FINAL REPORT

NASA CR70583

PROJECT A-855

STUDY FOR IMPROVEMENT OF GROUND TEST, INSTRUMENTATION SYSTEMS, AND METHODS - NEW METHODS FOR STAGE PROPELLANT TANK PROOF TESTING

JOHN H. BURSON, III

Contract NAS8-20110
Control No. DCN-1-5-60-00050-01 (1F)

2 April to 1 December 1965

N66-18324 (ACCESSION NUMBER)	1 (THRU)
56 (PAGES)	(CODE)
CR 70583 (NASA CR OR TMX OR AD NUMBER)	14 (CATEGORY)

FACILITY FORM 602

Prepared for
National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Huntsville, Alabama

1965

Engineering Experiment Station
GEORGIA INSTITUTE OF TECHNOLOGY
Atlanta, Georgia

GPO PRICE $ ______

CFSTI PRICE(S) $ ______

Hard copy (HC) 3.00

Microfiche (MF) .50

ff 653 July 65

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ABSTRACT

N66-18324

The objective of this research program was to develop high-density slurries suitable for use as pressure-transmitting media in hydrostatic testing of stage propellant tanks. This included a determination of the range of densities that could be obtained; determination of mixture stabilities; determination of compatability with stage and stage component materials; and the definition of pumping, storage, and other handling techniques.

Water-based slurries were formulated from a large number of materials and it was conclusively shown that specific gravities from two to six could be achieved with readily available materials and conventional chemical processing equipment. Lead oxide (litharge) was shown to be the most suitable material for producing stable slurries over a wide range of specific gravities. Laboratory data indicate that these slurries also act as pressure transmitting media. Additional studies are desirable in order to evaluate more fully and to improve the corrosion characteristics of the slurries when in contact with aluminum alloys.

A preliminary economic analysis favors the construction of an on-site plant for producing slurries in quantities of up to one million gallons. Additional prototype and pilot-plant studies are recommended, however, before construction of a plant is undertaken for large-scale production.

Author

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I. INTRODUCTION AND SPECIFIC OBJECTIVES

The liquid in the stage propellant tanks of a rocket is subjected to several times the normal gravitational attraction under operational conditions. The propellant tanks must be designed to withstand these additional stresses yet must also meet stringent weight limitations. Thus, proof-testing of propellant tanks should be performed under conditions closely approaching those of actual operation for an optimum matching of weight and performance.

Figure 1 illustrates the typical relationships between liquid depth and hydrostatic pressure for incompressible fluids. Curve "a" shows the hydrostatic pressure versus height for a fluid of unit density subject to normal gravitational attraction and exposed to the atmosphere. If this fluid is subjected to a ullage-pressure, the curve is simply shifted to higher pressures with no change of slope as is illustrated by curve "b". Curve "b" thus represents the pressure profile of a pressurized propellant tank before engine ignition and lift-off. When the gravitational attraction is increased from one to four, the slope of curve "b" is changed by a factor of 4 and the resulting pressure profile is depicted by curve "c". Therefore, curve "c" represents the true pressure conditions within a full propellant tank filled with unit density liquid under four times normal gravitational acceleration and normal ullage-pressure. To simulate the most extreme pressure condition which exists at the tank bottom with a unit density fluid that is subject only to normal gravitational attraction requires a very large ullage-pressure with the result that all of the tank except the bottom is stressed well beyond maximum operating limits. This condition is illustrated by curve "d". A tank that is proof-tested in this manner must have upper wall-thickness considerably greater than are required under actual operating conditions. However, if pressure testing

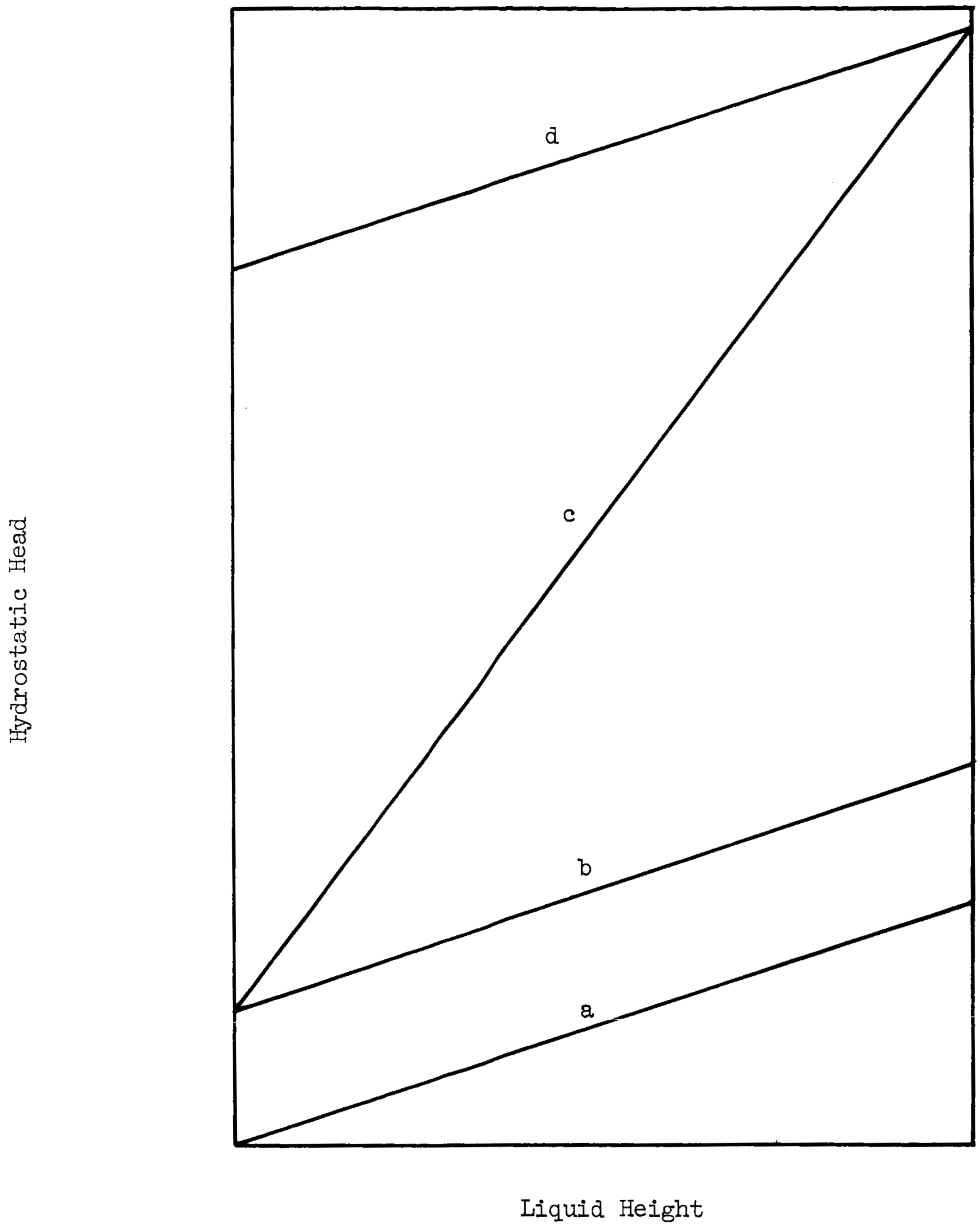

Figure 1. Hydrostatic Pressure for Various Combinations of Liquid Depth, Density, Gravitational Acceleration and Ullage-Pressure.

were accomplished with a fluid having a density of four, subject to normal gravitational attraction and the actual operating ullage-pressure, the pressure profile would be identical with that of curve "c". A considerable savings in weight should be possible because of the decreased wall thickness required for realistic proof-testing.

Because of the large quantities of high density fluid required for actual pressure testing and the general lack of such fluids it is technically and economically more feasible to consider the use of high-density slurries for this purpose.

The purpose of this study thus has been to develop high-density slurries which would be suitable for use as media for hydrostatic testing of stage-propellant tanks. The principal part of the study consisted of an engineering search for dense, granular solids from which slurries could be produced with a specific gravity range from two to about five or six. Slurry formulations meeting these density standards were also required to be chemically stable and non-settling for extended periods of time; to be inert to liquid oxygen, liquid nitrogen and hydrocarbon fuels; to transmit hydraulic pressure as does an in-compressible fluid; to be compatible with stage component materials; to be readily preparable and pumpable with conventional chemical processing equipment; and to be easily removable from tanks and piping by water flushing.

To be economically feasible, finished slurry formulations must be reasonably priced and composed of such readily available materials that purchases of large quantities would not seriously disturb existing markets. Recommendations of preparation, storage, and transfer techniques for suitable slurry formulations and a cost analysis for the materials and equipment required also formed a part of the study.

II. EXPERIMENTAL METHODS

An extensive survey of the chemical and physical properties of a large number of dense, granular solids revealed that oxides of lead, zinc and titanium and barium sulfate possessed many of the properties desired for the purposes of this study. Lead and zinc powder also appeared to be suitable for the higher specific gravities, i.e., from about four to six. Thoria, tungsten carbide, and several other dense materials were eliminated from consideration primarily because of their high cost and limited availability. Water was selected for initial use as the continuous medium in the slurry formulations, principally because it is relatively inert, cheap, and plentiful. The experimental research plan thus was to formulate water-based slurries from the materials outlined above using a variety of dispersing agents, solids loadings, and solids size distributions; to evaluate their suitability; and to refine the more promising formulations to meet contract specifications.

Initial experimental efforts were directed to a determination of the maximum amount of each of the materials selected that could be incorporated into a water slurry without excessive lumping or other signs of particle agglomeration or poor wetting. As expected, the amount of any given material that could be dispersed depended strongly on the particle size distribution and the specific surface area. The very fine powders required considerably more water to wet completely their large surface areas and a lower solids loading limit was established as opposed to the coarser, smaller specific surface area powders. Once dispersed, however, the powders with the finer size distributions exhibited much slower solids settling.

The most critical parameters for producing heavily loaded slurries that exhibit minimal settling are the size distribution of the suspended particles and the type and amount of dispersing agent present. A careful balance between the normal colloidal stability criterion of double-layer interactions and the principle of minimum void space among particles is required to achieve the very high solids loadings necessary for high-density slurries and still retain high fluidity. Maximum density for mechanical packing of particles requires a wide distribution of sizes to permit smaller-diameter particles to occupy the otherwise void spaces in the interstices among larger-diameter particles. Although a stable slurry is quite different from a simple mechanical mixture of particles the general principle of minimum void space among particles is, nevertheless, believed to be an important consideration in producing high-density slurries.[1]

The type and amount of primary dispersing agent used determines the polarity of the slurry, i.e., whether it is anionic, cationic, amphoteric, or non-ionic, and, to some extent, establishes the intensity of particle-to-particle interactions. The dispersing agent is also believed to liberate adsorbed water from the surface of the suspended solids thereby giving a somewhat more fluid suspension. A protective colloid is frequently used in conjunction with the primary dispersant. Casein solutions, methyl or ethyl cellulose and polyacrylates are most often used as protective colloids. Their purpose is to extend the range of particle-to-particle repulsive forces and thereby retard agglomeration and subsequent solids settling. Gelling agents may also be used to flocculate the slurry very slightly and thereby retard particle settling. By providing a weak but continuous gel structure, hard settling of particles is eliminated and redispersion is facilitated. Some other parameters affecting slurry stability are the order of addition of ingredients, pH, viscosity, specific surface area,

(1) R. K. McGeary, "Mechanical Packing of Spherical Particles," J. Am. Ceram. Soc., 44, 513-522 (1961).

surface treatment, and powder wettability. Detailed technical data including particle size distribution, specific gravity, surface treatment, specific surface area, availability and cost were obtained for a number of commercially available metal and metal oxide powders.

