BENTONE®, BARAGEL® rheological additives
Organoclay thickeners for the lubrication industry

Enhanced Performance Through Applied Innovation
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Overview

Elementis, Inc. is the world leader in the production and distribution of performance chemical products that are used to enhance many of the items that you and thousands of others rely on every day.

Elementis manufactures products on a worldwide basis, in facilities that are accredited under the international quality standard ISO 9000 series:

- Newberry Springs, California ISO 9002
- Charleston, West Virginia ISO 9002
- St. Louis, Missouri ISO 9002
- Livingston, Scotland ISO 9002

At Elementis, we take a proactive approach to quality. Our quality process is focused on providing consistent high-quality products and services which meet our customers’ needs. Some of the key elements of this process are:

Quality manual

The Elementis Quality Assurance Manual defines our Quality Policy.

Statistical process control

We employ Statistical Process Control techniques at all of our plants. Employees worldwide are specially trained to have a thorough understanding of its principles and procedures.

Rheology - Our core technology

Research and experimentation in the field of rheology (the science that deals with the flow of matter) led to pioneering the development of rheological additives. These are performance chemicals that affect the feel, flow and function of thousands of products used, such as grease, paint, ink and drilling fluids.

Technical service

Our teams of technical service specialists work closely with our customers and provide assistance from formulation through scale-up and production.

Analytical

Our analytical department provides support to research, development, technical service and manufacturing. We develop quality control analytical methods for our products. This multi-million dollar analytical capability includes some of the latest technology in X-ray Diffraction, Atomic Absorption, SEM, Fourier Transform Infrared Spectroscopy and Gas Chromatography/Mass Spectroscopy.
Clay chemistry

Elementis manufactures several different gellants for grease and other lubricating systems. The gellants are based on one of two types of layered clay from the smectite group shown in Figure 1. The montmorillonite (commonly called bentonite) type has a central layer of substituted aluminum oxide and has approximate dimensions of 8,000 x 8,000 x 10Å. The other clay type used is hectorite, which has a central layer composed of substituted magnesium oxide and has approximate dimensions of 8,000 x 800 x 10Å. Both clay types have outer layers of silicate.

Individual smectite clay platelets are quite flexible. The flexibility of the platelet is much like a piece of tissue paper, but with much greater strength. Because the individual platelets are so thin and flexible they do not interfere with the lubrication properties of a base fluid.

Ref:
Grim, R. E., Clay Mineralogy, Mc Graw-Hill, p. 79 (1968)
Hofmann, Endell, and Wilm, Marshall, and Hendricks
From natural clay to grease thickener

Natural smectites are highly efficient gellants for water and must be modified if they are to have utility in organic systems. Because of substitutions in the crystal lattice, cations (e.g., Na+, Ca++) occur at the surface to compensate the unbalanced charge. Replacement of the natural cation by a long chain organic cation (Figure 2) produces a highly efficient oil viscosity modifier and grease gellant. Equally important are the hydroxyl groups that occur along the platelet edges (Figure 3) because they provide sites for platelet-to-platelet association through hydrogen bonding. Water is shown as the hydrogen bonding bridge in Figure 3.
**BENTONE®, BARAGEL® thickening process**

As supplied to the customer, the dry BENTONE® and BARAGEL® additives are in the form of agglomerated platelet stacks (Figure 4a).

The process necessary to fully disperse the platelet stacks and then to “delaminate” the individual platelets within the stack requires a combination of mechanical and chemical energy. While heat is not essential in most systems, processing temperatures above 25°C (77°F) increase the rate at which the organoclay agglomerates wet out. The steps in the gelation process are illustrated in Figure 4. The agglomerated platelet stacks (Figures 4a and 4b) must first be wet out under the influence of low shear by base fluid penetration of the capillary spaces.

A polar additive is added, if a self-activating gellant is not used, forcing the platelets farther apart (Figure 4c). Application of high turbulent shear completely separates the platelets. The result is a randomly oriented, fully gelled organoclay rheological structure (Figure 4d) in the base fluid.

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**Recommended usage levels of polar activators**

The concentration of a polar activator will affect the consistency and shear stability of the grease. The optimum concentration varies according to the base oil and additives used in the formulation. Table 1 shows the recommended polar activator usage levels.

