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Cover Picture

WAVE-CUT FEATURES IN THE LUGERT GRANITE

In parts of the western Wichita Mountains, the Middle Cambrian Lugert Granite crops out where overlying Wichita and Hennessey Shales (Lower Permian) have been removed. The flutes, grooves, and potholes pictured here were carved by the Early Permian sea into the Lugert, which was exposed as islands and along the shoreline of that time. The wave-cut surface extends beneath the hill in the background of this view, but much of it is covered by debris from above.

Other evidences of wave sculpture in the area are caves along the old shoreline and wave-cut platforms on several hills in the Devil's Canyon physiographic group. These platforms range from 50 to 300 feet wide and are typically 2,200 to 2,250 feet above present mean sea level. Most are found only on the hillside which was toward the open sea; however, the ridge around Dome Mountain is continuous.

Many of the Lugert hills in this region were part of an archipelago, and, as the sea covered the islands during Wichita and Hennessey times, the shales which now blanket much of the granite were deposited.

The photograph is from Oklahoma Geological Survey Guide Book 5, *Field Conference on Geology of the Wichita Mountain Region in Southwestern Oklahoma*. The outcrops pictured are in NE¼ NW¼ sec. 7, T. 5 N., R. 18 W., Kiowa County.

—*P. W. W.*

Particle-Size Separation of Clays

Raymond L. Kerns, Jr.

INTRODUCTION

Particle-size separations are performed in clay-mineral studies to obtain quantitative information on the particle-size distribution within a sample or to determine the size range of a particular mineral phase. Such techniques also eliminate unwanted material from a sample, providing definitive information on a relatively pure mineral phase. Of great interest to clay mineralogists is the relationship between clay-mineral properties and particle size, or total surface area. Chemical composition, surface chemistry, crystallinity, high-temperature phase changes, DTA characteristics, and particle morphology are some of the parameters either known or suspected to vary with, or to be a function of, particle size.

This discussion is not intended as a detailed consideration of all available size-separation techniques, and derivations of fundamental equations are not considered essential herein. Such topics are dealt with in detail in more complete works on the subject (e.g., Krumbein and Pettijohn, 1938), whereas this paper is intended as a guide for students who plan to undertake size fractionation of clay material. Two methods, the siphon technique and the centrifugation technique, and their applications and limitations are considered.

The particle-size fractionation techniques ordinarily applied to clay-sized materials utilize differential settling velocities according to Stokes' law. Differential settling, followed by decantation, or siphoning, may be used to separate particles to a lower limit of 1-micron spherical diameter. Settling velocities for particles smaller than 1 micron are too slow for practical applications of this technique and such materials are more efficiently handled with high-speed centrifugation.

SIPHON TECHNIQUE

Stokes'-law settling.—The most commonly applied formula for calculating settling velocities of sedimentary particles is the Stokes' law equation.

$$V = \frac{2}{9}\,\frac{(d_1 - d_2)\,g r^2}{\eta} \qquad \textit{(Eq. 1)}$$

The velocity (V) of a falling sphere of radius r in a fluid may be calculated from the density of the sphere (d_1), the density of the fluid (d_2), and the viscosity of the fluid (η). The acceleration due to gravity (g) is 980 cm/sec^2, and deviations from this value are considered negligible. The variations in density (d_2) and viscosity (η) of the fluid are usually significant, even over a small range of temperatures, and ordinarily are taken into consideration.

Several assumptions are involved in applying Stokes' law. The more critical of these are that the particle must be spherical, smooth, and rigid and that there should be no slipping between it and the medium. Also, there must be no interference between particles in the medium, the terminal velocity must have been reached, and the settling velocity should not be too great.

Solutions of the Stokes' equation are available in several forms. One of the more useful is a nomograph by Schweyer (1952, p. 10), which permits rapid graphical solution of the equation. The only data needed are particle density and the temperature of the medium; a density of 2.7 gm/cc is a reasonable average for most sediments. Table I lists settling times through a distance of 10 cm, as interpreted from Schweyer's nomograph, for several particle sizes equivalent to whole-number phi-scale divisions.

