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AN EVALUATION OF HYDROCLONES FOR SIZE CLASSIFICATION OF THORIUM OXIDE

E. L. Youngblood
P. A. Haas

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CHEMICAL TECHNOLOGY DIVISION

Unit Operations Section

An Evaluation of Hydroclones for Size Classification of Thorium Oxide

E. L. Youngblood
P. A. Haas

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ABSTRACT

The performance of hydraulic cyclones or hydroclones was evaluated theoretically and experimentally for size classification of thorium oxide. Oversize material was removed from 1-3 μ mean diameter thorium oxide by use of commercial (modified TM-3 Dorr Clones) equipment. Batch and continuous classification were demonstrated with product rates of up to 1 kg of ThO_2 per minute and removal of over 95% of material three times the mean diameter. Good agreement was obtained between experimental results and calculations from literature correlations. Dispersion was not complete even with dispersing agents; therefore, effective removal of undersize ThO_2 was not practical. The advantages of hydroclones are that they require no dispersing agents and have an ability to classify below 7-μ material, as compared with hydraulic classification by sedimentation and air classification, respectively.

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1.0 SUMMARY

The single hydroclone studies show that removal of oversize particles from thorium oxide having a mean particle size from 1 to 5 μ can be accomplished in a 0.4-in.-dia hydroclone without the use of dispersing agents. The performance of a hydroclone for classifying thorium oxide particles can be illustrated by the relative concentration of the feed and product streams for various particle sizes shown in Fig. 1 for a single hydroclone. The shape of this curve is typical for all hydroclone classification performance. The shape and location of the curve in regard to particle diameter is dependent on the number of stages used in series and the d_{50} value* at which the stages are operated. In general an increase in d_{50} tends to shift the curve toward larger particle diameters while increasing the number of hydroclones in series tends to shift the curve toward lower particle diameters and increase the slope of the curve. Variations in operating conditions to change the value of d_{50} permit control over the classification range of a hydroclone system.

The particle size distribution of the product and the hydroclone efficiency for thorium oxide-water slurries can be estimated reasonably well by the use of equation (1) and a reduced efficiency curve.

$$d_{50} = 5 \times 10^3 \frac{D_c^{0.5}\ \mu^{0.5}}{(\Delta P)^{0.25}(\rho_s - \rho)^{0.5}} \quad ; \qquad (1)$$

D_c = the hydroclone inside diameter at the feed port, in.,
μ = the fluid viscosity, lb/ft.sec,
ΔP = feed to overflow pressure drop, ft. of fluid,
ρ = density of liquid phase, lb/ft^3,
ρ_s = density of solid phase, lb/ft^3.

This procedure gives good estimated values for particle size distribution for dilute slurries where particle interaction is not significant. At higher concentrations (above 50 g/liter), the estimated value of d_{50} for determining the overflow size distribution resulted in particle size distributions somewhat lower than the experimentally determined values. Dispersion of the ThO_2 is not complete, and the undispersed ThO_2 behaves as material of large particle size. Therefore, classification to remove undersize material is not as easily done as the removal of oversize material.

A modified Tm-3 Dorr Clone system described in this report appears suitable for removal of oversize particles on a production scale from thorium oxide feeds having particle size distributions in the range of 1 to 5 μ. With possible product withdrawal rates of from 0 to 20 liters/min and normal overflow

* d_{50} is the diameter of the particles (in microns) which are removed with a 50% efficiency.

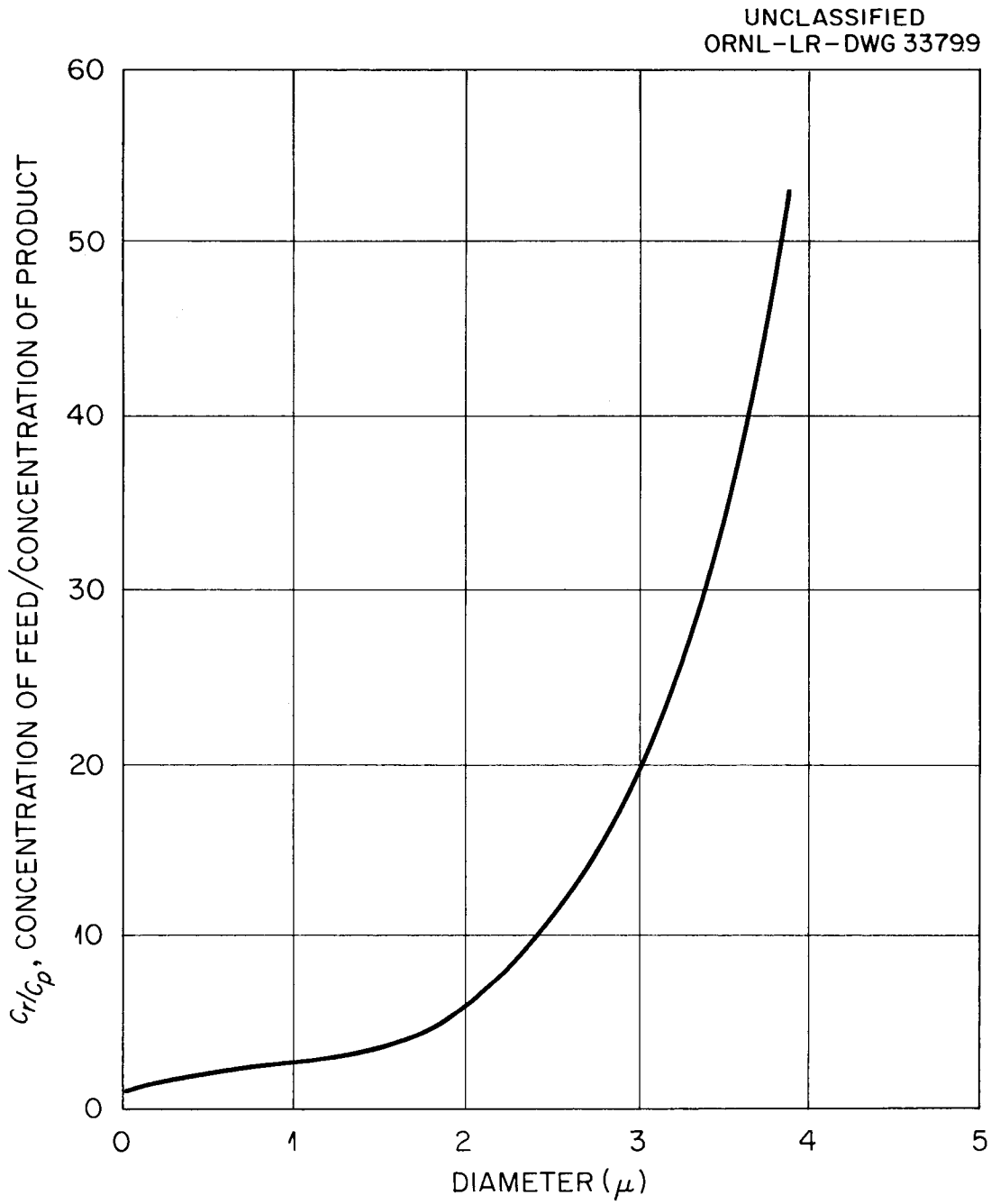

Fig. 1. Typical Separation Curve for a Single 0.4-in.-dia Hydroclone and a Thorium Oxide-Water Slurry.

concentrations of 10 to 50 g/liter, a production rate of up to 1 kg/min of product is possible. Both batch and continuous operation of the unit were acceptable from an operational standpoint; however, because of the holdup of thorium oxide in the continuous system, the batch system appears more suitable for processing small batches of thorium oxide while the continuous system is more suitable for processing large quantities of material all having the same particle size distribution.

The yield of product obtained from the hydroclone system is dependent to a large extent on the particle size distribution of the feed, the acceptable size, and the per cent of oversize material in the product. As is shown in Fig. 1, the greater the difference in the diameter of the particles the more easily they can be separated. Normally thorium oxide is precipitated to give a large per cent of the particles in the desired size range, and only a few per cent are larger than this value. The removal of the small per cent of particles more than 2 or 3 times the mean particle size of the feed has been accomplished with recovery of up to 70% of the fine particles available as product containing 99% of the material about twice the mean diameter. (The accuracy of the size analysis does not permit estimation of product purity above about 99%).

In both the batch and continuous systems the waste material contained a considerable amount of fine particles. Additional processing of this waste would be required to remove the fine particles in order to obtain high yields. The processing of this waste would mean a certain amount of recycling of material through the classification system; however, the hydroclone system compares favorably in this respect with gravity sedimentation and air classification (Appendix) which also require recycle of the waste streams for high product yield.

The 0.4-in.-dia hydroclone compares favorably with gravity sedimentation in classification ability and product yield for removing oversize particles from thorium oxide in the 1 to 5-in.-dia and offers the additional advantage that a dispersing agent is not required for classification. The air classification system described in the Appendix is useful for classifying thorium oxide particles above about 7 μ but does not classify particles below that size.

2.0 INTRODUCTION

Although the major application of the hydraulic cyclone or hydroclone has been as a liquid-solids separator, it is recognized to be a very effective particle classifier. The high shear forces and centrifugal forces developed in hydroclones are generally sufficient to disperse flocculated material without the use of dispersing agents which are required by some classification systems. Systems using hydroclones for size classification are relatively simple and the range of classification can be varied by changes in operating variables and by changes in hydroclone diameter.

Thorium oxide particles which have been produced by precipitation of thorium nitrate with oxalic acid and subsequently fired to 1600°C to decompose the oxalate normally contain several per cent oversize material (particles which

are considerably larger than the mean particle size). Since these oversize particles cause increased erosion in dynamic slurry systems, it is desirable to incorporate a classification step in the thorium oxide production system to remove them. The thorium oxide particle range of from 1.5 to 5 μ which has been found most desirable from the standpoint of erosion and yield strength (1) is below the range of most standard classifiers. In this report an investigation of the use of single and multiple hydraulic cyclones or hydroclones has been made to determine their suitability for thorium oxide classification. The multiple hydroclones used were commercially available TM-3 Dorr Clones which are 0.4-in.-i.d. at the feed port. Also presented for comparison is information on air classification and gravity sedimentation as a means of thorium oxide classification.

The two factors of major importance in the classification of thorium oxide particles are the removal of oversize particles from feeds having a particle size distribution in the range of 1 to 5 μ and the recovery of a large percentage of the available particles as product. Several variations in operating procedure and equipment arrangement were used to investigate the behavior of the hydroclone classifications system. The systems were operated on both continuous and batch basis.

The hydroclone studies were made by personnel of the Unit Operations Section. The air classification studies were made by the Barrier Development Section of the Oak Ridge Gaseous Diffusion Plant using a Sharples Super Classifier. The information on gravity sedimentation was obtained from the thorium oxide production facility of the Oak Ridge National Laboratory Chemical Technology Pilot Plant Section. The particle size analysis for the hydroclone studies were made by the Analytical Chemistry Division at Oak Ridge National Laboratory using a neutron activation technique in which the rate of particle sedimentation in water was determined by radiation counting.

3.0 HYDROCLONE THEORY

A considerable amount of information has been published on the theory of operation of hydraulic cyclones (2,3,4,5); however, it has not been developed sufficiently to permit hydroclone operation to be completely predicted for any given material without test operation. An understanding of the theory of operation of hydroclones is very helpful in determining how operating variables affect performance. An indication of how classification is accomplished in hydroclones and how their performance can be estimated is given in this section.

The hydroclone consists essentially of a conical chamber with exit ports located at the apex and base of the cone (Fig. 2). Liquid along with the particles to be classified is fed tangentially into the cone producing a spiral flow pattern in which the fluid and larger particles near the wall report to the underflow while the fluid and fine particles near the center of the hydroclone report to the overflow. In determining whether a given particle size will remain near the wall and be discharged to the underflow or will move toward the center and report to the overflow, the flow pattern in the system must be considered. After entering the feed port, the fluid has both tangential and

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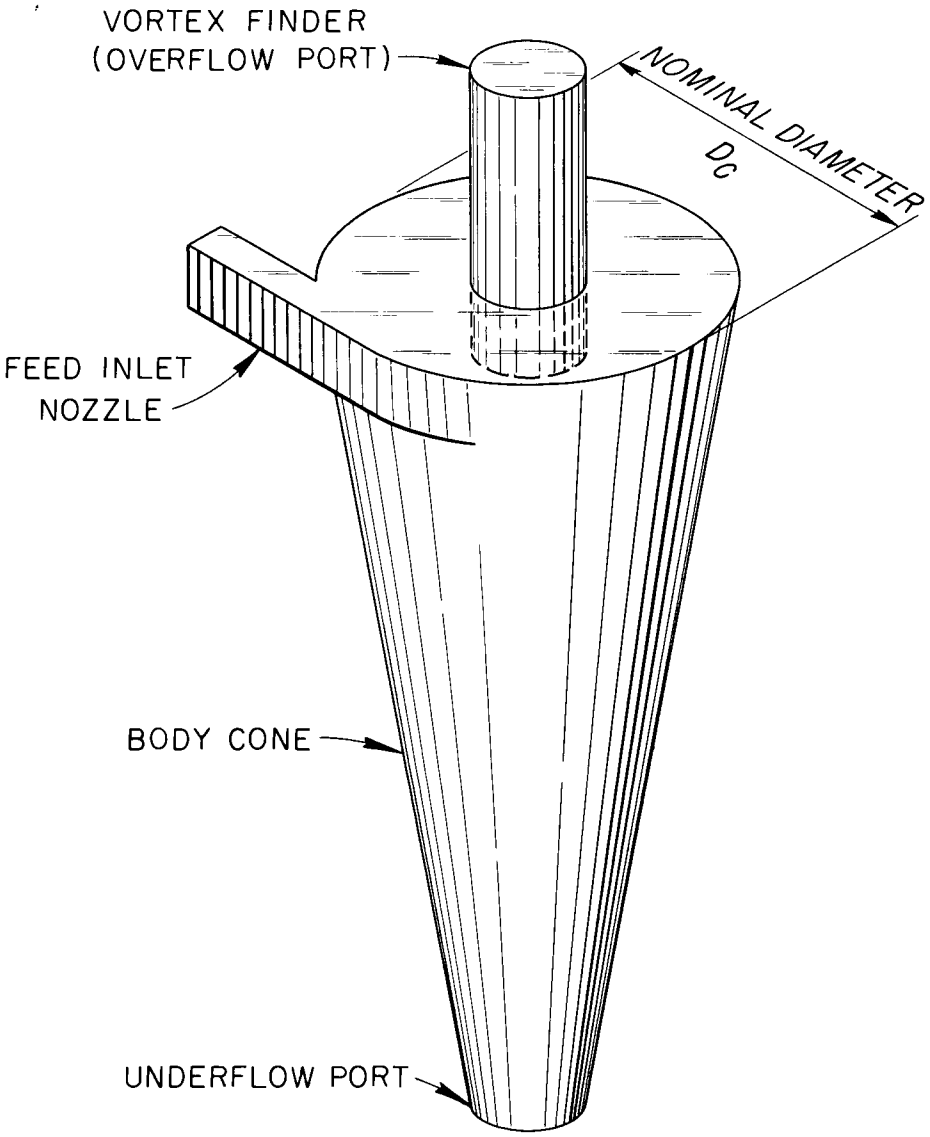

Fig. 2. Typical Hydroclone.

radial velocity components. The tangential component initially increases as the fluid moves toward the center of the hydroclone and then drops off rapidly near the center. During the period in which the tangential velocity is increasing the variation of the velocity with radius can be expressed as follows: (2)

$$Vr^n = K , \qquad (2)$$

where

V = tangential velocity,
r = radial distance,
n = constant,
K = constant.