**Propylene carbonate IS NOT RECOMMENDED for use in formulations that contain sodium nitrite. Sodium nitrite can react with propylene carbonate to form gaseous oxides of nitrogen and carbon dioxide. Be aware that sodium nitrite can also react with other ingredients in the grease, e.g., EP additives.**

For greases conforming to FDA or similar authority requirements, BARAGEL® 3000 activated with water or BENTONE® 34 activated with food grade ethanol are the recommended thickener systems.

An optimum amount of polar additive must be used to develop full gel strength. If insufficient polar additive is used, the platelets will remain tightly bound in stacks, resulting in soft grease consistency. An excess of polar additive will interfere with the hydrogen bond bridging mechanism of the water molecule, leading to reduced gel strength.

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**Table 1 Recommended polar additives and usage levels**

<table>
<thead>
<tr>
<th>Common Polar Additives</th>
<th>Typical Use Range of Polar Additive Based on Weight of Organoclay*</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Acetone</td>
<td>Cost Effective, But Low Flash Point. 25 - 40%</td>
</tr>
<tr>
<td>2. Methanol/H₂O (95/5)</td>
<td>Cost Effective But Low Flash Point. 7 - 20%</td>
</tr>
<tr>
<td>3. Food Grade Ethanol/</td>
<td>Must Be Used In Lubricants Conforming To FDA or Similar Authority Requirements. Low Flash Point. 12 - 28%</td>
</tr>
<tr>
<td>Water (95/5)</td>
<td></td>
</tr>
<tr>
<td>4. Propylene Carbonate</td>
<td>Better Gel Strength In Poor Wetting Base Stocks. High Flash Point. More Expensive. 8 - 25%</td>
</tr>
</tbody>
</table>
Milling equipment and shear manufacturing equipment

Organoclay greases are different from soap greases because they require high shear forces during production in order to properly delaminate the organoclay platelets and thicken the base stock. The shear forces required for proper dispersion must be produced by the equipment itself. Therefore, careful attention must be paid to equipment choice. Colloid mills (Figure 5) exert high shear to the grease as it moves through a narrow gap between the stator and high speed rotor. Homogenizers (Figure 6) produce high shear by forcing the grease through a narrow orifice at high velocity onto an impact ring. The high shear provided by a colloid mill or homogenizer establishes the organoclay matrix (Figure 4d) which results in the desired grease consistency.

Grease manufacturing processes

Easy to make

The following are three commonly used manufacturing procedures. Grease producers typically make modifications to conform to their individual equipment. Pump recirculation is recommended when using the Simple Mix Process to improve agitation.

Simple mix

In a mixing vessel of suitable size, add in the order listed:

1. All the base fluid - begin mixing
2. Organoclay - mix until dispersed (5 to 10 minutes)
3. Polar activator - mix 30 minutes minimum
4. Additives
5. Mill (Colloid mill or homogenizer)
6. Package

Cold concentrate

This process is more effective in cases when working with somewhat poorer wetting base oils, such as polyol esters and diesters.

In a mixing vessel of suitable size, add in the order listed:

1. 1/3 to 1/2 of the base fluid - begin mixing
2. Organoclay - mix until dispersed (5 to 10 minutes)
3. Polar activator - mix 30 minutes minimum
4. Remainder of base fluid
   (additives are usually blended in with this oil addition)
   - mix until fully blended.
5. Mill (Colloid mill or homogenizer)
6. Package

The Cold Concentrate Process typically produces harder greases than the other processes; however, greater strain is exerted on the equipment by the paste-like concentrate.
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Heated process

The Heated Process is used to drive out volatile polar activators and/or to aid in incorporating certain additives such as polymers. The Heated Process, although not necessary, aids in the dispersion of organoclay thickeners in high viscosity mineral oils, PAOs, vegetable oils and other fluids with low solvency.

It is important to add 0.1 percent water during the cooling cycle of the Heated Process if the temperature exceeded 212°F (100°C) to reestablish the hydrogen bonding necessary for maximum gel strength.

