LUOX® Colloidal Silica
Ceramic Shell Investment Casting

Table of Contents
Introduction 1
The Nature of LUOX® Colloidal Silica
The Function of LUOX® Colloidal Silica in Investment Casting
Advantages of LUOX® Colloidal Silica
LUOX® Colloidal Silica Grades for Investment Casting
Slurry Preparation 4/5
Maintenance of LUOX® SM or HS Slurries 6
Building Slurries with LUOX® SK 7
Zircon Slurries
Silica and Aluminosilicate Slurries
Alumina Slurries 8
Soluble Polymer vs. Latex Dispersions
Slurry Rheology
Slurry Maintenance 8
Dipping Procedures
Dewaxing 9
Shell Firing
Casting in the Ceramic Shell
Personal Safety and First Aid
Storage and Handling 10
Packages
Additional Information
Appendix 11
Slurry Testing of Difficult-to-Separate Slurries
Troubleshooting Guide 12
Advantages 14
Introduction

LUDOX® colloidal silica binders have been used in the investment casting industry for many years. Various researchers have done work to define the optimum use of LUDOX® colloidal silica products for the industry. The recommendations given in this booklet are based on that work.

The procedures described for slurry preparation and maintenance are our opinions of “best practices.” These “best practices” are based on the consensus thinking absorbed during many foundry visits, Investment Casting Institute training sessions, meetings, and conversations with investment casters.

The Nature of LUDOX® Colloidal Silica

LUDOX® colloidal silica is a water dispersion of extremely fine, spherical silica particles that can form bonds with all the refractory oxides used in the investment casting process and with each other. The fine particle refractories used in the industry are all made by grinding larger particles down to smaller particles. LUDOX® colloidal silica particles are grown from individual silica molecules in a polymerization process. The process used for polymerization allows us to carefully control both particle size and distribution. The particles in LUDOX® colloidal silica are very small. It would take about a million LUDOX® SM particles to equal the weight of one 325 mesh-fused silica grain. The particle size distribution of LUDOX® colloidal silica is very narrow when compared to most other manufacturer’s grades with the same average particle size. The uniformity of particle size and distribution demonstrates the superior process control that goes into LUDOX® colloidal silica manufacture and provides our customers with enhanced uniformity and control of their processes.

LUDOX® colloidal silica contains amorphous (noncrystalline) silica like the fused silica often used as a refractory in the industry. Because of this, it has a very low coefficient of expansion (like fused silica). Unlike silicate solutions that are thick, viscous liquids, LUDOX® colloidal silica is almost waterlike in its viscosity. Yet, most colloidal silica grades contain at least 25% (by weight) silica.

The particles in LUDOX® colloidal silica are kept from agglomerating or settling out because the surface of each particle is electrically charged. All of the grades discussed here have a negative charge. The particles repel each other and stay separated. The amount of the charge on most types of LUDOX® colloidal silica depends on the pH. Alkaline pH of about 9–10 is required to keep LUDOX® SM and HS-30 colloidal silica stable.

| Table 1: Typical Properties of LUDOX® Colloidal Silica for Investment Casting |
|-----------------------------------------------|----------------|------------|-----------|
| Stabilizing Counter Ion                       | SK Family   | HS-30     | SM        |
| Stabilizing Counter Ion                       | Sodium      | Sodium    | Sodium    |
| Particle Charge                               | Negative    | Negative  | Negative  |
| Avg. Particle Diameter (nm)                   | 12          | 12        | 7         |
| Specific Surface Area (m²/g)                  | 230         | 230       | 345       |
| Silica (as SiO₂) wt%                          | 25          | 30        | 30        |
| pH at 25°C (77°F)                             | 4-7         | 9.8       | 10        |
| Titratable Alkali (as Na₂O) wt%               | *           | 0.32      | 0.56      |

* Deionized sol contains about 0.05% Na₂O (occluded inside the particles).

The stability of LUDOX® SK colloidal silica, which is described in more detail below, is independent of pH.

The Function of LUDOX® Colloidal Silica in Investment Casting

LUDOX® colloidal silica serves two functions in the investment casting process. Because of its low viscosity, waterlike character, it makes an excellent vehicle in which to suspend refractory oxides with which the ceramic shell is built. This low viscosity allows high loadings of refractory to be suspended in LUDOX® colloidal silica before useful slurry viscosity is exceeded. Upon evaporation of the water, the particles bond to the ceramic particles and to each other and provide the “glue” that holds the shell together. Once formed, this bond is waterproof and the shell can be redipped. The bond is maintained and even improved when the shells are fired prior to being filled with metal.

Advantages of LUDOX® Colloidal Silica

Three types of binders are currently used in investment casting: colloidal silicas, ethyl silicate, and hybrid binders containing both. All give good bonding and refractory properties, but LUDOX® colloidal silicas are increasingly preferred because they:

- Are waterbased, present no flammability hazard, have a low order of toxicity, and are, therefore, more environmentally acceptable.
- Give longer slurry life, more consistent properties, and require less slurry maintenance. LUDOX® SK colloidal silica is especially stable and needs less slurry maintenance than alkaline slurries.
- Are less expensive.
LUDOX® Colloidal Silica Grades for Investment Casting

Grace offers many grades with a range of particle sizes and concentrations. Two of these have been widely used in the investment casting industry for years.

