.7 Analysis of Adobe by Microscopy

The binocular microscope, the polarizing microscope and the scanning electron microscope can be used to analyze the microfabric of adobes and soils. The term "fabric" refers to the arrangement of particles, particle groups and pores in an adobe or soil. The microfabric are those features which require a microscope for study. This includes information on the shape, packing, texture, and size distribution of particles. In addition, information on the type and distribution of minerals can be obtained.

The information which can be obtained from use of each of the three microscopes by an experienced petrographer is discussed in this section.

3.7.1 Binocular Microscope

The size, shape and arrangement of pores and voids, the presence of deposits in pores, the size, shape and packing of coarse and fine aggregates, and the presence of organic material such as twigs and straw are the main features that may be observed macroscopically by a low-power binocular microscope using reflected light. Many of the fine details of the microfabric of consolidated adobe specimens are obscured by the silt-clay matrix, and their

Table 8. X-ray Identification of the Principal Clay Minerals (<2 Microns) In An Oriented Mount of a Separated Clay Fraction from Sedimentary Material [1]

Mineral	Basal d spacings (001)	Glycolation effect; 1 hour, 60 °C	Heat effect, 1 hour
Kaolinite	7.15 Å (001); 3.75 Å (002)	No change	Becomes amorphous 550-600 °C
Kaolinite, disordered	7.15 Å (001) broad; 3.75 Å broad	No change	Becomes amorphous at lower temperatures than kaolinite
Halloysite, $4H_2O$	10 Å (001) broad	No change	Dehydrates to $2H_2O$ at 110 °C
Halloysite, $2H_2O$	7.2 Å (001) broad	No change	Dehydrates at 125-150 °C; becomes amorphous 560-590 °C
Mica, 2M	10 Å (002); 5 Å (004) generally referred to as (001) and (002)	No change	(001) becomes more intense on heating but structure is maintained to 700 °C
Illite, 1Md	10 Å (002), broad, other basal spacings present but small	No change	(001) noticeably more intense on heating as water layers are removed; at higher temperatures like mica
Montmorillonite Group	15 Å (001) and integral series of basal spacings	(001) expands to 17 Å with rational sequence of higher orders	At 300 °C (001) becomes 9 Å
Vermiculite	14 Å (001) and integral series of basal spacings	No change	Dehydrates in steps
Chlorite, Mg-form	14 Å (001) and integral series of basal spacings	No change	(001) increases in intenisty; >800 °C shows weight loss but no structural change
Chlorite, Fe-form	14 Å (001) less intense than in Mg-form; integral series of basal spacings	No change	(001) scarcely increases; structure collapses below 800 °C
Mixed-layer minerals	Regular, one (001) and integral series of basal spacings	No change unless an expandable component is present	Various, see descriptions of individual minerals
	Random, (001) is addition of individual minerals and depends on amount of those present	Expands if montmorillonite is a constituent	Depends on minerals present in inter-layered mineral
Attapulgite (palygorskite)	High intensity d reflections at 10.5 Å, 4.5 Å, 3.23 Å, 2.62 Å	No change	Dehydrates stepwise
Sepiolite	High intensity reflections at 12.6 Å, 4. 31 Å, 2.61 Å	No change	" "
Amorphous clay, allophane	No d reflections	No change	Dehydrates and loses weight

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[1] Compiled by Dorothy Carroll [26].

Table 9. Temperatures Required in the Identification of Clay Minerals [1]

Mineral	Temperature, 1 hour	Effect
Kaolinite, well-crystallized	575-625 °C	Replacement by amorphous meta-kaolin; no diffraction pattern
Kaolinite, disordered ("fire-clay")	550-562 °C	Similar to kaolinite
Dickite	665-700 °C	Similar to kaolinite
Nacrite	625-680 °C	Similar to kaolinite
Halloysite	125-160 °C 560-605 °C	Water removed Similar to kaolinite
Metahalloysite	125-150 °C 560-590 °C	Water removed Similar to kaolinite
Allophane	140-180 °C	Adsorbed water removed; amorphous at all temperatures
Mica, well-crystallized	700 °C	Shows gradual loss in weight, but does not break down below 700 °C; (001) spacings remain in diffractograms below 700 °C and up to 1000 °C
Illite, and clay micas	125-250 °C 350-550 °C 700 °C	Loss of hydroscopic water Reverts to mica structure Similar to mica
Glauconite	530-650 °C	Loss of interlayer water; reverts to mica structure
Celadonite	500-600 °C	Similar to glauconite
Biotite	700 °C	Phlogopite is similar to muscovite Biotite shows breakdown 700-1000 °C
Vermiculite	< 300 °C	Water removed in stages with 14 Å 13.8 Å 11.6 Å 9 Å. Initial (001) spacing controlled by humidity
Montmorillonite Group	300 °C	Original 15 Å spacing disappears; 9 Å spacing develops
Chlorite Group	600-800 °C	Show gradual weight loss, but no structural change
Mg-chlorite	650 °C	14 Å spacing is intensified; (004) at 3.54 not affected
Fe-chlorite	500 °C	14 Å spacing less intense and may become broad and diffuse
Mixed-layer clays	< 600 °C	Varies with kinds of minerals present and amount of each; check with X-rays
Palygorskite-Sepiolite Group		Water occurs in channels in structure
Sepiolite	< 200 °C	Rapid dehydration
	> 200 °C	Slow dehydration; spacing at 12 Å above 350 °C becomes weak and diffuse; a spacing at 9.8 Å appears and spacing at 7.6 Å becomes more intense; recrystallizes at 800 °C
Palygorskite	400-440 °C	Rapid dehydration, but no change in structure 400 °C; 400 °C 10.5 Å spacing becomes broad and diffuse; near 800 °C structure is destroyed

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[1] Compiled by Dorothy Carroll [26].

observation requires a binocular microscope with good resolving power.

The binocular microscope is a useful tool for adobe studies using specimens impregnated with a material such as epoxy, sectioned and polished. Consolidated samples which can be cut and polished are obtained by impregnation with an epoxy. Several commercially available epoxies have given excellent results. Methods of sample impregnation have been described by Morreland [28]. Sample preparation is also available from commercial laboratories at a nominal cost.