Since centrifugal force is proportional to V^2/r it also increases as the fluid moves toward the center of the hydroclone. The force moving a particle toward the center of the hydroclone is the viscous drag of the fluid caused by the radial velocity component. For the below-5-μ ThO_2 particles, this force may be expressed by Stokes' Law.(3) At some radius in the hydroclone the centrifugal force on larger particles increases sufficiently to overcome the viscous drag of the fluid and the inward motion of the particle is stopped. This radius may be expressed by the following equation (ref. 2):

$$U = \frac{V^2}{r} \cdot \frac{d^2(\rho_s - \rho)}{18\,\mu} \quad ; \qquad (3)$$

U = radial velocity,
d = particle diameter,
ρ_s = density of solid,
ρ = density of fluid,
μ = viscosity of fluid.

Since the radius at which a particle is stopped is a function of its diameter, larger particles stop nearer the hydroclone wall than smaller particles. If the inward motion of the particle is stopped sufficiently close to the wall to be in an area in which liquid reports to the underflow, the particle will also report to the underflow. However, if the particle is small enough to reach a region near the center of the hydroclone, it will be discharged to the overflow. The location at which the inward radial velocity of the particle is stopped (equilibrium location) has been determined by Kelsall(2) for a 3-in.-dia unit and is shown in Fig. 3.

When the equilibrium location of a particle lies at the boundary of the two discharge streams, it will have an equal chance of entering either stream. The particle size which will report equally to the overflow and underflow can be estimated by the following equation for small-diameter hydroclones.(4)

$$d_{50} = 5 \times 10^3 \frac{D_c^{0.5}\,\mu^{0.5}}{(\Delta\rho)^{0.25}\,(\rho_s - \rho)^{0.5}} \quad ; \qquad (1)$$

d_{50} = diameter of particle removed with 50% efficiency, μ,
D_c = hydroclone inside diameter at feed port, in.,
ΔP = feed to overflow pressure drop, ft of liquid,
μ = viscosity of fluid, lb/ft·sec,
ρ = density of liquid phase, lb/ft^3
ρ_s = density of solid phase, lb/ft^3.

The value of d_{50} for a hydroclone system is frequently used as a basis for estimating the performance of the unit.

The relationship between particle diameter and hydroclone efficiency (fraction of feed solids reporting to the underflow) can be estimated when d_{50} is known by the use of the reduced efficiency curves by Yoshioka and Hotta(5) shown in Fig. 4. This curve was obtained from data using a 6-in.-dia hydroclone. Other recently reported data gives a similar, but not identical, reduced efficiency curve for a 0.4-in.-dia hydroclone.(12) The reduced efficiency curve includes only the solids reporting to the underflow stream as a result of centrifugal separation and therefore does not consider fine solids reporting to the underflow as a result of entrainment in the underflow liquid. If 50% of the feed liquid is discharged through the underflow, this stream will contain 50% of all particle sizes plus the large particles centrifugally removed form the liquid reporting to the overflow stream. The total fraction of solids discharged to the underflow stream is related to the reduced efficiency by the following equation:

$$E = E_o + \frac{B}{F}(1 - E_o); \qquad (4)$$

E = total fraction of feed solids discharged to hydroclone underflow,
E_o = reduced efficiency, fraction of feed solids discharged to underflow due to centrifugal separation,
B = underflow liquid rate,
F = feed liquid rate.

The use of equations (1) and (4) along with the reduced efficiency curve in Fig. 4 enable efficiencies for various particle sizes to be estimated. Efficiencies for feeds which contain a distribution of particle sizes may also be estimated by considering small particle size groups within the feed distribution and summing the contribution from each group. The particle size distribution of the overflow and underflow streams may also be estimated by this method. Efficiencies and particle size distributions calculated by this procedure have been found to be in fair agreement with experimental results for thorium oxide slurries in 0.4-in.-dia hydroclones. Sample calculations for efficiency and particle size estimation are given in the appendix.

The use of the above method of calculating hydroclone performance assumes that the hydroclone is properly designed. A considerable amount of data has been published on hydroclone design(4), and this aspect has not been covered in this report.

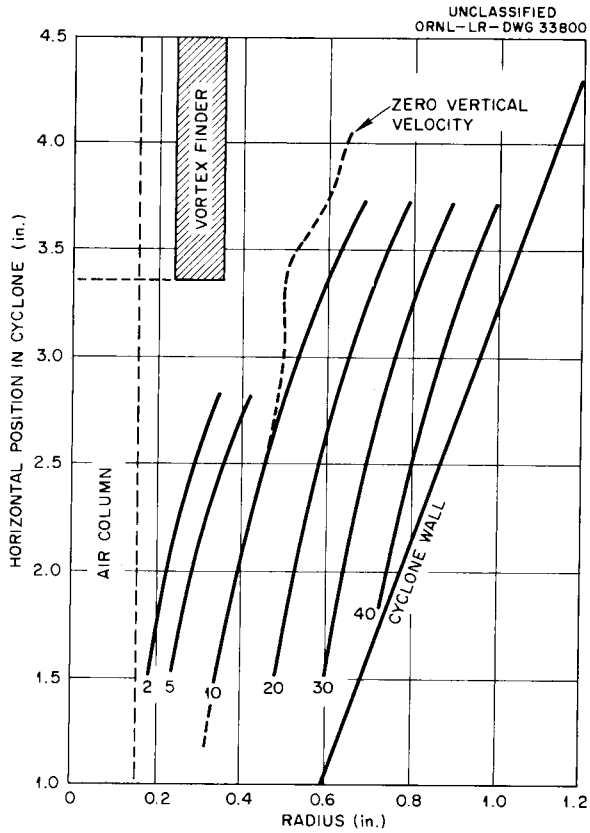

Fig. 3. Particle Equilibrium Locations in a 3-in.-Dia Hydroclone. Data from D.F. Kelsall.

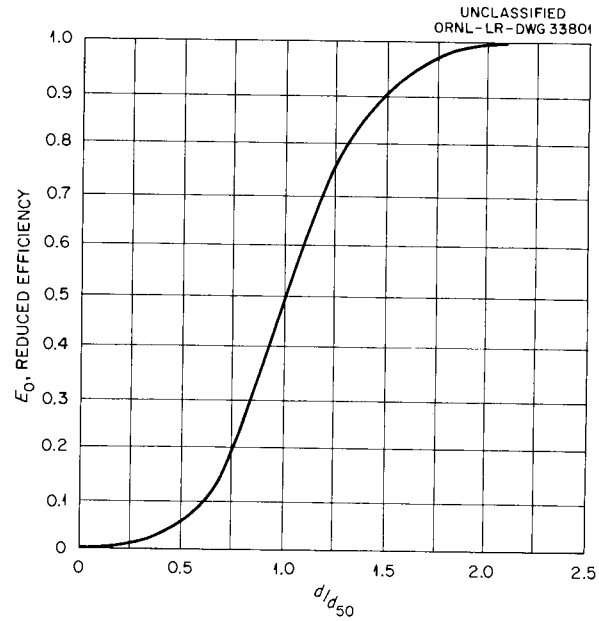

Fig. 4. Hydroclone Reduced Efficiency Curve. Data from N. Yoshioka and Y. Hotta.

4.0 HYDROCLONE CLASSIFICATION RANGE

The major variables effecting the range of classification of hydroclones, as indicated by equation (1), are the hydroclone diameter, the feed-to-overflow pressure drop, the density of the particles to be classified, and the density and viscosity of the liquid medium. For a particular slurry, such as thorium oxide and water at a constant temperature, the classification range is primarily dependent on the diameter of the hydroclone and feed pressure. The range can also be varied somewhat by changes in temperature of the system which effect the density and viscosity of the fluid.

The effect of feed pressure on d_{50} for a single 0.4-in.-dia hydroclone using a thorium oxide water slurry at 22°C and 70°C is shown in Fig. 5. The practical operating feed pressure for a hydroclone is limited at lower pressures by the minimum flow required to establish a vortex and the tendency for particles to settle out of suspension at low flow rates. At higher feed pressures the system is limited by the strength of the unit and the available capacity of the pumps; however, as shown in Fig. 5, the effect of feed pressure on d_{50} becomes less significant at higher feed pressures. In the case of plastic feeds higher feed pressures are sometimes desirable since the increased centrifugal forces tend to aid in causing particles to tear through the plastic structure of the solids. Higher feed pressures are also desirable from the standpoint of increased flow capacity. Hydroclones less than 1 in. in diameter which were designed for optimum performance have been reported to have flow capacities which vary with the feed to overflow pressure to the 0.44 power.(4)

The relationship between d_{50} and efficiency shown in Fig. 4 indicates that there should be essentially complete removal of particles larger than 2 or 3 times d_{50} from the overflow stream; therefore, it would not be practical from the standpoint of product recovery to recover particles larger than this value as fines to the overflow stream. This places a limitation of about 5 μ as the maximum diameter thorium oxide particle which can be effectively recovered in the overflow of a 0.4-in.-dia hydroclone when water is used as the suspending medium. Thorium oxide particles smaller than about one micron become difficult to remove from the overflow stream; therefore, classification of particles below this size is difficult. Thus the effective range of classification of the 0.4-in.-dia hydroclone for this system is from about 1 to 5 μ diameter. Inside this size range classification can be controlled by feed-to-overflow pressure drop and temperature. For classification outside this range other diameter hydroclones would be required. However, this range corresponds nicely with the desired thorium oxide particle size. The optimum feed pressure for maximum separation is dependent to a large extent on the particle size distribution of the feed and the desired cut point (diameter above which material is considered oversize).

An indication of the nominal range of classification of larger diameter hydroclones has been reported as follows:(6)

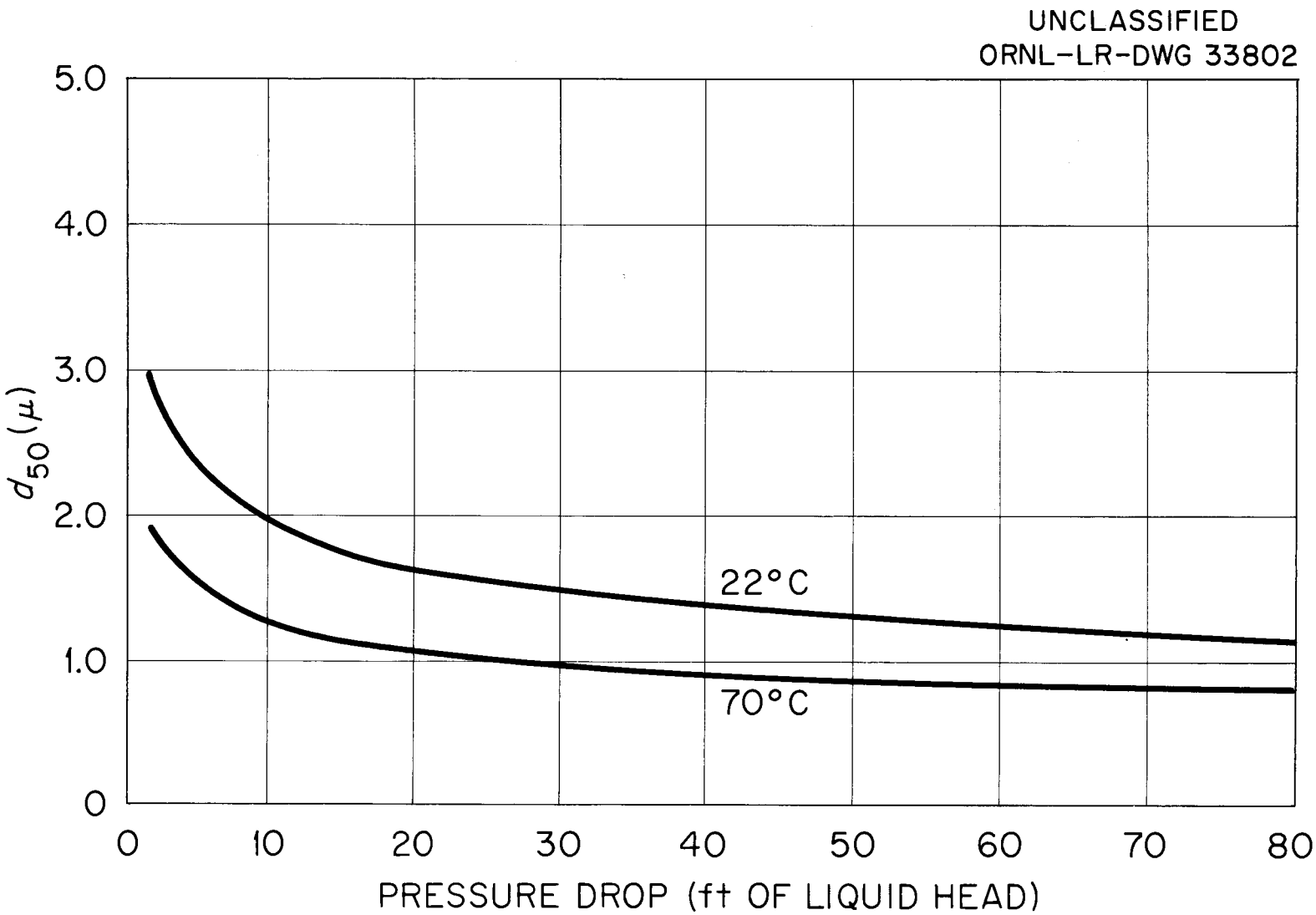

Fig. 5. Effect of Feed-to-Overflow Pressure on d_{50} for 0.4-in.-Dia Hydroclone Using a Thorium Oxide-Water Feed Slurry.

Hydroclone Diameter (In.)	Nominal Classification Size Range (μ)
3	5 - 20
8	20 - 40
14	100 - 325

The range of classification for thorium oxide would probably be limited to the lower end of the above ranges because of its relatively high density. Fitch and Johnson(7) have reported that the classification size of the hydroclone is influenced to some extent by the particle size distribution of the feed. Hydroclones operating under the same conditions give higher mesh of separation (size removed with 95% efficiency to the underflow) for feed containing larger size particles.

5.0 SINGLE HYDROCLONE STUDIES

The initial phase of the thorium oxide classification studies was made using a single 0.4-in.-dia hydroclone. The single hydroclone system was used to determine the effect of operating variables which are somewhat dependent on the nature of the feed material used and could not be extrapolated from existing data. Feed material used in all runs was 1600°C-fired thorium oxide obtained from the Chemical Technology Division Pilot Plant Section.

5.1 Equipment

The equipment consisted of a 0.4-in.-dia stainless steel hydroclone of the dimensions given in Table 1. Feed pressure was maintained by an Eastern Centrifugal Pump. Pressure gages and calibration tubes were used to measure pressure drops and flow rates. A flow diagram of the system is shown in Fig. 6. Runs were made by adjusting the throttling valves to give the desired pressures and flow distributions.

