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APPLICATION OF HINDERED SETTLING TO PARTICLE SIZE MEASUREMENT

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CHEMICAL TECHNOLOGY DIVISION

Chemical Development Section A

APPLICATION OF HINDERED SETTLING TO PARTICLE SIZE MEASUREMENT

J. M. DallaValle, J. P. McBride, V. D. Allred, E. V. Jones

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ABSTRACT

The settling of relatively concentrated suspensions of fine particles can be analyzed theoretically by analogy to a fluid moving up through a static bed of packed particles. Using this analogy an equation was developed describing the hindered settling phenomena in terms of a relation between two dimensionless groups--the Froude group and the Reynolds group--which correlated the data for the settling of suspensions of materials with densities from 1.1 to 4.0 g/cc and particle sizes from 8 to 1000 microns. The equation also permits the specific surface diameter of the suspended particles to be calculated from the hindered settling rate. Application of the equation to the settling of aqueous suspensions of a thorium oxide preparation calcined at various temperatures indicated the specific surface diameter of the suspended thoria increased with increasing calcination temperature from 4 microns at 650°C to 7.2 microns at 1300°C.

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1.0 INTRODUCTION

An initially uniform and relatively dense suspension of fine particles settles at a constant rate with a well-defined interface between supernatant and suspended particles. This form of settling is referred to as "hindered" settling. All particles move at the same rate, regardless of size, and the suspension does not change concentration until it reaches the settled mass of particles at the bottom of the container.

Usually, three zones of settling are observed. The initial zone, which describes the uniform motion of the interface (measured from its initial level), a compressive zone, and a final stationary state. These zones are shown in Fig. 1. Zone I terminates when the interface coincides with the ultimate settled mass of particles. It consists of a straight line, the slope of which is the rate of interface movement. The second zone is also a straight line with some degree of curvature in transition from Zone I, but its slope is much less than the initial settling line. The Zone II line measures the change in volume of the settled bed. This zone is not characteristic of all materials, but only those affected by hydrostatic head, adhered gas, or electrolyte atmospheres affecting their normal compaction behavior. Hard discrete particles which settle rapidly pass directly to Zone III wherein we have a permanently settled bed. Zone II is of special interest in most "thickener" operations. The region PP_1O has been called the delta region by McBride and co-workers (1957).