Examination of the polished specimens in reflected light gives a wealth of information. For example, many of the minerals and larger rock fragments may be identified and the size, shape, packing and colors of the grains observed. In addition, the types of organic material present, the residual structures of decomposed straw and twigs, the weathering of mineral fragments, angularity of the coarser silt particles, and the presence of soluble salts (precipitated as pore fillings after evaporation of pore water) are further examples of the types of information that may be obtained by this simple technique. Impregnation with a colored epoxy may be useful in studies of pore sizes and pore distributions. An alternative to polishing sawed specimens is to simply spray their surfaces with a colorless acrylic. This method frequently permits observation of greater detail and eliminates the danger of removal of the softer matrix that may occur in the polishing process. The photographic reproductions in figures 8-12 show the kinds of information which can be obtained with the binocular microscope. Adobe specimens from the mission church at Tumacacori are shown in figures 8-10. Figure 11 is a microscopic view of adobe from Escalante, and figure 12 shows the aggregate and coarse matrix of an adobe specimen from Fort Bowie. Low magnifications have been used (3-10x) to show the general aspect of these materials in figures 8-10 although magnifications of 80-100x are common with this type of microscope.

The binocular microscope is especially useful in assessing the effectiveness of the size fractionation process. The effectiveness of the sample dispersion method should be determined by examining the various size fractions microscopically, particularly for complete separation of the clay-silt fraction. In addition, individual minerals and rock fragments may be identified and studied in much more detail after being fractionated than was possible by a study of polished sections. By utilization of the binocular microscope, an experienced analyst should be able to obtain a fairly complete identification of the mineralogy of the adobe (with the exception of the clay-silt fraction), and an assessment of the homogeneity of the material.

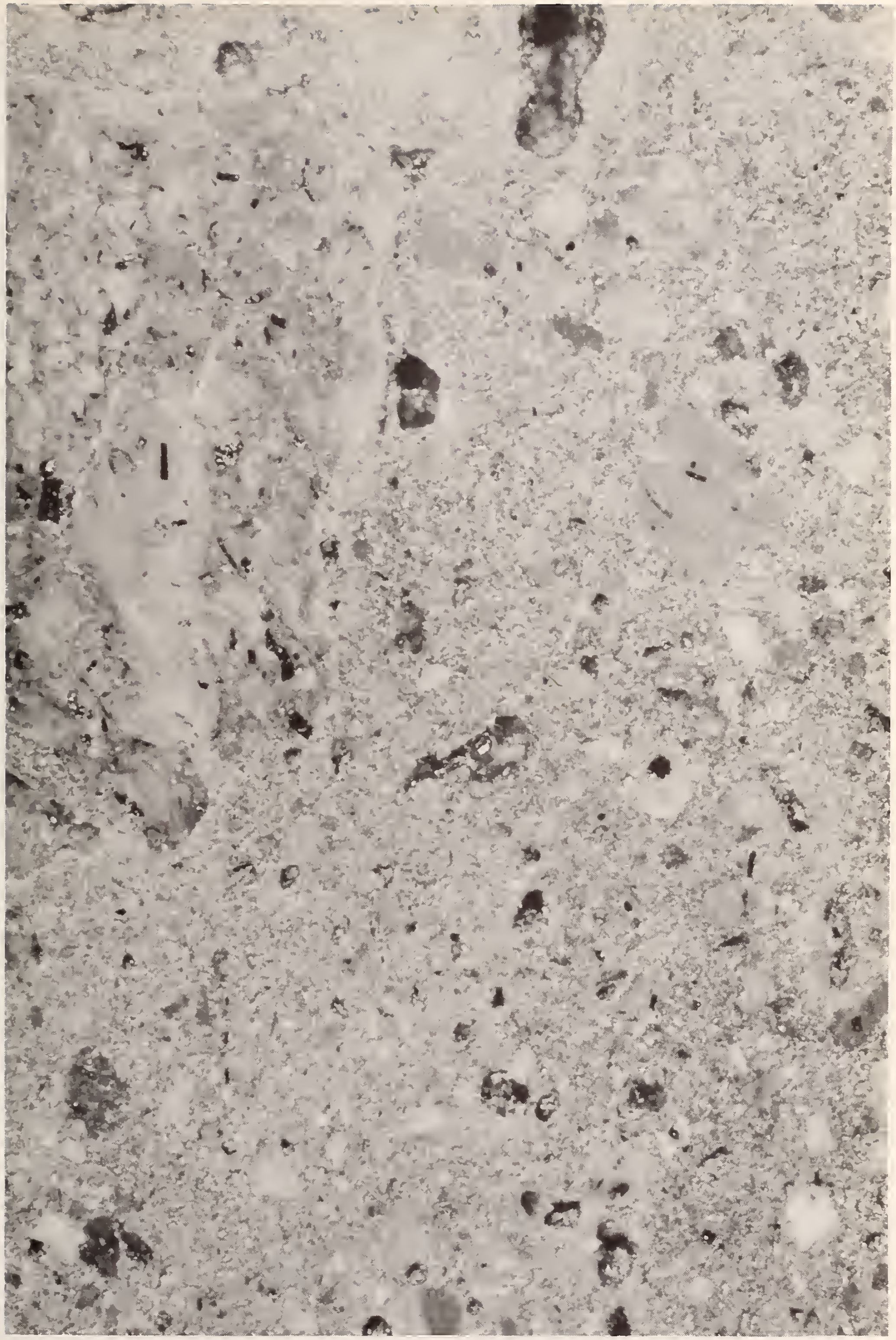

Figure 8. Microscopic view of epoxy-impregnated adobe specimen from Tumacacori. The specimen was viewed with a binocular microscope using reflected light (magnification of 3X).