Samples of these materials were acquired and numerous trial slurry formulations were prepared from each. The solids included six different grades of zinc oxide powder, two grades of barium sulfate powder, four grades of titanium dioxide powder, two grades of lead oxide (litharge), one grade of zinc powder, and two grades of lead powder.

A. Method of Preparation

A solution of hot water and dispersing agent was prepared by slowly adding dispersing agent to the water with mild agitation until all of the dispersant was dissolved and the solution was homogeneous. The degree of agitation was then increased to the highest level possible without inducing foaming or air entrainment by vortexing and the solid powder was added slowly in small quantities. The agitator speed was further adjusted to higher levels as the viscosity of the slurry increased and the rate of solids addition was decreased proportionately as the time required for wetting and dispersion increased. After about one-third to one-half of the total solids had been added, solids addition was stopped and the dilute slurry was mixed at high speed for about five minutes. This procedure was repeated several times during the addition of the remaining solids. This step-wise additions of solids permitted better wetting and dispersion of small agglomerates than could be achieved by continuous solids addition. After the addition of each solids increment, a few drops of dilute, silicone anti-foam emulsion were added to aid in the elimination of entrained air and to prevent further foaming.

Several different types of agitation systems were evaluated. These included propellors, paddles, turbine impellers, and high-shear, twin-blade homogenizers. A turbine-type impeller located very near the bottom of the mixing container gave the best combination of fluid shear and circulation needed for dispersing large quantities of solids. The initial water temperature was between 160 and 180^{O}F but generally decreased to about 100 to 110^{O}F by the end of solids addition. The use of hot water improved the rate of solids wetting and also resulted in lower power requirements for mixing because of decreased slurry viscosities at elevated temperatures.

In most instances, the finished slurry formulation was passed through a colloid-type dispersing mill to assure complete wetting and dispersion of the solids. By operating at low clearances between the conical rotor and stator of the colloid-type mill, uniform grinding into the lower micron size ranges was also possible.

The density, pH, viscosity, and per cent total solids were determined for each slurry. The most promising were further tested for particle size distribution, settling rate, cryogenic liquid compatability and corrosion characteristics.

B. Particle Size Distribution Measurements

The particle size distribution of the suspended solids was determined with a Coulter Counter, (Coulter Industrial Sales Co., Chicago, Illinois). This instrument electronically sizes and counts individual particles as they flow through a small aperture. A very dilute suspension in a weak electrolyte solution is prepared of the particles to be measured. The particle concentration of the suspension is sufficiently dilute that individual particles may pass through the aperture essentially one at a time. During operation, a known potential is established between two electrodes which are located on opposite sides of the aperture, and a pressure differential is establsihed across the aperture by unbalancing a mercury manometer connected to the electrolyte system. The dilute suspension flows from a beaker surrounding the aperture tube through the aperture and into a collecting vessel. As the particles pass through the aperture they momentarily change the effective conductivity of the aperture path by an amount that is proportional to the particle volume. The resulting electrical pulses are received by the electronic portion of the counter where they are amplified, scaled and counted. By varying the current across the aperture, the effective size limit of the aperture can be varied and counts can be obtained at a series of levels. In this manner a complete distribution of particle sizes may be obtained. As many as 6000 particles per second can be counted and sized with this device. The distributions thus obtained are generally much more significant from a statistical point of view than those obtained by microscopic or other direct measurement methods. Also, since the parameter measured with this method is the displaced particle volume, the resulting distributions are much more sensitive to particle agglomeration than are other methods such as sedimentation analysis. This is of particular importance when trying to produce stable slurries.

C. Particle Size Reduction Methods

Since the particle size distribution of many of the metal oxides as supplied by manufacturers was too coarse for stable slurry formulations, size reduction was accomplished either by grinding in a Tri-Homo, colloid-type mill, (Patterson Industries, East Liverpool, Ohio), or by wet ball-milling. The Tri-Homo mill is particularly useful in the preparation of slurries because the high-shear gradients established in it between two conical rotors are very effective in dispersing small agglomerates that may have developed during solids addition. Considerable solids grinding is also possible with the Tri-Homo although it will not approach the ball-mill in the ultimate fineness attainable.

D. pH Measurement

The pH of each slurry was measured by immersing standard glass and calomel electrodes of a Beckman pH meter (Beckman Instruments, Fullerton, California) directly in the slurry and recording the meter reading. After each measurement, the electrodes were carefully rinsed with distilled water to remove all residual slurry; they were kept immersed in clean, distilled water when not in use. The meter was standardized at least once each day with a buffer solution having a pH of 7.0.

E. Density Measurement

The specific gravity of each slurry formulation was determined by weighing a known volume of slurry to the nearest tenth of a gram.

F. Rheological Measurements

The rheological properties of the slurries were determined by measuring apparent viscosities with a Brookfield LVF viscosimeter at various shear rates and with several different spindle sizes. Since slurries are inherently non-Newtonian in character, apparent viscosities at various shear rates are often necessary to specify their flow properties. Measurements of this type are also frequently needed to define time-dependent flow properties such as thixotropic or rheopectic behavior wherein shear-stress varies with time when a constant shear rate is applied to the slurry. Controlled thixotropy is a very desirable characteristic for maintaining stability against particle settling under static conditions and yet permitting reasonable viscosities when a shear gradient is applied to the slurry. These are reversible phenomena; that is, it reverts to its original, gel-like structure when shear stresses are removed from such a material.

For routine comparisons of slurry properties, a single measurement of the apparent viscosity with a No. 4 spindle at 60 rpm was used. All apparent viscosities reported in this study are for the Brookfield LVF No. 4 spindle at 60 rpm unless otherwise specified.

G. Cryogenic Liquids Compatability

The compatability of selected slurry formulations with liquid oxygen was determined by depositing films of the slurries on the inner walls of stainless steel beakers and, after allowing the slurry to dry, filling the beakers with liquid oxygen. After all of the liquid oxygen had boiled off the beakers were allowed to warm to room temperature and were then re-filled with liquid nitrogen. Observations were made during these proceedings for any reactions or other apparent changes in the appearance of the dried films. When the beakers had again warmed to room temperature, the dried films were removed and examined with an optical microscope. The dried films were also re-dispersed in water and further examined for any apparent changes.

H. Corrosion Susceptibility Measurements

Corrosion studies with aluminum alloy stage components and stainless steel alloys were conducted with selected slurry formulations. These tests were performed by immersing carefully cleaned and weighed samples of the various alloys in the slurries and determining the general appearance and weight change of the samples at selected time intervals. Other samples were only partially immersed in the slurries to test the susceptibility of the samples to oxygen concentrations cells. Corrosion products formed were analyzed by X-ray and electron diffraction and by metallographic examination.

Types 304 and 316 stainless steel and aluminum alloy types 2219 and 2014-T6 were included in the corrosion-test program. The 2219 alloy was tested with no surface treatment and with an aerodite surface treatment. Samples of the 2014-T6 alloy were tested with no surface treatment and with anodized and alodine 1200 conversion coatings. The 304 and 316 stainless steels were tested with no surface treatment and after passivation.

I. Hydrostatic Pressure Measurements

A plexiglass container, 6 inches in diameter and 5 feet long, was equipped with a manometer leg for hydrostatic pressure testing. Slurries of various specific gravities were placed in the cylinder and the hydrostatic pressure developed was recorded as a function of slurry depth. Further qualitative studies of slurry compressibility were also made by subjecting small quantities of slurry in a steel cylinder to hydraulic pressures of several hundred pounds per square inch.

J. Dispersing Agent Studies

The type and amount of dispersing agent used in formulating highly loaded slurries determines, to a large extent, the zeta-potentials established for each particle and the mutual repulsive forces that thereby exist among them. The natural Brownian motion associated with very small particles and the interparticle repulsive forces established by dispersing agents may result in stable, non-settling slurries. When individual particles are completely dispersed in a highly loaded slurry, the viscosity of the slurry is generally quite low since any applied shear gradient is expended primarily in establishing a velocity gradient across the continuous medium. However the slurry viscosity is generally much higher if the particles in such a slurry are present in a flocculated condition. The higher viscosity results because a portion of the applied shear must be expended in breaking the long chains of particle aggregates which exist throughout the slurry. An ideal dispersing agent also should promote particle wetting and dispersability without significantly lowering the surface, or interfacial, tension of the continuous medium. Lowering the surface, or interfacial, tension often results in undesirable foaming and frothing.

For a given solids loading, the highest degree of dispersion is generally believed to coincide with the minimum slurry viscosity. The lower viscosities are desirable from an engineering viewpoint since energy requirements for pumping and agitation are less. Therefore, to establish the optimum amount of dispersant, a number of slurries were prepared, each having the same per cent solids but with varying amounts of dispersant. Slurry viscosities were measured under identical conditions and the quantity of dispersant that yielded the lowest viscosity was determined. Similar tests were conducted for each type of dispersing agent. Dispersing agents evaluated included Tamol 731, Tamol SN, Tamol 850,(Rohm and Haas Company, Philadelphia, Pa.), Darvan N, (R. T. Vanderbilt Company, Akron, Ohio), Igepal CO 630, (General Aniline and Film Company, New York, N.Y.), Tergitol (Union Carbide and Chemical Corporation, New York, N.Y.), and tetra sodium pyrophosphate.

Several thickening and gelling agents were also evaluated. The ultimate goal was to select a dispersant that would provide complete dispersion of solid particles and then select a second material which would establish a weak gel structure in the slurry. A weak gel structure established under static conditions would greatly impede solids settling and would result in a soft, readily re-dispersible sludge when significant settling had occurred after long-term storage. A slurry thus formulated would be rather viscous yet flow readily when shear was applied.

K. Total Solids Determination

The percentage of total solids present in a slurry was determined by weighing a known quantity of slurry to the nearest hundredth of a gram, drying to constant weight at 110^{o}C, cooling to room temperature, and re-weighing.

L. Slurry Abrasiveness

The abrasive characteristics of each slurry were evaluated qualitatively by examining the rotor of the Tri-Homo mill for indications of excessive wear after grinding batches of each slurry.

M. Settling Characteristics

The settling characteristics of slurries were qualitatively evaluated by determining the amount and degree of compaction of sediments formed after various storage periods, and the amount of clear liquid observed at the top of slurries after storage.