Particle-size separations of clays are most frequently used to eliminate unwanted mineral phases or to obtain monomineralic fractions so that differences in particle size may be considered on a relative, rather than absolute, basis; hence, the deviation of particle shapes from an ideal sphere is generally ignored. Under these conditions, particle sizes are simply reported as equivalent spherical diameters (ESD). If necessary, the classic Stokes' equation can be refined to take into account the platy nature of clay and mica particles.

Method.—The siphon technique is similar to the traditional decantation method. The material is disaggregated and dispersed in an aqueous medium. The suspension is allowed to stand undisturbed for the time necessary for settling out of all particles coarser than a chosen size; at the end of that time, the suspension is siphoned off down to a certain level. The material that has settled out is then redispersed and the settling and siphoning process is repeated.

TABLE I.—SETTLING TIMES FOR PARTICLES THROUGH A DISTANCE OF 10 CENTIMETERS*

PARTICLE DIAMETER (MICRONS)	ϕ SIZE	SETTLING TIME HR	MIN	SEC
62.5	4			25
31.0	5		1	40
15.6	6		6	40
7.8	7		27	
3.9	8	1	45	
2.0	9	6	50	
0.98	10	25		

* At 25 °C with assumed particle density of 2.7 gm/cc.

The number of times this process is repeated depends on the degree of separation desired in each particular case. Continued dispersals and withdrawals will, of course, result in a higher degree of separation. The amount of smaller sized material remaining in a bidisperse system after successive withdrawals will be $\frac{1}{2}$, $\frac{1}{2}^2$, $\frac{1}{2}^3$, . . . $\frac{1}{2}^n$ of the original amount, where the exponent n is the total number of withdrawals. A mathematical expression of the degree of separation by Stokes' settling law for this simple case is

$$\frac{w}{w_0} = p^{-2n} \qquad (Eq.\ 2)$$

where w is the amount of smaller sized material left after n treatments, w_0 is the original amount of fine material, and p represents the ratio r_l/r_s, r_l and r_s being the radii of the larger and smaller size grades, respectively (Krumbein and Pettijohn, 1938, p. 120). The equation shows that the smaller the value of p, the less efficient the separation will be in n treatments. Relatively good separation may be achieved, however, if the dispersion-siphon process is repeated often enough. The mathematical consideration is more complex in polydisperse systems. In most experiments with fine-grained sediments (less than 64 microns), an efficiency limit (w/w_0) is asymptotically approached after about ten withdrawals, assuming that the sample has been well dispersed and disaggregated each time.

Experimental considerations.—The first procedure in most cases, particularly with well-indurated samples, is to reduce the aggregate size for rapid and efficient disaggregation and dispersal. Rock specimens will ordinarily require crushing (avoid grinding) to pea size. The crushed rock is placed in a container, such as a 1-liter beaker, filled with distilled water. To avoid interference of the particles during settling, the concentration of solids should not exceed 10 percent by weight. Dispersal may be easily achieved with an ultrasonic generator; a Waring blender is equally effective, but this machine can be easily damaged by abrasive materials such as sand or silt. Fifteen minutes is usually sufficient for adequate disaggregation, although some materials require longer. Materials undergoing disaggregation by the ultrasonic method should be stirred frequently to facilitate the process.

After dispersion in distilled water, the suspension is stirred vigorously and allowed to stand undisturbed for a time sufficient to permit settling out of particles coarser than the desired size. The period of time needed can be selected from table I or from some other solution of Stokes' equation. If 1-liter beakers are used, a 10-centimeter settling distance is ideal. If the material flocculates, continued washing with distilled water may be necessary until the clay remains in suspension. The use of dispersants, such as Calgon (sodium hexametaphosphate), should be avoided if information concerning the cation-exchange capacity or the exchangeable cations present is desired.

After continued additions of distilled water, the volume of the suspension may exceed 1 liter; plastic wastebaskets of various sizes are available for handling larger volumes.

The siphon technique is suitable for separating particles at any interval desired, to an upper limit of 64 microns and a lower limit of 1 micron. Mechanical sieving is a more efficient method for separating particles coarser than 64 microns. Settling velocities of particles finer than 1 micron are so slow that settling techniques are impractical, and, if further differentiation or concentration is desired, one must resort to high-speed centrifugation. Table II may serve as a summary of the siphon method and as a laboratory guide.