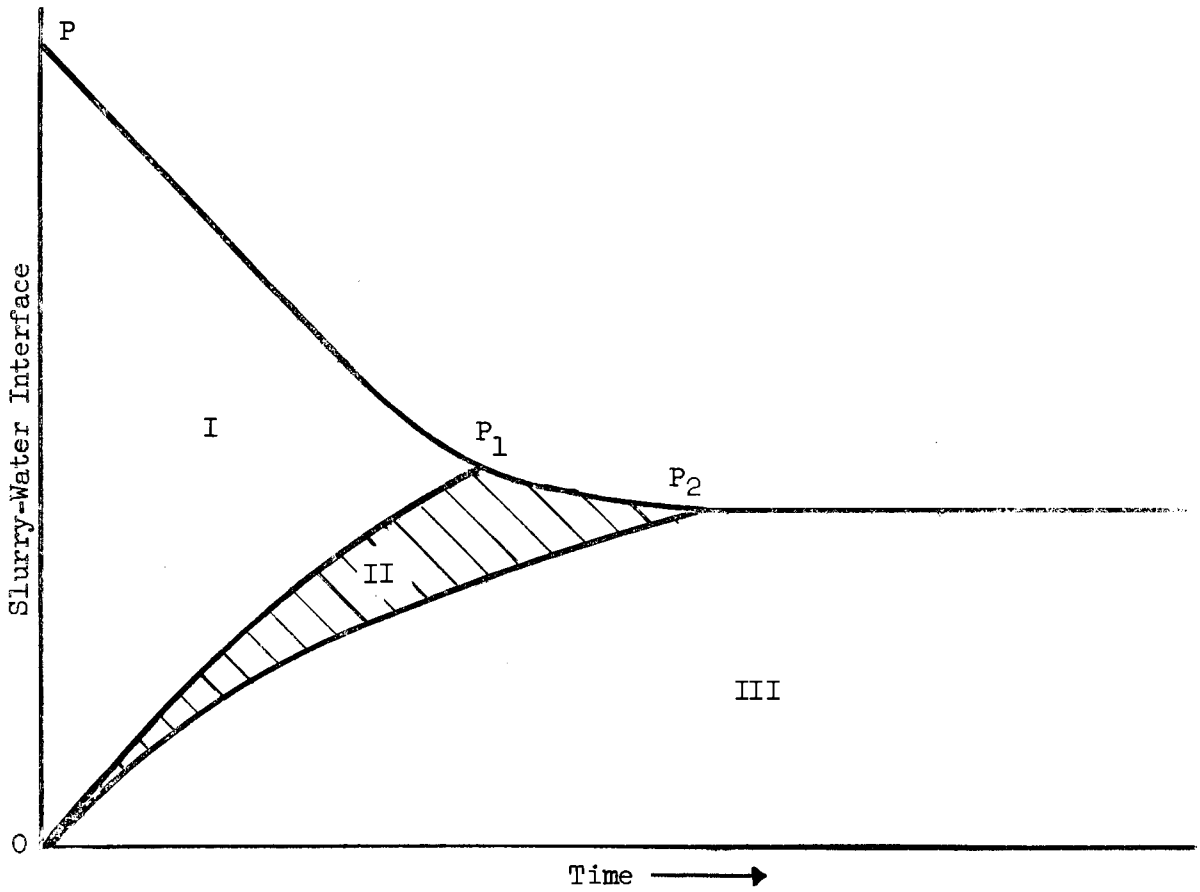

Fig. 1. Generalized Settling Zones for Moderately Concentrated Slurries.

Hindered settling takes place whenever there is mutual interference by particles in their motion. While mutual interference may occur at relatively low concentrations, it is customary to apply the term "hindered settling" only when the concentration is such that an observable interface exists between supernatant and suspended particles. It is further necessary to point out that the rate of settling is affected by the diameter of the container. Experience has shown that containers having a diameter of less than 25 mm produce marked wall effects.

The phenomenonof hindered settling has been studied rather extensively. Most efforts have been directed toward developing a suitable settling law in terms of measurable parameters.

2.0 PREVIOUS INVESTIGATIONS

Robinson (1926)[1] appears to have been the first to investigate hindered settling. He proposed modifications of the basic Stokes equation that would permit it to be used with concentrated suspensions. Essentially, Robinson's modification consisted in using density and viscosity of the suspension in place of the liquid density and viscosity. This approach has been invalidated on purely physical grouds, although fortuitous agreement with experiment has been obtained in many instances.

Eglof and McCabe (1937)[2] confirmed Robinson's equation for very high concentrations of suspensions but did not stipulate precisely the limiting conditions. These investigators recognized the three settling zones mentioned earlier and proposed two empirical equations, the first for the true hindered settling and the second for the compressive zone. Both relations gave fairly good results and are still widely used.

It remained for Steinour (1943)[3,4,5] to present the best analysis to date on the phenomenon of hindered settling. Steinour's contributions are contained in a series of three papers and were intended to develop an equation that would accurately describe such phenomena as the "bleeding" of cement pastes. In his first article, Steinour[3] applied the concept of hydraulic radius to Stokes' free fall equation and derived from his analysis an equation very much like the Kozeny-Fair-Hatch equation for flow through a packed bed of particles. Steinour's equation was:

$$u_o = g\,(\rho_s - \rho_o)D_s^{\,2}\,e^3.F(e)/18\,\mu\,(1 - e) \qquad (1)$$

where u_o is the velocity of the interface, e the porosity of the suspension, ρ_s and ρ_o the density of the particles and liquid, respectively, μ the

viscosity of the liquid, F(e) a shape factor depending on the porosity, and g the gravitational acceleration constant. Due to the nature of the phenomenon, if the above equation applies, then F(e) must approach zero as e approaches infinity (infinite dilution). However, at high concentrations, F(e) may remain practically constant. If u_s denotes Stokes' velocity, then the equation above is essentially nothing more than

$$u_o = u_s \, e^3 F(e)/(1 - e) \qquad (2)$$

In a series of carefully conducted experiments with many types of uniform spherical particulate suspensions, Steinour[3] was able to show from his data that F(e) = 0.123. For spherical particles, the phenomenon of hindered settling conformed to the empirical relation

$$u_o = u_s \, e^2 \, 10^{-1.82(1-e)} \qquad (3)$$

The form of F(e) is thus evident by comparison with the preceding equation.

In his second paper,[4] Steinour confined himself to investigating irregular particles and found that a certain amount of "immobile" water was associated with the fall of the particles. For this condition, an expression quite different from equation 3 was developed, but in a form such that when there was no immobile water, the equation reduced to equation 3.

Steinour's third paper[5] concerned itself with flocculated suspensions. Here again equation 3 was modified and a general expression for all types of hindered settling obtained. It required the determination of the immobile water.

Ward and Whitmore (1950)[6] criticized Steinour's investigations with regard to the constancy of the immobile water layer with particle size. These investigators, however, verified the general conclusions reached by Steinour with regard to hindered settling.

Hawksley (1950)[7] assumed that an "equilibrium" particle arrangement was established during hindered settling. Previous investigators regarded the particles as randomly distributed during settling. Hawksley's view is that while the distribution is undoubtedly random at the very beginning of settling, an orientation effect quickly sets in. This effect is attributed to the upward rise of displaced liquid. He argues that this upward flow of liquid causes a rearrangement of successive layers so as to effect a minimum overall resistance or a maximum rate of settling. Hawksley, working from these assumptions, derived a rather complex expression in terms of measurable parameters which fitted most hindered (nonflocculated) settling data. However, the basic assumptions made by Hawksley seem unnecessary since his results are, in a sense, quite empirical.