III. EXPERIMENTAL RESULTS AND DISCUSSION

The range of specific gravities theoretically attainable with water-based slurries of titanium dioxide, barium sulfate, zinc oxide, and lead oxide (litharge) is shown by Figure 2. As a general rule, solids loadings greater than about 80 to 90 per cent by weight are very difficult to achieve and still maintain good flow properties. At very high volume percentages of solids, particle-to-particle contact prevails and a thick paste results. Such a mixture is not truly a fluid, either Newtonian or non-Newtonian. It is two continuous phases, each capable of flowing and transmitting pressure. The specific gravities indicated at about 85 to 90 weight per cent solids may be considered as practical upper limits for fluid slurries that transmit pressure as a homogeneous fluid.

Obviously, titania, zinc oxides and barium sulfate are poorer choices than lead oxide from a density standpoint; however, they are of of considerable interest because of their ready availability in a wide range of particle sizes and the considerable technical knowledge in formulating slurries of these materials that has been acquired by the paint and rubber industries.

A. Titanium Dioxide Slurries

At the beginning of this study, titanium dioxide powder was the only material on hand. Therefore, the initial, exploratory slurries were prepared using this material. Pigment-grade titanium dioxide is a very finely-divided material, generally having a mass mean diameter less than 0.10 micron and with a relatively large specific surface area. Stable slurries were prepared with this material at up to 80 weight per cent solids. Solids loadings above 80 weight per cent gave very viscous slurries and wetting of the solids was quite difficult. Slurries containing less than 80 weight per cent solids were

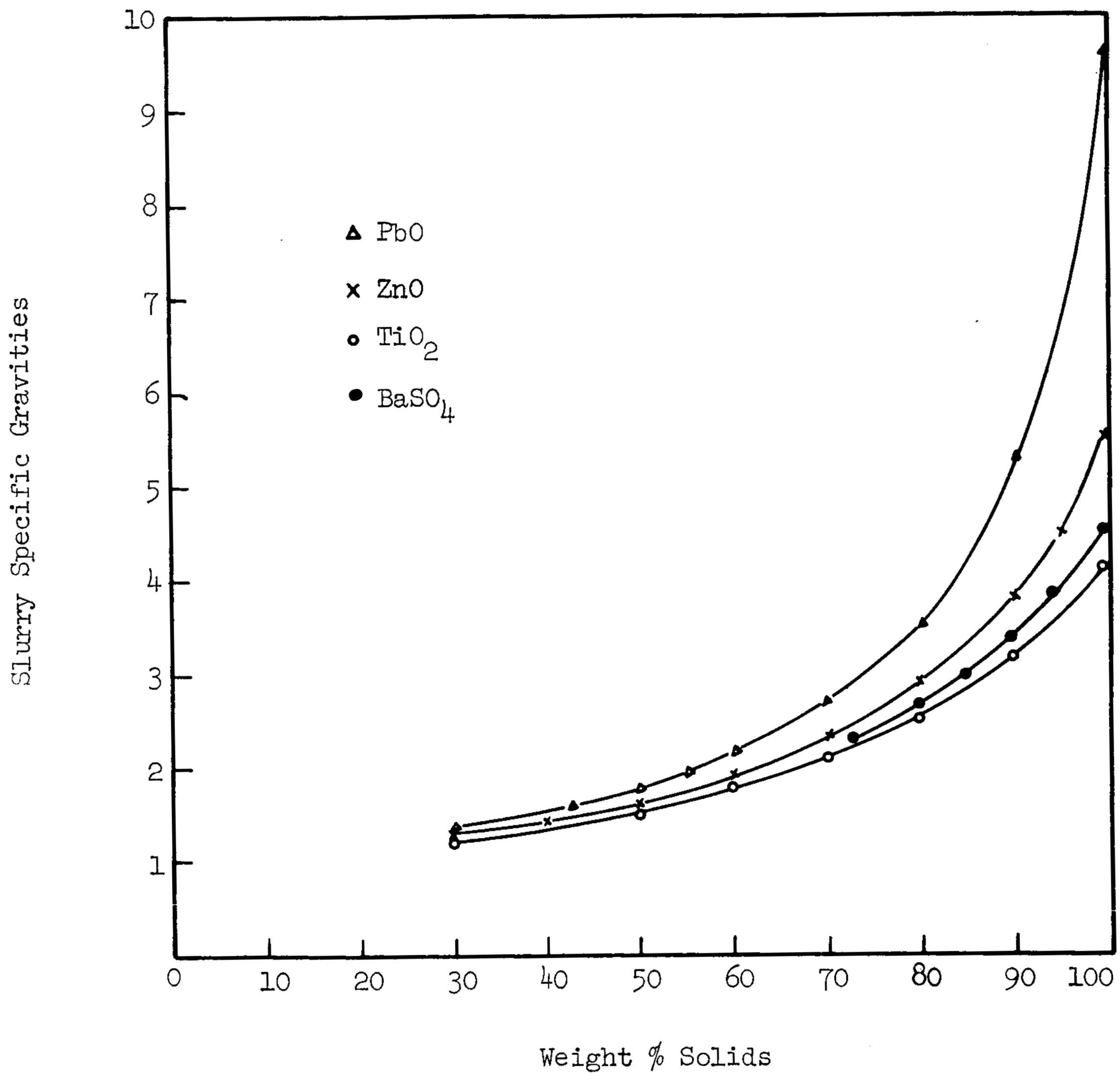

Figure 2. Specific Gravities of Water-Based Slurries for Various Solids Loadings.

readily preparable with moderate-speed agitation and needed no further grinding for stabilization against settling. Table I lists the necessary ingredients and the procedure for preparing a slurry of titanium dioxide with a specific gravity of 2.5. Slurries with lower specific gravities can be produced simply by increasing the amount of water and adjusting the final viscosity with Benagel EW, (National Lead Company, New York, N.Y.), to that specified in Table I. Physical properties, stability, and compatability results for these slurries are also presented in Table I.

B. Zinc Oxide and Zinc Slurries

Zinc oxide is a relatively dense material that is moderately priced and readily available in a wide range of particle size distributions. Materials having been given several surface treatments are also available. Slurry specific gravities to 3.5 with good flow properties were obtained with this material. The best slurry settling characteristics were obtained when a combination of three different particle size grades of zinc oxide were used. Particle size distributions for the three grades and the composite distribution are shown in Figures 3 and 4, respectively. These results confirm that the principle of maximum packing as applied to a wide range of particle sizes rather than a narrow distribution of sizes yields the best combination of slurry settling and flow characteristics. Table II lists the ingredients and preparation procedure for producing a stable zinc oxide slurry with a specific gravity of 2.85. Slurry properties and test results are also shown in Table II. Specific gravities higher than about 2.85 required high-shear grinding in a Tri-Homo mill to wet and disperse completely all of the solids. High speed agitation with a turbine-type impeller was adequate to prepare zinc oxide slurries with specific gravities less than about 2.85.

TABLE I

PREPARATION PROCEDURE FOR A TITANIUM DIOXIDE SLURRY AND PROPERTIES OF THE FINISHED SLURRY

Materials	% Solids	Dry Weight	Wet Weight
Titanium Dioxide*	100.00	100.00	100.00
Tamol 850	30.00	.50	1.67
Water	--	--	24.00
Benagel EW	100.00	.10	.10

Procedure: Add the Tamol 850 solution to the required amount of hot water with mild agitation. After the Tamol 850 has mixed, increase the agitation to the highest possible level without causing a vortex to form and slowly add the powder at the point of maximum turbulence. When the mixture begins to become rather viscous, stop the solids addition and allow the slurry to mix at high speed for several minutes. At the end of this high-speed mixing period, decrease the agitation to a very slow speed and add 5 drops of Dow Corning Anti-foam B per gallon of mixture. Repeat this sequence as many times as required until all of the Titanium Dioxide has been added. Add the required amount of Benagel EW with maximum agitation. Let mix for at least 10 minutes to disperse the Benagel EW.

Properties

Specific Gravity	2.5
Weight Per Cent Titanium Dioxide	80.0
Volume Per Cent Titanium Dioxide	47.0
Viscosity	850 cps
pH	10.5
Mass mean equivalent spherical diameter	0.10
Cryogenic Compatability	Very good
Water Washability	Very good
Volume Per Cent Sediment after:	
1 Day	0
7 Days	4
14 Days	5

* Anatase Grade A-440 supplied by New Jersey Zinc Company, New York, New York.

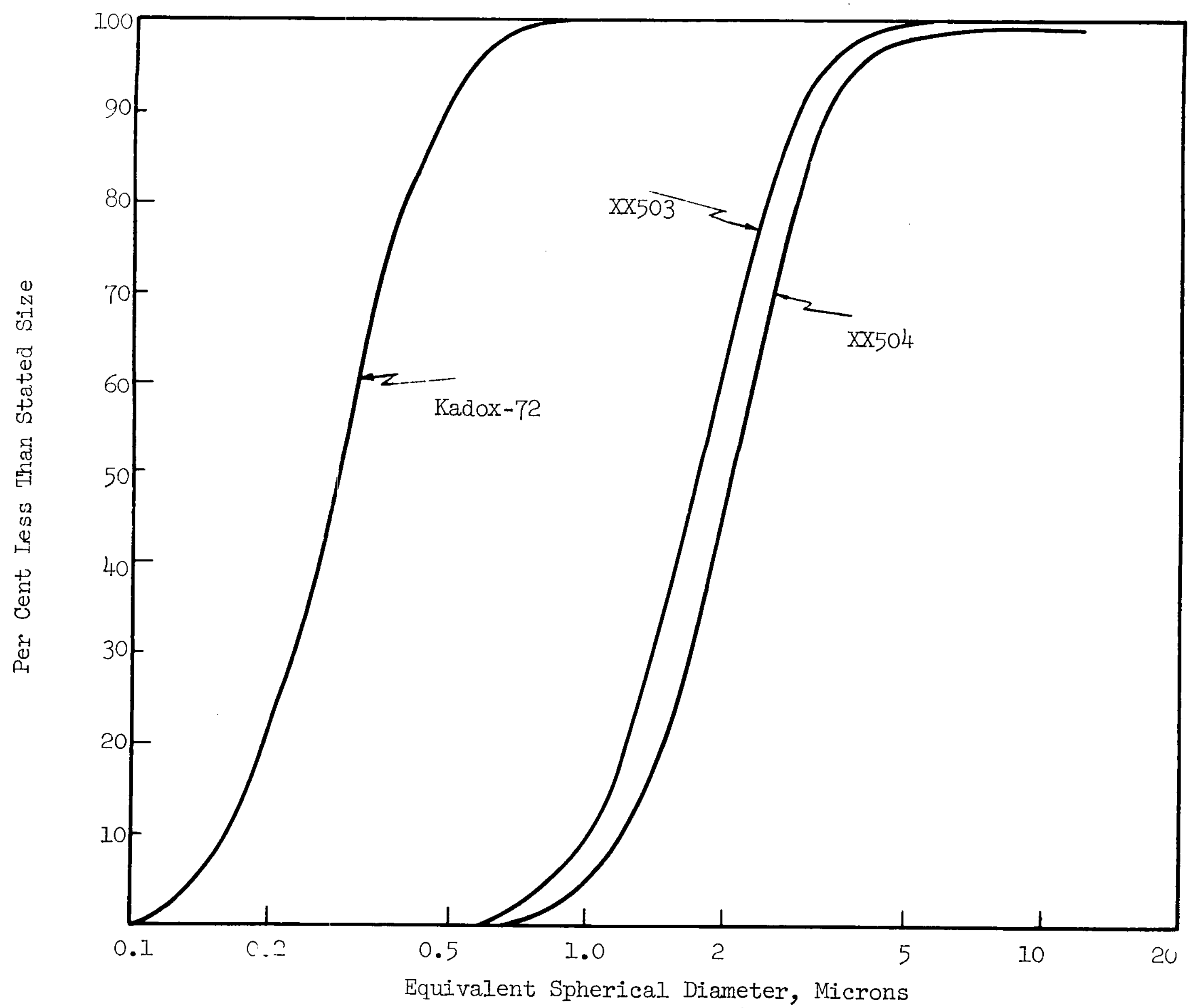

Figure 3. Particle Size Distributions for Commercially Available Zinc Oxide Powders.