LUDOX® HS-30 colloidal silica

Provides ease of formulation and excellent fired strength.

LUDOX® SM colloidal silica

Gives greater economy when properly diluted with water. Because of its small particle size, maximum green strength is obtained using SM diluted to about 22% silica.

Both of these binders require routine pH maintenance in order to remain stable when used as a slurry binder. Typically, alkali must be added periodically in the form of sodium, potassium, or ammonium hydroxide in order to maintain the pH level above about 9.3.

LUDOX® SK colloidal silica

Is a family of ultrastable binders designed for investment casting. SK binders are formulated products that contain water-soluble polymers based on patented technology. This technology encompasses making a stable colloidal silica without alkali and combining it with water-soluble polymers to give a binder with excellent stability and strength. All SK binders are supplied ready to use, without the need for additives.

Currently four LUDOX® SK binders are available. They are LUDOX® SK, SK-B, SK-F, and SK-R colloidal silica. LUDOX® SK and SK-B colloidal silica are similar, except that SK-B is formulated without surfactant and is intended as a backup binder. SK and SK-B were the original offerings in the SK family.

SK-F and SK-R are similar to each other, but SK-R contains no surfactant and is intended as a backup binder. These newer, improved products differ from SK and SK-B by virtue of the differences in the water-soluble polymers they contain. The polymers in SK-F and SK-R give somewhat higher green and fired strength than SK and SK-B and also appear to set somewhat more rapidly than the original offerings.

In the manufacture of LUDOX® SK colloidal silica, the surface of each particle is modified so that they take on a negative charge independent of the pH. The sodium is removed by deionization. This modified and deionized colloidal silica is combined with water-soluble polymer for enhanced green strength and film-forming ability. LUDOX® SK and SK-F colloidal silica are formulated as face coat binders and contain a wetting agent and an antifoam and are somewhat turbid due to the presence of the surfactant.

Backup binder versions formulated without surfactant are labeled LUDOX® SK-B and SK-R colloidal silica. These grades are quite clear compared to SK and SK-F colloidal silica.

Slurries made with LUDOX® SK colloidal silica require no pH maintenance to remain stable. It is merely necessary to replace water lost by evaporation. Distilled or deionized water is preferred, and very hard water should be avoided because the accumulation of calcium or magnesium ions will destabilize all grades of LUDOX® colloidal silica.

The typical pH of LUDOX® SK colloidal silica is 4–7. Most colloidal silicas would gel rapidly in this pH range, but the colloidal silica in SK has been modified to be stable across a wide pH range. Therefore, it has been possible to eliminate alkaline stabilizers from the system and pH adjustments are not necessary.

WARNING! LUDOX® SK products should not be mixed with standard colloidal silicas.

Low Sodium in LUDOX® SK colloidal silica

The elimination of sodium from LUDOX® SK colloidal silica results in its having less tendency to soften and flow at high temperatures, and this may result in less high temperature shell deformation. The low sodium content also decreases the rate of conversion to crystalline silica.

The stability of LUDOX® SK slurries extends beyond the mere resistance to gelation. SK slurries that are maintained at constant viscosity by water replacement exhibit:

- Constant plate weight over time.
- Constant binder silica content.
- Fast strength development — shorter times between dips.
The consistency of the plate weight characteristics yields more consistent shells and provides better control over shell weight.

The constant binder silica is indicative of the excellent stability built into the particles. This results in long slurry life and very reproducible results.

SK gains strength during the drying process faster than conventional binders. Although shells do not dry faster, the polymer in SK contributes to strength development during the earlier stages of drying. The result is that one can redip shells on a shorter time schedule, speeding up production rates. Figure 1 illustrates the difference in the rate of strength build.

In these experiments, the dips were made every one or two hours and compared with a control sequence in which each dip was allowed to fully dry before being redipped. The test specimens were all allowed to dry 24 hours after the last dip. Weight measurements made in a similar experiment indicated that the slurry pickup and water loss rates were about the same for all slurries.

High Shear Mixers

High shear mixers can be used as described above for the initial dispersion of the refractory powder. High shear mixing will significantly decrease the time for dispersion and wet-in. After the initial wet-in period, agitation should be reduced to normal levels to allow air to be eliminated. It may take 24 hours to eliminate all the entrapped air. Be sure to watch for excessive heating during the high shear phase of the mixing.

Jar Mixing

One excellent technique that ensures virtually no change in slurry makeup due to evaporation or overheating is to combine the components in sealed jars and roll them in order to accomplish the dispersion and wet-in. During the preparation, no monitoring of the slurry will be necessary. As with normal mixing in a tank, 24–48 hours should be sufficient to produce a stable, usable slurry that can be transferred as needed to the dipping pots.