TABLE II.—THE SIPHON METHOD

Step 1.	Crush sample to pea size.
Step 2.	Place in 1-liter beaker with distilled water; suspension should be less than 10% solids by weight.
Step 3.	Disaggregate and disperse with ultrasonic generator or Waring blender.
Step 4.	Allow to stand for sufficient time to remove particles of desired size; determine time from table I; 10-cm settling distance is best for 1-liter beakers.
Step 5.	Repeat steps 2, 3, and 4 ten times for adequate separation.
Step 6.	Save material smaller than 1 micron for concentration or further size fractionation by centrifugation.

CENTRIFUGATION TECHNIQUE

Mathematical consideration.—Centrifugation is usually necessary to concentrate or to size-fraction dispersed materials of less than 1-micron ESD. The fundamental equation for the application of centrifugal force to particle-size separation is developed completely in other works (e.g., Krumbein and Pettijohn, 1938). The basic equation is a special form of Stokes' law (equation 1).

$$V = \frac{2}{9}\,\frac{(d_1 - d_2)\omega^2\,(x + a)r^2}{\eta} \qquad \textit{(Eq. 3)}$$

The variables V, d_1, d_2, r, and η are the same as in equation 1. The only difference is that the term for gravitational acceleration (g) has been replaced by the term $\omega^2\,(x + a)$, where ω is the angular velocity, a the distance of the particle from the axis of rotation before fall, and x the distance of fall. The velocity (V) may be written as dx/dt, and equation 3 may be written as a differential equation with dx/dt substituted for V (Krumbein and Pettijohn, 1938). Arranging for integration, one obtains:

$$r^2\int_0^t dt = \frac{9\eta}{2\omega^2(d_1-d_2)}\int_0^x \frac{dx}{x+a} \qquad \textit{(Eq. 4)}$$

and after integration:

$$t = \frac{9\eta \log_e \frac{x+a}{a}}{2\omega^2\,(d_1 - d_2)\,r^2} \qquad \textit{(Eq. 5)}$$

Method.—The Lourdes Super Centrifuge, model LCA-1, may be operated at high speeds with a continuous flow of suspension through the apparatus. Figure 1 is a schematic cross-sectional view of the container. It is conical with an outside diameter of 22 cm at the base, tapering to 15 cm at the top. The total volume is about 1 liter, but when operated under continuous-flow conditions there is no liquid in the inner chamber (A). The steady-state volume of the liquid in the container is 600 milliliters. The container has a removable plastic liner and a lid that may be tightened with a special wrench. The liquid enters the centrifuge at the center (B), leaves through holes in the lid (C), and enters the cap (D). The escape velocity must be sufficient to cause the liquid to circle the inside periphery of the cap at least

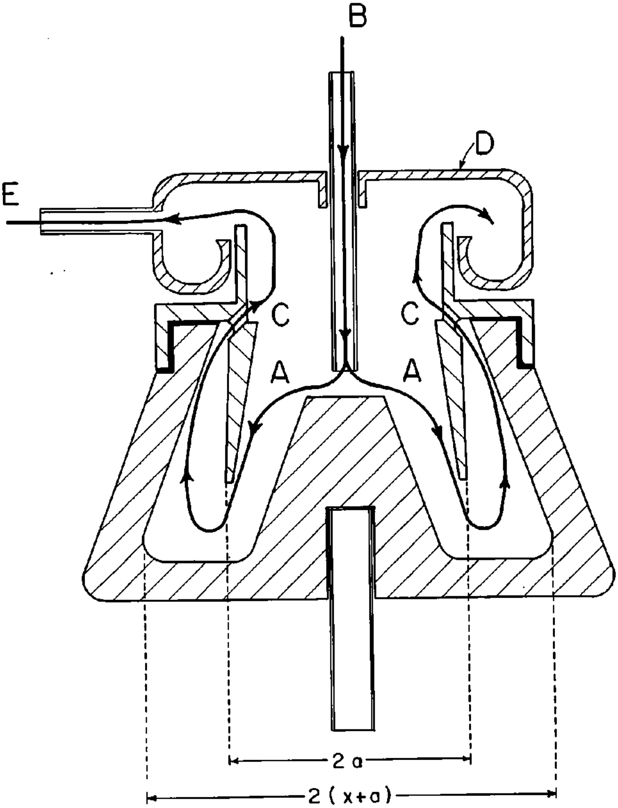

Figure 1. Cross-sectional view of centrifuge container for the Lourdes Super Centrifuge, model LCA-1. Diameter of inner chamber (2a) is 9.5 cm; internal diameter of container [2(x + a)] is 16 cm.