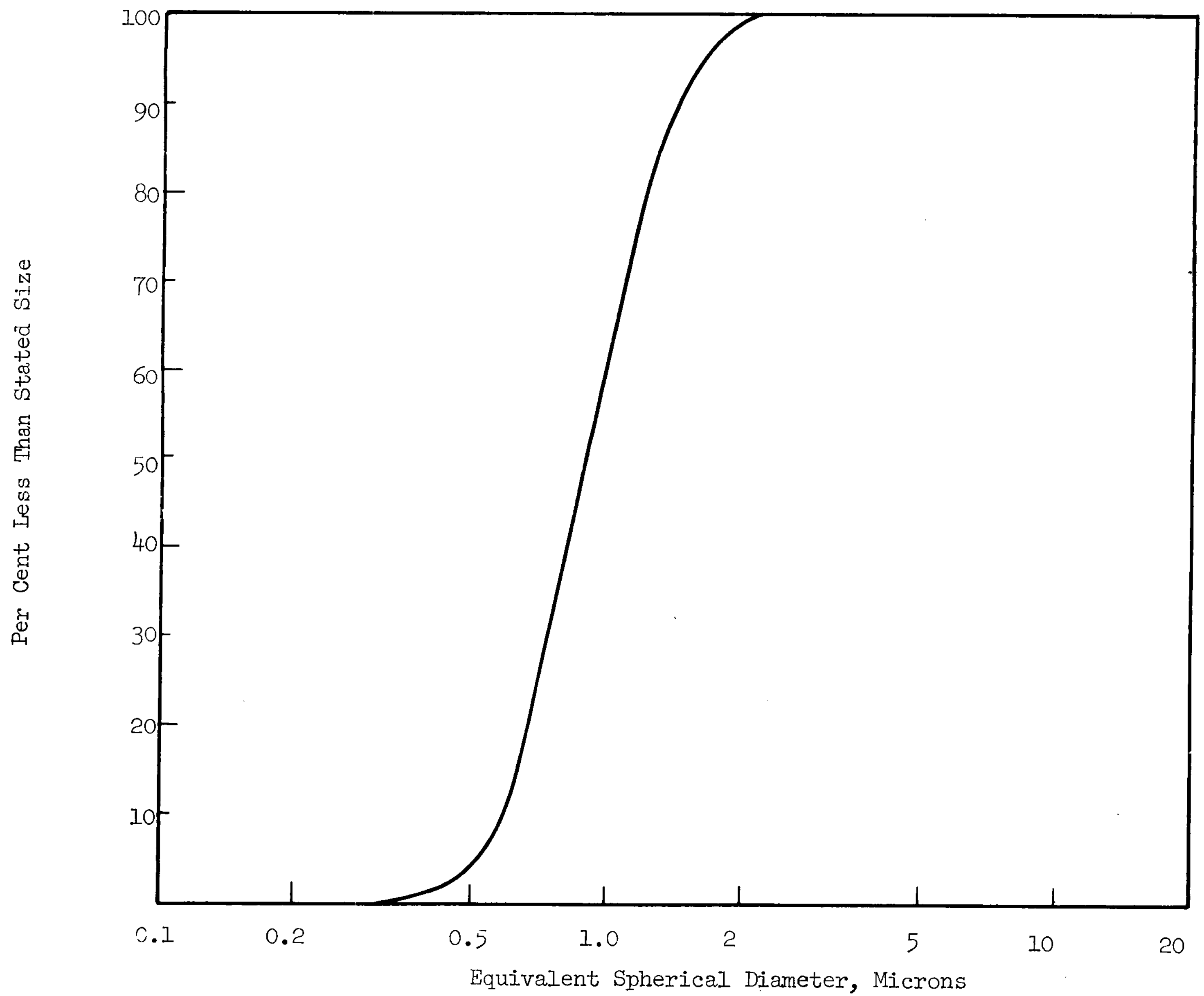

Figure 4. Particle Size Distribution for a Composite Mixture of Three Grades of Zinc Oxide.

TABLE II

PREPARATION PROCEDURE FOR A ZINC OXIDE SLURRY AND PROPERTIES OF THE FINISHED SLURRY

Materials	% Solids	Dry Weight	Wet Weight
Kadox 72 Zinc Oxide*	100.00	40.00	40.00
XX503 Zinc Oxide*	100.00	30.00	30.00
XX504 Zinc Oxide*	100.00	30.00	30.00
Tamol 850	30.00	.75	2.50
Hot Water	--	--	22.50
Benagel EW	100.00	.10	.10

Procedure: Add the Tamol 850 solution to the required amount of hot water with mild agitation. After the Tamol 850 has mixed, increase the agitation to the highest possible level without causing a vortex to form and slowly add the powder at the point of maximum turbulence. When the mixture begins to become rather viscous, stop the solids addition and allow the slurry to mix at high speed for several minutes. At the end of this high-speed mixing period, decrease the agitation to a very slow speed and add 5 drops of Dow Corning Antifoam B per gallon of mixture. Repeat this sequence as many times as required until all of the zinc oxide has been added. Add the Benagel EW with high speed agitation and mix at least 10 minutes to assure dispersion.

Properties

Specific Gravity	2.85
Weight Per Cent Zinc Oxide	80.0
Volume Per Cent Zinc Oxide	41.0
Viscosity	750
pH	10.9
Mass mean equivalent spherical diameter	.90
Geometric Standard Deviation	2.90
Cryogenic Compatability	OK
Abrasiveness	Low
Water Washability	Very Good
Volume Per Cent Sediment after:	
1 Day	0
7 Days	2
14 Days	6

* Supplied by New Jersey Zinc Co.

Slurries prepared with elemental zinc powder were generally unsatisfactory. The particle size distribution was difficult to control and excessive settling was experienced with all trial formulations. The highly reactive nature of finely-divided zinc resulted in a reaction of zinc particles with the Tamol 850 dispersing agent in the relatively high pH slurries. Considerable gas evolution and a general de-stabilization was noted after storage for several days. Non-ionic dispersants proved to be ineffective and no further efforts were attempted with this material.

C. Lead Oxide and Lead Slurries

Slurry specific gravities to slightly above 3.9 were achieved with lead oxide. For the purposes of this report, all references to lead oxide are specifically to litharge, i.e., PbO, unless otherwise identified. The commercially available grades of fumed litharge are too coarse to permit formulation of non-settling slurries. The particle size distribution of the finest commercially available grade is shown by Figure 5. Many trial formulations were prepared with this material using various types and amounts of dispersing agents. Rapid solids settling was experienced at all solids loadings with the as-received material. When the lead oxide was wet-ground in a ball-mill or colloid-type mill to a specified size distribution, fluid slurries could be produced that showed minimal settling after two weeks storage. Figure 6 shows the particle size distribution of such a slurry. Comparison of Figures 5 and 6 indicates the degree of size reduction required.

Lead oxide powder is a very difficult material to wet and deagglomerate. Slurries produced by simply adding lead oxide powder to water and dispersing agent with moderate-speed turbine agitation have a characteristically high viscosity (3,000-10,000 cps). If this same slurry is ball-milled or processed

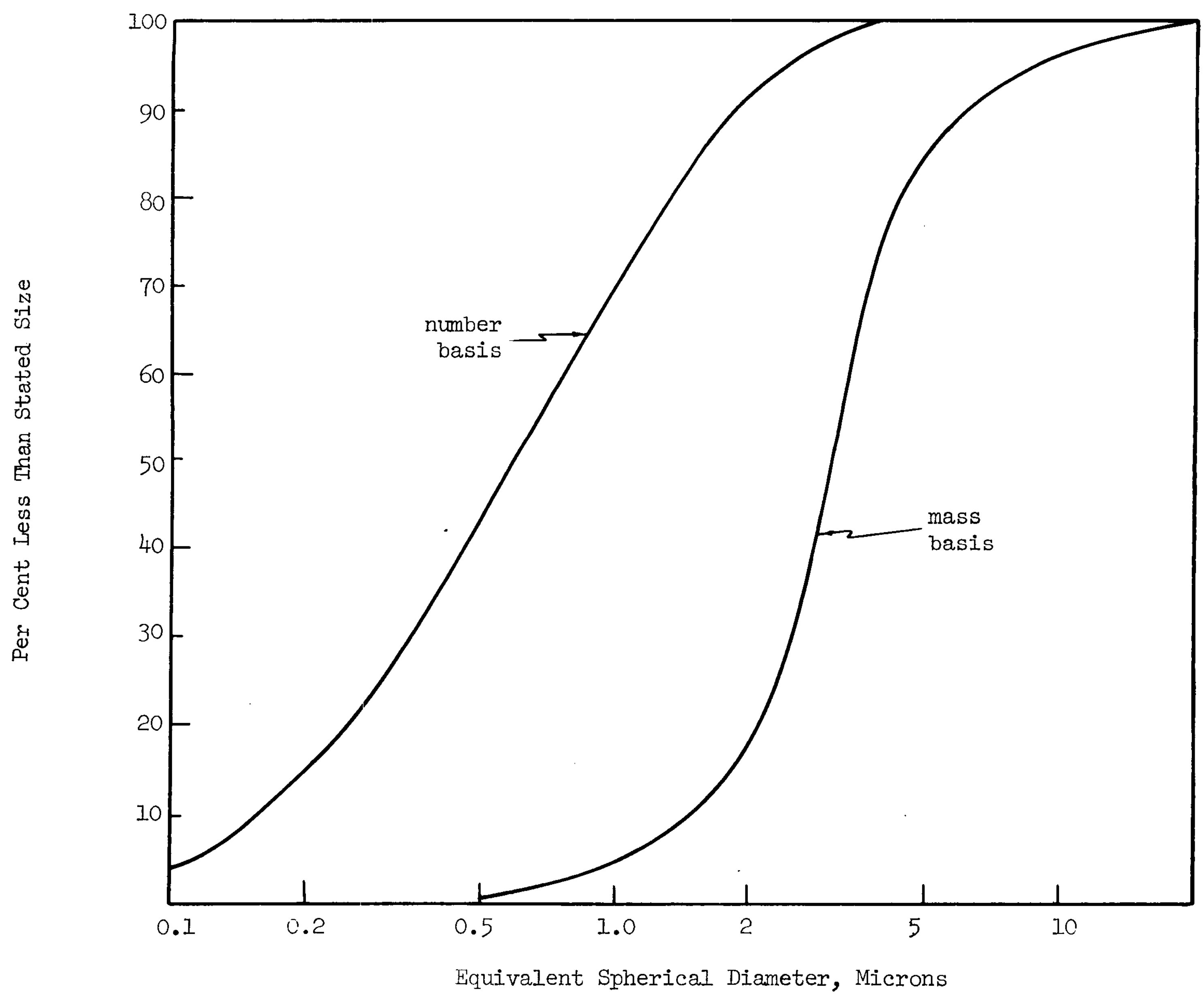

Figure 5. Particle Size Distribution for Commercially Available Fumed Litharge.