Optimum Silica Concentration

Extensive laboratory work has been done to determine the correct ratio of binder to grain for optimum shell strength. R. L. Rusher reported his work, “Strength Factors Influencing Ceramic Shell Molds,” in the Cast Metals Research Journal, Vol.10, No. 4 and Vol. 11, No. 1. Figure 2 is based on Rusher’s findings. “Factors Affecting Shell Strength,” presented by W. O. Roberts at the 25th Annual Meeting of the Investment Casting Institute, presents an approach somewhat simpler to implement in actual practice. Figure 3 is based on Roberts’ work. Reprints of these articles are available from Grace upon request.

Slurry Preparation

- Add the colloidal silica and water to the mix tank.
- Slowly add the refractory grain and agitate well. The slurry viscosity will at first be high but will fall as the binder wets out the refractory grain and air is eliminated from the slurry.
- After all the grain has been added, continue to agitate the slurry until all the air has been eliminated and the viscosity has stabilized. This will probably take 24–48 hours.
- Adjust the slurry to final viscosity by adding grain or LUDOX® colloidal silica-water mixture as needed. It is best to make the initial formulation so that the viscosity comes out high, requiring the addition of the LUDOX®-colloidal silica water mix, to avoid air being introduced when adding more grain. When the slurry is stable, it may be added to the dip tanks as needed.

- Add the colloidal silica and water to the mix tank.
- Slowly add the refractory grain and agitate well. The slurry viscosity will at first be high but will fall as the binder wets out the refractory grain and air is eliminated from the slurry.
- After all the grain has been added, continue to agitate the slurry until all the air has been eliminated and the viscosity has stabilized. This will probably take 24–48 hours.
- Adjust the slurry to final viscosity by adding grain or LUDOX® colloidal silica-water mixture as needed. It is best to make the initial formulation so that the viscosity comes out high, requiring the addition of the LUDOX®-colloidal silica water mix, to avoid air being introduced when adding more grain. When the slurry is stable, it may be added to the dip tanks as needed.

- Add the colloidal silica and water to the mix tank.
- Slowly add the refractory grain and agitate well. The slurry viscosity will at first be high but will fall as the binder wets out the refractory grain and air is eliminated from the slurry.
- After all the grain has been added, continue to agitate the slurry until all the air has been eliminated and the viscosity has stabilized. This will probably take 24–48 hours.
- Adjust the slurry to final viscosity by adding grain or LUDOX® colloidal silica-water mixture as needed. It is best to make the initial formulation so that the viscosity comes out high, requiring the addition of the LUDOX®-colloidal silica water mix, to avoid air being introduced when adding more grain. When the slurry is stable, it may be added to the dip tanks as needed.

- Add the colloidal silica and water to the mix tank.
- Slowly add the refractory grain and agitate well. The slurry viscosity will at first be high but will fall as the binder wets out the refractory grain and air is eliminated from the slurry.
- After all the grain has been added, continue to agitate the slurry until all the air has been eliminated and the viscosity has stabilized. This will probably take 24–48 hours.
- Adjust the slurry to final viscosity by adding grain or LUDOX® colloidal silica-water mixture as needed. It is best to make the initial formulation so that the viscosity comes out high, requiring the addition of the LUDOX®-colloidal silica water mix, to avoid air being introduced when adding more grain. When the slurry is stable, it may be added to the dip tanks as needed.
On the basis of these investigations, it has been shown that the optimum silica concentration required to achieve the best possible shell strength is dependent on the colloidal silica particle size. The smaller the particles, the lower the optimum concentration.

At a particle size of 12 nanometers, the binder concentration needed to optimize green strength is about 30% silica. LUDOX® HS colloidal silica, which has this particle size, is packaged at both 30 and 40% silica concentration. The optimized silica concentration of LUDOX® HS-30 colloidal silica allows it to be used directly without dilution. LUDOX® HS-40 colloidal silica diluted to 30% silica would work equally well.

LUDOX® SM colloidal silica has a smaller particle size and must be diluted to approximately 22% silica solids in order to optimize its bonding characteristics. LUDOX® SM colloidal silica is sold at 30%. LUDOX® SM colloidal silica can be conveniently diluted to 22% solids by combining two volumes of SM with one volume of water.

Used at these concentrations, optimum green strength will be obtained regardless of the refractory grain chosen. Therefore, in practice, it is only necessary to use either binder at its optimized silica concentration and add sufficient grain to obtain a stable working viscosity between 7 and 40 seconds (No. 5 Zahn Cup).

Table 2:
Correlation of Viscosity Readings* – Different Flow Cups

<table>
<thead>
<tr>
<th>Zahn No. 4 (Seconds)</th>
<th>Zahn No. 5 (Seconds)</th>
</tr>
</thead>
<tbody>
<tr>
<td>31</td>
<td>16</td>
</tr>
<tr>
<td>27</td>
<td>14</td>
</tr>
<tr>
<td>26</td>
<td>14</td>
</tr>
<tr>
<td>25</td>
<td>14</td>
</tr>
<tr>
<td>23</td>
<td>13</td>
</tr>
<tr>
<td>22</td>
<td>13</td>
</tr>
<tr>
<td>19</td>
<td>11</td>
</tr>
<tr>
<td>18</td>
<td>10</td>
</tr>
<tr>
<td>12</td>
<td>7</td>
</tr>
<tr>
<td>10</td>
<td>6</td>
</tr>
<tr>
<td>9</td>
<td>6</td>
</tr>
<tr>
<td>9</td>
<td>5</td>
</tr>
<tr>
<td>7</td>
<td>5</td>
</tr>
</tbody>
</table>

* Zircon/colloidal silica mixture was used to calibrate the viscometers.