A inner chamber **C escape ports**
B inlet pipe **D cap**
E outlet pipe

once. An opening in the side of the cap (E) is fitted with a hose which carries the overflow to an outside container.

The diameter of the inner compartment ($2a$) is 9.5 cm. The inner diameter of the centrifuge container is 16 cm at the widest part; the inside radius, therefore, is 8 cm and corresponds to the value of ($x + a$) in equation 5. The value of x is 3.25 cm and the value of a is 4.75 cm for this system.

This centrifuge is equipped with a tachometer which registers the centrifuge speed in revolutions per minute. To establish working conditions for particle-size separations using equation 5, rpm values must be converted to angular velocities in radians per second. The relationship is:

$$\omega = \frac{2\pi\theta}{60} \qquad \textit{(Eq. 6)}$$

where ω is angular velocity in radians per second, π is 22/7, and θ is the centrifuge speed in revolutions per minute. Equation 6 may be substituted into equation 5 to yield:

$$t = \frac{(9\eta)\left(\log e \frac{x+a}{a}\right)(3600)}{8\pi^2\theta^2(d_1 - d_2)r^2} \qquad \textit{(Eq. 7)}$$

Substitution of viscosity (η) and density (d_2) of water at 25 °C, an average density of 2.7 gm/cc for the solid phase, an x value of 3.25 cm, and a value for a of 4.75 cm, equation 7 reduces to:

$$t = \frac{1.0149}{\theta^2 r^2} \text{ sec} \qquad \textit{(Eq. 8)}$$

For given values of r and θ, t is the time in seconds required for all particles of radius r and larger to fall through the distance x. After falling through the distance x they are trapped within the centrifuge. Particles of radius less than r are carried out of the system with the overflow.

A speed regulator is built into the centrifuge; with a mechanism for controlling the length of time that a given volume of liquid remains in the centrifuge it is possible to do particle-size separations. A simple apparatus for controlling the rate of flow of liquid into the centrifuge is diagrammed in figure 2. This gravity-feed mechanism is equipped with a stopcock (A) by which the flow is adjusted by trial and error until the desired rate is obtained. The height of the reservoir (B) should be at least 4 feet above the centrifuge (C) and overflow container (D). If fluctuations of the hydrostatic head are within a range of ±3 inches, deviation in flow rate will not be more than a few percent.

Flow rate is a function of the time (t) obtained by solving equation 8 and of the volume of liquid in the centrifuge during steady-state operation (600 ml). The flow rate (F_R) in liters per minute for any solution of equation 8 may be calculated as:

$$F_R = \frac{600(\text{ml}) \times 60(\text{sec/min})}{t(\text{sec}) \times 1000(\text{ml/l})} \qquad \textit{(Eq. 9)}$$

which reduces to:

$$F_R = \frac{36}{t} \text{ (l/min)} \qquad \textit{(Eq. 10)}$$

It is more convenient to work with the reciprocal of the flow rate, referred to as the flow time in minutes per liter:

$$F_t = \frac{1}{F_R} \qquad \textit{(Eq. 11)}$$

or, combining equations 10 and 11:

$$F_t = \frac{t}{36} \text{ min/l} \qquad \textit{(Eq. 12)}$$

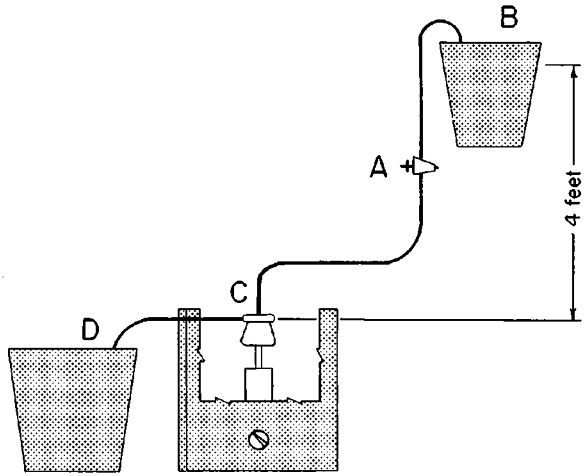

Figure 2. Diagram of apparatus for controlling flow rate into a centrifuge.