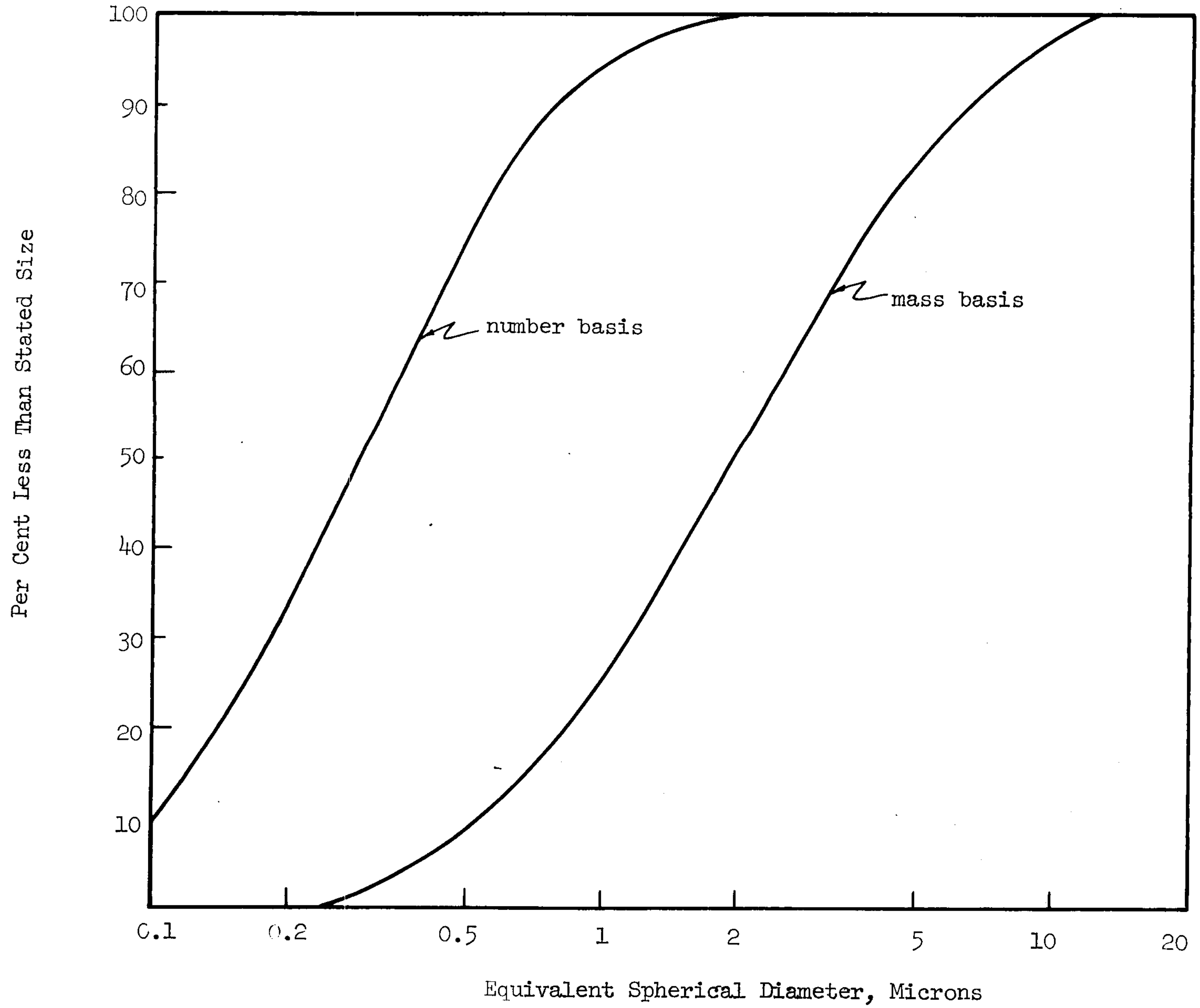

Figure 6. Particle Size Distribution for a Stable Lead Oxide Slurry.

in a high-shear dispersing device, the viscosity drops to only 100-200 cps and the slurry is very smooth and has a homogeneous appearance. Care must be exercised during the solids addition to avoid excessive air entrapment. Entrained air gives rise to much higher viscosities and can result in a quasi-compressible fluid. Much of the grinding energy expended in producing suitable slurries from this material is utilized in the breaking of agglomerates and only a relatively small fraction goes into actual particle size reduction. Slurries produced from lead oxide powder that had been dry-ground to the required particle size distribution were still very difficult to wet and required high-shear dispersing action to achieve high fluidity. Some type of high-shear grinding mill or a ball-mill is considered a necessity for producing highly loaded slurries with this material, particularly for specific gravities above 3.5. Several passes through a laboratory model Tri-Homo mill were required to achieve the dispersion and size reduction required. Industrial models of high-shear grinders are generally more efficient, however, and a single pass through such a device equipped with an abrasive rotor material operating at a close clearance may achieve the desired result. Wet-grinding in a ball mill gave the best overall slurry properties. Solids loadings to about 90 weight per cent were possible when a ball mill was used to grind and disperse the materials. Ball mills, however, are not desirable for large-quantity production, primarily because of their low output rate. Further studies at the pilot plant level are needed to determine the suitability of high-volume, high-shear dispersing devices for large-scale production of lead oxide slurries with densities above 3.5.

Table III lists the ingredients and procedure to prepare a lead oxide slurry with a specific gravity of 4.0. Slurry properties and test results are also presented in Table III.

TABLE III

PREPARATION PROCEDURE FOR A LEAD OXIDE SLURRY AND PROPERTIES OF THE FINISHED SLURRY

Materials	% Solids	Dry Weight	Wet Weight
Lead Oxide*	100.00	100.00	100.00
Tamol 850	30.00	1.00	3.33
Hot Water	--	--	16.00
Benagel EW	100.00	.15	.15

Procedure: Add the Tamol 850 solution to the required amount of hot water with mild agitation. After the Tamol 850 has mixed, increase the agitation to the highest possible level without causing a vortex to form and slowly add the powder at the point of maximum turbulence. When the mixture begins to become rather viscous, stop the solids addition and allow the slurry to mix at high speed for several minutes. At the end of this high-speed mixing period, decrease the agitation to a very slow speed and add 5 drops of Dow Corning Antifoam B per gallon of mixture. Repeat this sequence as many times as required until all of the lead oxide has been added.

Grind the completed slurry formulation in a Tri-Homo or equivalent type disperser-homogenizer operating at a rotor clearance of 0.001 inch. After the slurry has passed through the mill, return it to the original container and, with the highest level of agitation possible, add the required amount of Benagel EW. When the slurry is homogeneous and shows no signs of lumping, process it through the Tri-Homo mill three more times.

Properties

Specific Gravity	4.0
Weight Per Cent Lead Oxide	84
Volume Per Cent Lead Oxide	35
Apparent Viscosity (LVF #4 @60 rpm)	1150 cps
pH	11.9
Number mean equivalent spherical diameter	0.28μ
Mass mean equivalent spherical diameter	1.95μ
Geometric Standard Deviation	2.33
Cryogenic Compatability	O.K.
Abrasiveness	Low
Water Washability	Fair, use cold water
Volume Per Cent Sediment After:	
1 Day	0
7 Days	1.5
14 Days	2.0

*Fumed Litharge supplied by National Lead Corporation.

Specific gravities to 6.1 were obtained with mixtures of lead and lead oxide. The resulting slurries had good flow properties but settled into a hard mass after about three days storage. Further laboratory studies are needed to produce stable, non-settling slurries with specific gravities above about 5.0. The pressure transmitting characteristics of such high density slurries should also be studied carefully.

Direct exposure of lead oxide slurries to sunlight caused a series of color changes from bright yellow to a dull red-brown. In some instances, the color further changed to black upon prolonged exposure to sunlight. These color changes occurred only within a very thin layer at the surfaces of the sample containers. X-ray diffraction of the surface film revealed a wide range of oxides of lead, from lead sub-oxide to lead dioxide. These darker films were somewhat more difficult to remove by water-rinsing than the usual film, but, otherwise presented no difficulties. No measurable density alterations were noticed after these changes had occurred. Closed containers of slurry sometimes turned a light shade of yellow-brown on prolonged storage but showed no other changes in properties. These changes are probably due to the large number of lead oxides that are thermodynamically possible within the pH range of these slurries. Examination of a Pourbaix diagram for lead and water reveals that litharge is the most stable state for lead at these pH levels, so very little over-all change in oxidation states would be predicted.

Wet films of lead oxide slurry were best removed from aluminum and glass surfaces by rinsing with cold water. However, if the films were allowed to dry, removal was considerably more difficult and some abrasive action was often required. Complete removal from porous surfaces was quite difficult since the fine particle size of the lead oxide resulted in a penetration of the surface. Tamol 850 concentrations of one per cent and higher improved washability of these slurries.

Lead oxide slurries with specific gravities of 3.0 and 4.0 were used in the hydrostatic testing device described previously. A comparison of theoretical and experimental results is shown in Figure 7. The experimental results and theoretical predictions agree within the limits of experimental error. More extensive laboratory-scale testing in fully instrumented prototype test equipment is recommended to verify the full range of hydrodynamic properties.

D. Barium Sulfate Slurries

Specific gravities to 3.2 were achieved with barium sulfate powder. Slurries produced with this material had rheological properties vastly different from any of the other slurry materials tested. The apparent viscosity of barium sulfate slurries containing more than about 80 weight per cent solids increased markedly with increasing rate of shear. Thus, this slurry was dilatant as opposed to the pseudoplastic or thixotropic nature of slurries produced from lead oxide, for example. This property is most undesirable from an engineering point-of-view because of the excessive power requirements to mix and pump the material. Quite possibly, slurries could be produced from this material that would not exhibit this type behavior. Further studies are desirable to explore this possibility if barium sulfate slurries are deemed otherwise suitable.

Barium sulfate powder up to 80 weight per cent solids wets and disperses readily in water with moderate-speed turbine agitation. However, early efforts to produce stable slurries from this material were discouraging because of rapid solids settling. Several new, smaller particle size grades of this material were received during the latter part of this study and better results were obtained using approximately the same formulations as with lead oxide.