Fig. 2: LUDOX® Colloidal Silica

Fig. 3: Green Strength vs. Binder Silica

The weight of refractory grain required will vary with its density. For example, it will take about twice the weight of zircon as fused silica for a given weight of binder, because the zircon is twice as dense as fused silica.
Maintenance of LUDOX® SM or HS Slurries

Monitoring Slurries
Slurries based on LUDOX® SM or HS colloidal silica should be routinely monitored for pH, viscosity, and binder solids (binder silica). Each binder has a different optimum binder solids concentration depending on its particle size. SM should be used at 22–25% and HS at about 30% binder silica solids for best results. Viscosity and pH should be monitored on a daily basis as a minimum. Binder solids should be measured at least weekly.

pH Control
Slurries produced with LUDOX® SM or HS colloidal silica require monitoring and maintaining pH in a range preferably between 9.3 and 10 in order to keep the colloidal silica stable.

pH Measurements
When measuring pH, we recommend using a pH meter instead of pH test papers. pH test paper strips are only accurate to about 0.5 pH units and, therefore, do not give good pH control.

The pH meter should be standardized using standard buffer solutions according to the manufacturer’s directions prior to each measurement or series of measurements. Two buffers, such as 7 and 10 or 7 and 9, should be used to standardize the meter, not just one.

pH Adjustments
pH adjustments are best made with ammonium hydroxide. Ammonium hydroxide evaporates with the water and will not change the chemistry of the binder. Adding alkaline hydroxides such as sodium or potassium hydroxide will change the binder chemistry and are difficult to add without causing local gelation of colloidal silica. This local gelation may later redisperse, but, in an opaque liquid such as a slurry, it is impossible to know for sure.

Viscosity Measurements
The most common method in the industry to measure viscosity is to use a viscosity cup such as a Zahn cup. The measurements are quick and reproducible as long as the same technique is used for each measurement. The cups are sturdy and well suited for use in the dip room. Zahn cup measurements are not good indicators of more subtle changes in rheology, however.

In plate weight measurements, a plate of known surface area and weight is dipped and drained for a predetermined time and then weighed to determine the slurry weight retained. This type of measurement will indicate changes in the flow characteristics of the slurry that may occur even though the Zahn cup reading is constant.

More sophisticated devices, such as Brookfield viscometers, measure the viscosity at different shear rates. However, these instruments are delicate and best used in a separate laboratory setting.

Binder Solids Determination
- Take a sample of the slurry and allow the refractory to settle out and separate from the binder phase. If available, a centrifuge will speed the process.
- Collect at least 10mL of binder and weigh a known volume in either a 10mL volumetric flask or a 10 ml graduated cylinder. Do this determination as close to 25°C (77°F) as possible.
- Determine the specific gravity by dividing the weight by the volume.
- Use Table 3 or Figure 4 to determine the silica content.

Addition of Water
Water continually evaporates from slurries and must be replaced to maintain slurry viscosity and to avoid the binder silica level from becoming too concentrated to give optimum performance. We recommend the use of distilled water for slurry additions. Optimally, this distilled water should have enough ammonium hydroxide added to it to raise its pH to about 9.5–10. Please note the use of pH-adjusted water does not apply to LUDOX® SK slurries.
Building Slurries with LUDOX® SK

We suggest that you use LUDOX® SK colloidal silica as received, without dilution and without pH adjustment. The product contains both a wetting agent and an antifoam, and additional amounts should not be necessary.

However, if either foam or wetting should become a problem, small additional amounts may be added. Not all wetting agents or antifoams are compatible with LUDOX® SK colloidal silica, even if they are recommended for use with other grades. You should call your distributor or Grace for help with your selection.