Table 1 Hydroclone Dimensions

All dimensions in inches

Hydroclone Diameter	0.4
Feed Port	0.1 x 0.11
Underflow Port Diameter	0.1
Overflow Port Diameter	0.1
Hydroclone Length (from vortex finder to underflow port)	2.4

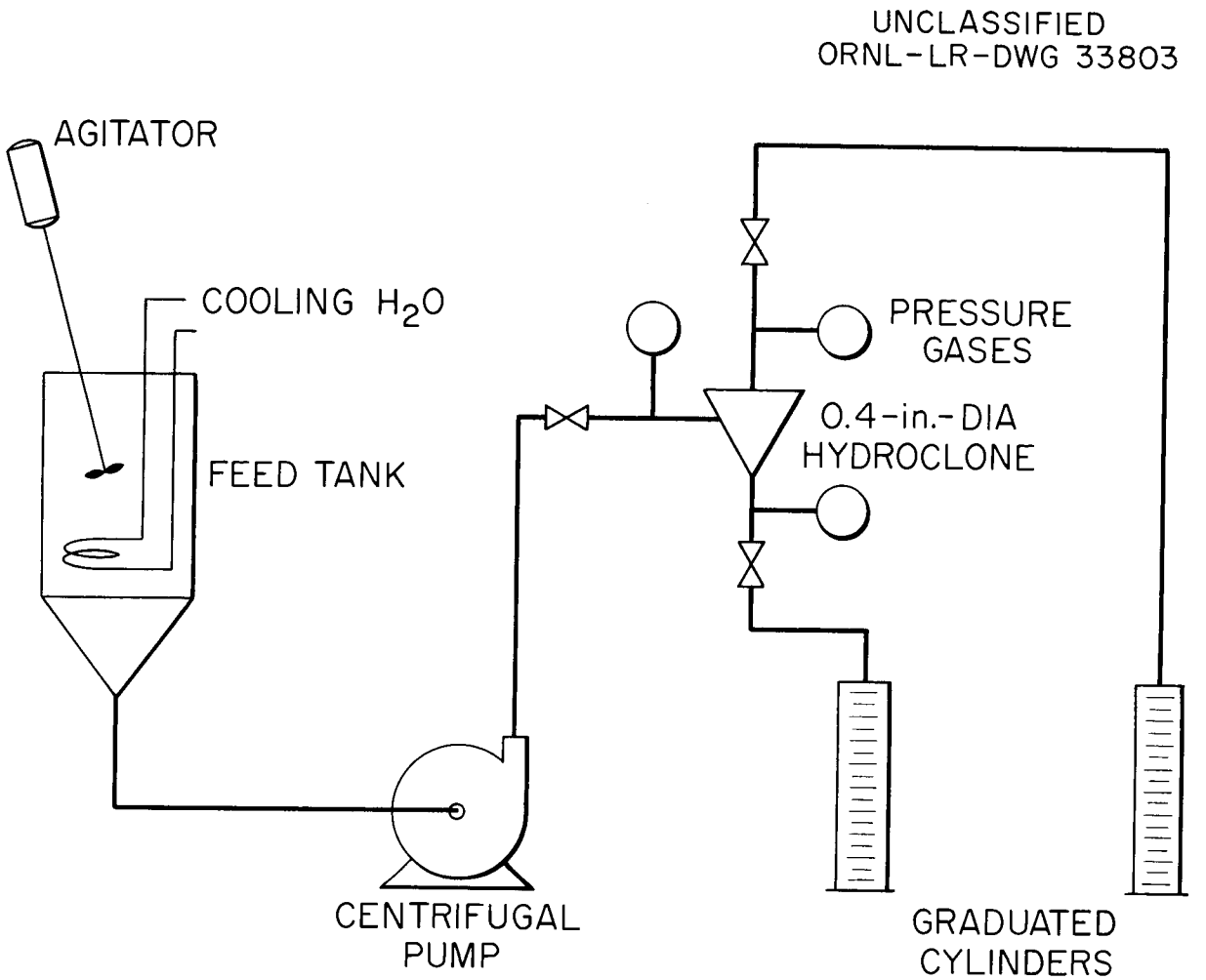

Fig. 6. Equipment for Single Hydroclone Test.

5.2 Effect of Dispersing Agents

The high shear and centrifugal forces developed in the hydroclone makes it suitable for classifying most flocculated material without the use of dispersing agents. It has been reported that hydroclones are capable of dispersing all but the most tightly bound flocs.(7) Slurries of thorium oxide and water normally have a tendency to flocculate, thus requiring a dispersing agent such as oxalic acid or sodium pyrophosphate for gravity classification.

Particle size distribution for the hydroclone overflow stream for both dispersed and undispersed feed shown in Figs. 7 and 8 indicate that comparable classification is accomplished in hydroclones for undispersed feeds as compared with dispersed feeds. This is in agreement with other reported data on thorium oxide slurries.(8) Figure 9 shows that results for classification of methyl alcohol slurries of thorium oxide are very similar to those for water slurries. The calculated particle size distribution based on the method previously outlined is also given.

Although the particle size distribution of the overflow stream is not greatly changed by the addition of dispersing agents, the over-all recovery of fine particles to the overflow stream is increased by dispersed feeds as shown in the reduced efficiency curves in Fig. 10. This increase is probably attributable to incomplete deflocculation without the dispersing agent which results in some of the flocculated material behaving as larger particles and thus reporting to the underflow stream.

5.3 Effect of Feed Concentration

As the solids concentration of the feed increases the interaction of the particles becomes greater and the effectiveness of the hydroclone as a classifier is decreased. The literature reports that this effect can usually be neglected for slurries in which the solids-to-liquid volume does not exceed a 1 to 8 ratio.(7) The effect of feed concentration on the particle size distribution from the hydroclone overflow stream is shown in Fig. 11. These results show an increase in the mean particle size of the fine particles from the overflow stream as the feed concentration is increased. There is also a decrease in hydroclone efficiency (fraction of feed solids reporting to underflow) with increased feed concentration as shown in Fig. 12. In general, feed concentrations up to 300 to 400 g/liter thorium oxide do not greatly reduce the effectiveness of the hydroclone as a classifier.

5.4 Effect of Flow Distribution

A certain fraction of the feed liquid must be discharged through the underflow stream to insure complete removal of large particles. If an insufficient amount of liquid is discharged through this stream, the hydroclone may become plugged or large particles may be forced into the overflow stream. However, in operating a single hydroclone, it is not desirable to discharge an excessive amount of liquid through the underflow since this increases the loss of fine particles to this stream. It would normally be expected that higher underflow rates would be required for more concentrated feeds to insure adequate removal

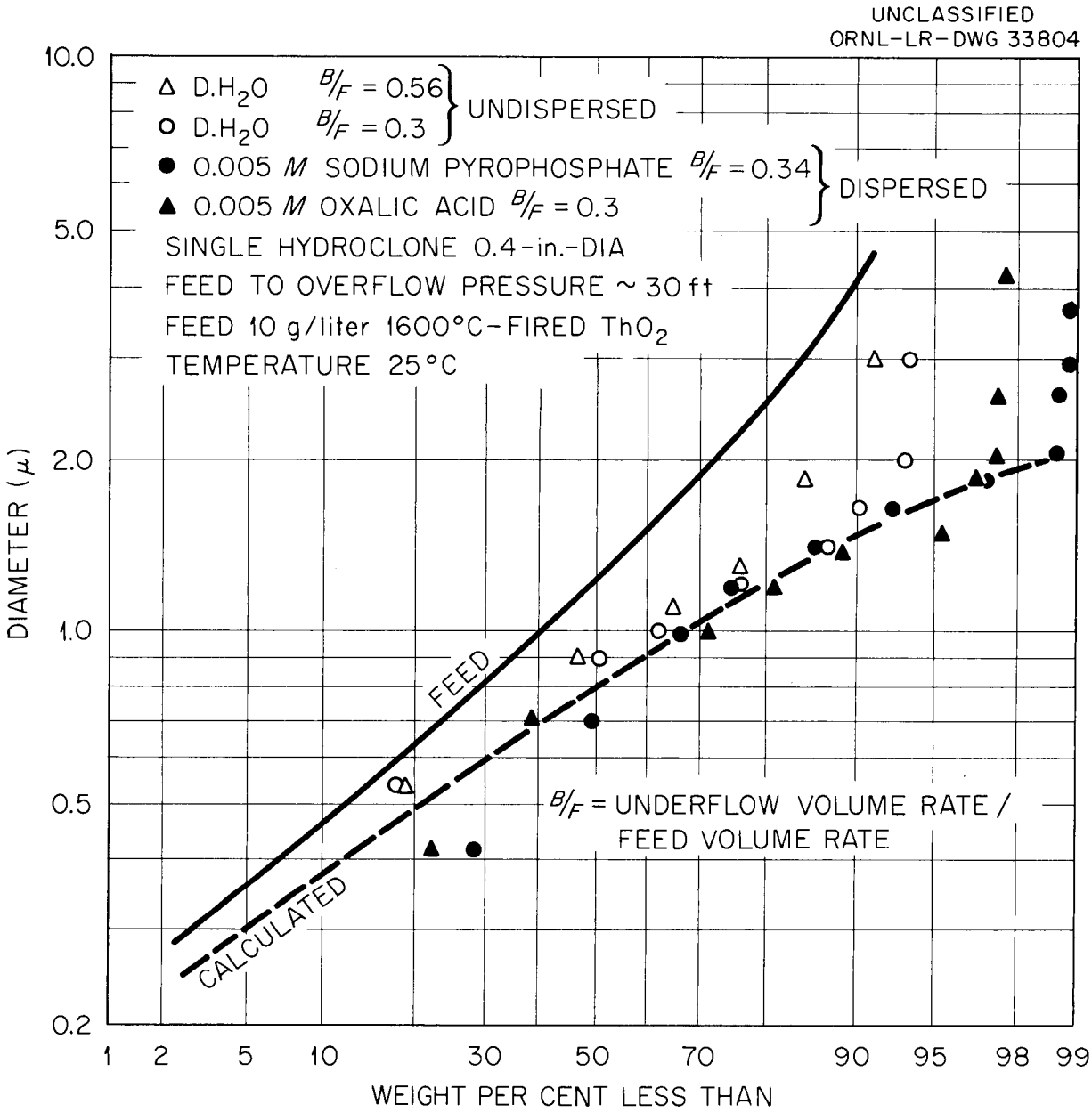

Fig. 7. Particle Size Distribution from Hydroclone Overflow for Dispersed and Undispersed Thorium Oxide Slurry.

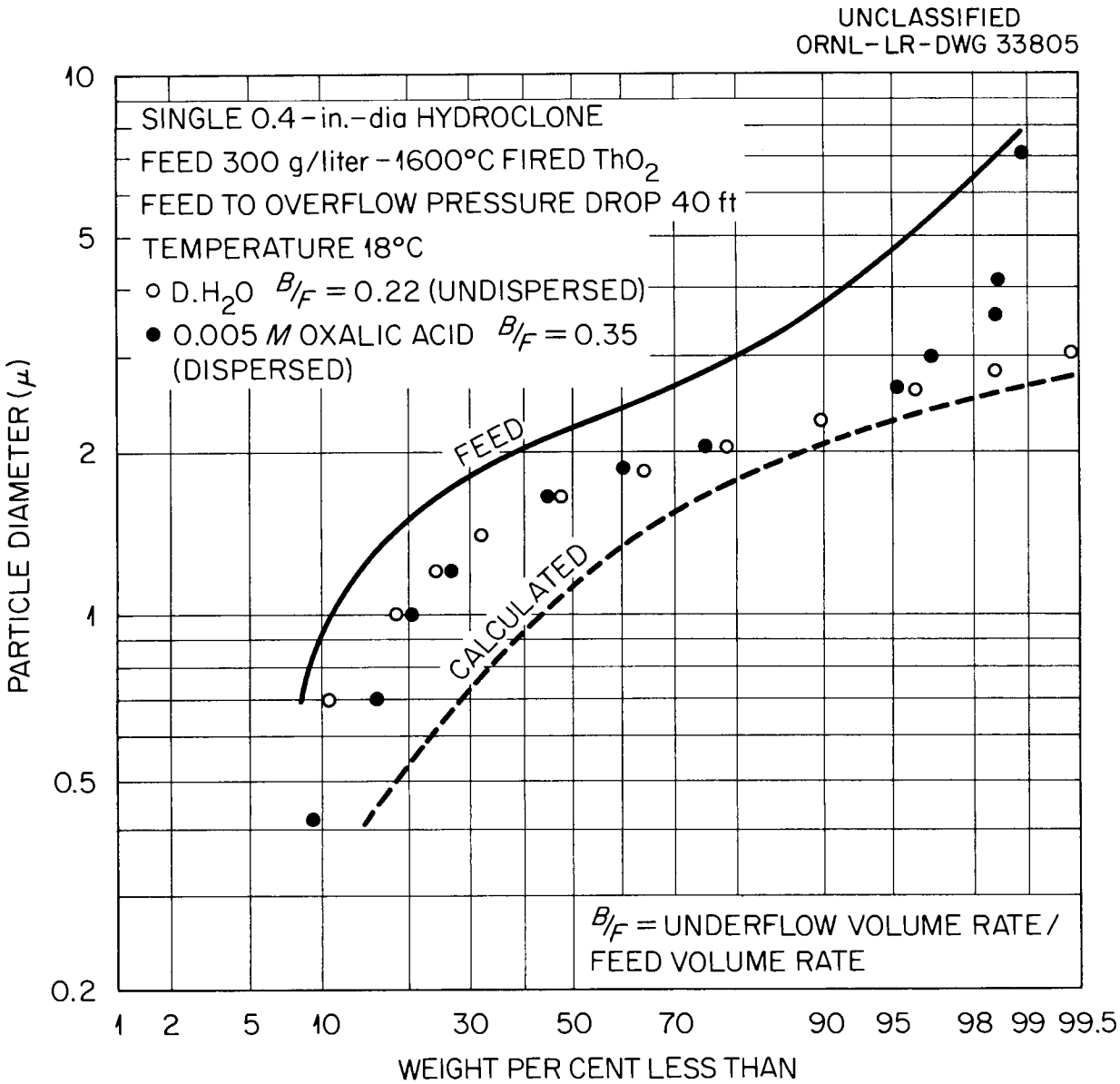

Fig. 8. Particle Size Distribution of Hydroclone Overflow for Dispersed and Undispersed Thorium Oxide Slurry.

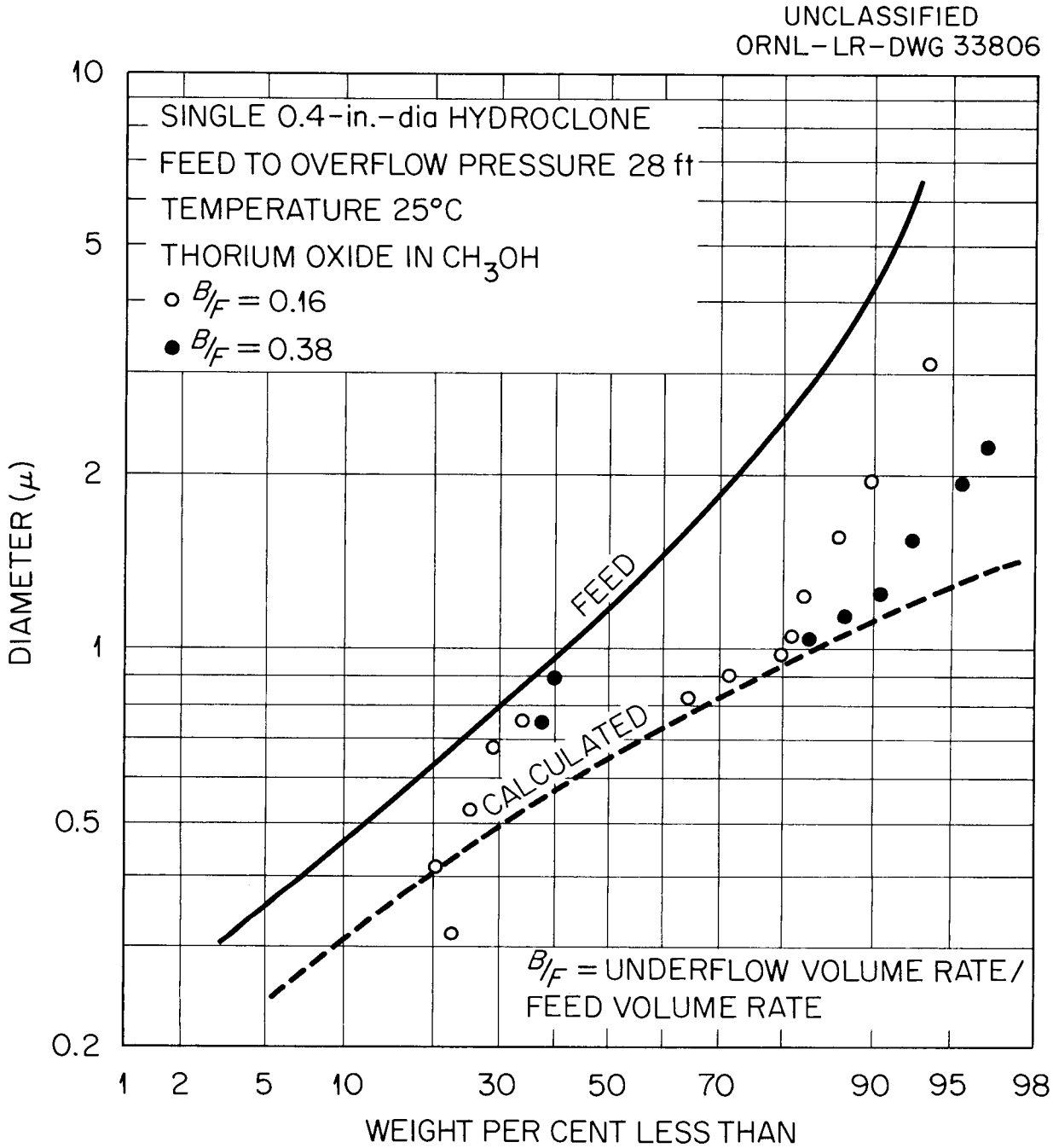

Fig. 9. Particle Size Distribution from Hydroclone Overflow for Thorium Oxide-Methyl Alcohol Slurry.

