Technical Information MF-TI 1

Thickeners for Lubricating Greases
Thickeners for Greases

Phyllosilicates

Phyllosilicates are clay minerals of natural origin. They have a platelet structure and are often stacked (figure 1). The individual platelets are extremely thin (approx. 1 nm) and have a diameter of 500–1000 nm. Because of their chemical structure the platelets are negatively charged at their surface but positively charged or neutral at their edges.

Positively charged ions (cations) are embedded between the individual platelets; often these are sodium or calcium cations.

The phyllosilicates are available as commercial products in powder form. In their natural form, they are highly hydrophilic and therefore extremely compatible with and easily dispersable in water.

Organophilic Phyllosilicates (Organoclays)

To increase the compatibility of the phyllosilicates with organic solvents such as oils, these silicates are organically modified and become organophilic phyllosilicates, also known as “organoclays”. The process involves replacing the phyllosilicate’s cations (e.g., Na⁺, Ca²⁺) with complex, organic cations. These organic cations can have various long-chained terminal groups, thereby giving the compound different polarities. By selecting the cation, you can significantly influence the compatibility of the organoclay with the solvent, which means it is possible to produce organoclays with optimum compatibility for various solvents/oils (figure 3). Depending on the choice of base oil, a suitable product must be selected to produce the lubricating grease.

Organoclays are also available as powders and, just like unmodified layered silicates, have a platelet structure (figure 2).

Example of the Compatibility of some Organoclays

<table>
<thead>
<tr>
<th>Low polarity</th>
<th>High polarity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aliphatic hydrocarbons</td>
<td>Aromatic hydrocarbons</td>
</tr>
<tr>
<td>CLAYTONE-40</td>
<td>TIXOGEL-VP or TIXOGEL-MP or TIXOGEL-MP100*</td>
</tr>
<tr>
<td>CLAYTONE-AF* or CLAYTONE-HT or CLAYTONE-HY*</td>
<td>TIXOGEL-MPZ</td>
</tr>
</tbody>
</table>

* self-activating grades
Products

BYK sells organoclays that can be used as thickeners for lubricating greases under the trade names CLAYTONE and TIXOGEL. Figure 3 provides an overview of the suitable organoclay grades. Additional materials (e.g. suitable for food contact) are available on request.

Thickening of Oils

To thicken an oil, thereby creating a lubricating grease, the phyllosilicate platelets first need to be separated from each other. This process is also called delamination. Completely dispersing the organoclay ensures optimum swelling and therefore high viscosity/gelling. For this reason, dispersion should be performed with an aggregate that can deliver high shear rates and a high energy input. The higher the introduced shear energy, the better the gel formation. Higher temperatures also make dispersion easier.

Once the platelets are separated from each other, they arrange themselves in the solvent like a house of cards. The platelets coordinate via hydrogen bonds and form the “house of cards”/network. This network ensures gel formation/thickening and therefore increases the viscosity. The higher the concentration of the organoclay, the higher the achieved viscosity and thereby the NLGI classification of the lubricating grease.

Dispersion and Network Formation of Phyllosilicates

Dispersion Addition of an activator, gel formation via hydrogen bonds

figure 4
Activator

Polar activators positively influence gel formation/thickening. Firstly, they ensure better platelet delamination and therefore better dispersion. Secondly, they settle between the platelets and form hydrogen bonds, which increases the cross-linking and thereby the viscosity/thickening of the lubricant (figure 5).

Standard activators are:
- Propylene carbonate or propylene carbonate/water (95/5)
- Methanol or methanol/water (95/5)
- Ethanol or ethanol/water (95/5)
- Other short-chain alcohols or acetone

Adding 5% water to the activator increases its ability to form hydrogen bonds, making the activator more effective.

The optimum proportion of the activator depends on the formulation. We recommend starting with an activator proportion of about 10–20%, based on the organoclay being used. Please note that too much of the activator can cause the viscosity to decrease again (figure 6).

Most organoclays need the addition of an activator. However, there are also “self-activating” products that already contain an activator. Nevertheless, small quantities of an activator can help to further optimize the performance of self-activating organoclays. In many cases, it is enough to add a little water.
Lubricating Grease Production

Lubricating greases are often produced according to the following basic process:
- The oil is provided and stirred.
- 5–7 % of the clay is added and stirred at high shear rates for approx. 5–10 min.
- The polar activator is added (10–20 % based upon the clay) – it is stirred for a further approx. 25–30 min.
- The remaining additives (e.g. antioxidants, anti-corrosives) are added whilst stirring.
- The mixture is processed via a colloid mill.