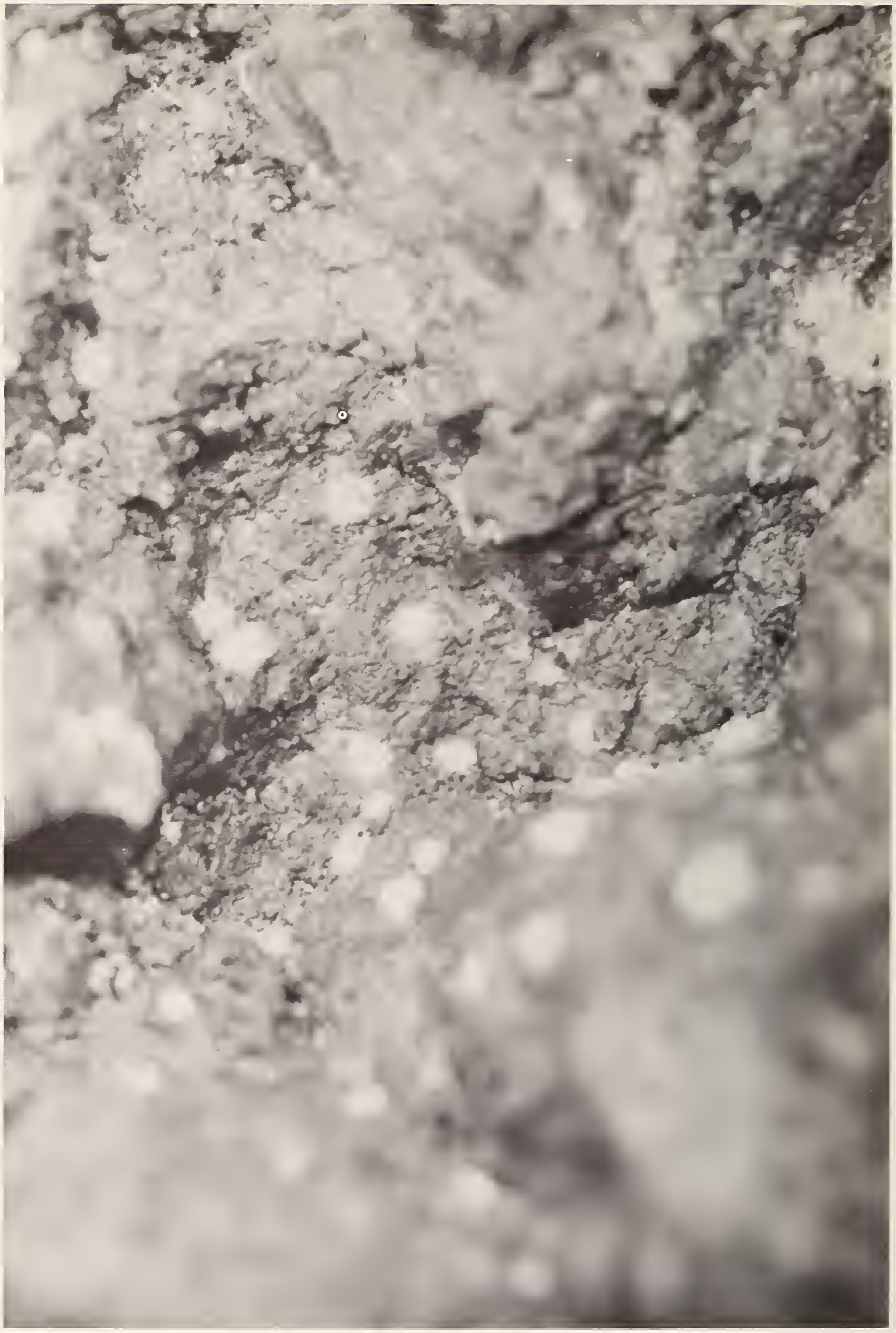

Figure 9. Gypsum in pore space of adobe specimen from Tumacacori viewed with a binocular microscope using reflected light (magnification of 10X).

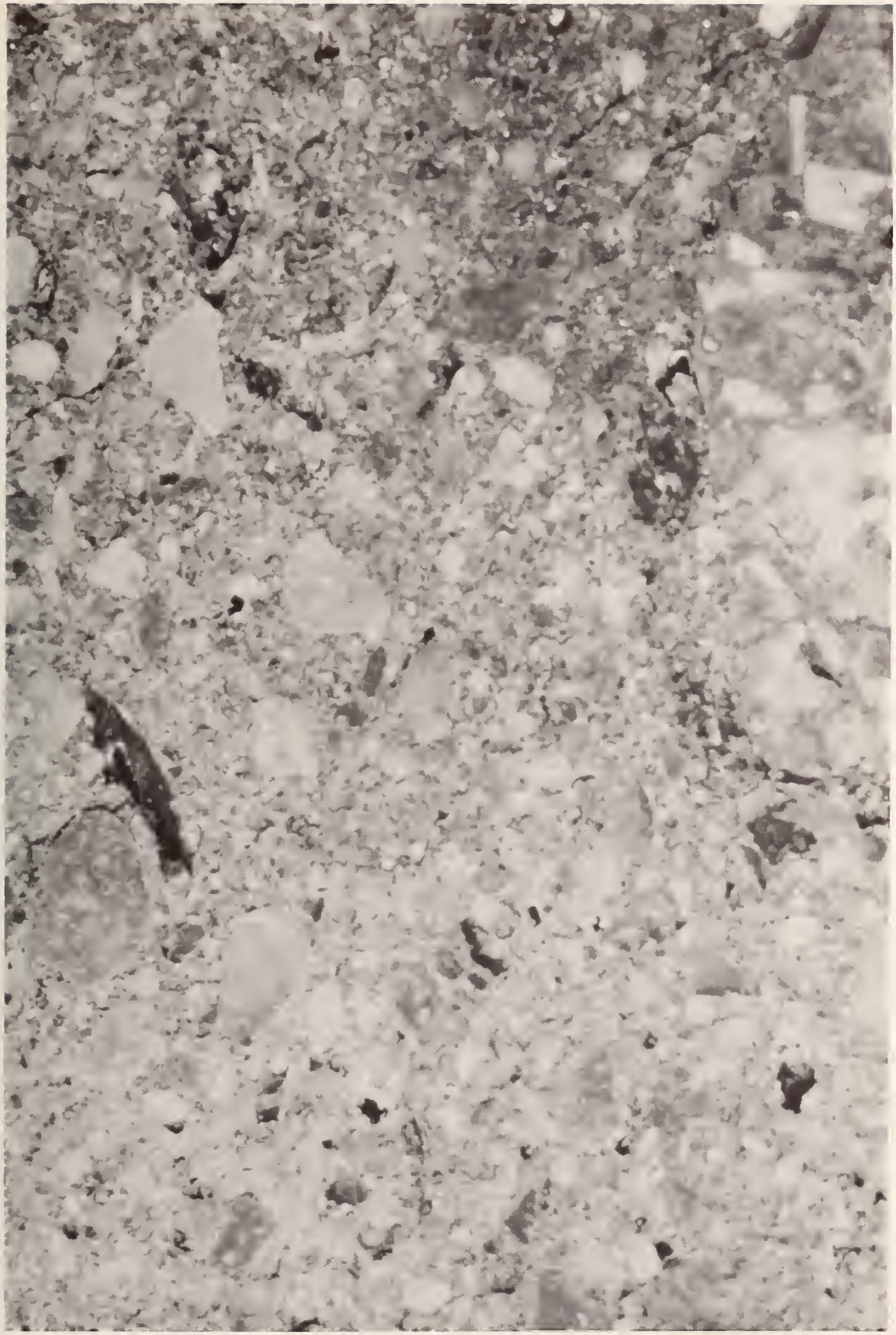

Figure 10. Microscopic view of epoxy-impregnated and polished section of adobe sample from Tumacacori. The fabric of the sand and the coarse matrix are shown. The specimen was viewed with a binocular microscope using reflected light (magnification of 10X).