<table>
<thead>
<tr>
<th>Specific Gravity</th>
<th>LUDOX® SM</th>
<th>LUDOX® HS</th>
<th>LUDOX® SK</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.12</td>
<td>18.23</td>
<td>18.70</td>
<td></td>
</tr>
<tr>
<td>1.125</td>
<td>18.86</td>
<td>19.37</td>
<td></td>
</tr>
<tr>
<td>1.13</td>
<td>19.49</td>
<td>20.04</td>
<td></td>
</tr>
<tr>
<td>1.135</td>
<td>20.12</td>
<td>20.72</td>
<td></td>
</tr>
<tr>
<td>1.14</td>
<td>20.75</td>
<td>21.21</td>
<td>21.39</td>
</tr>
<tr>
<td>1.145</td>
<td>21.38</td>
<td>21.83</td>
<td>22.07</td>
</tr>
<tr>
<td>1.15</td>
<td>22.01</td>
<td>22.44</td>
<td>22.74</td>
</tr>
<tr>
<td>1.155</td>
<td>22.64</td>
<td>23.06</td>
<td>23.42</td>
</tr>
<tr>
<td>1.16</td>
<td>23.27</td>
<td>23.68</td>
<td>24.09</td>
</tr>
<tr>
<td>1.165</td>
<td>23.90</td>
<td>24.29</td>
<td>24.76</td>
</tr>
<tr>
<td>1.17</td>
<td>24.53</td>
<td>24.91</td>
<td>25.44</td>
</tr>
<tr>
<td>1.175</td>
<td>25.16</td>
<td>25.52</td>
<td>26.11</td>
</tr>
<tr>
<td>1.18</td>
<td>25.79</td>
<td>26.14</td>
<td>26.79</td>
</tr>
<tr>
<td>1.185</td>
<td>26.42</td>
<td>26.75</td>
<td>27.46</td>
</tr>
<tr>
<td>1.19</td>
<td>27.05</td>
<td>27.37</td>
<td>28.14</td>
</tr>
<tr>
<td>1.195</td>
<td>27.68</td>
<td>27.99</td>
<td>28.81</td>
</tr>
<tr>
<td>1.2</td>
<td>28.31</td>
<td>28.60</td>
<td>29.48</td>
</tr>
<tr>
<td>1.205</td>
<td>28.94</td>
<td>29.22</td>
<td>30.16</td>
</tr>
<tr>
<td>1.21</td>
<td>29.58</td>
<td>29.83</td>
<td>30.83</td>
</tr>
<tr>
<td>1.215</td>
<td>30.21</td>
<td>30.45</td>
<td>31.51</td>
</tr>
<tr>
<td>1.22</td>
<td>30.84</td>
<td>31.07</td>
<td>32.18</td>
</tr>
<tr>
<td>1.225</td>
<td>31.47</td>
<td>31.68</td>
<td>32.86</td>
</tr>
<tr>
<td>1.23</td>
<td>32.10</td>
<td>32.30</td>
<td>33.53</td>
</tr>
<tr>
<td>1.235</td>
<td>32.91</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.24</td>
<td>33.53</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.245</td>
<td>34.14</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.25</td>
<td>34.76</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 3: Silica Content vs. Specific Gravity

- The weight of refractory and binder used should be approximately the same as are currently being used.
- Add the LUDOX® SK colloidal silica to the mix tank.
- Slowly add the refractory while agitating. The slurry will initially be thick, but will thin with time as air is eliminated. High shear mixing equipment may be used to prepare SK slurries.
- Allow the slurry to equilibrate under normal agitation (about 24 hours).
- Add additional binder or refractory to make any final viscosity adjustment needed.

Note: It is best to make up the slurries at high viscosity requiring the addition of binder to thin them rather than too thin requiring more powder. The addition of powder will require more time to eliminate air.

Fig. 4: Silica Content vs. Specific Gravity

Zircon Slurries

Because LUDOX® SK colloidal silica is insensitive to pH, the rapid pH drop, often observed in zircon slurries, has no effect on slurry stability.

The very slight acidity of SK is not sufficient to solubilize impurities such as iron that can happen with highly acid hybrid or ethyl silicate binders.

Silica and Aluminosilicate Slurries

Excellent stability is also observed with silica and aluminosilicate slurries.
Alumina Slurries

Alumina flours having low (<200 ppm) boron present should be selected. Boric acid is sometimes used to wash alumina. Even moderate levels of boric acid or borates coagulate the water-soluble polymer in LUDOX® SK colloidal silica, resulting in high thixotropy or actual gelling. Alumina flours with very low levels of borates work very well, as do fused aluminas.

Soluble Polymer versus Latex Dispersions

The polymeric film former in LUDOX® SK colloidal silica is more efficient as a binder because of its molecular size, which more closely matches colloidal silica dimensions than the much larger latex particles commonly added to slurries. Because it is a soluble polymer, it is not as prone to mechanical destabilization as are latices. *High shear mixers may be used to prepare LUDOX® SK slurries.*

Where high shear mixers are used, we recommend that LUDOX® SK-B colloidal silica be used and surfactant added later, as needed, in order to minimize the time required to allow for air to escape from the slurry. Because SK-B does not contain a surfactant, it will release air faster and foam much less readily than SK. Please note that not all surfactants are suitable and we suggest you consult your distributor or Grace for the proper choice.

LUDOX® SK slurries have excellent stability. One laboratory slurry was operated continuously for well over a year. Makeup additions were made from time to time, but no pH adjustments were ever made.

Slurry Rheology

Rheology-related characteristics such as flow and plate weight remain surprisingly constant with age in LUDOX® SK slurries. However, the plate weight for a given Zahn cup reading may be different from that found when using alkaline colloidal binders. *Some adjustment of the working viscosity of the SK slurry may be needed to give plate weights similar to what you are used to.*

Slurry Maintenance

- Routinely add water (preferably distilled) to compensate for evaporation.
- At least once a week, check the silica content by separating the binder phase from the refractory by settling or centrifuging. Determine the specific gravity of the SK binder and use Table 3 or Figure 4 to determine the silica content. *Note: LUDOX® SK slurries are more difficult to separate than standard alkaline slurries. This is due to the better suspending capabilities and lower pH of the SK binder. A modified test method for silica determination in hard to separate slurries can be found in the appendix.*
- Maintaining the SiO2 content of the binder in the 24–26% range is preferred, but slightly higher contents (up to 28%) would be expected to operate satisfactorily.
- Do not make pH adjustments.
- Additions of extra wetting agents or antifoams should be made using only those recommended by Grace for use with LUDOX® SK colloidal silica.