The following modifications can also help to improve incorporation:
- It may be useful to heat the oil to 50–60 °C and perform the process with warm oil. This has a positive influence on the organoclay dispersion.
- If a lubricating grease is produced based on a base oil that has poor wetting properties, it can help to use half the volume of oil to incorporate the organoclay. This increases the shear and can have a positive influence on the organoclay dispersion. The remaining oil quantity can be added later in the process.

Benefits of Lubricants Produced Using Organoclays

Lubricants produced using organoclays feature significant benefits:
- Good performance at high temperatures
- Good performance at low temperatures
- High dropping point
- Very low “bleeding” of the oil from the lubricant
- Very good adhesion to metal

For these reasons they are used in a wide variety of industrial applications.


Lubricating greases are classified according to ASTM D 217. Measuring the cone penetration, the results of which categorize the lubricating greases into NLGI classes. The lubricating grease is first processed in a grease worker (60 double strokes – 60 x).

Figure 7 shows how the penetration depth of the cone and therefore the varying NLGI classification depends on the used quantity of organoclay. In this case, CLAYTONE-40 was tested in various oils.

In figure 9, the viscosity of the lubricating grease and the base oil is plotted at different shear rates. The base oil shows Newtonian behavior at constant viscosity, both at high and low shear rates. In comparison, the lubricating grease behaves thixotropic. The viscosity at low shear rates is extremely high and decreases once the shear rates are increased.

Incorporating the organoclay also significantly influences the NLGI class of the lubricating grease. The better the organoclay is sheared and therefore the individual platelets separated from each other, the higher the NLGI class.

### Effect of CLAYTONE-40 in Different Solvents

<table>
<thead>
<tr>
<th>Addition in %</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>NGLI class</td>
<td>0</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Shear rate (s⁻¹)</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
</tbody>
</table>

In figure 7.

- Naphthenic, high viscosity
- Paraffinic, low viscosity
- Polyalphaolefinic, low viscosity

### Dynamic Viscosity of Base Oil with and without CLAYTONE-40

- Group I base oil
- Group I base oil with 6 % CLAYTONE-40

In figure 9.

<table>
<thead>
<tr>
<th>Viscosity (mm²/s)</th>
<th>API class</th>
</tr>
</thead>
<tbody>
<tr>
<td>40 °C</td>
<td>II Paraffinic</td>
</tr>
<tr>
<td>32</td>
<td>66</td>
</tr>
<tr>
<td>100 °C</td>
<td>5</td>
</tr>
<tr>
<td>Viscosity index</td>
<td>99</td>
</tr>
<tr>
<td>Pour point (°C)</td>
<td>-15</td>
</tr>
</tbody>
</table>

In figure 8.
Figure 10 shows how the NLGI class is influenced by the quantity of base oil used in the first mixing step and the effect the quantity of the activator has. If the organoclay is initially mixed with just 50% of the base oil, its concentration is therefore increased. The mixture is more viscous and the organoclay experiences a better shear as a result of the increased concentration. The remaining 50% of the base oil is added together with the other additives, leaving the total formulation unchanged. Only by selecting the process parameters is it possible to achieve a significantly higher NLGI class through better shear. The optimization of the used activator quantity results in an additional improvement.

### Product Overview

<table>
<thead>
<tr>
<th>21 CFR 178.3570</th>
<th>Activation</th>
<th>API I-III</th>
<th>API IV</th>
<th>API V (Naphthenic)</th>
<th>API V (Ester)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLAYTONE-40</td>
<td>Activator required</td>
<td>+</td>
<td>(+)</td>
<td>+</td>
<td>+</td>
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<tr>
<td>CLAYTONE-AF</td>
<td>Self-activating</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
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<tr>
<td>CLAYTONE-APA</td>
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<td>+</td>
<td>+</td>
<td>+</td>
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</tr>
<tr>
<td>CLAYTONE-HT</td>
<td>Activator required</td>
<td>+</td>
<td>(+)</td>
<td>+</td>
<td>+</td>
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<tr>
<td>CLAYTONE-HY</td>
<td>Self-activating</td>
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<td>+</td>
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<td>+</td>
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<tr>
<td>TIXOGEL-MP</td>
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<td>+</td>
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<td>TIXOGEL-MP 100</td>
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<td>+</td>
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<tr>
<td>TIXOGEL-MP 250</td>
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<tr>
<td>TIXOGEL-MPZ</td>
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<td>TIXOGEL-VP</td>
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<td>TIXOGEL-VP V</td>
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<tr>
<td>TIXOGEL-VZ</td>
<td>Activator required</td>
<td>(+)</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
</tbody>
</table>
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