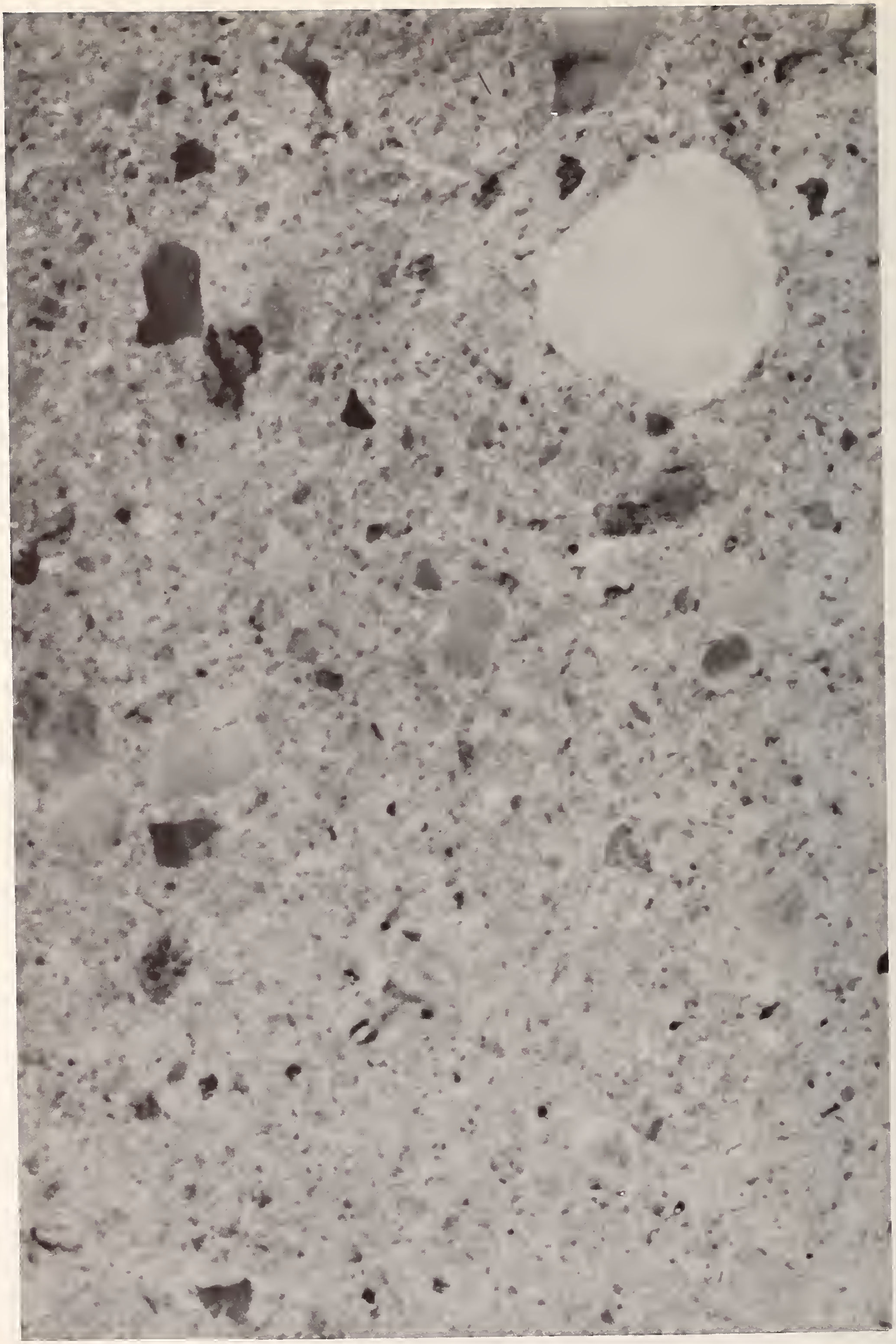

Figure 11. Microscopic view of epoxy-impregnated and polished section of adobe sample from Escalante. The specimen was viewed with a binocular microscope using reflected light (magnification of 10X).