Dipping Procedures

The exact sequence varies among foundries. *Table 4 shows a sequence of increasing grain size as the shell is built. For the prime coat, the pattern assembly is dipped into the slurry being certain that all surfaces are evenly coated. Excess material is drained back into the mixing tank. The operator should make sure the coating does not fold or sag; this may cause surface imperfections in the final casting.*

Table 4

<table>
<thead>
<tr>
<th>Cost</th>
<th>Grain Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prime Slurry Grain</td>
<td>200 - 325 mesh</td>
</tr>
<tr>
<td>Prime Stucco Grain</td>
<td>50 - 100 mesh</td>
</tr>
<tr>
<td>Backup Slurry Grain</td>
<td>120 - 200 mesh</td>
</tr>
<tr>
<td>Backup Stucco Grain</td>
<td>20 - 50 mesh</td>
</tr>
</tbody>
</table>
The wet pattern is then stuccoed with refractory grain. The particle size of the stuccoing grain is generally 50–100 mesh on the prime (inner) coats and 20–50 mesh on the backup coats.

After stuccoing, the coated pattern is allowed to dry at 24–27 °C (75–80 °F) and 40–60% relative humidity. If the humidity is too low, the rapid drying may cause cracking of the shell; if too high, the drying rate may be uneconomically long.

Subsequent coats are applied by dipping in the slurry, stuccoing, and drying. Four to nine coats are generally required, depending upon the casting size and metal pressures to be encountered. The last coat normally is not stuccoed. After the final coat, the shell is dried until the final moisture content is 2% or less. This final drying takes about 24 hours depending on the shape and number of coats; therefore, overnight drying or “curing” is normally recommended before dewaxing.

**Dewaxing**

After the shell is dried, the wax pattern is removed. This is usually accomplished by rapid heating in a steam autoclave at 550–830 kPa (80–120 psi), so that the outer surface of the wax is liquefied and drained first. If the pattern is not rapidly heated in this manner, the bulk of the wax will expand and crack the shell.

Recommended autoclaving guidelines are:

- Steam pressures of 550–830 kPa (80–120 psi) or higher should be used. Lower pressures may cause shell cracking.

- The autoclave should be pressurized as rapidly as possible, preferably in 15 seconds or less. Slow heat transfer to the wax pattern will cause the shell to crack.

- After the autoclaving cycle is complete, the pressure should be released slowly (30–60 seconds). Delamination of the outer layers of the shell may result if the pressure is released too rapidly.

**Shell Firing**

The shell is fired in an oxidizing atmosphere at 980 – 1100 °C (1800–2000 °F) for about 30 minutes to 1 hour to burn out the remaining wax and any carbon residue, while bringing the shell to the casting temperature. This is normally done just prior to casting and the shell is maintained hot until ready to pour.

**Casting in the Ceramic Shell**

The temperature of the shell must be 870–980 °C (1600–1800 °F) when the molten metal is poured into it to prevent the metal from solidifying too rapidly. This also ensures the absence of moisture. The shell, if large, can be supported by packing in warm, dry sand or metal shot.

When the metal has solidified, the shell is removed using an air hammer. The finished castings are then removed from the gates with abrasive cutting wheels. These parts are usually cleaned or finished by grinding, sandblasting, or caustic leaching at temperatures up to 540 °C (1000 °F), or a combination of these.

**Personal Safety and First Aid**

All persons handling LUDOX® colloidal silica should be thoroughly familiar with the Grace Material Safety Data Sheet.

**Health Hazards**

Contact with LUDOX® colloidal silica may cause transient irritation of the skin and eyes. Dust from spills or deposits may be considered to be amorphous silica, although the particles are normally larger than respirable size. However, when aerosols are intentionally produced, such as in spray applications, respirable droplets or dust may form that may cause irritation of the respiratory system with coughing. Studies have shown that when applied within the recommended limits, these materials are unlikely to produce health hazards, such as pulmonary fibrosis, as observed with crystalline materials. Users should review the Material Safety Data Sheet provided with each shipment for the various exposure limits.
**Safety Precautions**

Avoid contact with skin or eyes. Avoid circumstances that cause a mist or spray of the liquid or dust of dried particles.

**First Aid**

In case of skin or eye contact, flush with plenty of water for at least 15 minutes. Call a physician. If large amounts are inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

**Storage and Handling**

LUDOX® colloidal silica should be stored above 2°C (35°F) and below 43°C (110°F) to avoid irreversible gel formation or silica precipitation.

Bulk storage tanks or drum storage areas should be located in heated buildings. Lines to and from storage tanks must also be protected from freezing. Outdoor storage facilities can be used, if suitably insulated, heated, and protected from the weather.

Storage tanks of cross-linked polyethylene or fiberglass-reinforced plastic (FRP) have proven suitable. Metal tanks of 304 stainless steel or mild steel lined with resin should also be satisfactory.