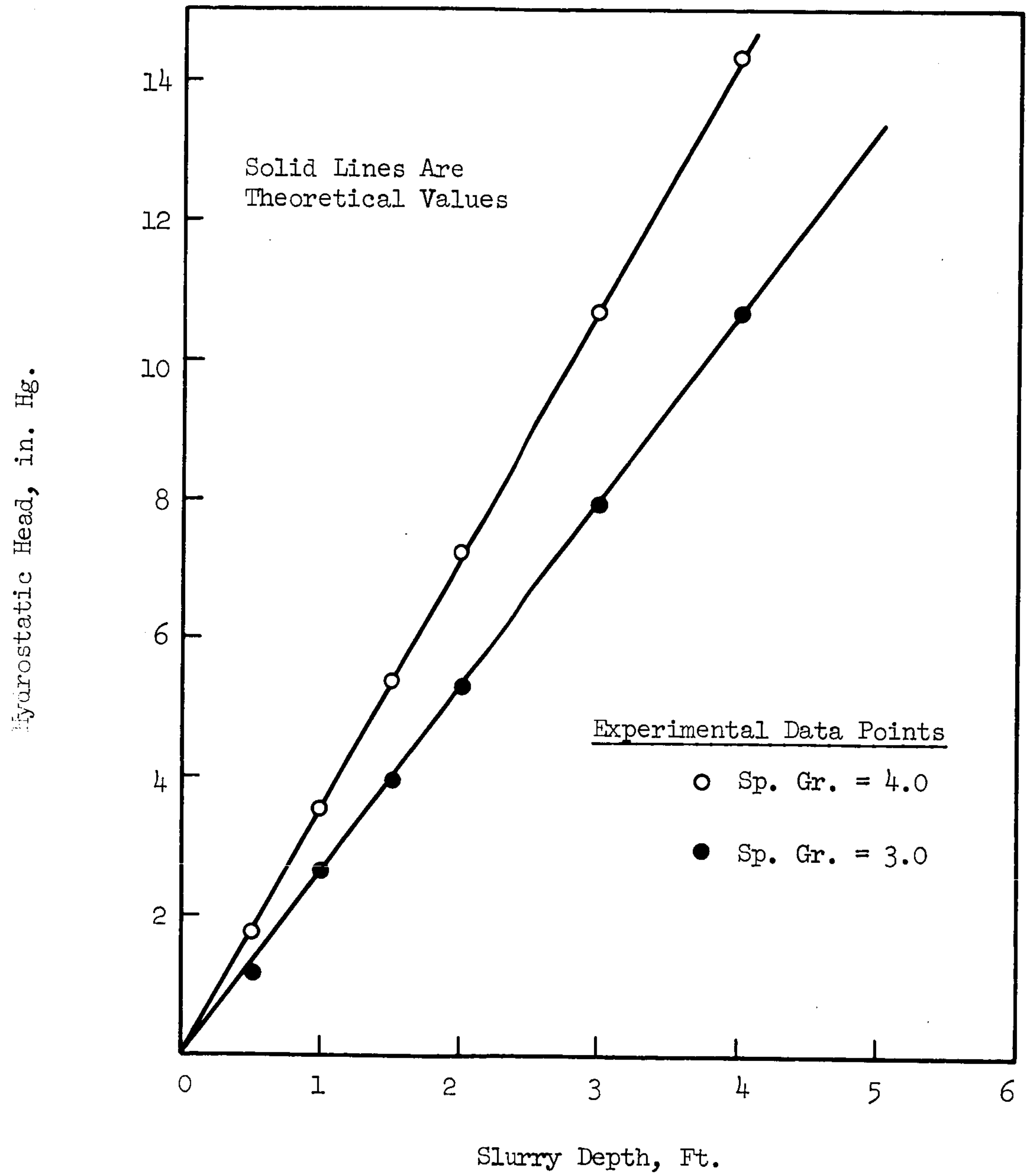

Figure 7. Comparison of Predicted and Measured Hydrostatic Pressures for Lead Oxide Slurries.

Specific gravities to 3.2 were achieved with the newer material without any additional grinding. Solids settling remained somewhat of a problem, although it is believed that selection of a proper dispersant will minimize the settling. Barium sulfate is acidic whereas the other materials tested were basic, therefore a dispersant with opposite polarity to that of Tamol 850 may be most effective. Table IV lists the preparation procedure and properties for a barium sulfate slurry with a specific gravity of 3.0.

E. Corrosion Test Results

Active and passivated samples of types 304 and 316 stainless steel showed no noticeable attack after total and partial immersion in lead oxide slurries for 12 days. Each of the aluminum alloys, i.e., 2014-T6 and 2219, with and without protective oxide coatings showed noticeable corrosive attack after 12 days immersion in lead oxide slurry. The unprotected alloys each reacted to give a black crust which was shown by X-ray diffraction to consist of a mixture of lead and lead oxide (PbO). The extent of reaction was greatest at the freshly cut edges of the sample.

The 2014-T6 with an alodine 1200 conversion coating gave a glossy black finish after three weeks immersion. Electron diffraction confirmed that the tightly adherent film was composed of lead and lead oxide (PbO). Metallurgical examination of the coatings interface revealed that very little pitting attack had occurred. The corrosion sample showed a slight weight increase after the three-week immersion.

An anodized sample of 2014-T6 showed some slight pitting attack but no continuous black film formation as was observed on the alodine coated sample. The pitting attack occurred only at isolated spots on the sample; probably at imperfections in the anodized coating. Metallurgical examination and electron

TABLE IV

PREPARATION PROCEDURE FOR A BARIUM SULFATE SLURRY
AND PROPERTIES OF THE FINISHED SLURRY

Materials	% Solids	Dry Weight	Wet Weight
Barium Sulfate*	100.00	100.00	100.00
Tamol 850	30.00	1.00	3.33
Hot Water	--	--	11.60
Benagel EW	100.00	--	--

Procedure: Add the Tamol 850 solution to the required amount of hot water with mild agitation. After the Tamol 850 has mixed, increase the agitation to the highest possible level without causing a vortex to form and slowly add the powder at the point of maximum turbulence. When the mixture begins to become rather viscous, stop the solids addition and allow the slurry to mix at high speed for several minutes. At the end of this high-speed mixing period, decrease the agitation to a very slow speed and add 5 drops of Dow Corning Antifoam B per gallon of mixture. Repeat this sequence as many times as required until all of the Barium Sulfate has been added.

Properties

Specific Gravity	3.0
Weight Per Cent Barium Sulfate	86.0
Volume Per Cent Barium Sulfate	57.0
Viscosity	4800 cps
pH	10.3
Mass mean equivalent spherical diameter	3.1
Washability	Very good
Volume Per Cent Supernatant Liquid after:	
1 Day	0
7 Days	5

* Barimite XF, supplied by Thompson, Weinman and Co., Cartersville, Georgia.

microprobe analysis showed slight pits with the formation of some elemental lead.

A sample of aerodite coated 2219 reacted very similarly to the anodized 2014-T6, that is, only slight pitting was observed after a three week exposure to lead oxide slurry. Metallurgical examination showed only slight pits but no definite analysis of the corrosion products could be made with the electron microprobe.

Slurries formulated with zinc oxides gave the same general results. No extensive testing was done with them, however, since lead oxide appeared to be the most likely choice for a test medium.

Apparently the corrosion mechanism consists essentially of a reduction of the lead oxide present as a high pH slurry in contact with the aluminum surface coating to yield a surface film of lead and lead-oxides. The most obvious way to minimize this type of corrosion with active metals such as aluminum alloys is to adjust the slurry pH to neutral. All attempts to lower the slurry pH and to formulate slurries with a finished pH of about neutral resulted in destabilization and rapid solids settling.

F. Optimization of Dispersant Level

A large number of different types of dispersing agents were evaluated. Tamol 850 was found to be the most effective dispersant for all of the solids materials with the possible exception of barium sulfate. Lead oxide was the most difficult of all to disperse, consequently most of the dispersant evaluation tests were conducted with it. Figure 8 shows the apparent viscosity of lead oxide slurries containing 80 weight per cent solids and with varying amounts of Tamol 850 and Tamol SN dispersant and no gelling agent. Each of the slurries was passed through a laboratory model Tri-Homo mill four times while operating at a rotor clearance of 0.001 inch. In all cases the

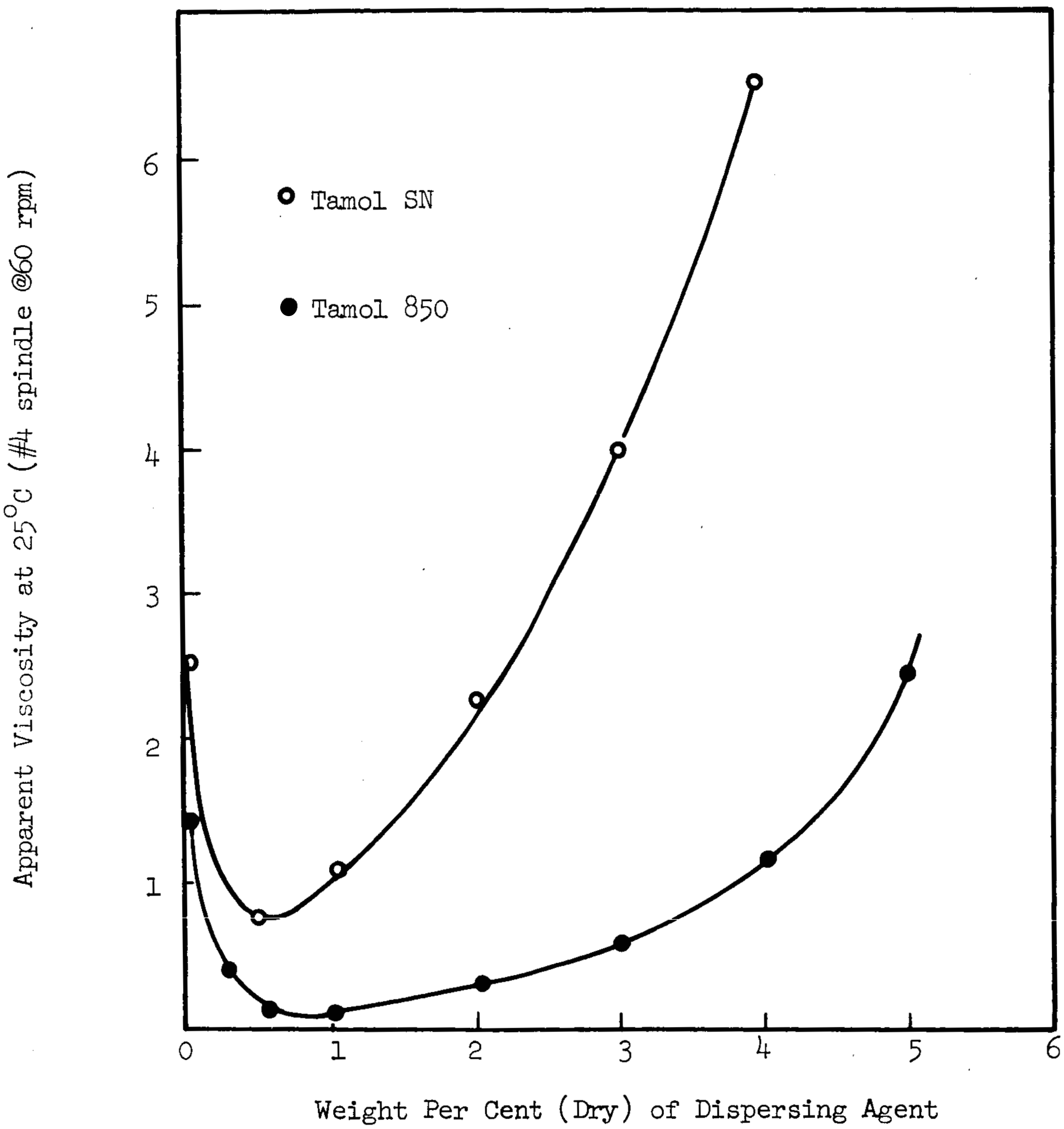

Figure 8. Apparent Viscosities for Lead Oxide Slurries Containing 80 Weight Per Cent Solids and Varying Amounts of Dispersing Agent.

dispersant concentration is reported as the percentage of dry dispersant based on 100 grams of dry slurry solids. Slurries with low apparent viscosities resulted for Tamol 850 concentrations from about one-half to two per cent. Although one-half per cent dispersing agent produced the maximum degree of fluidity, a concentration of one per cent was chosen as the optimum percentage of dispersant for lead oxide slurries containing up to 90 weight per cent solids; primarily because the ease of water washability was significantly improved with only a slight increase in slurry viscosity.