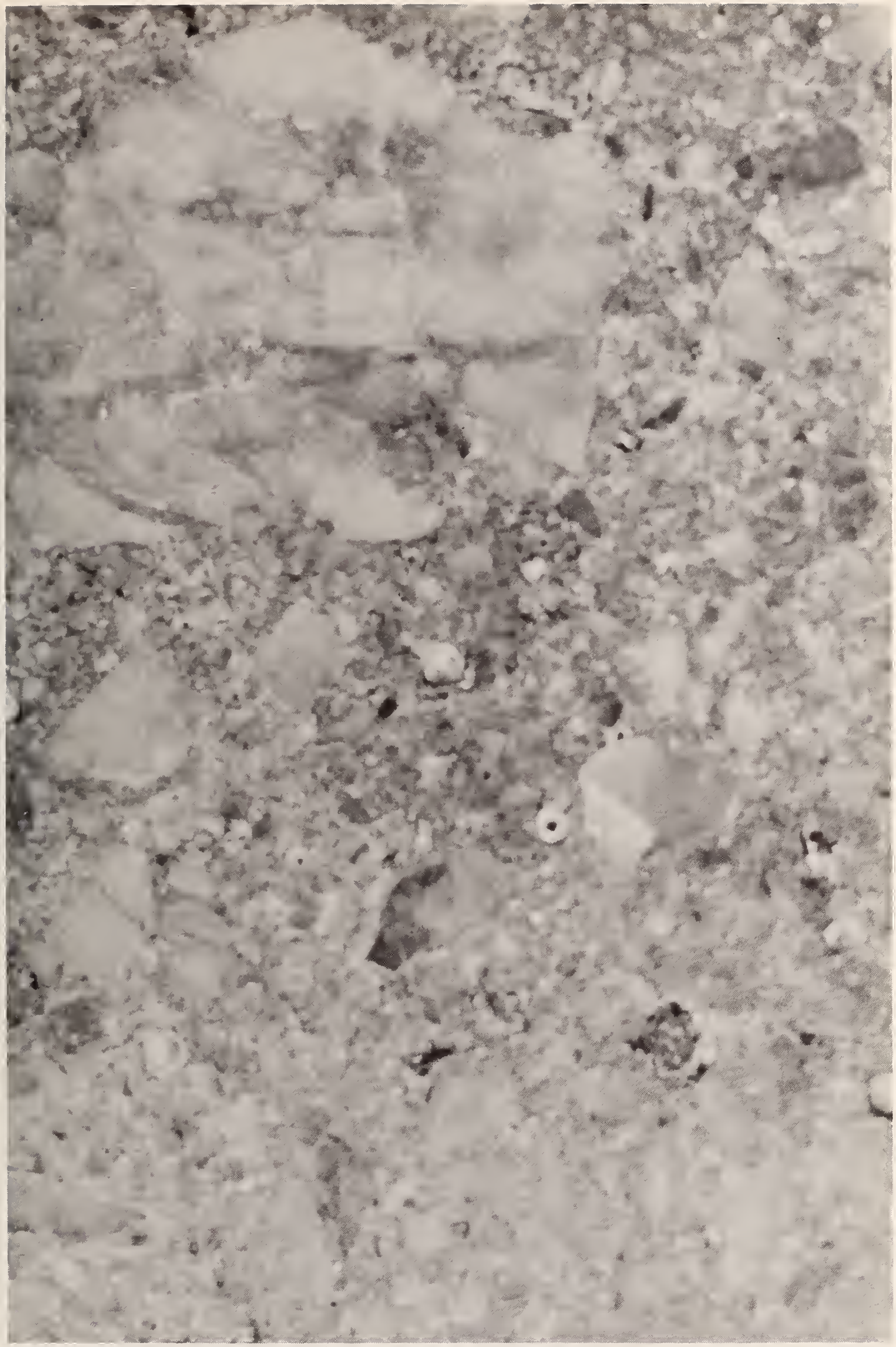

Figure 12. Microscopic view of epoxy-impregnated and polished section of adobe sample from Fort Bowie. The specimen was viewed with a minocular microscope using reflected light (magnification of 10X).

3.7.2 Polarizing Microscope

Thin sections of adobe (standard thickness 30 microns) may be studied with a petrographic microscope in transmitted polarized light. With a magnification capability of 1000x, this instrument permits observation of much finer microfabric detail than the binocular microscope. Figure 13 is an example of a photomicrograph of a thin section of adobe taken in transmitted polarized light at very low magnification (50X). Even at this low magnification considerable detail is revealed by the contrasting interference colors, which will assist the microscopist in selecting areas for examination at higher magnifications.

The polarizing microscope is commonly used to identify the mineral present in a soil. All crystals have a characteristic index or indices of refraction which are unique for a given compound and serve as a means of identifying it. In practice the unknown mineral, in powder form, is immersed in a series of oils of known index of refraction until a match is observed under the microscope. The index of the oil is then taken as the index, or one of the primary indices, of the mineral. A crystal may have one, two or three primary indices of refraction depending upon its symmetry. Classification of the crystal into one of three symmetry classes is made, indices measured and reference tables listing mineral refractive indices searched until the compound is identified. This method is particularly useful when the material of interest is present in minute amounts or when X-ray or SEM equipment is not available. There are many excellent texts on the use of the polarizing microscope and this technique is discussed in detail in reference [29]. Sets of oils for refractive index determinations are commercially available. Refractive indices of the more common minerals are given in references [30,31]. Additional information on the optical properties of minerals is given in the series of Deer et al. [32].

3.7.3 Scanning Electron Microscope

The scanning electron microscope (SEM) has a magnification range of 20 to about 150,000 and a depth of field some 300 times greater than that of the optical microscope. With this instrument, electrons strike a sample and secondary electrons are emitted from the sample surface to form what appears to be three dimensional images. Cost of SEM's are in the same range as X-ray diffraction equipment. Because of the valuable information which can be obtained, this instrument is now found in many university and research laboratories. It may be used to study polished or fractured surfaces, individual crystals or powders. The SEM