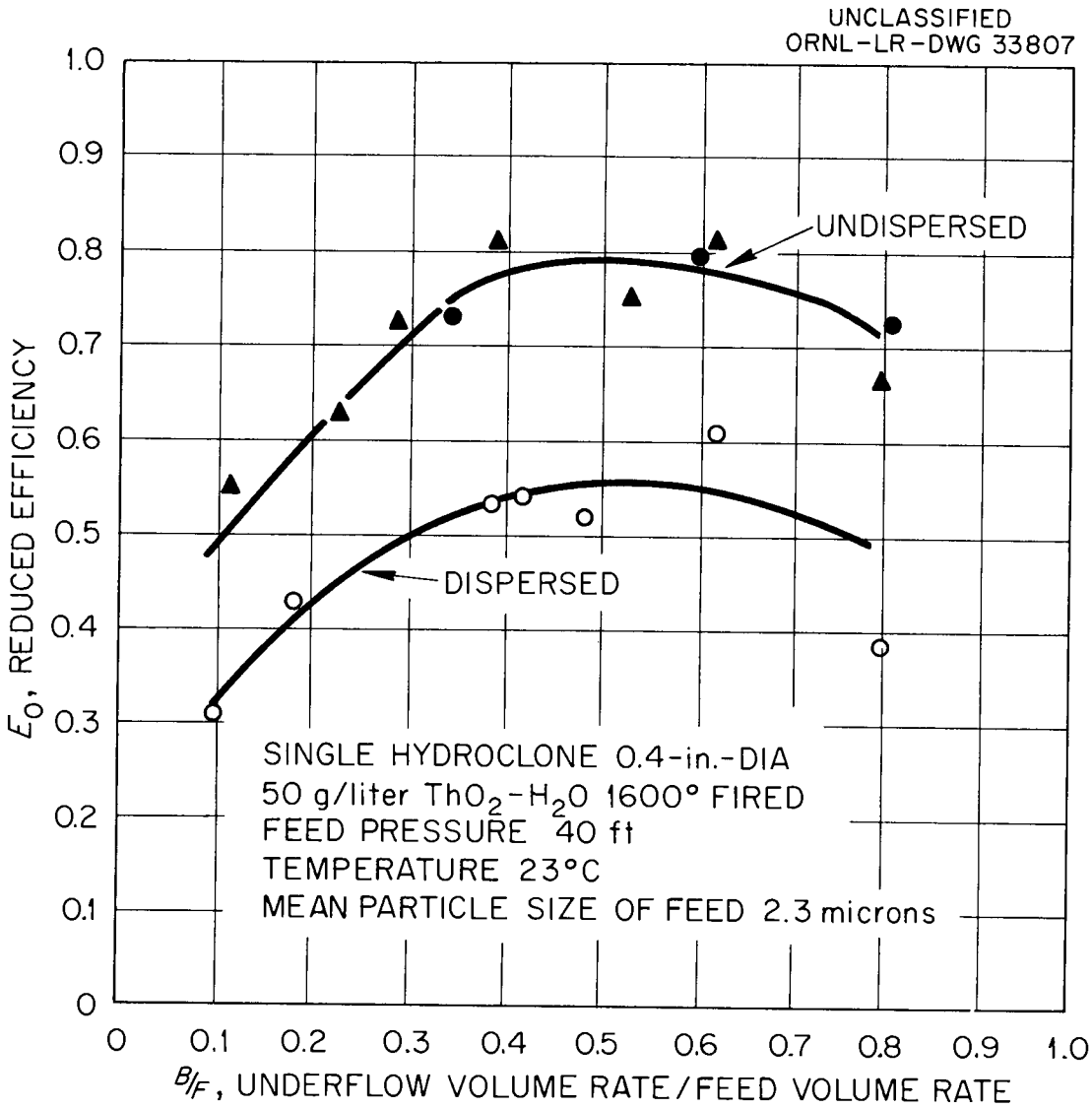

Fig. 10. The Effect of Underflow Flow Ratio and Dispersing Agents on Hydroclone Efficiency.

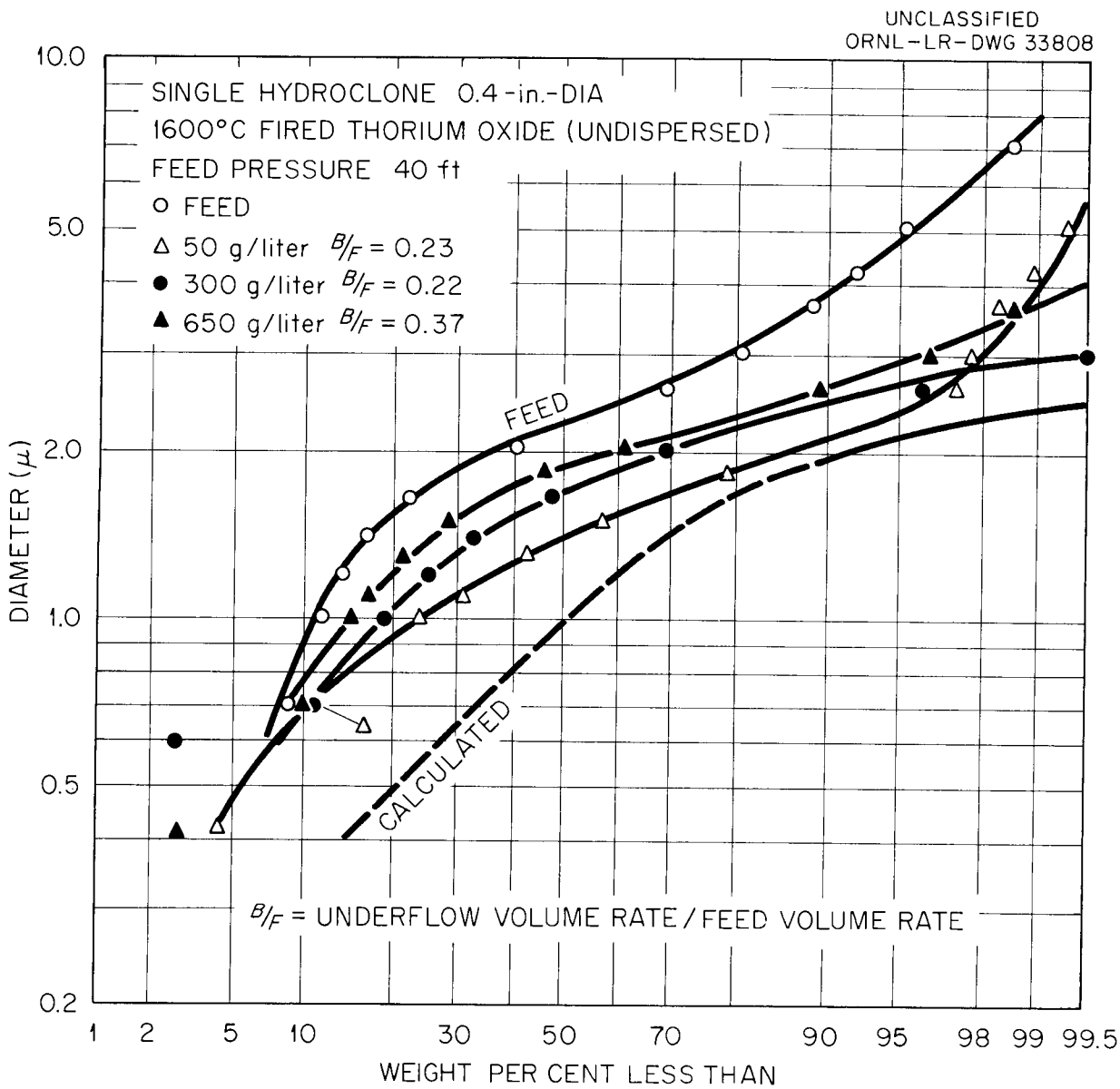

Fig. 11. Effect of Feed Concentration on Thorium Oxide Particle Size Distribution from Hydroclone Overflow.

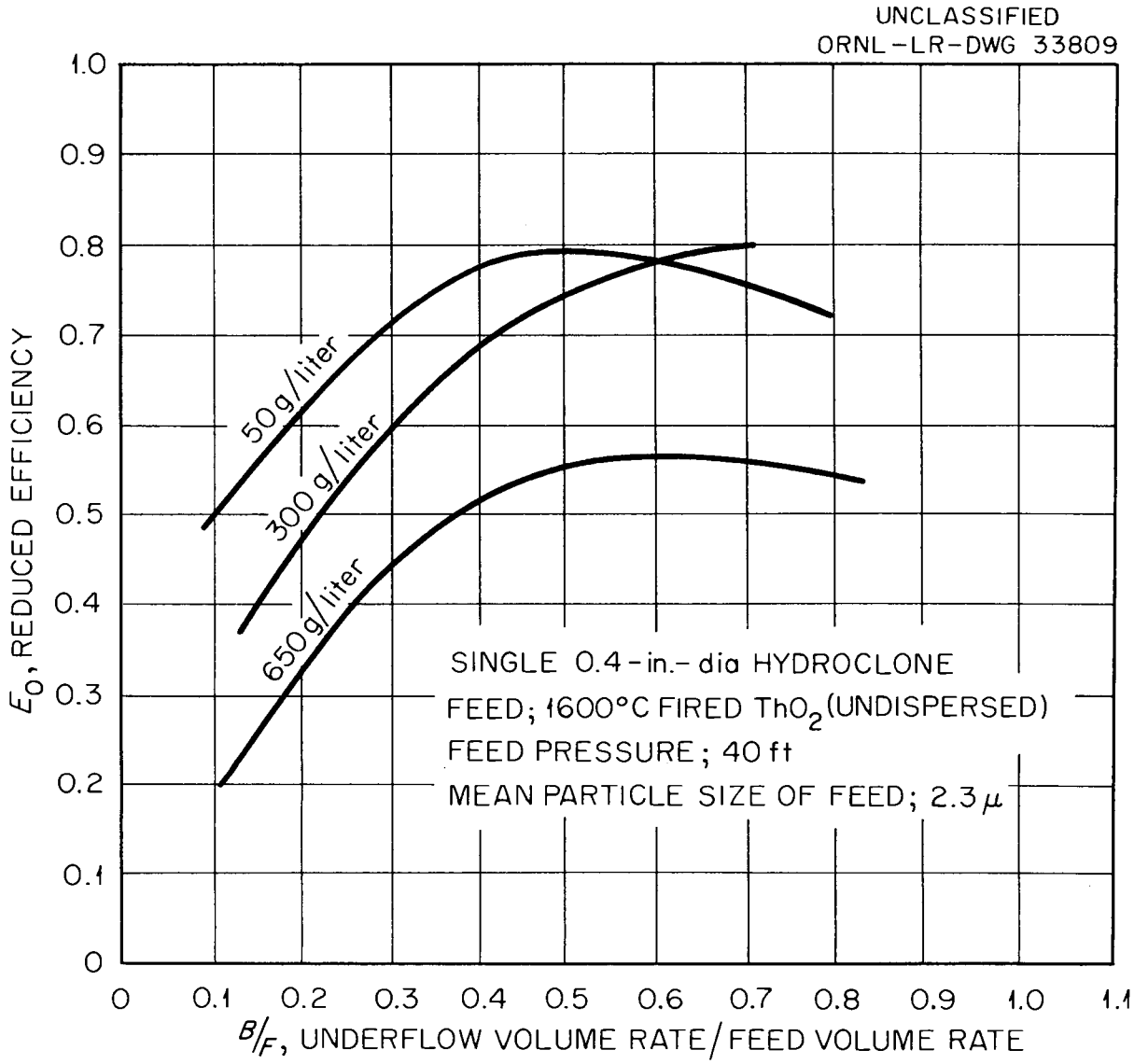

Fig. 12. The Effect of Underflow Ratio and Feed Concentration on Hydroclone Efficiency (Based on ThO_2 Feed Distribution Shown in Fig. 11.).

of large particles. This effect is shown in Fig. 10 and 12 where hydroclone reduced efficiency for various feed volume-to-underflow volume ratios is given. These values were determined for a constant feed-to-overflow pressure-drop and using feed having the same particle size distribution. It will be noted that the reduced efficiency also decreases somewhat at higher feed-to-underflow ratios. The particle size distribution of the overflow stream given in Fig. 13 shows a lower value for the mean particle diameter for intermediate flow ratios.

5.5 Effect of Repeated Classifications

A run simulating cascade operation was made by collecting the overflow stream and giving the material a second and third pass through the hydroclone. The underflow from each pass was discarded. This operation gives a final product which has a lower mean particle size than can be accomplished by one pass through a hydroclone. Also the probability is greatly reduced that large particles will enter the final overflow stream due to short circuiting which occurs when a portion of the feed stream flows along the wall of the hydroclone and goes directly to the overflow stream without going through the classification zone. The over-all effect of this method of series operation is to increase the sharpness of the particle distribution of the fine product at the expense of some additional loss of fine material to the underflow. The particle size distribution of thorium oxide from the overflow stream after three passes through the hydroclone is shown in Fig. 14. This distribution may be compared with the results for a single pass as shown in Fig. 7 since the feed material and operating pressures were similar for each run. A comparison of the calculated portion of the feed distribution which would be recovered in the hydroclone overflow stream from one and three hydroclones in series is given in Fig. 15.

6.0 MULTIPLE HYDROCLONE STUDIES

Scale-up of a hydroclone system is accomplished by placing single units in parallel rather than increasing the size of a single hydroclone since the classification range is dependent on hydroclone diameter. In the case of thorium oxide classification it is normally desirable to obtain a sharp classification of particles; therefore, it is desirable to place several units in series. Series operation serves to increase the removal of large particles from the overflow stream and also greatly reduces the probability of large particles entering the overflow stream as result of short circuiting or temporary malfunctioning of a single unit. For this reason the unit selected for testing the effectiveness of hydroclones for classifying 1-5-μ thorium particles on a production scale consisted of a three-stage unit, each stage of which contained thirty-two 0.4-in.-dia hydroclones in parallel to provide the desired flow capacity (~15 gpm at 30-ft liquid head).