All the above studies on hindered settling have held to a general viewpoint that as settling progresses, the porosity of the "actively settling portion" of the suspension remains constant. Also, a portion of the suspension that does not actively engage in the process is supposed to exist at the bottom of the settling chamber. This inactive zone is of zero height initially and builds up as the top level of the suspension subsides. When the top level of the suspension and the top level of the inactive zone meet, the "critical" point is reached (see Fig. 1). Any additional settling is then due to the compression of this now totally inactive zone.

An opinion quite different from the above has been expressed in a recent monograph by Allison and Murray (1951).[8] They view the settling of a suspension as the gradual decrease in height of the suspension accompanied by a decrease in the porosity of the suspension. The porosity is assumed to be constant throughout the suspension at any given instant and all the suspension is viewed as "inactive", that is, in the sense of resistance of flow of the fluid displaced. Utilizing this concept and applying the Kozeny-Fair-Hatch equation (below), the investigators were able to evaluate the specific surface areas of a number of powders. Their procedure was to obtain the customary sedimentation rate data. The curve of interface height vs. time was graphically differentiated to obtain the velocity of fall of the interface. For each value of velocity, corresponding values of the porosity, e, and the density of the suspension, ρ_{sus}, were calculated based on the foregoing assumption. By plotting velocity vs. the porosity function, $e^3 \rho_{sus}/(1 - e)^2$, straight lines were obtained from whose slopes the value of the specific surface could be obtained in accordance with the Kozeny-Fair-Hatch equation in the form

$$u_o = g \ \rho_{sus} \ e^3/5 \ \mu \ S^2(1 - e)^2 \qquad (4)$$

where S is the specific surface area (volume basis).

While the initial concentration affects the initial settling rate (in most experiments with hindered settling), the effect on the calculated specific surface area was found to give a maximum difference for the same material of 14%. In general, Allison and Murray obtained good agreement with surface area measurements made by the usual permeametric methods.

More recently, Loeffler and Ruth (1953)[9] re-examined the whole problem of hindered settling and showed that it is closely related to the phenomenon of fluidization and showed that the Kozeny-Fair-Hatch equation is the logical starting point to deal with both problems. The results were correlated in special plots, taking into account immobile or adhered water, but are rather complicated and probably more involved than warranted, considering the scatter of the plotted data.

Of the various investigations cited, those of Steinour appear most pertinent to the present discussion. Aside from a wealth of experimental data, Steinour's contributions offer a rational basis for analyzing the problem of hindered settling. Steinour applied Stokes' law with a modification. This approach is rather curious inasmuch as he seems to have been fully aware that the Kozeny-Fair-Hatch relation for flow through a packed bed might equally well be used. The end results are the same since it is immaterial whether one considers the motion of the particles or the motion of the fluid. In either case, the law of resistance is the same. Loeffler, as we have seen, preferred to use the Kozeny-Fair-Hatch relation as a starting point.

Steinour's inclusion of adhered or occluded liquid as affecting motion in hindered settling is of particular importance with aggregated or flocculated materials. Steinour assumes and later shows that adhered liquid markedly affects the density of the settling solids as well as their diameter. The situation is enhanced with aggregates which carry along liquid in their interstices as they settle. Fortunately, most materials carry little adhered liquid and necessary corrections for both density and diameter need not be considered.

3.0 DEVELOPMENT OF HINDERED SETTLING RELATIONS

3.1 Basic Relations

We begin with the Kozeny-Fair-Hatch equation for the pressure drop of a fluid moving through a bed of packed particles:

$$\Delta p/L = 6\,\mu\, S_v^2\, u\, (1-e)^2/e^3\, g \qquad (5)$$

where L is the depth of the packing having a specific surface S_v and porosity e, and μ and u are the viscosity and velocity of the fluid, respectively. The velocity is computed on the basis of the approach area to the packing. The factor g is the gravitational acceleration constant (= 981 cm/sec^2). The specific surface area is the area of the particles per unit volume of particles. The specific surface area can be expressed in terms of a diameter,

$$S_v = 6/D_s \qquad (6)$$

In order to apply equation 5 to hindered settling, it is necessary to obtain an expression for the driving force, $\Delta p/L$, which complies with the phenomenon. This can be achieved by regarding the downward motion of the suspension to be equivalent to an upward fluid motion just sufficient to keep the particles in suspension. Essentially, this is the procedure used in the analysis of fluidization and is contained in Loeffler's discussion of the analogy between hindered settling and fluidization. Proceeding along these lines, we may convert the pressure drop per unit length of capillary to an equivalent hydrostatic head:

$$\Delta p/L = (1 - e)(\rho_s - \rho_o)\ L/L \qquad (6)$$

where ρ_s is the density of the solid particles and ρ_o the density of the fluid. Inserting this expression in equation 1, and then multiplying through by u_o (first changing u to u_o the settling velocity), we obtain the relation

$$u_o^2(1 - e)/g\ D_s e^3 = K\ (\rho_s - \rho_o)D_s u_o/\mu \qquad (7)$$

in which K = 1/180 (approximately).