A stopcock **C centrifuge**
B reservoir **D overflow container**

A formula for the direct calculation of flow time for any combination of values of θ and r in centimeters may be obtained by combining equations 8 and 12. This results in the formula:

$$F_t = \frac{1.0149}{36\,\theta^2 r^2}\ (\mathrm{min/l}) \qquad \textit{(Eq. 13)}$$

or:

$$F_t = \frac{0.02819}{\theta^2 r^2} \qquad \textit{(Eq. 14)}$$

where θ and r have the same significance as in equation 8.

Solutions of equation 14 are plotted graphically in figure 3. Values of flow time (F_t) in minutes per liter are plotted on the ordinate and centrifuge speeds (θ) in revolutions per minute are plotted on the abscissa. Each curve represents a different equivalent spherical diameter (ESD).

Experimental considerations.—The curves in figure 3 are plotted over a range of centrifuge speeds from 3,000 to 12,000 revolutions per minute. At speeds less than 3,000 rpm the centrifugal force is insufficient to expel all the water from the container through the overflow outlet, and some fluid leaks out between the centrifuge cap and the containers. Speeds greater than 12,000 rpm cause excessive motor wear. Flow times less than 5 minutes per liter should be avoided because small errors in adjustment in this range lead to large errors in the separated particle sizes; flow times longer than 60 minutes per liter are impractical. Centrifuge speeds and flow times for the more

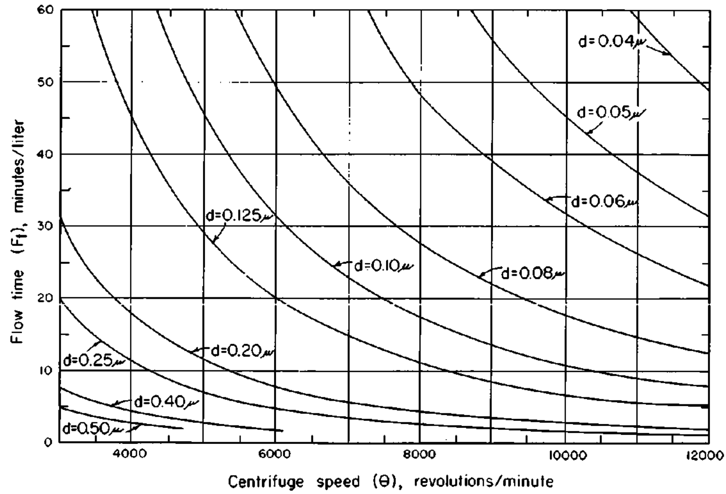

Figure 3. Calibration curves of flow time, in minutes per liter, against centrifuge speed, in revolutions per minute, for the Lourdes Super Centrifuge, model LCA-1.

common particle-size ranges are listed in table III. Instructions for operating the Lourdes Super Centrifuge, model LCA-1, are outlined on a plate on the underside of the machine cover.

TABLE III.—CENTRIFUGE OPERATION SETTINGS FOR MORE COMMON PARTICLE SIZES

PARTICLE DIAMETER (MICRONS)	Ø SIZE	FLOW TIME (MIN/LITER)	CENTRIFUGE SPEED (RPM)
0.500	11	5	3,000
0.250	12	5	6,000
0.125	13	5	12,000
0.0625	14	20	12,000

If the centrifuged material were redispersed and recycled several times, particles sizes could be more completely separated; however, this operation would be long and tedious. For routine separations, thorough and rigorous disaggregation and dispersal of the clay should produce a reasonable separation.

References Cited

Krumbein, W. C., and **Pettijohn, F. J.**, 1938, Manual of sedimentary petrography: New York, Appleton-Century-Crofts, Inc., 549 p.

Schweyer, H. E., 1952, Sedimentation procedures for determining particle size distribution: Univ. Fla., Coll. Engineering, Engineering Progress, vol. 6, no. 6, Bull. ser. no. 54, 19 p.