The three stages were connected to give a countercurrent flow pattern similar to that of a distillation column (Figs. 16 and 17). The underflow stream from the center stage was fed to the feed stream of the top stage while the underflow stream went to the bottom stage feed stream. A portion of the overflow stream from the bottom stage underflow and the top stage overflow stream were recycled to the feed ports as shown in the flow diagram. A portion of the recycle streams was removed as product stream containing the fine-

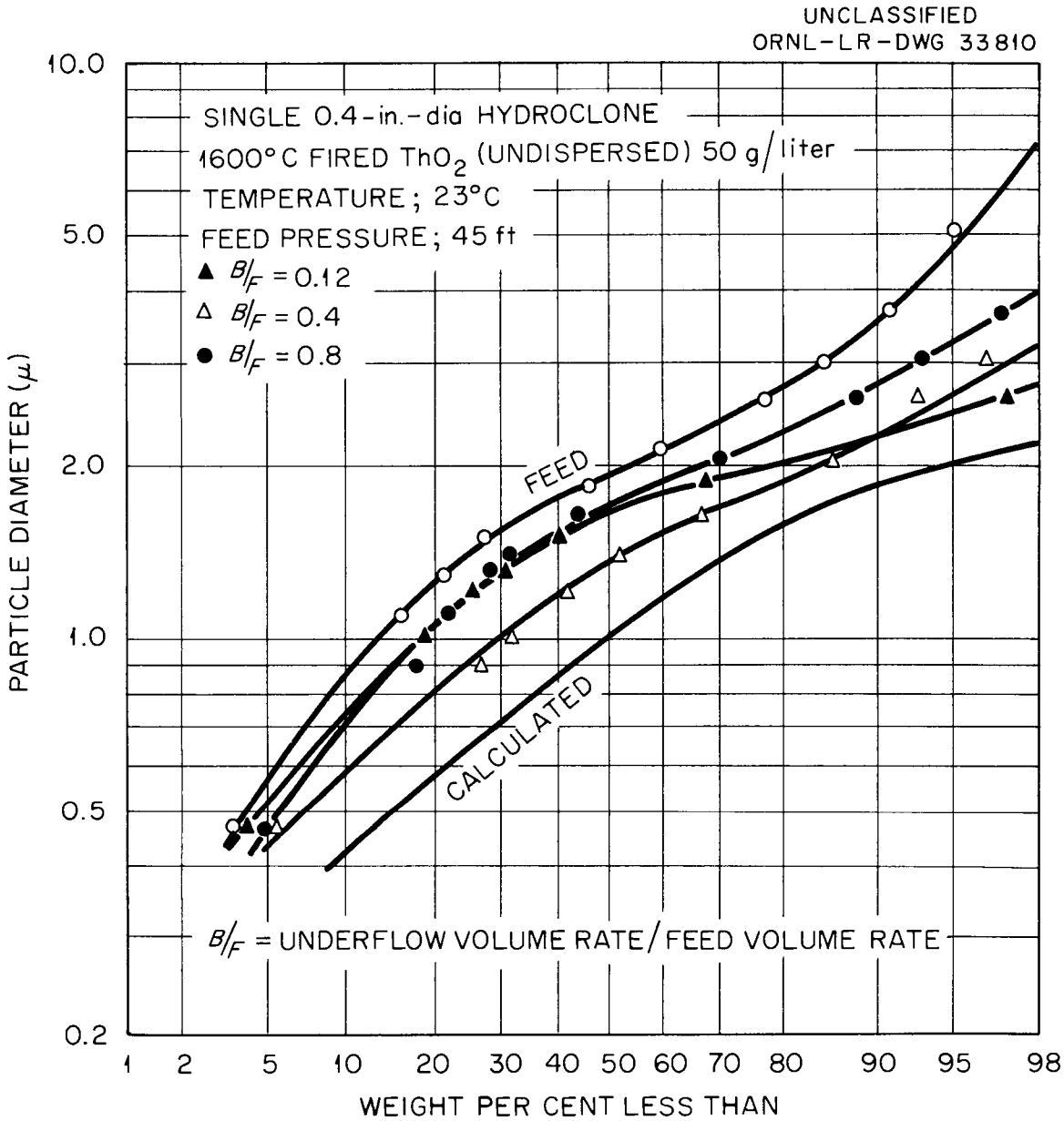

Fig. 13. Effect of Fraction of Feed Volume Discharged to Hydroclone Underflow on Particle Size Distribution of Overflow for Thorium Oxide-Water Slurry.

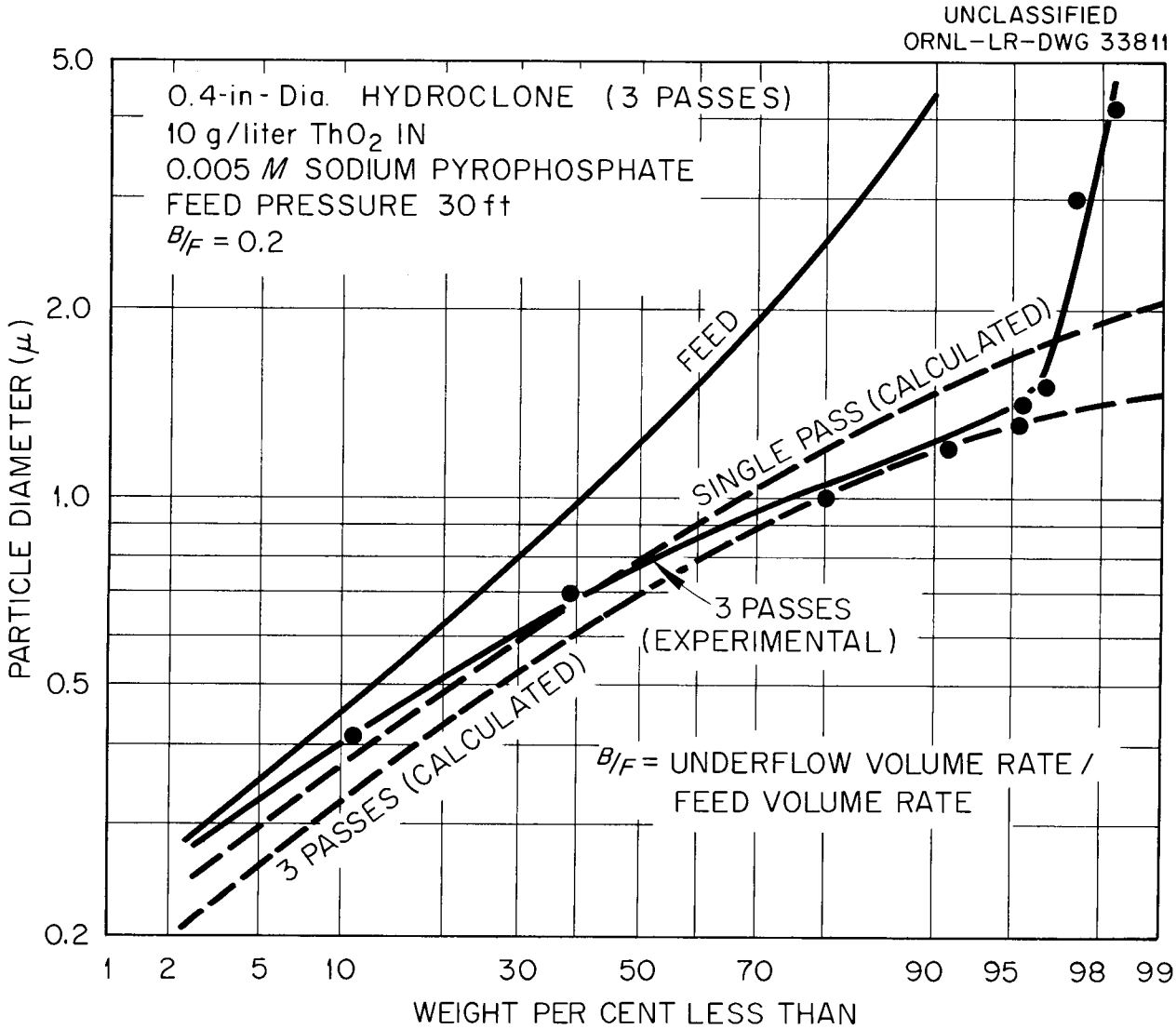

Fig. 14. Particle Size Distribution for Overflow from Three-Stage Hydroclone System for Thorium Oxide Slurry.

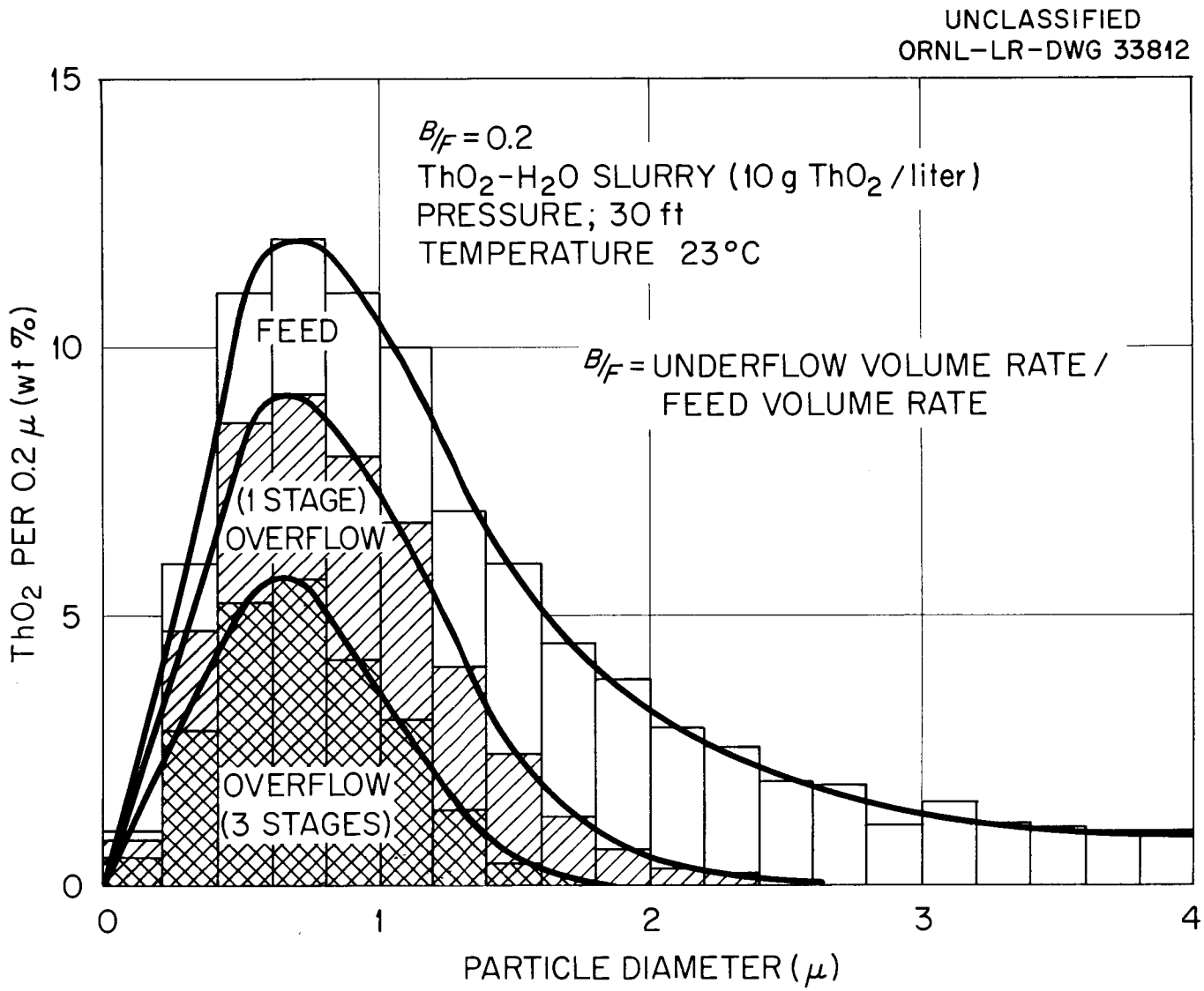

Fig. 15. Calculated Overflow Recovery for a One-and Three-Stage Hydroclone System using a Thorium Oxide-Water Slurry.

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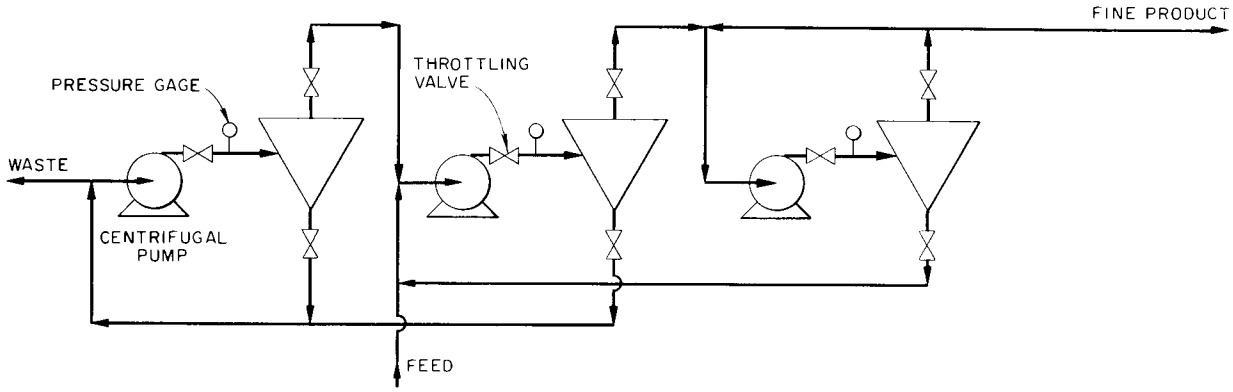

Fig. 16. Flow Diagram of Continuous 3-Stage Hydroclone Classification System.

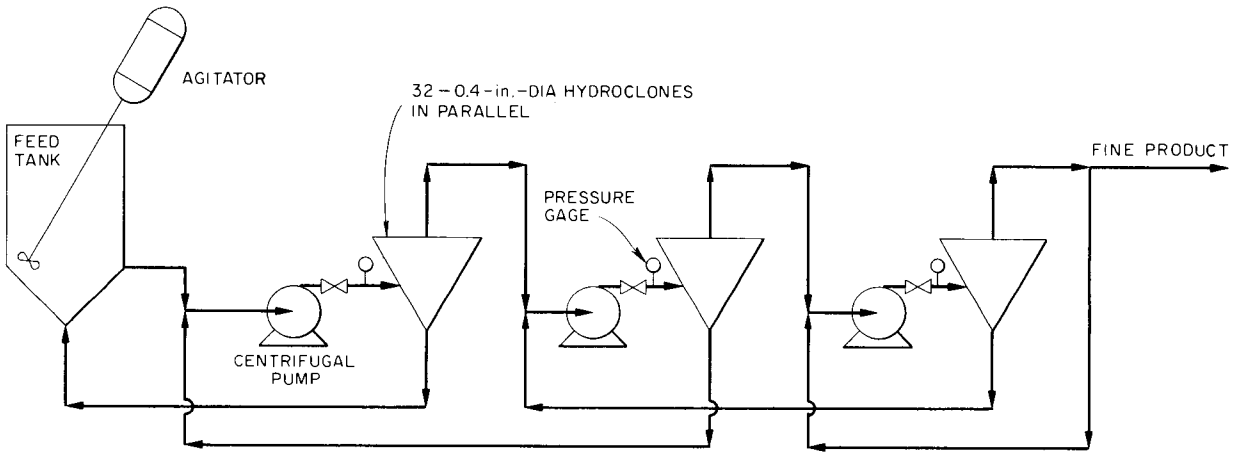

Fig. 17. Flow Diagram of a Batch 3-Stage Hydroclone Classification System.

particle fraction and waste containing coarse particles. Centrifugal pumps equipped with throttling valves on the discharge lines were placed on the feed streams to each stage to maintain the required pressure head. In this system the flow rate and pressure drop through each stage may be varied independently of the system feed rate by internal recycle. This feature is designed to permit control over the classification range of the system by variation of the pressure drop through each stage, which is controlled by the throttling valves located on the pump discharges. This method of operation also eliminates the necessity for close manual control over the flow distribution between underflow and overflow streams which is required with systems in which no recycle is used. A system somewhat similar to the one described above has been reported to give improved classification over a single stage unit.(9)

6.1 Equipment

The equipment consisted of commercially available TM-3 Dorr Clones which were modified to permit recycle of the underflow and overflow streams. Worthington centrifugal pumps were used between stages. These pumps were equipped with mechanical shaft seals which were not designed for long term slurry operation. Some difficulty was experienced with leakage of these seals; however, they were adequate for the experimental studies.