Equation 7 is similar to the equation for Stokes' settling of a single particle except for the factor given by equation 2 for which we write

$$F(e) = 18/180 = 0.10$$

Thus, we have the following equations relating hindered settling velocity to Stokes' velocity:

$$u_o/u_s = 0.1\ e^e(1 - e)(\text{Kozeny-Fair-Hatch}) \qquad (8)$$

$$u_o/u_s = e^2\ 10^{-1.82(1 - e)}(\text{Steinour}) \qquad (3)$$

It is quite clear that the first of these equations gives $u_o/u_s = 0$ when e = 1, which is incompatible with the actual situation since $u_o/u_s = 1$ for e = 1. Also, there is a difference in the diameters used in the Kozeny-Fair-Hatch equation and Stokes equation for a single particle. This may amount to an appreciable difference, although in this analysis the diameters are considered identical. If we write the first of the two equations immediately above as

$$u_o/u_s = 0.1\ e^3/(1 - e + 0.1) \qquad (8a)$$

we comply with the necessary conditions. The error in this adjustment is rather large and seems unwarranted in view of the calculations made in Table 1 below.

Recently, Richardson and Zaki (1954)[10] have examined the problem of hindered settling by assuming two ideal configurations of suspended particles and have applied straightforward hydrodynamic principles to the flow resistance of the fluid moving past the particles. Using the first configuration of Richardson and Zaki, a correction factor, $\beta\ t^{-1}$, is applied to Stokes' settling of an average particle in order to predict hindered settling velocity for any void fraction e of a suspension. Table 1 shows the calculated values of $\beta\ t^{-1}$ and compares them with those calculated from equations 3 and 8 . Considering that Richardson and Zaki are concerned with an ideal geometric system of uniform spheres, the agreement with Steinour's equation 3 and the Kozeny-Fair-Hatch correction, equation 8 , is rather striking.

Table 1. Ratio of Hindered Settling Rate, u_o, to Stokes' Settling, u_s, for Various Slurry Porosities

	u_o/u_s		
Porosity (e)	Richardson and Zaki, $\beta\ t^{-1}$ (Configuration I)	Equation 3	Equation 8
0.4	0.006	0.013	0.011
.5	.026	.031	.025
.6	.066	.067	.054
.7	.132	.140	.114
.8	.241	.199	.256
.9	.474	.530	.729

3.2 Hindered Settling Correlation

Equation 7 is a relation between two dimensionless groups--the Froude group and the Reynolds group. In general, we would expect for hindered settling that if

$$Fr = f(Re)$$

the necessary requirements for hindered settling are complied with, provided the assumption made immediately above is pertinent.

Figure 2 shows a plot of a representative collection of available data on hindered settling. The modified Froude number is plotted as the ordinate against the Reynolds number in which the apparent density of the suspension is used. The values of D_s are for the most part obtained by permeametry, although in some instances they were derived from size distribution data obtained from microscopic measurements.

The data included in Fig. 2 include materials of different shapes with densities ranging from about 1.1 to 4.0 and particle sizes from about 8 to as high as 1000 microns. The suspending liquids were mostly water, but, as may be seen, ethylene glycol (ρ_o = 1.1, and μ = 15.5 cp) and, in the special case of tapioca, a special oil (ρ_o = 0.89, μ = 7.13 p) have been used. The data are chiefly those of Steinour, Loeffler, and Gilliland.

Figure 2 indicates a fairly good correlation, even with Steinour's flocculated materials for which he indicates slight adhered-water corrections. The slope of the points plotted is unity, as called for by equation 7. The intercept called for in that equation is 5.56×10^{-3} or approximately 6.0×10^{-3} within the limitations of the scatter.

4.0 DETERMINATION OF PARTICLE DIAMETERS BY HINDERED SETTLING

It is obvious from the linear relation of data plotted in Fig. 2 that it permits the use of equation 7 for the computation of the specific surface diameter, D_s, of the suspended particles, provided, of course, that their motion is hindered. Rearranging equation 7 and using the intercept value of 6×10^{-3} (for convenience), we obtain

$$D_s = 13 \left[\frac{u_o \mu}{g\,(\rho_s - \rho_o)} \cdot \frac{1 - e}{e^3} \right]^{1/2} \qquad (9)$$

in which the dimensions are in cgs units (D_s in cm; u_o in cm/sec, μ in poise). This equation provides a simple means for determining a measure of particle diameter which corresponds to that obtained by the usual permeametric techniques. It is a very simple matter to observe the rate of fall of an interface. The other constants involved are usually available from handbooks or are easily measured.