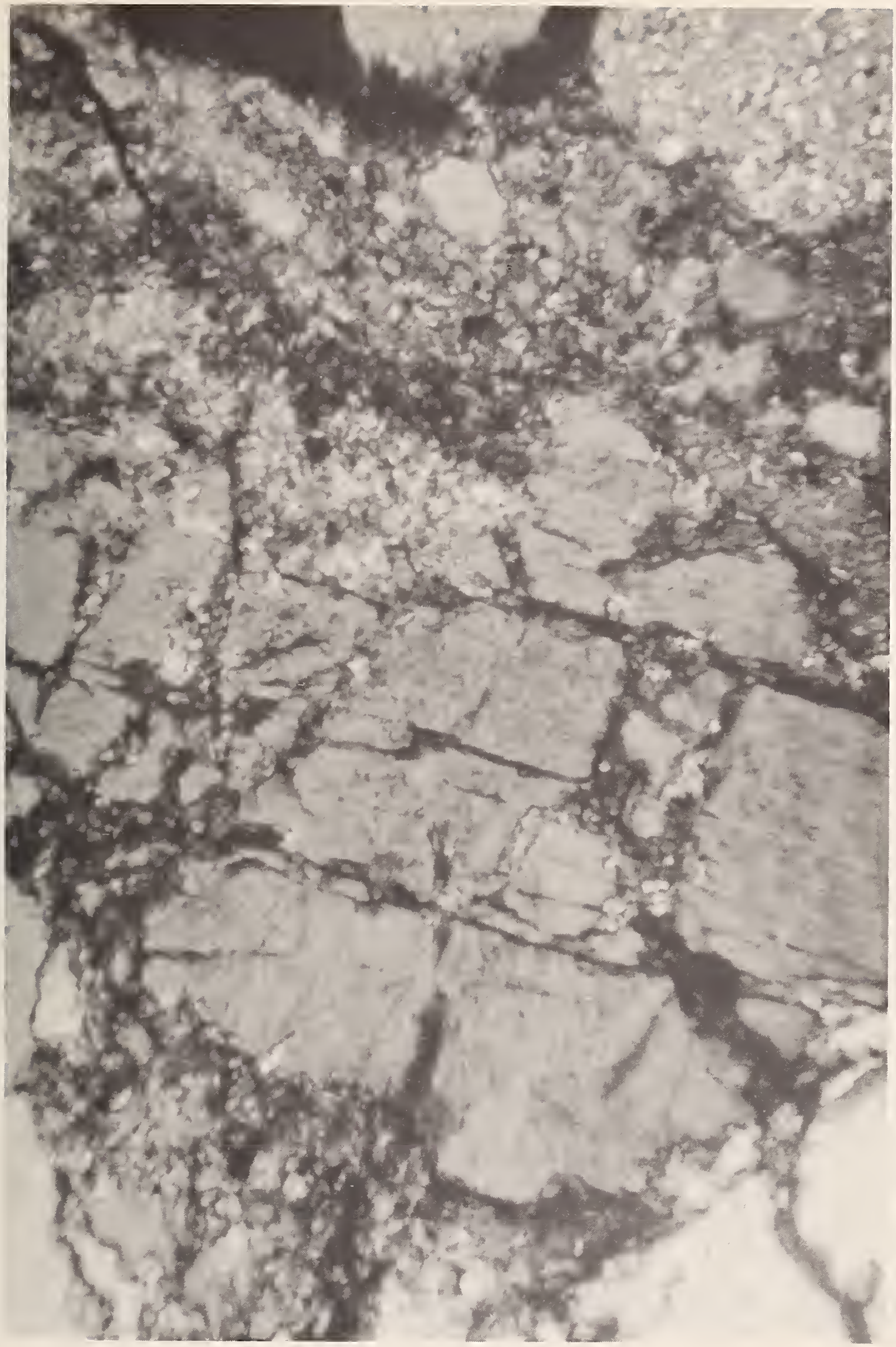

Figure 13. Thin section of adobe sample from Fort Bowie. The specimen was viewed using polarized transmitted light (magnification of 50X).

is used for examining the microfabric of adobes and soils, observation of clay particles which are too small to be observed with optical microscopes, and the arrangement and porosity of the silt and clay particles. If equipped with an energy dispersive X-ray analysis attachment, SEM may also be used for chemical or elemental analysis of adobes and soils.

Figures 14-18 show examples of some applications of this technique to adobe studies. Figure 14 is a scanning electron micrograph of a polished section of adobe from the church at Tumacacori. The micrograph was taken at very low magnification (30X) to show the general fabric of the coarse and fine aggregate and the clay-silt matrix. Figure 15 shows an elemental map of a fine particle in the adobe from Tumacacori. Because the particle was suspected to be gypsum the area was mapped for calcium and sulfur, and the two maps were superimposed. Gypsum, which has filled a pore, is shown by the white areas in the photograph (magnification 500X). The energy distribution of this light region, obtained by energy dispersive X-ray analysis (EDXA), is shown in figure 16. The prominent sulfur peak on the left and the calcium peak on the right suggests the presence of gypsum ($CaSO_4 \cdot 2H_2O$).

During size fractionation of the adobe specimens, the soluble salt fractions can be separated for elemental and other analysis. Figure 17 shows the energy distribution of a bulk salt sample from Tumacacori, and indicates the elements potassium, magnesium, sulfur, chlorine and calcium. This material is extremely hygroscopic. A small amount of the powder previously dried at 110 °C was placed on a microscope slide and exposed to the laboratory atmosphere, with a relative humidity of about 60 percent, for approximately one-half hour. The resulting material was viewed through a polarizing microscope in transmitted light at a magnification of 65X. The large volume of water absorbed by and eveloping the grains is clearly evident (figure 18).

The elemental analyses that have been discussed in this section are qualitative determinations. Computers can be interfaced with SEM's [33] so that quantitative elemental analyses can be made. Quantitative determinations of individual soluble salts in adobe could provide valuable information in the investigation of the factors causing the deterioration of adobe.

Figure 14. Scanning electron micrograph of adobe sample from Tumacacori (magnification of 30X).