The hydroclones used in each stage were 0.4-in.-dia Bakelite cyclones which had essentially the same dimensions as those given in Table 1 with the exception that the length from the vortex finder to the underflow port was slightly longer (3.14 in.). Erosion of the Bakelite hydroclones was low and no replacement was required due to wear during the duration of the study.

Throttling valves and pressure gages were installed on the discharge of each pump to permit control over flow rates through each stage. The connections between stages were made with rubber hose. With the exception of the rubber hose and gaskets and the Bakelite hydroclones, the system was constructed of stainless steel.

Feed slurry for the system was prepared in a 55-gal drum having a cone shaped bottom. The tank was equipped with an agitator for maintaining solids in suspension. The 0.4-in.-dia hydroclones were designed for feeds containing mostly particles smaller than 325 mesh. A screen was placed on the discharge line from the feed tank to remove particles larger than about 1200 μ (14 mesh) which might plug the hydroclone openings. For runs with a continuous product and waste withdrawal, a pump was placed on the feed tank to maintain a positive feed pressure on the system and to aid in agitation of the feed tank by recirculation of a by-pass slurry stream from the bottom to the top of the feed tank. A Wilfley sand pump was used for this purpose. This pump gave satisfactory operation in the slurry system. A photograph showing the TM-3 Dorr Clone and the pumps is shown in Fig. 18.

6.2 Feed Preparation

In all runs the feed slurry was prepared by agitating 1600°C-fired thorium oxide in demineralized water until a uniform slurry was obtained. In some cases

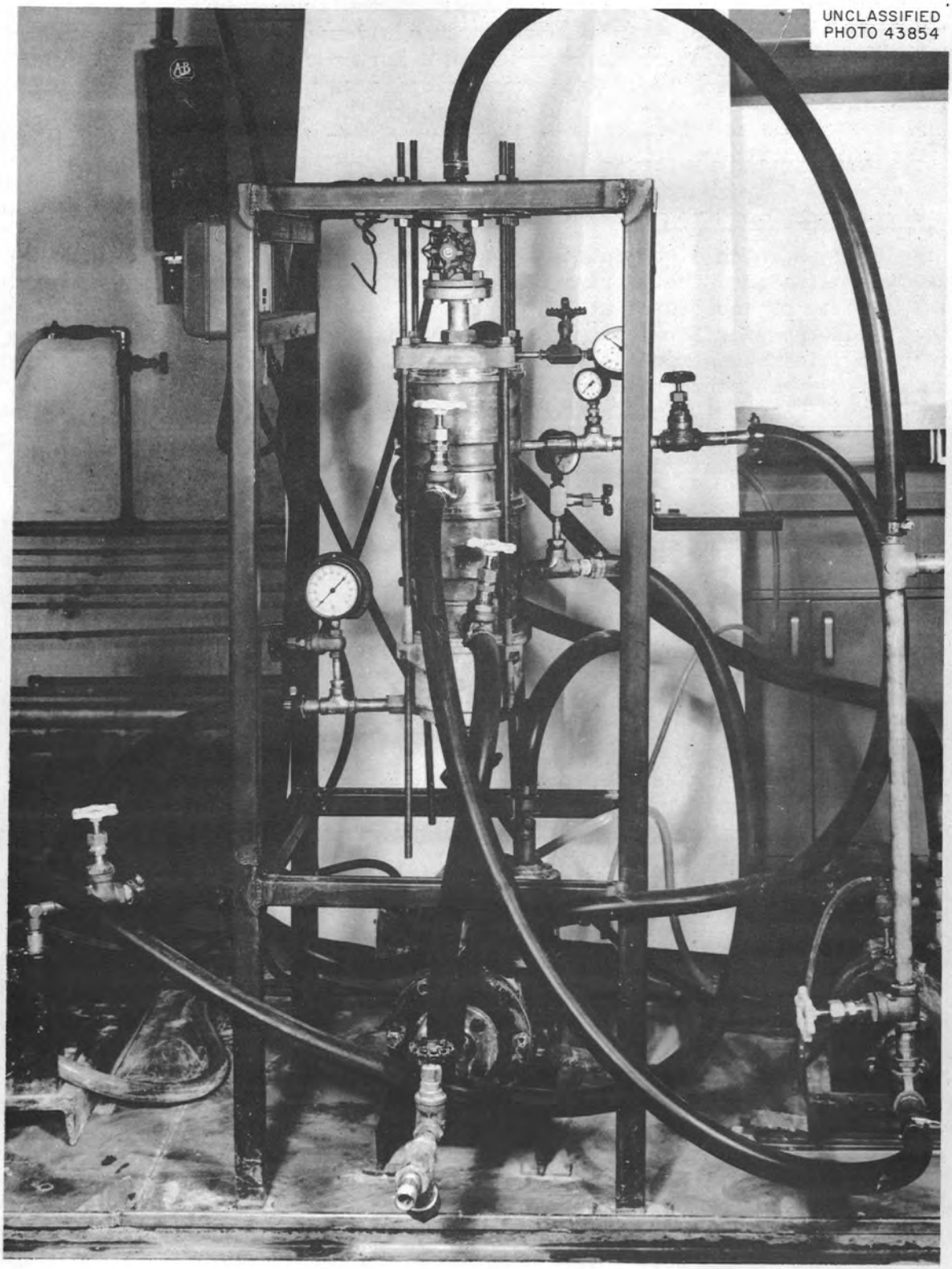

Fig. 18. TM-3 Dorr Clone Classification Systems.

the firing operation used in the production of the thorium oxide had caused sintering of the particles into lumps which could not be sufficiently dispersed by agitation. Powdering of these lumps in a mortar and pestle was required prior to slurry preparation. Additional dispersion of these lumps was obtained from pumping and circulating the slurry through the system during classification. Material which was not dispersed by this action behaved as larger particles and was separated by the system as waste.

6.3 Continuous Operation

In runs in which a continuous feed addition and product and waste withdrawal was maintained the thorium oxide slurry from the feed tank was introduced at the suction of the center stage pump. Product containing fine particles was removed from the overflow stream of the top stage while the waste containing the oversize particles was removed from the underflow stream of the bottom stage. In this system the center and top stages serve to remove oversize particles from the product stream while the bottom stage served to recover additional fine particles from the waste stream and return these particles to the center stage for further classification.

Continuous runs were made by preparing approximately 55-gal batches of slurry in the feed tank and feeding this slurry through the system and collecting the product and waste in separate containers. The thorium oxide was 1600°C-fired material having a mean particle size of 1.5 to 2 μ. Feed concentrations of 50 to 100 g/liter and product withdrawal rates of 4 to 12 liters/min were used. Using feed pressures of about 15 psi, the concentration of the overflow stream ranged from 10 to 50 g/liter while the waste stream ranged from 400 to 800 g/liter. The particle size distribution typical of continuous operation are shown in Fig. 19; additional results are included in the Appendix (Figs. 26-28).

The relatively high concentration of the slurry in the bottom stage coupled with a system volume holdup of about 20 liters resulted in a considerable holdup of thorium oxide in the classification system (~6 Kg). For runs of short duration such as these experimental runs, this holdup represents a fairly large fraction of the feed material and thus results in a reduction in product recovery since a considerable amount of fine particles remain in the system. This holdup would become less significant in processing larger quantities of thorium oxide.

6.4 Batch Operation

The batch system was designed for more efficient processing of smaller quantities of thorium oxide than could be efficiently handled by the continuous system. In this system the feed tank was placed in the underflow recycle stream of the bottom stage of the system as shown in Fig. 17. The batch system was operated in much the same way as a batch distillation column. The product containing the fine particles was removed from the overflow stream of the top stage while the larger particles concentrate in the feed tank. All three of the stages serve to remove oversize material from the product stream. Smaller particles are removed preferentially from the system and, as product is removed

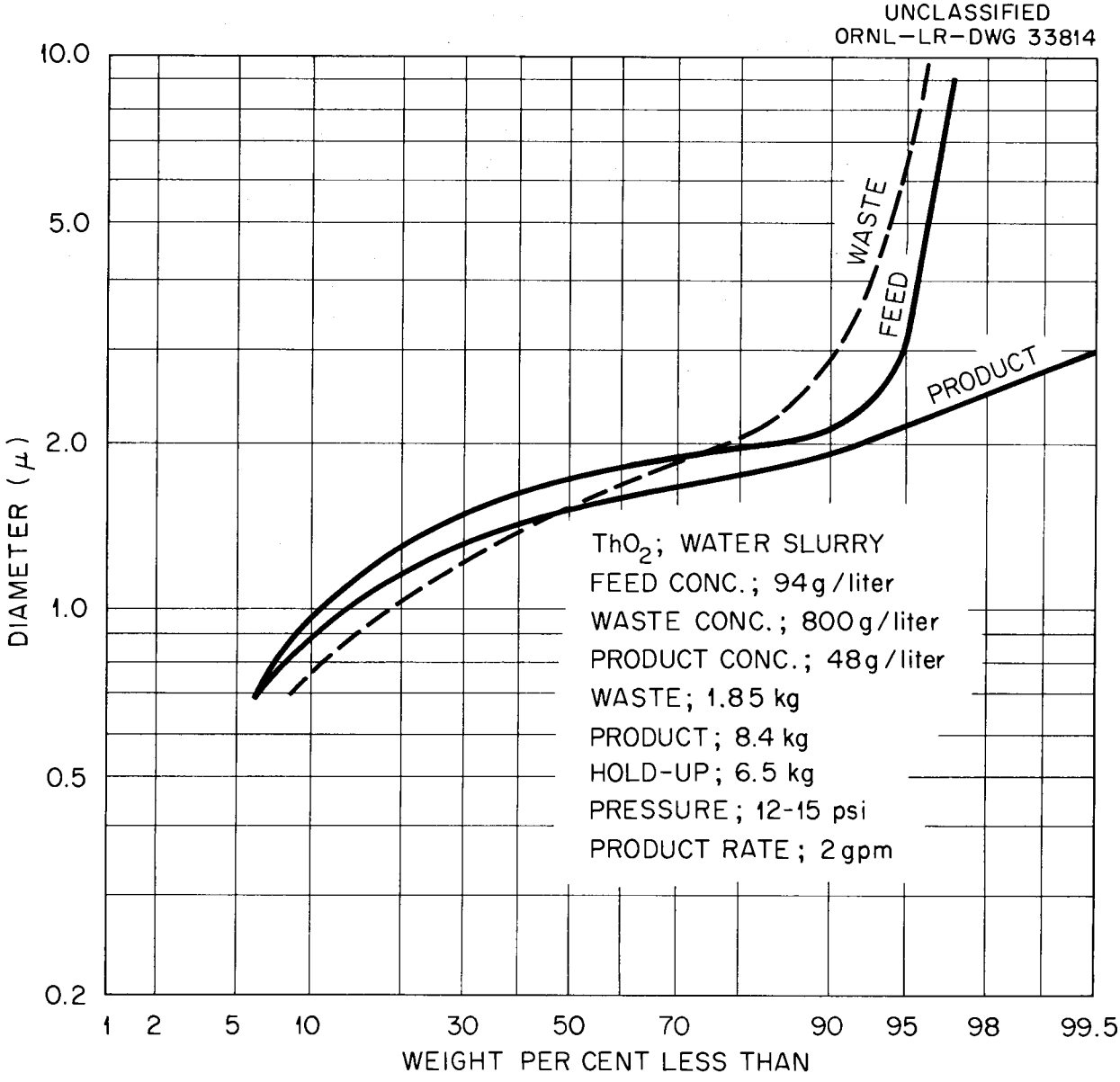

Fig. 19. Particle Size Distribution Curves from TM-3 Dorr Clone Classification System (Continuous Operation).

from the system, the feed tank becomes more depleted in smaller particles and the average particle of the product stream increases. The point at which product removal is terminated depends on the acceptable product size distribution.

Since the product stream is always considerably more dilute than the initial feed, there is a tendency to concentrate the slurry in the feed tank. To prevent this concentration from becoming excessive to the point at which classification efficiency of the unit is reduced, a water stream must normally be added to the feed tank. For runs in this study in which the product was collected as a slurry the make-up water was supplied from a demineralized water line at a rate sufficient to maintain a constant liquid volume in the feed tank. In a production unit in which a centrifuge is used to remove the thorium oxide from the product stream the water from the centrifuge can be returned to the feed tank. The concentration in the tank can be varied during operation by adjusting the rate of water addition. Results for a typical run of this type system using a 1600°C-fired thorium oxide with a mean particle size of about 3-μ is shown in Fig. 20. The variation of the particle size of the product with time is also given in this figure. Additional results for batch classification studies are included in the Appendix (Fig. 29 and 30).

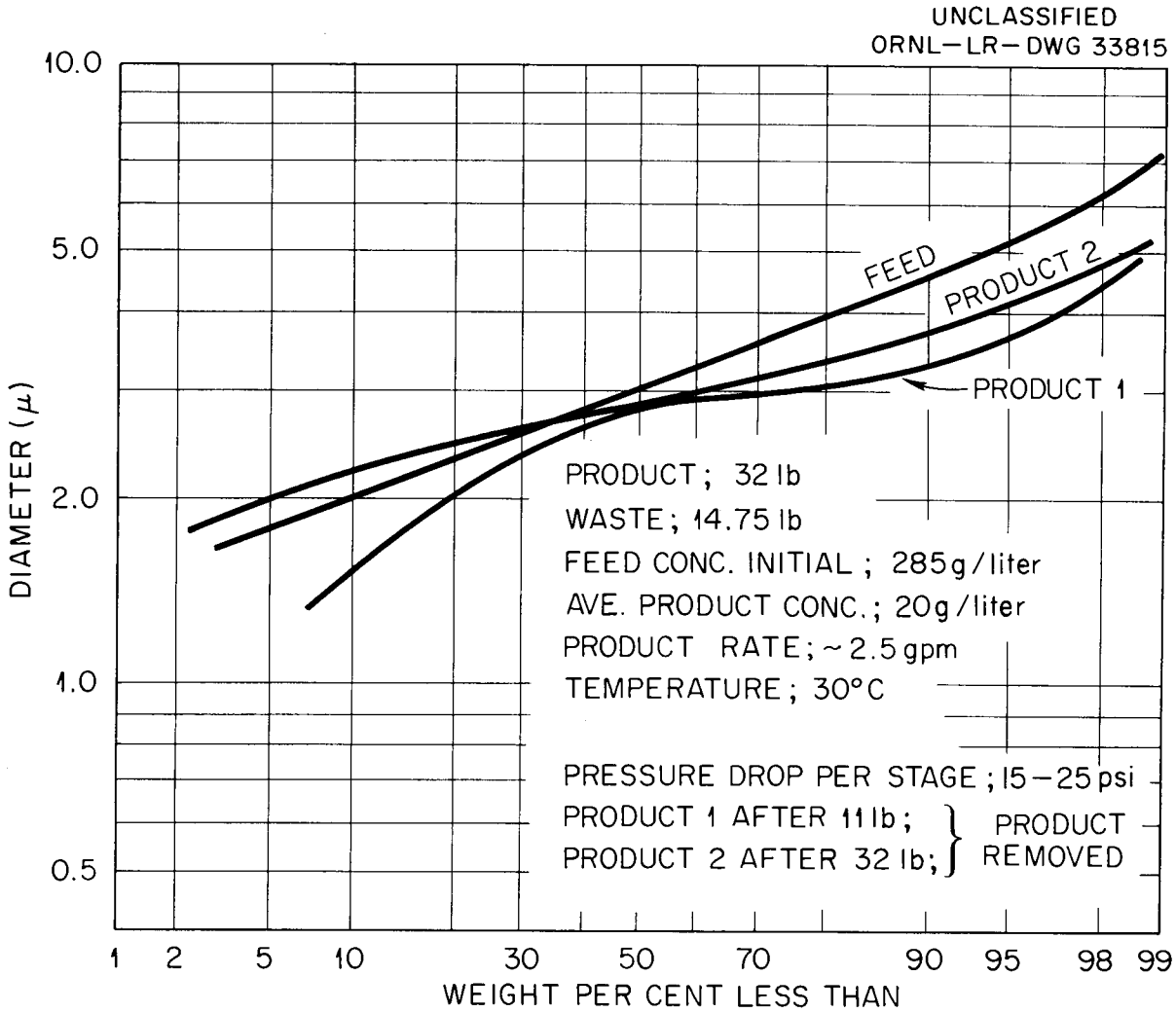

Fig. 20. Particle Size Distribution of Thorium Oxide Classified in a TM-3 Dorr Clone (Batch Operation).