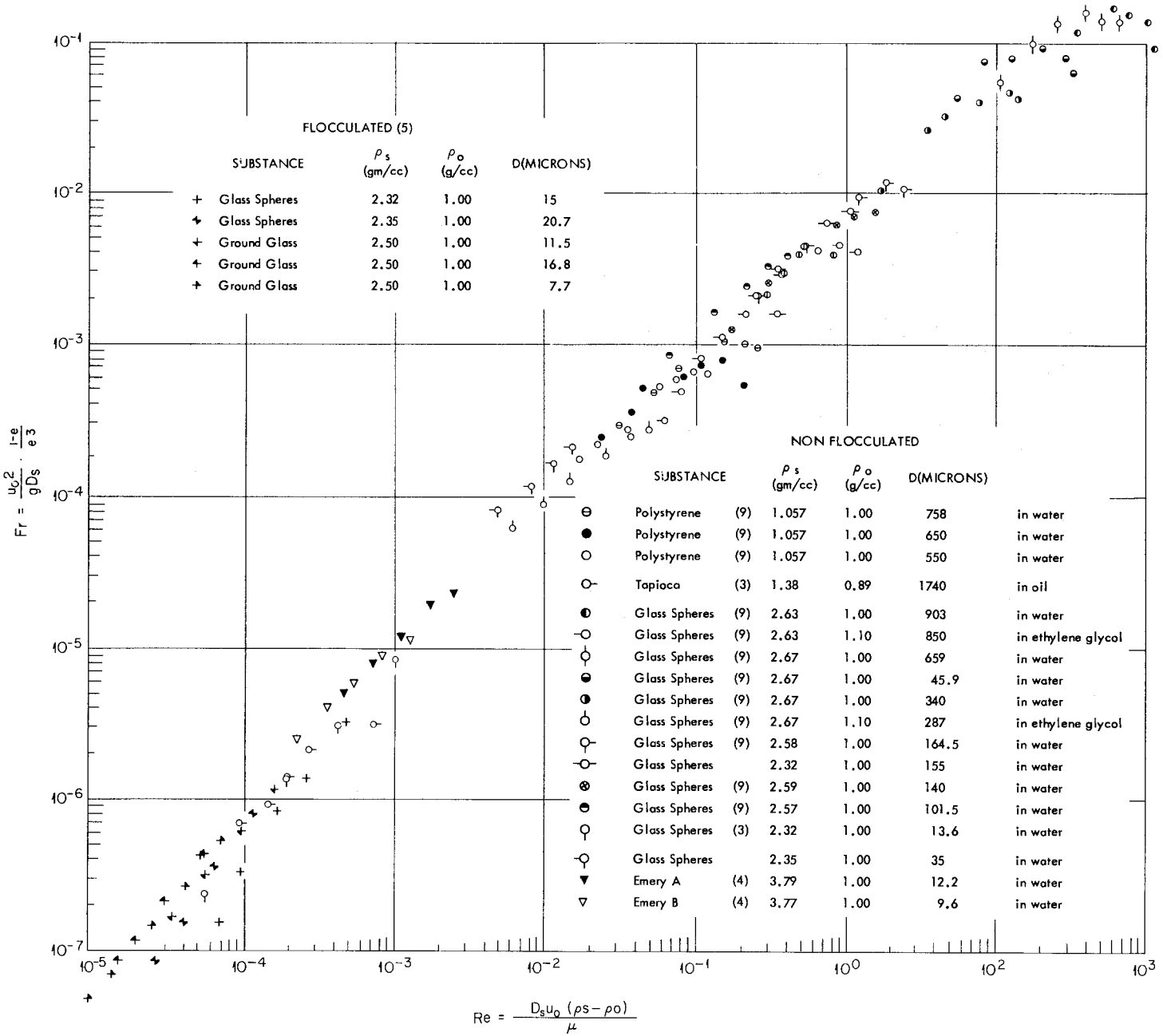

FIG. 2. FROUDE-REYNOLDS RELATION FOR HINDERED SETTLING.

5.0 EXPERIMENTAL RESULTS

Equation 9 was used to calculate the specific surface diameter of the particles in aqueous thorium oxide suspension (Table 2). The settling data were obtained with 250,500, and 650 g Th/kg H_2O suspensions of thorium oxide calcined at various temperatures from 650 to 1300°C (cf Figs. 3, 4, and 5). Diameters were calculated only from the hindered settling rates since equation 9 does not apply to suspensions which are in compaction. Suspensions of the thorium oxide calcined at 650 and 800°C were in compaction at concentrations above 500 g Th/kg H_2O, suspensions of the higher fired oxides at concentrations above 650 g Th/kg H_2O.

The specific surface diameters, calculated using equation 9, increased with increasing calcination temperature, from 4.0 μ at 650°C to 7.2 μ at 1300°C (cf Table 2). The diameters were much larger than those obtained using the ordinary sedimentation techniques in dilute, dispersed systems, which for the thorium oxide used gave a particle diameter of about 1 μ. Since the systems were flocculated the diameters calculated by equation 9 may have a rheological significance and could represent the actual "working" particle in a suspension.