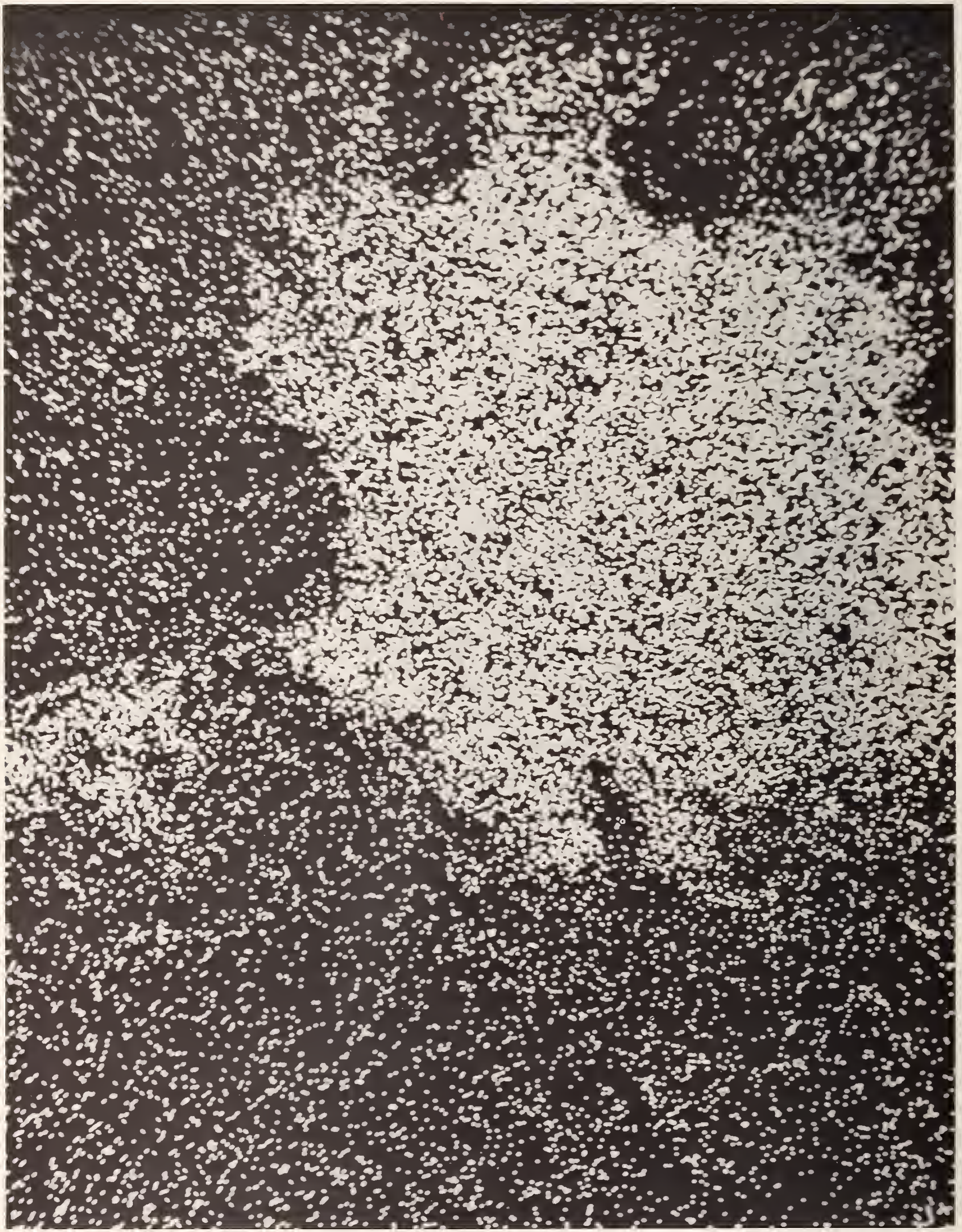

Figure 15. Energy dispersive X-ray element map of adobe sample from Tumacacori (magnification of 500X). The white regions indicate the presence of calcium and sulfur.

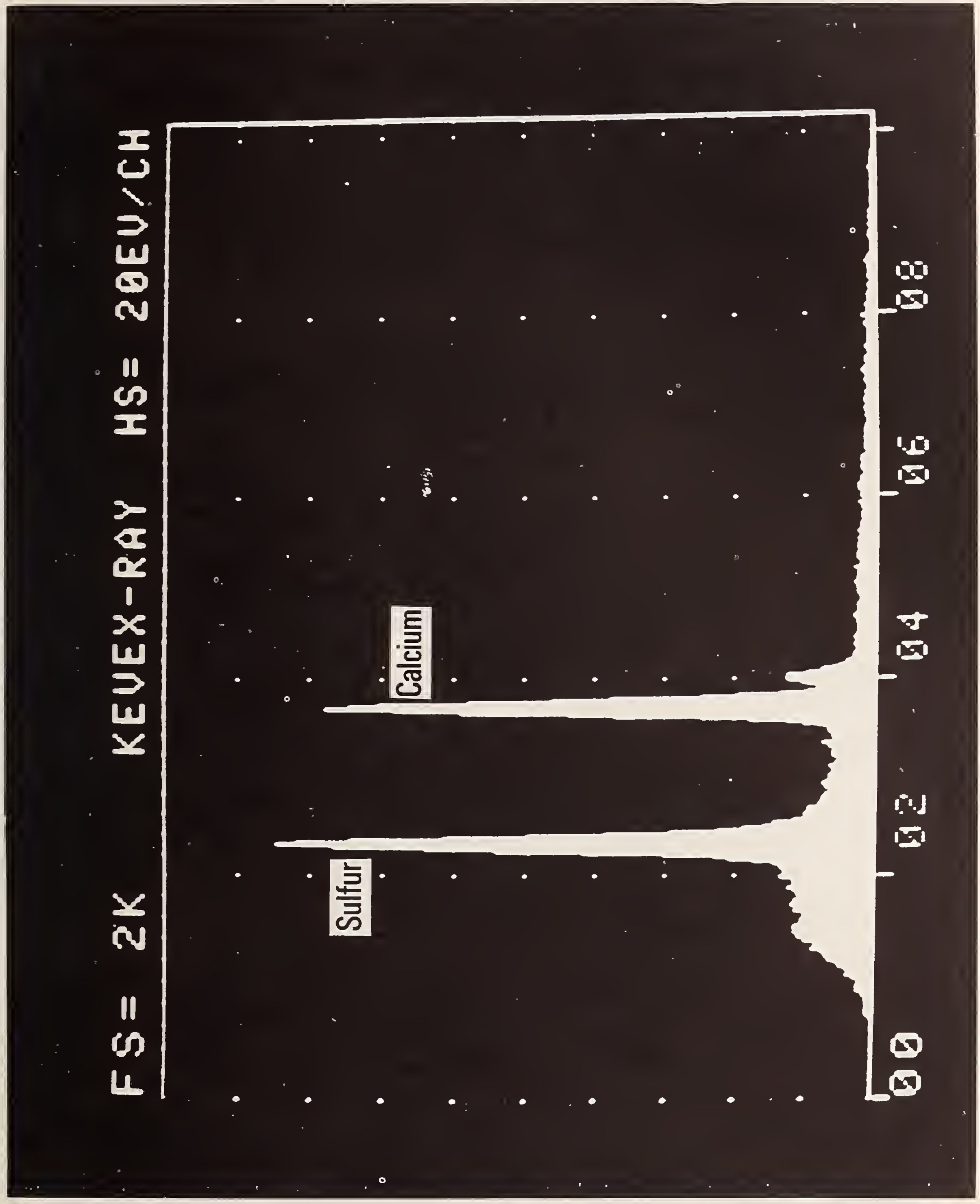

Figure 16. Energy dispersive X-ray spectrom of white material shown in figure 15. The two major peaks indicate the presence of a material containing calcium and sulfur, probably gypsum ($CaSo_4 \cdot 2\ H_2O$).