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8.0 APPENDIX

8.1 Air Classification of ThO_2

Two batches of 1600°C-fired, micropulverized, thorium oxide having mean particle sizes of 6.4 and 2.6 μ were classified using a Sharples XD-10 Super Classifier to determine the suitability of air classification of thorium oxide powders.(10) The unit chosen for the test had the lowest noted range of classification of any air classifier readily available (from 5 to 26 μ) for thorium oxide. The classifier had a feed capacity of 400 lb of powder per hr.

The Sharples Super Classifier operates on the air vortex principle in which centrifugal force imparted to the particle to be classified is opposed by the viscous drag of air. The unit has the characteristic of giving a relatively sharp cut point (size separation point).

The cut point of the unit was set at the lowest value (about 5 μ). The only significant difficulties encountered in operating the unit with thorium oxide were difficulties in feeding and recovering the powder due to the tendency of the powder to stick to the feed funnel and walls of the classifier.

The classifier gave essentially complete removal of particles larger than 7 μ. No additional reduction in size could be achieved by a second pass of the fine product through the classifier. The yields for each run and the particle size distribution of the feed, fine and coarse fractions are given in Table 2 and Figs. 21 through 24.

Table 2 Air Classification of Thoria Powders*

Powder	Cut	Cut-Point (μ)	Yield (%)		Average Diameter (μ)		
			Fine	Coarse	Feed	Fine	Coarse
Run 1	First	5.0	70.1	29.7	2.7	2.2	3.9
	Second	5.0	73.5	26.5	2.2	2.3	2.4
Run 2	First	5.0	40.6	44.9	6.6	2.3	35.5
	Second	5.0	30.5	58.9	2.3	2.5	2.2

*W. E. Tewes and A. Gosen, Classification of Thoria Powder, KL-116 Oak Ridge Gaseous Diffusion Plant, May 6, 1958.

8.2 Gravity Sedimentation Classification

Gravity sedimentation, which takes advantage of the faster settling rates of larger particles in liquids, has been used successfully by the Chemical Technology Pilot Plant Section to classify thorium oxide particles in the

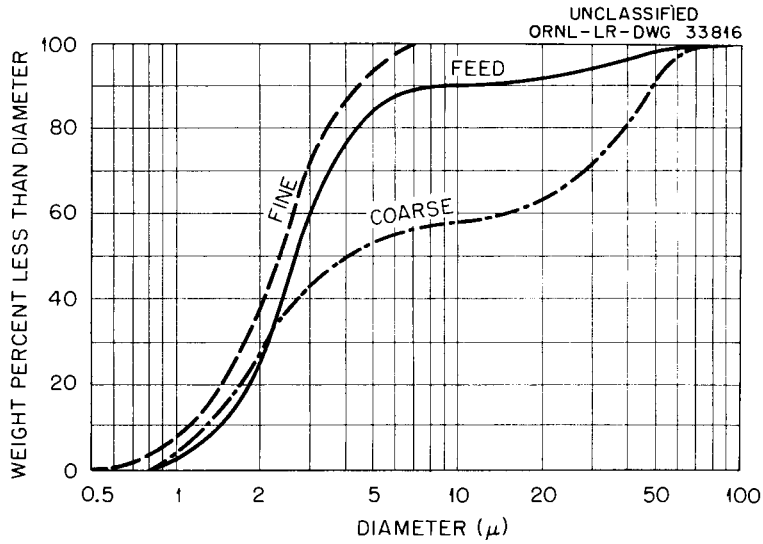

Fig. 21. Particle Size Distribution of Thorium Oxide Powder Classified in a Sharples Super Classifier, (Run 1). W.E. Tewes and A. Gosen, Classification of Thoria Powder, KL-116 Oak Ridge Gaseous Diffusion Plant, May 6, 1958.

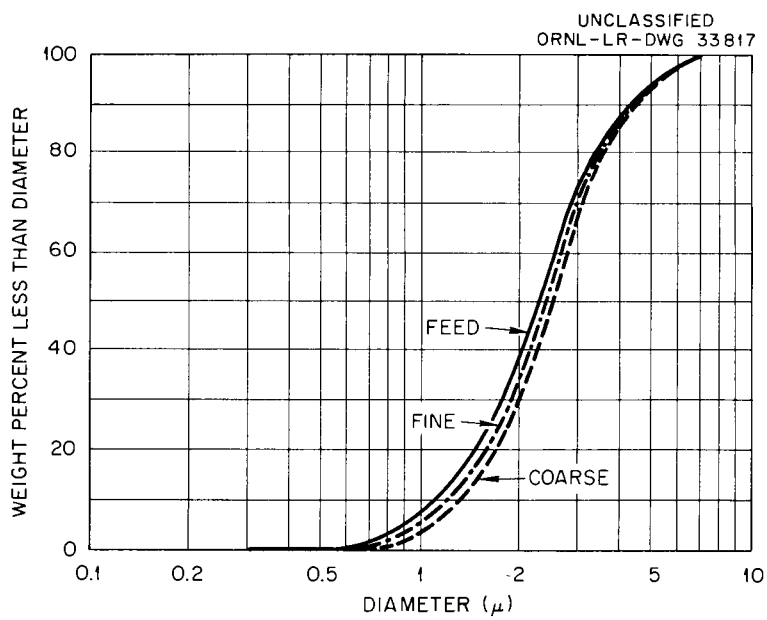

Fig. 22. Particle Size Distribution of Thorium Oxide Powder Classified in a Sharples Super Classifier. (Second Cut-Feed Fine Fraction from Run 1). W.E. Tewes and A. Gosen, Classification of Thoria Powder, KL-116 Oak Ridge Gaseous Diffusion Plant, May 6, 1958.

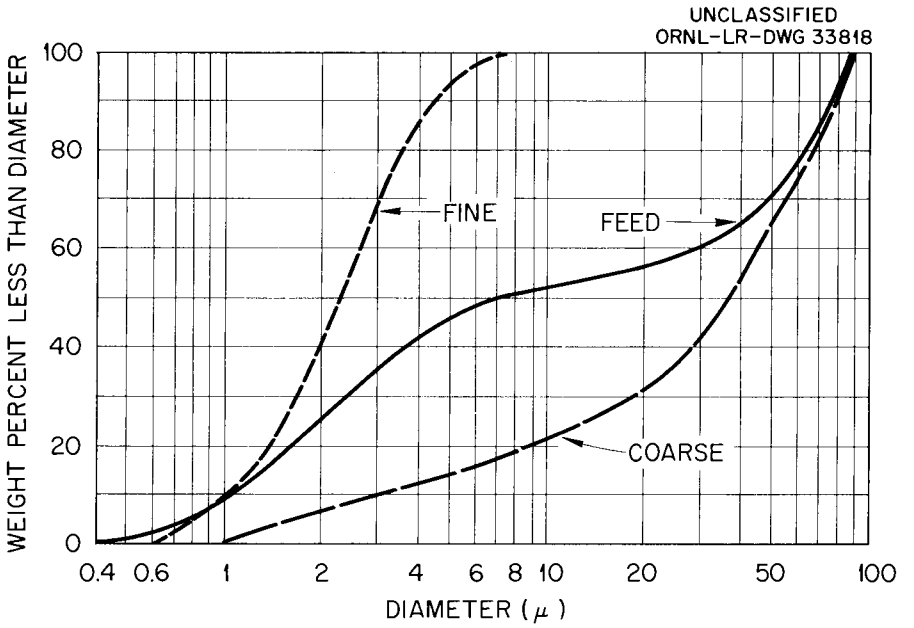

Fig. 23. Particle Size Distribution of Thorium Oxide Powder Classified in a Sharples Super Classifier (Run 2). W. E. Tewes and A. Gosen, Classification of Thoria Powder, KL-116 Oak Ridge Gaseous Diffusion Plant, May 6, 1958.

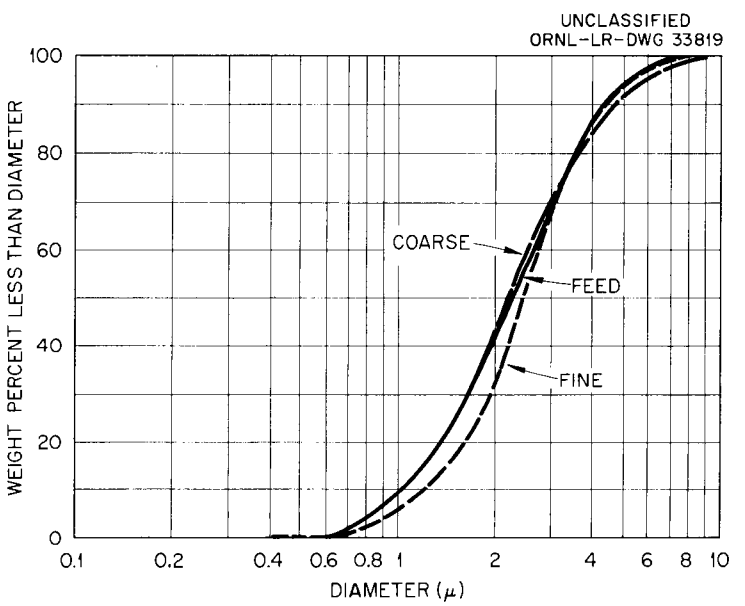

Fig. 24. Particle Size Distribution of Thorium Oxide Powder Classified in a Sharples Super Classifier. (Second Cut-Feed Fine Fraction from Run 2). W. E. Tewes and A. Gosen, Classification of Thoria Powder, KL-116 Oak Ridge Gaseous Diffusion Plant, May 6, 1958.

1-5-μ diameter range.(11) The procedure used entails mixing 20-kg batches of thorium oxide (which has been powdered to remove large lumps) with 27 gal of demineralized water in a 55-gal drum equipped with discharge taps down the side. The slurry which is at a concentration of about 200 g/liter is adjusted to a pH of 2.6 with oxalic acid to disperse the thorium oxide particles. Classification is accomplished by agitating the slurry and then allowing time for the larger particles to settle below the slurry discharge taps before the liquid containing fine particles is decanted, The decanted slurry is collected in a second drum where it is again classified as described above. Additional fine material is recovered from the thorium oxide remaining in the first drum by resuspending it in demineralized water and repeating the classification step. By a total of 4 such steps 70 to 75% of the feed material can normally be recovered as product. This value is increased to 80 to 85% recovery by classifying the large particles fraction collected from 4 or 5 batches to feed. The oxalic acid used as a dispersing agent must be removed from the product thorium oxide; this is accomplished by firing the thorium oxide to 650°C. Curves which show the particle size distribution of the feed and product for a typical run is shown in Fig. 25.

8.3 Sample Calculation

The fraction of feed solids reporting to the overflow stream and the particle size distribution of the overflow from a hydroclone may be calculated as shown by the following illustration. For operation of a 0.4-in.-dia hydroclone with a feed to overflow pressured drop of 30 ft and using a dilute thorium oxide-water feed slurry, the d_{50} is calculated from equation (1). The particle size distribution of the feed is given in Fig. 7. The temperature of the system is 22°C and the B/F ratio is 0.2.

From equation (1)

$$d_{50} = \frac{5 \times 10^3 (0.4)^{.5} \times (.000672 \times .96)^{.5}}{(30)^{.25} \left[(9.7 - 1)\ 62.4\right]^{.5}} = 1.46 \text{ microns} .$$

The hydroclone performance is calculated as shown in Table 3 from the feed particle size distribution in Fig. 7. The per cent of the feed in 0.2-μ size groups is determined (column 2). Using average values of d/d_{50} the reduced efficiency is determined for each size group from Fig. 4. The summation of reduced efficiency times the per cent of material present for each size group gives the total reduced efficiency for the feed distribution (column 5) the summation of $(1-E_o)$ feed composition gives the quantity of material on each size group which reports to the overflow stream (column 6). The quantities in column 6 are corrected to total 100% and the cumulative size distribution of the overflow stream is given in column 7.

8.4 Additional Experimental Data

In addition to the typical results picked to support the conclusions presented, many other results were obtained to verify the reproducibility of the data, to check mechanical operation of equipment, and for other purposes.