The effect of sulfuric acid additions on the settling rates and specific surface diameter of 900°C fired thorium oxide in aqueous suspension is shown in Table 3. It is to be noted that up to about 2500 ppm of sulfate, the rate of initial settling is the same at all sulfate concentrations. Above this amount, the rate of settling is very much affected by the presence of sulfuric acid. The dispersion and fineness of particles (as implied by the decrease in hindered settling) is enhanced as the pH falls below a value of 5.7.

Table 2. Rate of Hindered Settling and Specific Surface Diameters in Aqueous Suspensions of ThO_2 Calcined at Various Temperatures

Concentration, g Th/kg H_2O	Hindered Settling Rates, in cm/sec, and Specific Surface Diameters, in microns (for indicated oxide calcination temperature)									
	650°C		800°C		900°C		1000°C		1300°C	
	u_o	D_s	u_o	D_s	u_o	D_s	u_o	D_s	u_o	D_s
250	0.05	3.2	0.06	3.5	0.075	3.9	0.10	4.5	0.123	5.0
500	.022	4.7	.029	5.7	.038	6.5	.085	9.6	.032	10.7
650	---	---	---	---	.016	5.8	.024	6.4	.024	6.0
		4.0 (avg.)		4.6 (avg.)		5.4 (avg.)		6.8 (avg.)		7.2 (avg.)

D_s, specific surface diameter, calculated from equation 9.

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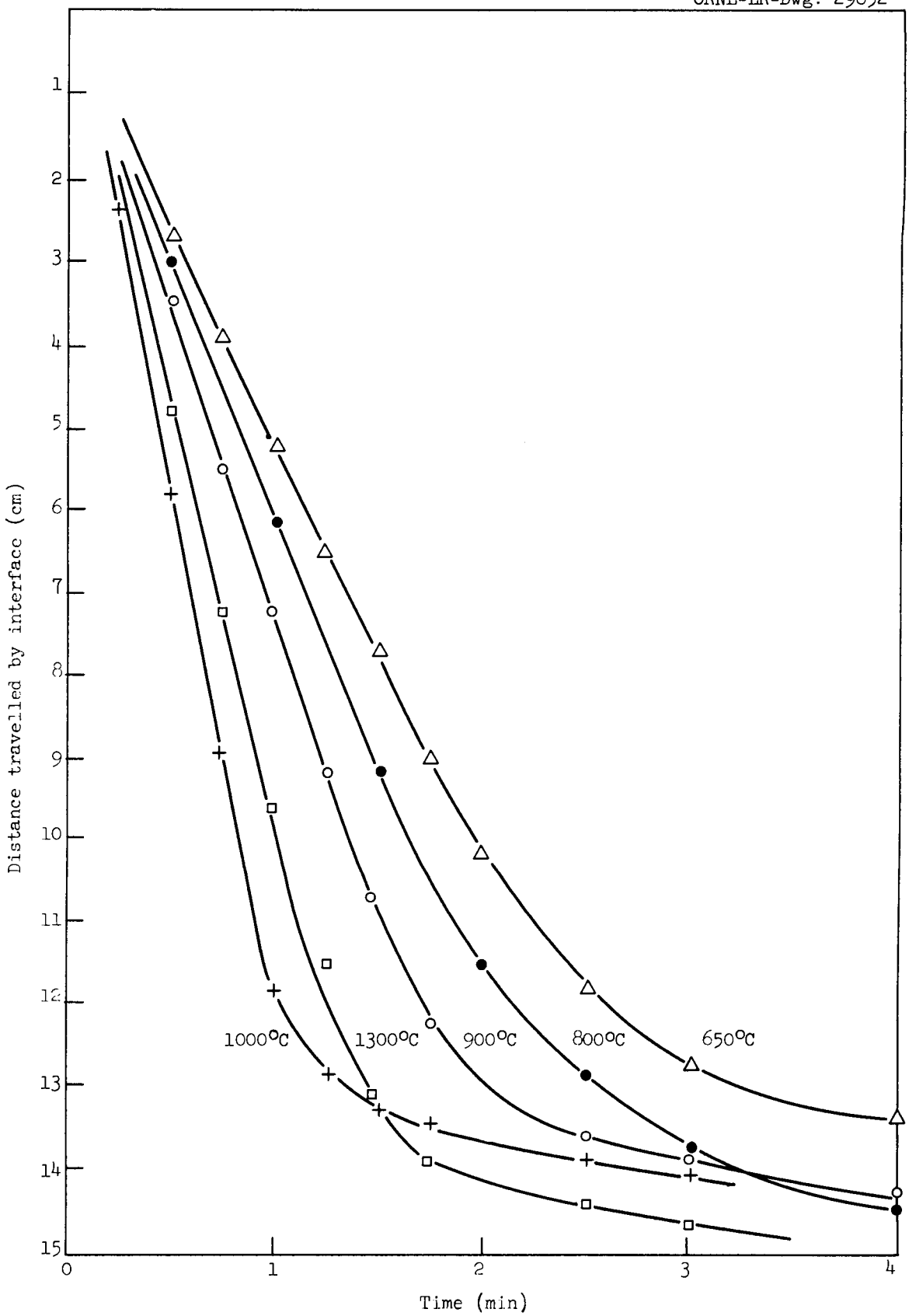