In a mixing vessel of suitable size, add in the order listed:
1. All the base fluid - begin mixing
2. Organoclay - mix well
3. Polar activator - mix 30 minutes minimum
   Heat to 150°F - 250°F (66°C-121°C) for 20 - 30 minutes
4. Add remaining additives
5. Mill (Colloid mill or homogenizer)
6. Package

Notes on grease production methods

Additives to enhance grease performance are generally added before milling with sufficient mixing to ensure homogeneity.

Application of organoclay grease

Because of their excellent performance under extreme environmental conditions, the BENTONE® and BARAGEL® rheological additives have wide application as gellants for lubricants used in:
- Mining Equipment
- Foundry and Steel Fabrication
- Rolling Mills
- Textile Mills
- Cement Manufacture
- Paper Mills
- Food Processing Equipment
- Farm Machinery
- Off-Highway Construction Equipment
- Aviation
- Aerospace
- Metalworking Fluids
- Mold Release/Drum Release
- Railroad (Wheel-Rail and Switch Lubricants)

Figure 7

Plant schematic which can be used for any of the processes mentioned above.
Features of Elementis organoclay additives

- Produce non-melting, high temperature greases
- Provide bleed resistance
  - long life without syneresis
  - also used to enhance the bleed resistance of nonclay greases
- Approved for FDA applications (BARAGEL® 3000, BENTONE® 34)
- Self-activating gellant (BARAGEL® 3000)
- Effective for environmentally friendly systems (BARAGEL® 10)
- Low temperature pumpability, low starting torque
- Base oil versatility:
  - Petroleum Oils
  - Vegetable Oils
  - Silicone Fluids
  - Synthetic Oils and Fluids

BARAGEL® 3000 does not require the use of chemical activators that are used with conventional organoclay gellants (e.g., alcohols, ketones, carbonates). After the dry organoclay powder has been incorporated into the oil, addition of 0.1 percent water by weight of the total grease is recommended to obtain maximum gelling efficiency. Water addition will normally boost the gelling efficiency of BARAGEL® 3000 about 10 percent. If water is undesirable, additional BARAGEL® 3000 may be used to offset the effect of the water.

Figure 8 shows the gelling efficiency of BARAGEL® 3000 in three commonly used base oils. Actual grease consistencies depend on oil, additives and process equipment.

Self activated BARAGEL® 3000

Self activating BARAGEL® 3000 represents a major breakthrough in the way organoclay greases are made. Polar activators are no longer required to achieve full dispersion of organoclay platelets. This eliminates a serious fire hazard and reduces out-of-specification products. Fewer raw materials plus reduced fire hazard = money saved.

### Figure 8

**BARAGEL® 3000 Efficiency in 3 Common Oils**

<table>
<thead>
<tr>
<th>Oil Type</th>
<th>600 Solvent Neutral</th>
<th>150 Bright Stock</th>
<th>1200 MVI Naphthenic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity, cSt</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>@40°C</td>
<td>113</td>
<td>500</td>
<td>270</td>
</tr>
<tr>
<td>@100°C</td>
<td>12.1</td>
<td>31.5</td>
<td>15.2</td>
</tr>
<tr>
<td>Viscosity Index</td>
<td>95</td>
<td>96</td>
<td>51</td>
</tr>
<tr>
<td>Pour Point, °C</td>
<td>-9</td>
<td>-9</td>
<td>51</td>
</tr>
<tr>
<td>Flash Point, °C</td>
<td>249</td>
<td>299</td>
<td>230</td>
</tr>
</tbody>
</table>

Self activating BARAGEL® 3000 represents a major breakthrough in the way organoclay greases are made. Polar activators are no longer required to achieve full dispersion of organoclay platelets. This eliminates a serious fire hazard and reduces out-of-specification products. Fewer raw materials plus reduced fire hazard = money saved.
Biodegradable greases
BARAGEL® 10

BARAGEL® 10 is recommended for vegetable oils and synthetic esters used to produce biodegradable greases. The efficiency of BARAGEL® 10 in four vegetable oils is illustrated in Figure 9. Greases produced with BARAGEL® 10 may be manufactured by one of the typical procedures described on page 3.