Excellent flow properties and minimal solids settling were obtained when a lead oxide slurry with the particle size distribution shown by Figure 6 was prepared using one per cent Tamol 850 and subsequently thickened to about 1150 cps with Benagel EW. The thixotropic nature of this slurry before addition of Benagel EW is shown by Figure 9. The apparent viscosity is seen to decrease with increasing shear rate for each of the two spindle sizes shown.

G. Toxicology

Lead oxide is the only one of the materials tested that poses any significant toxicity hazard. Lead oxide is classified as a semi-toxic material; however, there appears to be little hazard involved in its use unless the material is ingested or inhaled in significant quantities. According to the National Lead Company, the only precaution taken during its manufacture is the use of respirators by workers handling bulk quantities of the material.

H. Economic Considerations

The raw-material costs for various solid materials from which slurries might be prepared are shown in Figure 10. Careful consideration must be given to the minimum specific gravity which would accomplish the desired results.

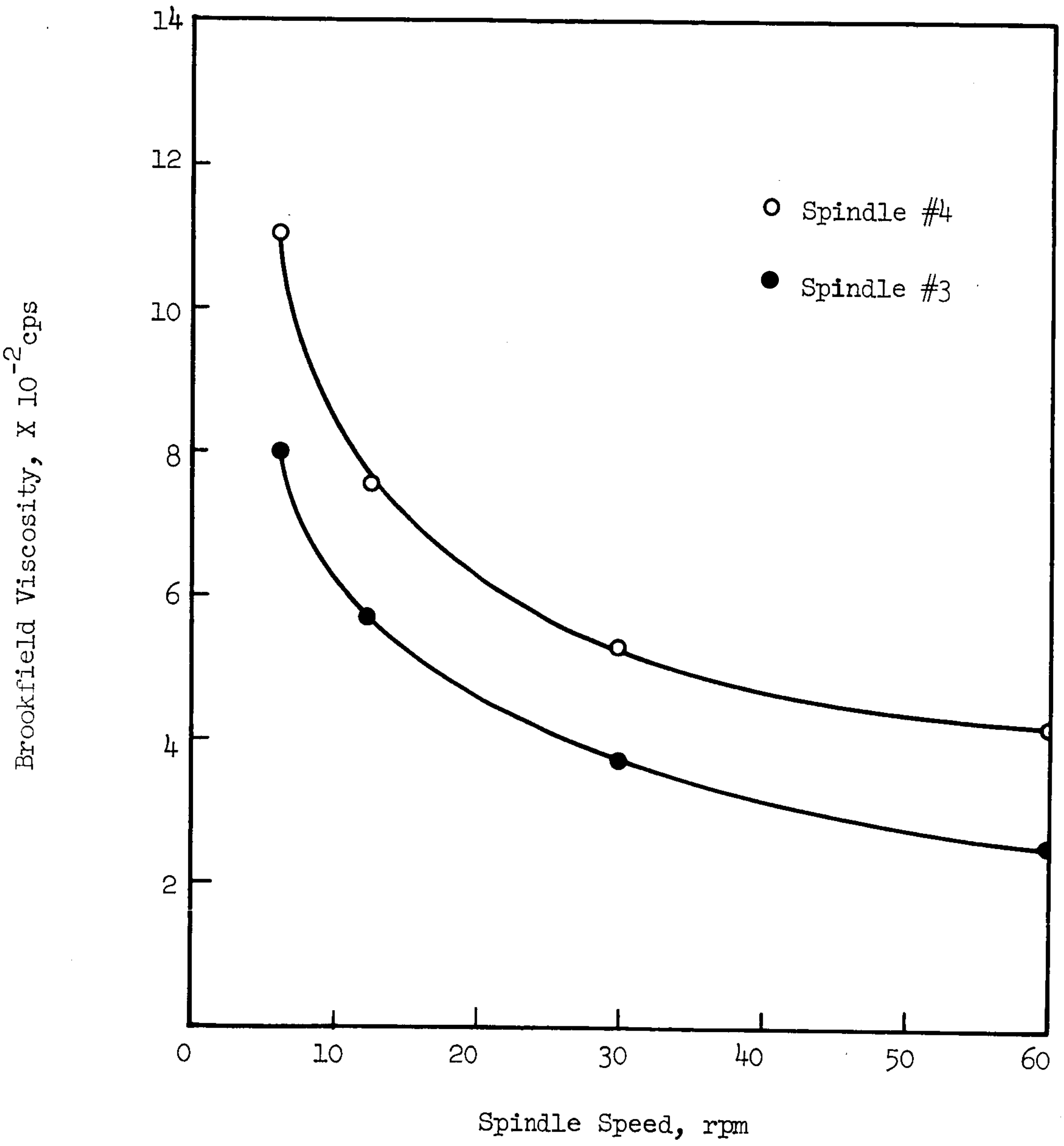

Figure 9. Variation of Apparent Viscosities for a Lead Oxide Slurry (Sp. Gr. = 4.0) with Shear Rates.

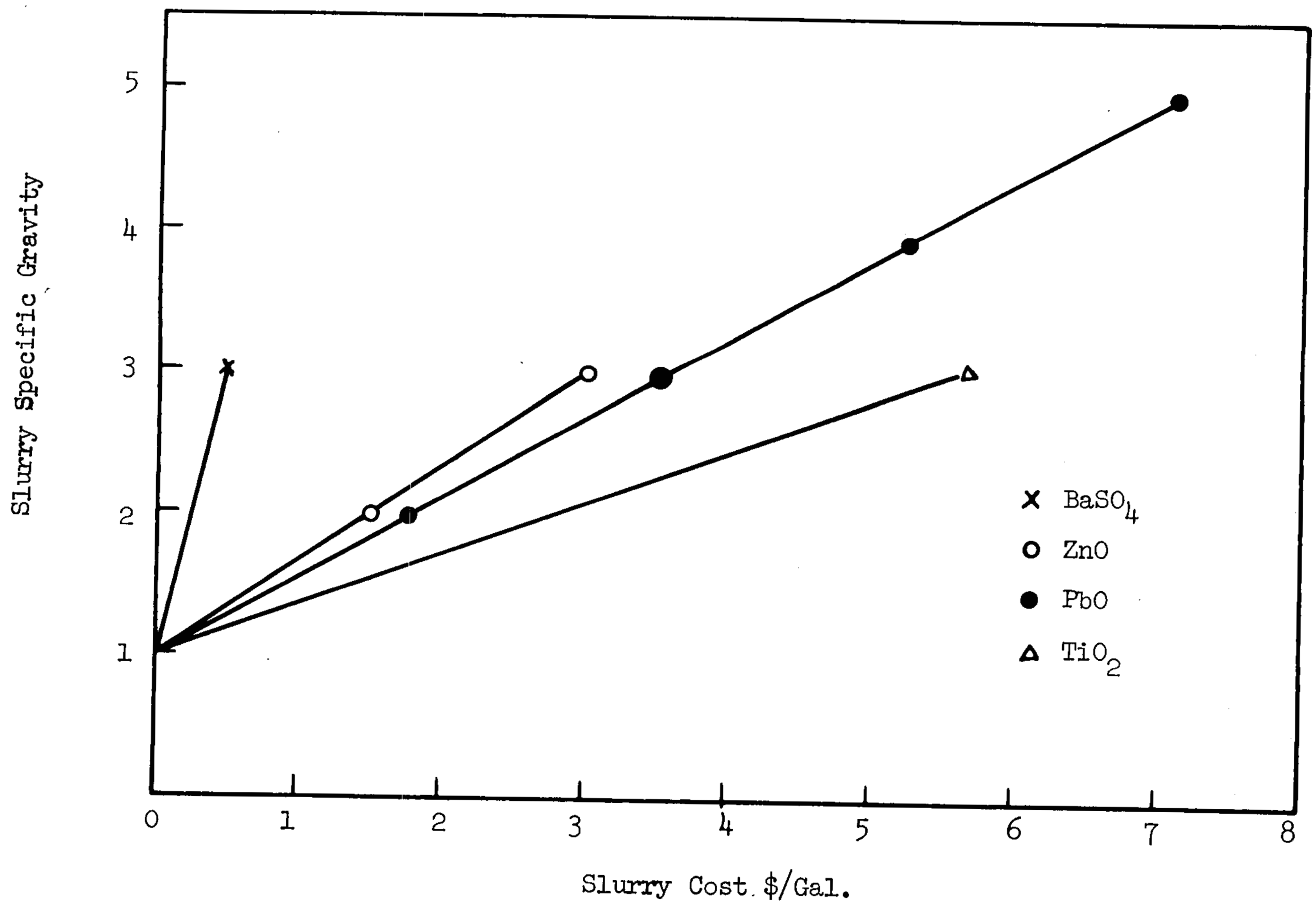

Figure 10. Approximate Raw Materials Costs for Water-Base Slurries.

For example, if a specific gravity of 3.0 would permit a reasonable approach to the desired goal, the economic advantages afforded by a barium sulfate slurry are considerable. However, if higher specific gravities are required, lead oxide is recommended for the slurry solids.

The usual mark-up for the pigment dispersions above materials cost as quoted by commercial suppliers ranges from 50 to 100 per cent, depending upon the quantity sold. Assuming that this mark-up could be reduced to 25 per cent for quantities of approximately one million gallons, the quoted price for a lead oxide slurry with a specific gravity of 4.0 would be about $6.60 per gallon. Purchase of one million gallons at this price would require an expenditure of about $1.3 million above raw materials costs. Quoted prices from commercial suppliers are generally F.O.B. their plant, so the additional expense of shipping and possible demurrage charges must be added to this figure. Shipment of high density materials also poses unique problems. Conventional railway tank cars have a capacity of 10,000 gallons but a gross weight limitation of about 140,000 lbs. Thus, a tank car could carry only 4200 gallons of a slurry with a specific gravity of 4.0. Sloshing of 4200 gallons of such a high-density material in a 10,000 gallon enclosure could have unfortunate consequences. Also the problem of receiving and unloading some 240 tank cars within a short period of time is a formidable task. If the slurries are purchased from a commercial supplier, some arrangement should be negotiated with the supplier to repurchase and dispose of the slurry when it is no longer needed. One commercial supplier of slurries was contacted and quoted a price of $8.25 per gallon, F.O.B. their plant, for lead oxide slurry with a specific gravity of 4.0 in million-gallon quantities. This supplier indicated that the material would be repurchased for $1.00 per gallon, F.O.B.

their plant, after use.