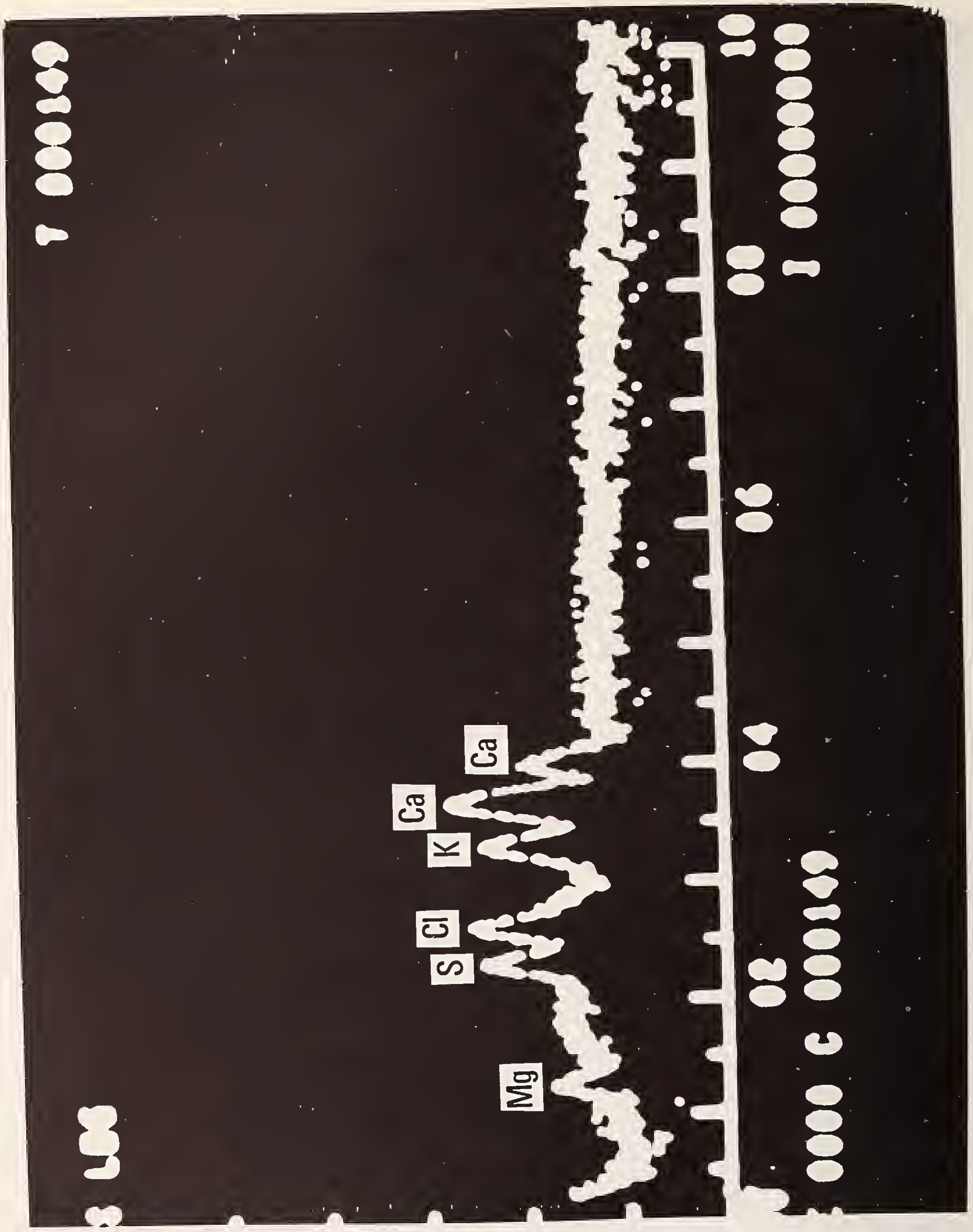

Figure 17. Energy dispersive X-ray spectrum of salts extracted from adobe sample from Tumacacori. The major peaks indicate the presence of the elements potassium, magnesium, sulfur, chlorine, and calcium.

Figure 18. Microscopic view of hygroscopic salts extracted from adobe sample from Tumacacori. The salts were viewed with polarized transmitted light (magnification of 65X).

4. SUMMARY AND RECOMMENDATIONS

Methods are described in this report for the characterization of those physical properties and mineralogical features of adobes which appear to have the most significant effects on adobe durability. These features include particle size distribution, mineral composition of particle size fractions, presence of soluble salts, the microfabric of the silt-clay matrix, and the rheological response of adobes to moisture. In addition, methods are given for assessing the compatibility of repair materials and preservation techniques with the original adobe of historic structures.

It is recommended that the analysis of an adobe should include the following determinations; color, pH, amount of soluble salts, particle size distribution, liquid and plastic limits, and the X-ray diffraction "fingerprint." In addition, viewing an adobe specimen through a binocular microscope can provide interesting information on the gross fabric of the adobe. This complete analysis can be carried out within a week using equipment available to most testing laboratories.

The definitive analysis of the mineral composition and the proper analysis of the fine microfabric features of an adobe are more difficult than the preceeding determinations, and require more sophisticated techniques. However, at times such analyses are justified. For example, if there is any indication that the soil selected for making repairs to an adobe structure contains a swelling clay, e.g., montmorillonite type, then a definitive identification should be performed.

The characterization of the physical properties and mineralogical features of adobe should become a part of the standard methodology developed for the preservation of adobe structures. The methods described in this report can form the basis for this contribution to the standard methodology.

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APPENDIX - DESCRIPTION OF SOURCES OF ADOBE SAMPLES

Escalante - An indian farming community inhabited from about 900 to 1450 A.D. It was located along the Gila River, in Arizona.

Fort Bowie - An Army fort constructed on the Apache Pass in Arizona. The adobe test samples were taken from the ruins of the 2nd fort, constructed in 1868.

Tumacacori National Monument - A Jesuit Missionary settlement, of which the mission church is the most important building. The church was constructed between 1802 and 1822. Tumacacori National Monument is located about 40 miles south of Tucson, Arizona.

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Methods are described for the characterization of those physical properties and mineralogical features of adobe which appear to have the most significant affect on the durabilities of adobes. These methods include determinations of color, pH, soluble salts, particle size distribution, liquid and plastic limits, and the X-ray "fingerprint" of adobe. In addition, methods are given for the identification of the mineralogy of adobe soils and for the examination of the microfabric of adobe.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)* Adobe building materials; adobe soil; color determination; microfabric analysis; mineralogical analysis; particle size distribution; pH; plastic and liquid limits; soluble salts.

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