Piping and fittings of 304 stainless, polyvinyl chloride (PVC) Type 1, Grade 1 or Derakane® FRP should be used. Avoid brass or copper pipe or fittings.

Self-priming centrifugal pumps constructed of stainless steel or nylon are recommended for service with LUDOX® colloidal silica. Durco® Mark II 316 stainless steel pumps have proven satisfactory. Mechanical seals are recommended, but if experienced mechanics are not available, packing should be used. A Viking® HL 724 pump run at low speeds would minimize packing problems.

Diaphragm-type pumps made of stainless steel and equipped with PTFE fluoropolymer resin parts (e.g., pumps of the Wilden Pump and Engineering Company) have given good service with LUDOX® colloidal silica.

Valves should be of the same material as the piping. System design should minimize the number of valves, although each line should be valved at both ends. This permits flow control at point of use, yet prevents a massive spill in case of a ruptured line and also permits work on a line even when the storage tank is full.

The piping system should be flushed thoroughly to remove any silica that could form dried deposits between pumpings. Spills of LUDOX® colloidal silica should be flushed with plenty of water. Watch for slipping conditions in the spill area.

To obtain assistance in designing bulk storage facilities, contact your Grace representative or your LUDOX® colloidal silica distributor.

**Packages**

LUDOX® colloidal silica is available in 55-gallon polyethylene nonreturnable drums and 275-gal bulk drums. In North America, also available in tank cars and tank trucks.

**Additional Information**

Other information available from Grace or your LUDOX® colloidal silica distributor to assist with use of LUDOX® colloidal silica for investment casting:


---

1 Ashland Licensing & Intellectual Property LLC.
2 Flowserve Management Company
3 Viking Pump Inc., Houdaille Industries
Appendix

Slurry Testing of Difficult-to-Separate Slurries
This procedure will facilitate testing of slurries that are difficult to separate. LUDOX® SK slurries tend to be more difficult to separate than standard alkaline slurries.

Summary of the Method
Weighed portions of the slurry are diluted with known amounts of water to provide additional liquid for separation from the refractory. The diluted binder is separated and the silica content determined.

One portion of the slurry is dried to determine the original water content.

Using this data, the binder silica content of the original, undiluted slurry can be calculated.

The method described below uses two dilutions, and the results of the two experiments are compared for accuracy and averaged.

It is also suggested that the specific gravity of the slurry be determined as a control parameter.

Method
After making sure that the slurry is well mixed and uniform:

1. Weigh out about 100 grams of slurry into a small pan. Record the weight as Slurry Wet Weight (SWW).
2. Dry in an oven at 60°C (140°F) – preferably overnight. Record the weight of the dried slurry as Slurry Dry Weight (SDW).
3. Calculate the fraction water:
   \[ W = 1 - \left( \frac{SDW}{SWW} \right) \]
4. Weigh out two 500 gram aliquots of the slurry into jars with tight fitting lids and add 50 grams of water to one, 100 grams of water to the other. Label A and B, respectively.
5. Shake the water-slurry mixtures until thoroughly blended and then allow to settle.
6. After the slurries have settled somewhat, remove about 15 mL of liquid from the top of A and B, and centrifuge to further separate the binder from the refractory. The goal is to get at least 10 mL of binder phase to work with.
7. Measure out about 10 mL of each binder phase from A and B, and weigh each. Record the weight and volume from each as BWA, BVA, BWB, and BVB (Binder Weight A, Binder Volume A, etc.).
8. Calculate the Specific Gravity of each as:
   \[ \text{Sp. G. (A)} = \frac{BWA}{BVA} \]
   \[ \text{Sp. G. (B)} = \frac{BWB}{BVB} \]
9. Calculate the fraction of colloidal SiO$_2$ in each as:
   \[ f_{\text{SiO}_2} = \text{Sp. G.} \cdot 1.25 - 1.2125 \]
10. If \( X \) = the grams of Binder Silica in the original 500 gram sample of slurry and \( fA \) and \( fB \) = the fractions of Binder Silica in samples A and B, respectively, then:
   \[ fA = \frac{X}{50 + 500 \cdot W + X} \]
   and
   \[ X = \frac{fA \cdot (50 + 500 \cdot W)}{1 - fA} \]
   \[ fB = \frac{X}{100 + 500 \cdot W + X} \]
   and
   \[ X = \frac{fB \cdot (100 + 500 \cdot W)}{1 - fB} \]

Note: The 50 and 100 are the amount of water added to dilute the original sample in each case.