A BARAGEL® 10/canola oil grease proved to be readily biodegradable exhibiting 72.1% degradation in 28 days.*

*Tested at 20mg/l, according to OECD Method 301B “Ready Biodegradability: Modified Sturm Test.”
Synthetic and FDA grease

The versatility of organoclay gellants is demonstrated in Figure 10. Recommended usage levels for NLGI consistency Nos. 1-3 are shown for PAO, FDA white oil, polybutene and silicone fluid.

Additives

Additives vary tremendously in their effect on organoclay grease consistency from little or no effect to complete and irreversible destructuring of the grease. Any additive should be screened for degelling effects before it is used in a production batch. This is easily done in the lab by preparing an organoclay grease containing no additives then mixing in each additive and observing for degelation.

A complete reference grease formulation is shown in Figure 11.

The following is a partial list of specialty additives that has been successfully used in organoclay greases:

Extreme pressure/antiwear additives
- Graphite
- Molybdenum disulfide

Water resistance/tackiness additives
- Polysisobutylene
- Styrene-isoprene copolymer
- Natural latex
- Ethylene-propylene copolymer

Antioxidants
- p,p’-dioctyldiphenylamine
- Octylated N-phenyl-1-naphthylamine
- Zinc diamylthiocarbamate
- Butylated hydroxytoluene (BHT)
- Alkylated diphenylamines

Corrosion inhibitors
- Sodium Nitrite
- Disodium Sebacate

Warning: Certain additive types, (e.g., sulfonate, naphthenate, imidazoline, dithiophosphate, etc.) associate with the organoclay and disrupt the hydrogen bonding required for gel formation which was described on page 2. Concentrations as low as 0.1 percent of these additives can cause complete destructuring of the grease (i.e., the grease becomes liquid).

Sodium sulfonate is shown blocking hydrogen bond formation in Figure 12.
Rheology

Elementis rheological additives generally produce thixotropic behavior (thin when sheared, recover when shearing is decreased). This behavior is illustrated in the following two figures. In Figure 13, the viscosities of two greases approach the viscosities of their base oils, canola and TMPT (trimethylol propane trioleate) ester, at very high shear rates. Figure 14 shows the rapid viscosity recovery of the same two greases when shear is relaxed. Time interval A-B shows viscosity is low when moderate shear is applied. At B the shear force is reduced by a factor of 104 and the viscosity rebuilds dramatically and remains constant over the interval B-C.

Greases are influenced by temperature much like their base oil component. Figure 15 shows the viscosities of a bright stock (high temperature grease) and a PAO (low temperature grease) over the range -60°C to 180°C.

Base oils also influence the yield values of greases. Figure 16 shows that a 6cSt PAO grease with 8 percent thickener has a lower yield value at 25°C than a similar bright stock oil grease with 4.5 percent thickener.
**Trouble shooting guide**

**Poor gel strength**

**Check in order:**
1. Gellant concentration
2. Processing shear
3. Polar activator and/or water concentration
4. Compatibility of additives
5. Oil temperature

**Poor age stability**

**(syneresis/softening)**

**Corrective Actions:**
1. Use antioxidant
2. Manufacture toward the hard side of the NLGI grade
3. Use more viscous base oil
4. Use additives with better compatibilities

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**Color change/gassing**

Repeat the formulation in the lab omitting one ingredient at a time and age test at the conditions under which color change occurred (temperature, UV, moisture, pH). Replace the additive responsible.

Oxides of nitrogen (NO, NO₂, N₂O) have been detected in sodium nitrite containing greases where propylene carbonate and/or metal sulfides were used. Carbon dioxide has also been detected in greases containing propylene carbonate.

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**Product selector**

<table>
<thead>
<tr>
<th>Product Feature</th>
<th>BARAGEL® 10</th>
<th>BARAGEL® 20*</th>
<th>BARAGEL® 3000</th>
<th>BENTONE® 27</th>
<th>BENTONE® 34</th>
<th>BENTONE® 38</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paraffinic Petroleum</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Naphthenic Petroleum</td>
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<tr>
<td>FDA White Oil</td>
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<tr>
<td>Synthetic Hydrocarbon</td>
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<tr>
<td>Vegetable Oils</td>
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<tr>
<td>Esters</td>
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<tr>
<td>Silicone Fluid</td>
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<tr>
<td>Polyalkylene Glycol</td>
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</table>

* Eastern Hemisphere only
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