Purchase of slurries from commercial suppliers would, of course, preclude any appreciable capital investment or the necessity for acquiring and training new personnel in rather specialized skills. However, the added flexibility afforded by on-site manufacture and the very considerable economic advantages recommend the construction of an on-site processing plant for producing the required quantity of high density slurry.

Current prices and delivery information are listed in Table V for the materials most likely to be used in the preparation of high density slurry formulations.

TABLE V

PRICE AND DELIVERY INFORMATION FOR MATERIALS USED IN SLURRY FORMULATIONS

Material	Price per Pound
Titanium Dioxide (Anatase grade)(3)	$0.2500(1)
Zinc Oxide (Kadox 72)(3)	$0.1525(1)
Zinc Oxide (XX503 and XX504)(3)	$0.1475(1)
Lead Oxide (Fumed Litharge)(4)	$0.1835(2)
Barium Sulphate (No. 22 Barytes)(5)	$0.0015(2)
Barium Sulphate (Barimate XF)(5)	$0.0030(2)
Tamol 850 (30% solution)(6)	$0.1950(1)
Ben-A-Gel EW(4)	$0.8600(2)

(1) Price quoted is F.O.B. shipping point with minimum freight paid or allowed.

(2) Price quoted is F.O.B. manufacturing point.

(3) As supplied by New Jersey Zinc Company.

(4) As supplied by National Lead Company.

(5) As supplied by Thompson, Weinman and Company

(6) Supplied by Rohm & Haas Company

IV. SUMMARY AND CONCLUSIONS

The purpose of this study was to develop high-density slurries with specific gravities from two to about six that would be suitable for use as pressure transmitting media in the hydrostatic testing of stage, propellant tanks. Slurries meeting specific gravity requirements were to be thoroughly tested and characterized, and recommendations were to be made for suitable types of equipment for the preparation, pumping, and storage of the slurries.

Water-based slurries were formulated from a large number of materials, and it was conclusively shown that specific gravities from 2.0 to 6.0 could be achieved with conventional chemical processing equipment. Lead oxide was shown to be the most suitable of the solids tested for producing stable slurries with a wide range of specific gravities. The necessary particle size distribution, the optimum type and amount of dispersing agent, and a suitable preparation procedure are therefore specified for producing lead oxide slurries with specific gravities to 4.5.

A preliminary economic analysis indicated that barium sulfate slurries are highly desirable for specific gravities to about 3.0. Lead oxide slurries are recommended for specific gravities above 3.0. The economics of purchasing the required quantities of the slurries from commercial suppliers were compared with those of building an on-site processing plant for producing the slurries. This preliminary analysis favors the construction of an on-site facility. However, a comprehensive pilot-plant study is strongly recommended before construction of a plant is undertaken for full-scale production. Pilot-plant studies are needed to determine the best method of production and storage, the suitability of specific types of equipment, and materials of construction.

Additional studies are also needed to improve the corrosion characteristics exhibited by the slurries when in contact with aluminum alloys. A comprehensive prototype testing program utilizing the principles of similarity is also strongly recommended. Such a program would test exhaustively the pressure transmitting characteristics of high-density slurries under conditions dynamically similar to those of actual operation.

V. RECOMMENDATIONS FOR FUTURE WORK

Now that potentially feasible slurries have been developed, a prototype testing program is next recommended to determine the feasibility of hydrostatic testing with high-density slurries under conditions dynamically similar to actual operational ones. Bench-scale, laboratory tests support the feasibility of such a testing procedure; however, a more complete analysis, particularly at high pressures, is desirable before a full-scale test program is begun.

Several small-scale models of stage propellant tanks should be tested under conditions as similar as possible to those at maximum operational stress levels. These models should be fabricated from the same materials as the full-scale tanks and instrumented with sensitive, pressure-measuring transducers and strain gages. The strain and pressure profiles should be continuously monitored while the tank was under static loads. These strain data would be particularly useful in determining if metal creep might present problems under long-term static loads, i.e., several hundred hours, at or near the yield strength of the alloys used. Studies might also include the effects of vibration on the induced stresses and strains. Controlled vibrational energy could be transmitted to the prototype propellant tanks from external sources such as high-frequency oscillators or mechanical vibrators and the increased stresses and strains monitored continuously.

The high-density slurries developed in the present study should also be examined periodically for possible de-stabiliziation or stratification with formation of a density gradient. A prototype testing study such as the one proposed would best be performed by a laboratory group with a strong background in instrumentation and physical testing and a capability for analyzing stress response in various types of Non-Newtonian fluids. A nine-month to one-year effort at a cost of about $40,000 is estimated to be sufficient for the recommended study.

The unfavorable corrosion tendencies of high pH slurries in contact with aluminum alloys remains a possible disadvantage to their use as proof-testing media. Preliminary attempts to produce suitable slurries with a neutral pH have been unsatisfactory. The production of highly-loaded, non-settling slurries with a neutral pH seems unlikely since the large zeta-potentials needed for slurry stabilization are generally associated with either a high or a low pH. Studies to date have shown that the corrosion problem is not so much an attack on the aluminum alloys as it is a decomposition of the slurry solids at the slurry-alloy interface. Corrosion inhibitors added directly to the slurry may alleviate this problem, or direct metal contact may be prevented by applying a very thin film of a protective material, such as a silicone oil, to the tank walls before addition of the slurry. A simultaneous investigation of the basic corrosion mechanism and an engineering approach to determine control methods should do much to solve this problem. Additional corrosion studies are thus recommended to solve the aforementioned problems and to determine the suitability of materials of construction for possible use in slurry production facilities.

A laboratory that is equipped with instruments such as a research metallograph, potentiostats, an electron microscope, an electron microprobe, and X-ray, electron and neutron diffraction facilities is highly desirable for study of complex corrosion problems such as encountered in this work. A well-equipped laboratory and a research staff with a strong background in physical metallury and corrosion mechanisms is essential in the performance of this study. A one year effort at a cost of about $25,000 is believed to be a reasonable estimate for this phase of the recommended program.

Ordinary scale-up methods assume that the effectiveness of agitation will remain approximately the same if the same power per unit volume of fluid is supplied to the impeller. This, in effect, assumes dynamic similarity from one system to the other. Thus the design of full-scale plant equipment for producing slurries in large quantities requires pilot-plant or laboratory experience for the agitation part of this problem with equipment which would be geometrically and dynamically similar to that of any resulting installation. Large agitator power requirements will be necessary for batch production of slurries in quantities as large as 5,000 gallons. This suggests that additional studies should be made to test the feasibility of using a continuous production process rather than batch methods. If the required degree of shear and viscosity control can be achieved with moderate-power, high-output continuous agitators, much higher production rates with lower capital expenditures could be realized. There are several on-line, commercial mixing units available which might be modified to perform this task. If, however, batch agitators are shown to afford the most practical design, additional information such as optimum liquid depth, tank diameter, type and number of baffles and the feasibility of using steam-jacketed tanks should be obtained from pilot-plant studies. Although it is not possible at the present to determine with any degree of accuracy the scaled-up power requirements to accomplish the desired result, an approximation may be made from the following empirical relationship:[2]

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(2) J. H. Perry, Chemical Engineers Handbook, 3rd Edition, p. 224, (1950).

$$\text{Horsepower/cubic foot} = \frac{(\text{Specific Gravity})(10+\mu^{1/4})}{320}$$

where μ is the slurry viscosity in centipoise. For a slurry with a specific gravity of 4.0 and a maximum viscosity of 10,000 cps, the requirement for a 5,000 gallon tank would be about 170 H.P.

Storage of the slurry would probably best be accomplished in large-diameter, agitated tanks with a low length-to-diameter ratio. Tank height should be kept low to avoid large hydrostatic heads but, even so, thick walls would be required to support the large stresses. Consideration should also be given to providing a continuous rotary vacuum filter and a tunnel dryer, or perhaps a spray drier, so that all, or at least a part, of the slurry solids could be stored as a dry powder. Storage of part of the total slurry solids as a dry powder and the remainder as a slurry would permit more flexibility in the range of specific gravities readily attainable. If, for example, it were desired to increase the specific gravity of a slurry from 3.0 to 4.0, this would be much more easily accomplished by adding dried solids to a low-viscosity slurry already at a specific gravity of 3.0 than by having to re-wet and re-disperse the entire quantity of solids.

A small, laboratory-size centrifugal pump was shown in the present investigation to be effective for pumping lead oxide slurries with specific gravities up to 4.0. The high flow rate and high pressure requirements imposed by full-scale processing, however, require further study at the pilot-plant level before firm pump recommendations can be made. The additional shearing action of high speed centrifugal pumps may also decrease the agitation requirements. Pilot plant studies are the best way to determine interrelated requirements such as these. Diaphragm and screw-type pumps are also quite effective in handling slurries, but they are limited to rather low flow rates and relatively small heads. Preliminary results suggest the glandless-type centrifugal slurry pumps to be the most likely choice. Special design modifications to conventional slurry pumps may be required, however, because of the high head and flow rate requirements. The maximum head that can be generated practically with single-impeller, turbine pumps is about 250 to 300 feet of water. For higher heads, two or more impellers are generally placed in series. Heads to 1000 feet may be generated with multi-stage turbine pumps.

Plug-type valves with teflon liners are recommended for use in slurry handling systems. Other types of valves are more susceptible to blockage and fouling and are rarely satisfactory when handling suspended solids.

Proper choice of materials of construction may make possible a considerable reduction of capital expenditures. For example, if mild steel instead of stainless steel is suitable a large savings would be realized. Since lead pigments are widely used as corrosion inhibitors for steel, it is likely that mild steel may be acceptable. Only further corrosion studies with typical materials of construction will provide these answers.

A number of equipment manufacturers conduct pilot plant studies for equipment scale-up and process optimization. The recommended pilot plant study could be performed by an equipment manufacturer or a more impartial study might be accomplished by a non-profit, university sponsored research group. A pilot-plant study conducted by a non-profit group such as a School of Chemical Engineering or an Engineering Experiment Station could probably be completed in nine to 18 months at a cost of from $50,000 to $70,000, depending upon the amount of equipment that would have to be purchased and the availability of trained research personnel. A comparable study by a commercial manufacturer would probably cost 50 to 100 per cent more but could likely be completed in less time.

A comprehensive research program in which all three of the recommended study areas are performed simultaneouly could be accomplished more economically since there would be less duplication of equipment and effort and a better coordinated program should result. A combined program could probably be completed in 12 to 18 months at a cost of about $90,000 to $100,000.

Respectfully submitted:

John H. Burson, III

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Approved:

Frederick Bellinger

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