Fig. 3. Room Temperature Settling Curves for Aqueous Slurries Containing 250 g Th/kg H_2O of D-16 Thorium Oxide Calcined at Indicated Temperatures (slurries tumbled 24 hr before settling in 100-ml graduated cylinders)

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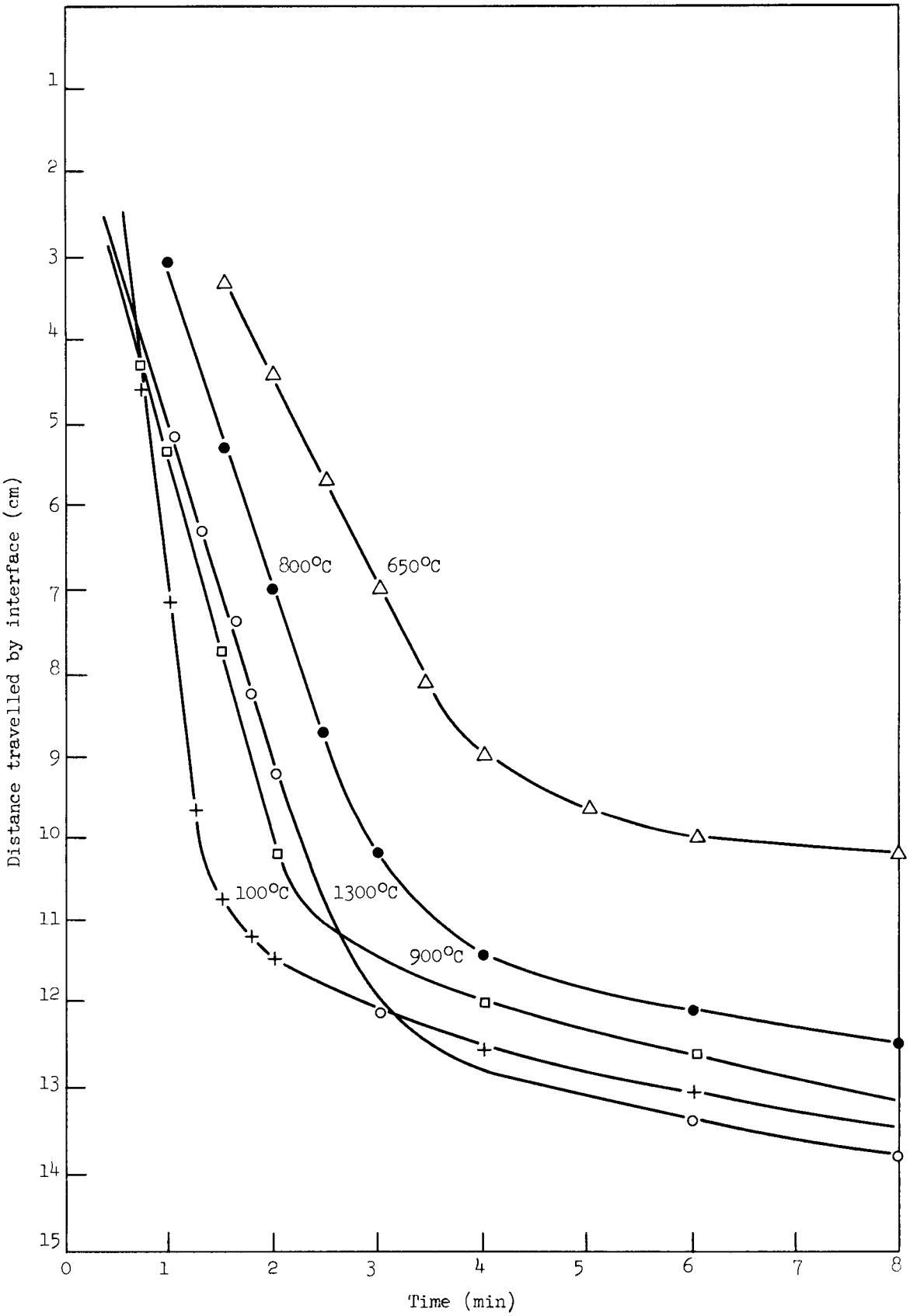

Fig 4. Room Temperature Settling Curves for Aqueous Slurries Containing 500 g Th/kg H_2O of D-16 Thorium Oxide Calcined at Indicated Temperatures (slurries tumbled 24 hr before settling in 100-ml graduated cylinders)