Robert L. DuBois Named Kerr-McGee Professor in Geology and Geophysics

Dr. Robert L. DuBois, named Kerr-McGee Professor in Geology and Geophysics, has joined the faculty of the School of Geology and Geophysics at The University of Oklahoma; in addition to teaching at the University, he will set up a magnetic-research laboratory on one of the Norman branches of the campus and will be in charge of the Earth Sciences Observatory at Leonard. An acknowledged authority on paleomagnetism and archaeomagnetism, Dr. DuBois brings to this position experience as a professor at the University of Arizona and as a consultant to Kaiser Steel Corporation, American Smelting and Refining Company, and other major mining companies, as well as to Jet Propulsion Laboratories in Pasadena, California.

Of inestimable value to both geologists and archaeologists would be certain knowledge of the orientation and intensity of the earth's magnetic field for any period of time in the past; knowing this, data on the magnetic orientation and intensity of petrologic and archaeological specimens could be compared with those on major time periods in the past, and the ages of the specimens could be determined. The principal objective of studies conducted by the magnetic-research facility will be to gather data for a complete chronicle of field reversals, magnetic polar wanderings, and other changes in the earth's magnetic field through time.

When completed, the laboratory will contain equipment for measuring the direction and intensity of magnetism in archaeological and petrologic samples, as well as the stability of magnetic properties of the samples. At the University's Earth Science Observatory in Leonard, measurements will be made and data recorded on local and distant earthquakes, earth tides, changes in the tilt of the Earth on its axis, magnetic micropulsations, geomagnetic field variations, earth-current fluctuations, and microbarometric pulsations. The observatory, presented to the University in 1965 by Humble Oil & Refining Company, is at an elevation of 843 feet and is well away from most sources of electrical and sound interference.

Dr. DuBois received two bachelor's degrees and a master's degree in engineering and a Ph.D. in geology from the University of Washington. His memberships in professional organizations include the American Geophysical Union, Geological Society of America, Mineralogical Society of America, American Association for the Advancement of Science, Sigma Xi, Arizona Geological Society, and the Arizona Academy of Science. Dr. DuBois is a past chairman of the geological section of the Arizona Academy of Science and has also edited the *Arizona Geological Society Digest*.

A. A. P. G. Mid-Continent Regional Meeting

The convention theme for the Mid-Continent Regional Meeting of the American Association of Petroleum Geologists reads: "Sound imagination plus bold exploration will find future Mid-Continent reserves"; with a program designed to encourage imaginative exploration, the meeting will be held September 27-29 at the Broadview Hotel in Wichita, Kansas. Registration will begin at 9:00 A.M. Wednesday, September 27, and papers will be presented beginning at 9:00 A.M. and 1:30 P.M. on both Thursday and Friday.

The objectives of the convention, according to co-chairmen R. J. Gutru and E. J. McNeil, are "to stimulate geologic imagination, to encourage more daring exploration, and to provide the opportunity for professional friends to meet and discuss the challenges ahead in exploring for the 'elusive' future Mid-Continent petroleum reserves." Several papers dealing either directly or indirectly with Oklahoma geology will be presented with these objectives in mind*; among them are: *Stratigraphic Applications of Dipmeter Data in the Mid-Continent,* Ray Campbell; *Early Paleozoic Overlap in the Southern Mid-Continent,* P. A. Chenoweth; *Some Interesting Aspects of Carbonate Oil Accumulation in the Mid-Continent Area,* J. F. Harris; *Use of Clay Mineralogy in Determining Source of Deep Basin Sands,* F. H. Manley, Jr.; *The Depositional Environments of the Spiro Sands in the Arkoma Basin,* R. H. Potts, Jr.; *Application of Trend Analysis to Pre-Morrow Surface, Southeast Hugoton Embayment Area,* M. W. Schramm, Jr.; and *A Basis for Red Fork Sand Exploration in Northwest Oklahoma,* P. C. Withrow.

Housing information can be obtained from Wayne L. Brinegar of K & E Drilling, Inc., 719 Union Center Bldg., Wichita, Kansas, 67202; prior to September 10, all applications for housing must be received by the Convention and Visitors Bureau, Wichita Chamber of Commerce, 121 North Broadway, Wichita, Kansas, 67202.

* Abstracts will be published in the October issue of the Notes.

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