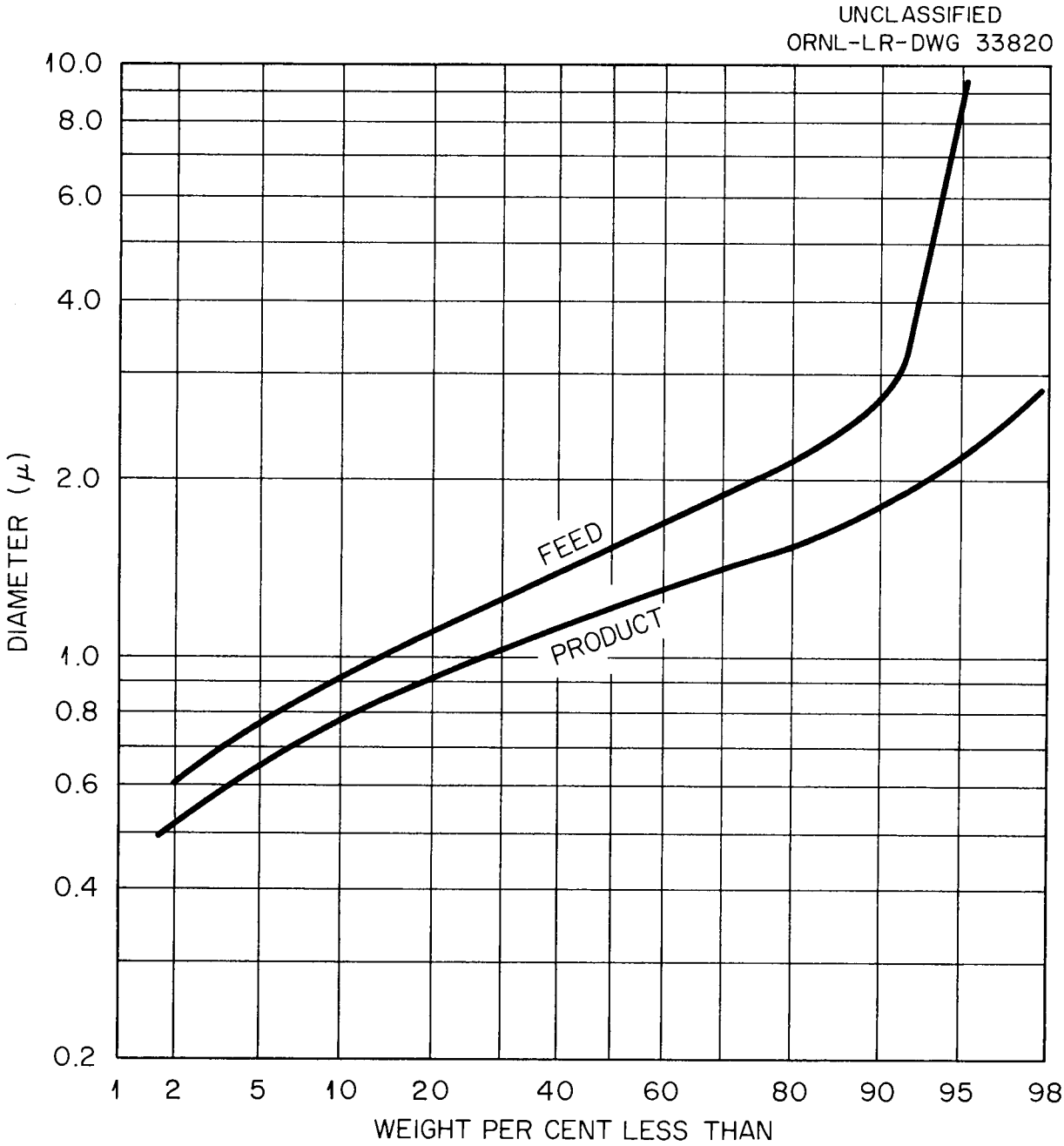

Fig. 25. Particle Size Distribution Curves for Thorium Oxide Classified by Gravity Sedimentation. Personal communications W. T. McCarley and C. V. Ellison, ORNL chemical technology pilot plant section and R. L. Pearson, ORNL chemical technology chemical division section A, July 19, 1958.

Table 3 Calculated Hydroclone Performance

$B/F^{*} = 0.2$
Temperature 22°C
$\Delta P = 30$ ft
$E_o = .44$
$E = E_o + B/F(1-E_o)$
$E = .44 + .2(.56)$
$E = .44 + .112 = .552$ fraction to underflow

Size Range	Feed Composition Per Cent/.2 micron	d/d_{50} ave.	E_o	(E_o) Feed Comp. / 0.2 micron	$(1-E_o)$ Feed Comp. / 0.2 micron	Overflow Composition Per Cent/.2 micron	Cumulative Particle Distribution of Overflow
0 - 0.2	1	0.07	0.0	0.0	1.0	1.78	1.78
0.2 - 0.4	6	0.21	0.01	0.06	5.04	8.94	10.82
0.4 - 0.6	11	0.34	0.025	0.28	10.72	19.02	29.84
0.6 - 0.8	12	0.48	0.055	0.66	11.34	20.15	49.99
0.8 - 1.0	11	0.62	0.10	1.1	9.9	17.55	67.54
1.0 - 1.2	10	0.75	0.18	1.8	8.2	14.54	82.08
1.2 - 1.4	7	0.89	0.33	2.31	4.69	8.32	90.40
1.4 - 1.6	6	1.03	0.55	3.3	2.7	4.89	95.19
1.6 - 1.8	4.5	1.16	0.695	3.12	1.38	2.45	97.64
1.8 - 2.0	3.8	1.3	0.80	3.02	0.78	1.38	99.02
2.0 - 2.2	2.9	1.44	0.88	2.55	0.25	0.44	99.46
2.2 - 2.4	2.6	1.57	0.92	2.39	0.21	0.37	99.83
2.4 - 2.6	1.9	1.71	0.96	1.82	0.08	0.14	99.97
2.6 - 2.8	1.8	1.85	0.98	1.77	0.03	0.05	100.00
2.8 - 3.0	1.1	1.99	0.99	1.09	0.01	0.02	--
3.0 - 3.2	1.6	2.12	~ 1.0	1.6	0.0	0.0	--
above 3.2	16.8	all above 3.2 removed to underflow		16.8	0.0	0.0	--
				43.67	56.33		

* β = underflow liquid rate.

E = total fraction of solids discharged to hydroclone underflow.

E_o = reduced efficiency, fraction of solids discharged to underflow.

F = feed liquid rate.

Additional results for continuous operation of the TM-3 cascade (Figs. 26, 27, 28) may be compared with Fig. 19. Batch operation of a two stage TM-3 cascade (Figs. 29, 30) may be compared with three stage batch operation (Fig. 20).

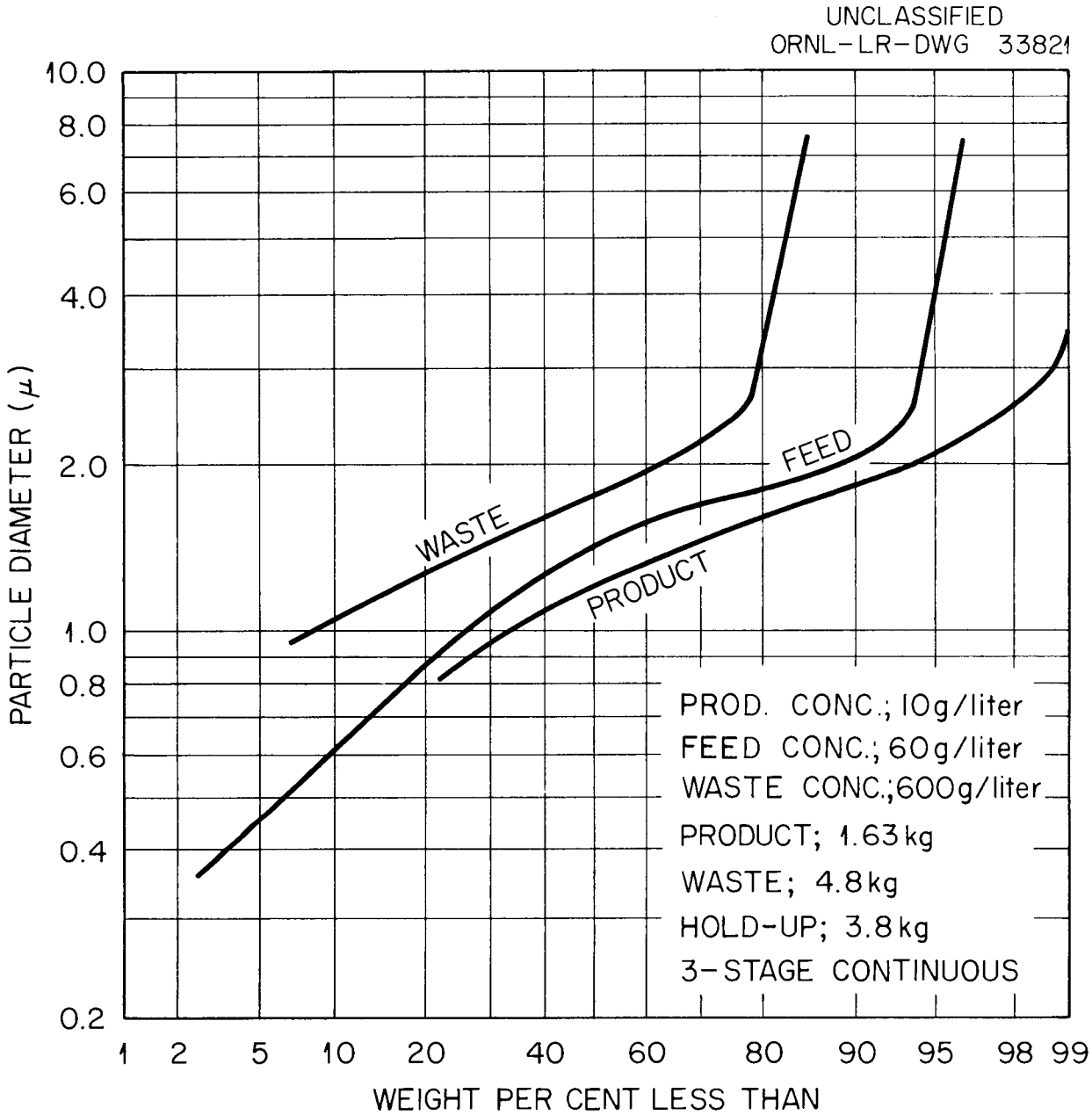

Fig. 26. Particle Size Distribution Curves for Thorium Oxide Classified in a TM-3 Dorr Clone System (Continuous Operation).

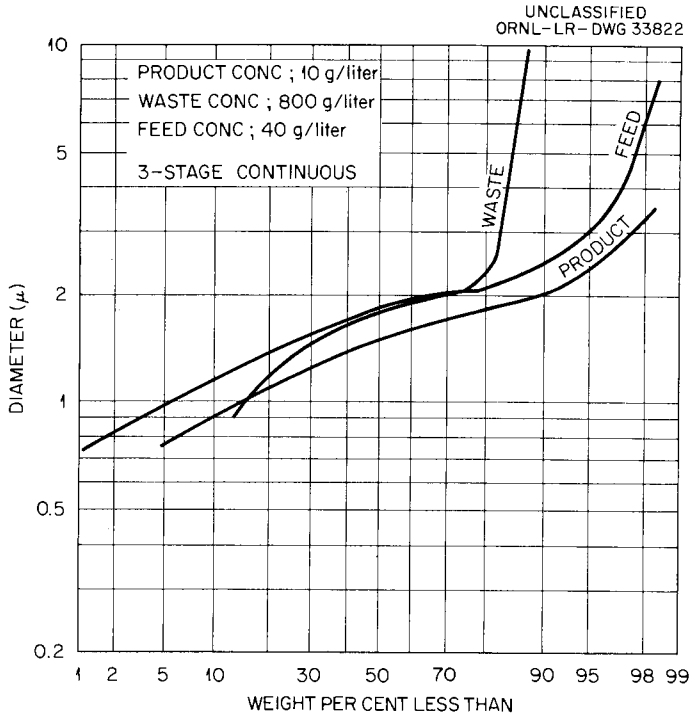

Fig. 27. Particle Size Distribution of Thorium Oxide Classified in a TM-3 Dorr Clone (Continuous Operation).

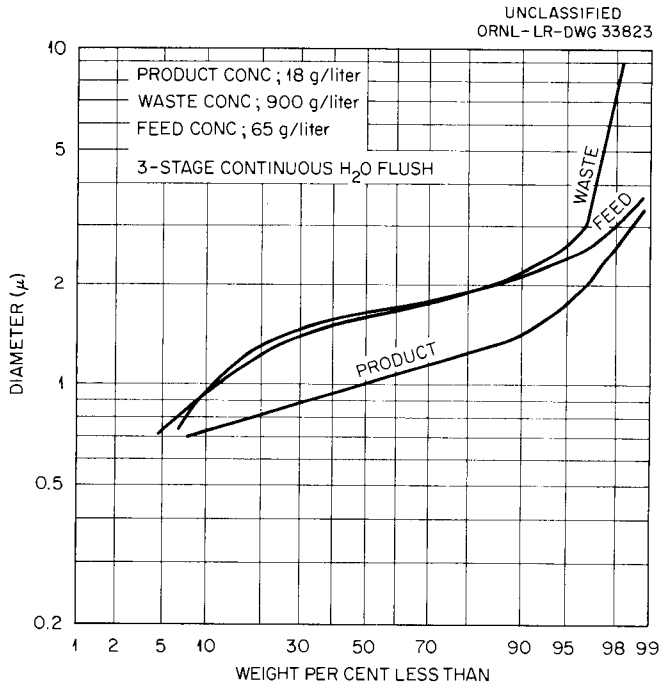

Fig. 28. Particle Size Distribution of Thorium Oxide Classified in a TM-3 Dorr Clone (Continuous Operation with Water Injection at Bottom Stage).

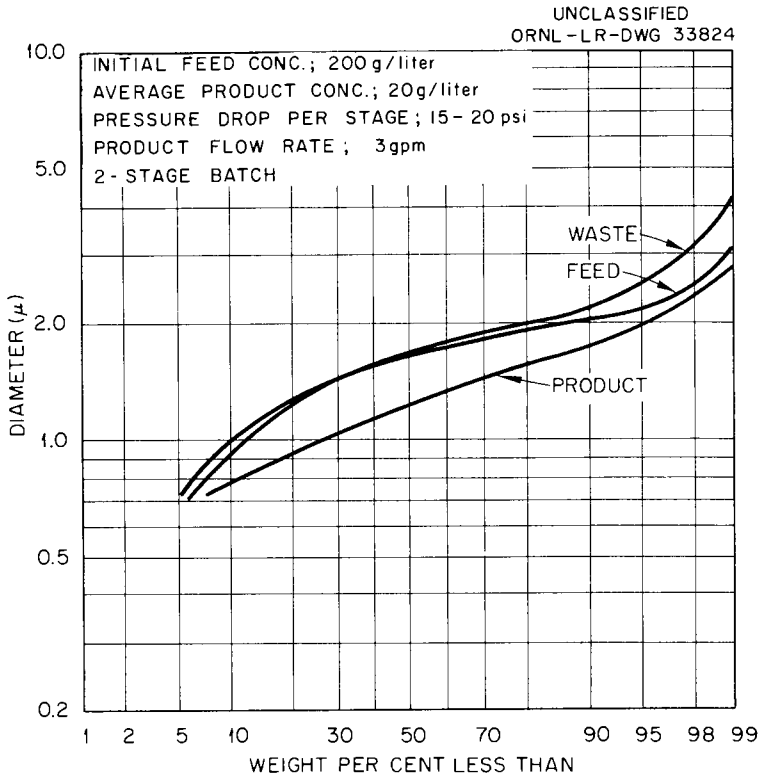

Fig. 29. Particle Size Distribution for Thorium Oxide Classified in a 2-Stage Dorr Clone System. (Batch Operation)

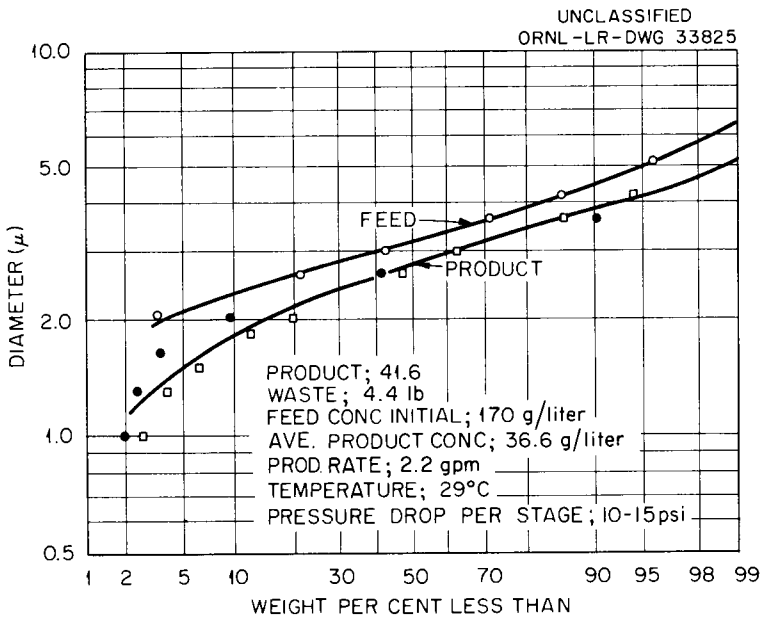

Fig. 30. Particle Size Distribution of Thorium Oxide Classified in a 2-Stage Dorr Clone (Batch Operation).

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