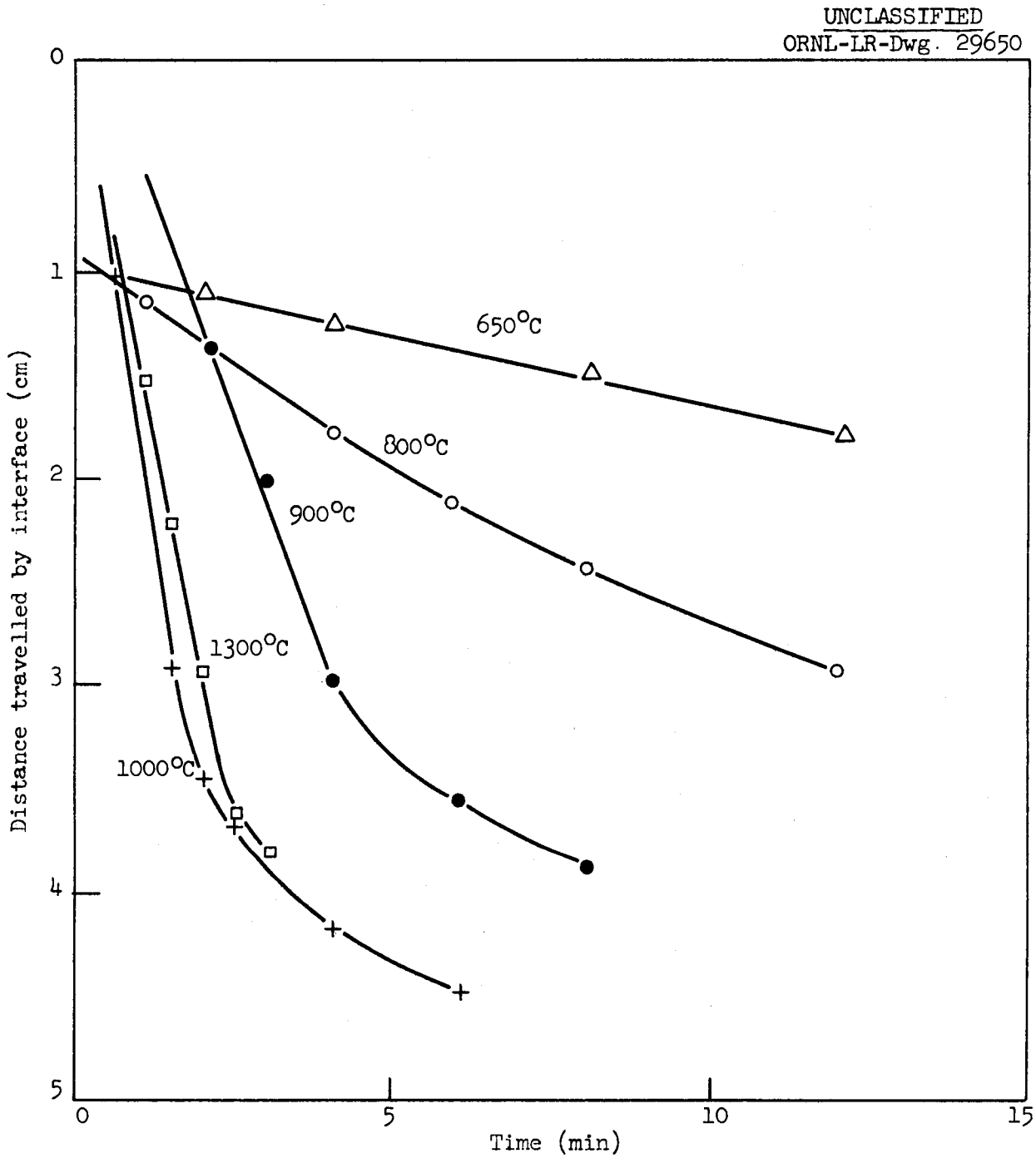

Fig. 5. Room Temperature Settling Curves for Aqueous Slurries Containing 650 g Th/kg H_2O of D-16 Thorium Oxide Calcined at Indicated Temperatures (slurries tumbled 2 hr before settling in 100-ml graduates)

Table 3. Settling Rates and Specific Surface Diameters in Thorium Oxide Slurries Containing Sulfuric Acid

Conditions: 900°C fired ThO_2

500 g Th/kg H_2O

Room temperature

Sulfuric Acid Concentration[a] (ppm)	Slurry pH	Solution pH	Settled Density (g/liter)	Settling Rate, u_o (cm/sec)	Specific Surface Diameter, D_s[b] (μ)
0	9.7	8.7	1670	0.0565	7.94
200	9.7	8.7	1660	.0514	7.56
500	9.7	8.7	1655	.0694	8.56
1000	9.0	8.2	1630	.0644	8.46
2500	7.4	5.7	1700	.0489	7.36
5000	3.3	3.1	1865	.00667	2.71
20,000	1.6	1.5	1450	.00079	0.82

[a]Based on ThO_2.

[b]D_s, specific surface diameter, calculated using equation 9.

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Professor DallaValle passed away during the final stages of the preparation of this report for publication. The remaining authors express their regret at the loss of a good friend and worthy scientist.

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