11. The original % Binder Silica in the undiluted slurry, %SiO$_2$, can be solved for:
   \[ \%\text{SiO}_2 = \frac{100 \cdot X}{500 \cdot W + X} \]
## Troubleshooting Guide

<table>
<thead>
<tr>
<th>Problem</th>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Shell Delamination and Spalling</strong></td>
<td>Rapid vaporization of steam condensed in shell wall.</td>
<td>More complete drying of shell. Depressurize autoclave slowly.</td>
</tr>
<tr>
<td>■ Spalling of prime coat on dewaxing (“flash firing”).</td>
<td></td>
<td></td>
</tr>
<tr>
<td>■ Delamination of outer layer on autoclave dewaxing</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Shell Cracking</strong></td>
<td>Weight of wet coating deforms cantilever assembled pattern parts.</td>
<td>Redesign pattern assembly to avoid cantilevered extremities.</td>
</tr>
<tr>
<td>■ Cracks form during the drying period.</td>
<td>Uneven drying causes gel shrinkage stress development in shell.</td>
<td>Begin drying immediately after final coat.</td>
</tr>
<tr>
<td>■ Cracks form at end of drying period, but prior to dewaxing.</td>
<td>Wax expansion because of an increase in temperature.</td>
<td>Rotate pattern assembly while drying or dry in turbulent air.</td>
</tr>
<tr>
<td>■ Cracks form on dewaxing in: Steam autoclave.</td>
<td>Wax warms and expands prior to liquefying.</td>
<td>Reduce $\Delta T$ ($\Delta T$ is the dry bulb temperature minus the wet bulb temperature).</td>
</tr>
<tr>
<td>■ Gas furnace.</td>
<td>Shell too weak to withstand dewaxing pressure.</td>
<td>Load, close, and pressurize autoclave more rapidly.</td>
</tr>
<tr>
<td>■ Cracks form on cooling after firing.</td>
<td>Shell too dense to permit wax penetration.</td>
<td>Increase thickness; more coats or larger stucco.</td>
</tr>
<tr>
<td>■ Use of crystalline silica as refractory powder.</td>
<td>Devitrification of amorphous to crystalline silica.</td>
<td>Increase modulus of rupture. Use higher autoclave pressure.</td>
</tr>
<tr>
<td>■ Use of amorphous silica; i.e., fused silica.</td>
<td></td>
<td>Increase shell permeability.</td>
</tr>
<tr>
<td>■ Impurities in stuccoes or refractory powders.</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Abnormal Slurry Viscosity</strong></td>
<td>Grain not fully wetted.</td>
<td>Let mix overnight.</td>
</tr>
<tr>
<td>■ Viscosity too high or low as prepared.</td>
<td>Improper composition, i.e., $R_w$.</td>
<td>Measure density and adjust composition accordingly.</td>
</tr>
<tr>
<td>■ Entrained air (high viscosity, low density).</td>
<td></td>
<td>Stir slowly to allow air to escape; add antifoam agent only if necessary.</td>
</tr>
<tr>
<td>Problem</td>
<td>Cause</td>
<td>Remedy</td>
</tr>
<tr>
<td>---------</td>
<td>-------</td>
<td>--------</td>
</tr>
</tbody>
</table>
| Viscosity increases slowly with use:  
  - Density high.  
  - Density normal. | Evaporation loss.  
  Binder gelling because of:  
  Instability with refractory powder.  
  High slurry temperature. | Add water.  
  Use only recommended refractory powder.  
  Reduce stirring speed; cool slurry. |
| Casting Defects  
  - Positive defects or “plus metal.” | Prime dip viscosity too low. | Increase prime slurry viscosity to give better casting surface. |
| Mold buckle. | Weak shells because of:  
  Insufficient grain.  
  Too thin shell or coats. | Check viscosity and density of slurry.  
  Check for slurry gelation (high viscosity, low density).  
  Add additional coats, increase backup slurry viscosity, or use coarser stucco. |
| “Scabs” or irregular cavities. | Second coat penetrates cracks in prime coat, caused by too fast drying. | Check humidity in drying room to be sure it is near 40–60% (never below 30%). Also temperature fluctuations and air draft causing irregular drying. |
| Metal flash or “fins” (“plus metal”). | Shell cracking because of:  
  Wax expansion.  
  Low burnout temperature or slow autoclave pressurization. | Control dip room temperature and humidity. Dry and store at dip room temperature.  
  Increase temperature.  
  Higher steam pressure or larger steam manifold. |
| Metal breakthrough (bubbles of “plus metal”). | Insufficient wetting of wax pattern.  
  Incomplete removal of silicone mold release agent.  
  Bubbles in the prime coat. | Use adequate wetting agent in prime dip slurry.  
  Use prewet solution of LUDOX® colloidal silica plus wetting agent.  
  Improve pattern cleaning with better solvents and/or detergents and/or procedure.  
  Use light air blast (or blowing) to remove bubbles before stuccoing. |
| Generally rough surface. | Prime dip viscosity too low. | Increase viscosity to the extent possible while still filling mold cavities adequately. |
| Inclusions (nonmetallic). | Poor mold handling practices: stucco in mold. | Do not stucco final dip. Get full coverage with seal coat. |
Advantages

- Predictable uniform slurries for easier production control
- High “green” strength for better mold quality during dewaxing
- Strong ceramic shells with greater dimensional stability for closer tolerances
- High bonding strength to prevent shell failure and costly rejects
- A low-cost source of silica binder with uniform properties from batch to batch

If you aren’t using Grace LUDOX® colloidal silica binder to improve your ceramic shell process... consider specifying LUDOX® colloidal silica on your next order.

Ordering Information

To order Grace LUDOX® colloidal silica, additional literature, or a product sample, call Customer Service toll-free at 888-659-1716. For locations outside the United States, contact the local Grace representative in your country.