Caprolactam –
Innovation of a Classic Material
Caprolactam – a versatile basis for modern products

Products made of caprolactam-based polyamide 6 always cut a good figure...

... let dreams of white wedding gowns come true – at reasonable prices...

... and lend a touch of elegance to everyday products...
From carpets to tires, gearwheels to dowels, swimsuits to socks: caprolactam is one of the materials that have changed our world.

Caprolactam is the basis for manufacturing polyamide 6. Due to its versatile properties, polyamide 6 forms an ideal starting point for a very wide range of products:

- Carpeting, domestic textiles, furnishing fabrics
- Industrial textiles such as tire-cord fabric, fishing nets, bristles, cables and ropes
- Industrial plastics such as gearwheels, dowels, moldings and products in the tool sector
- Clothing textiles such as tights, swimwear, protective clothing, blouses and dresses
- Packaging film
Almost three-quarters of a century have passed since high molecular weight compounds became a focus of research at BASF. Our laboratories witnessed the birth of new polymerization processes, the creation of new monomers and their conversion to valuable polymers. The first plastics grades took shape in 1930, and the development of fiber intermediates began in 1938. This cleared the way for the production of what is perhaps the most exciting type of polymer for synthetic fibers and plastics: polyamide 6.

Today BASF supplies a full range of plastics, fiber intermediates, solid polymers, polymer dispersions and solutions. But most of all, we are the world’s largest producer of caprolactam. More than a fifth of the total annual production originates from the three BASF-group production facilities which are located in Ludwigshafen, Antwerp, Belgium, and Freeport Texas, USA.

By 1995 BASF was able to send more than 8 million tons of caprolactam on its way to buyers across the globe.

Want to know more about BASF and caprolactam?

More than you will find in this brochure or in our data bulletins? About how caprolactam is shipped, unloaded and stored? About technologies for spinning nylon 6 or the safe handling of polyamide 6 products? Just ask us. For we want to do more than give our customers the right goods at the right time in the right quantity and quality. We also want to help them to transform those goods into exactly the right things.

We will gladly place our entire expertise at your disposal, anywhere in the world. On pages 14 and 15 you will find the address of a knowledgeable contact person in your very neighborhood.
Conserving resources and protecting the environment: the new BASF technology production

Caprolactam and BASF were a genuine success story. But even so, our researchers were still not satisfied. The result of their ongoing commitment was a new technology for manufacturing caprolactam on the basis of butadiene instead of benzene. This considerably shortened the path from the starting material to the final product. It also meant far fewer by-products – a major contribution to resource conservation and environmental protection.

Caprolactam and BASF are a combination that still holds great promise for the future.
ASF produces caprolactam from benzene and ammonia using a mature technology that has been further perfected in recent years. Our research department developed a new caprolactam method based on butadiene and successfully tested the resultant caprolactam in the ultra-sensitive manufacture of the finest textile fibers.

With both techniques our priorities are the same: top-quality raw materials, modern production equipment and precise processing. The result is caprolactam of extreme purity and consistent quality, as reflected in its extraordinarily narrow specification limits (see page 14). These are exactly the properties needed in caprolactam’s main area of application: polyamide 6.

To reach and maintain this top level of quality we at BASF introduced a comprehensive quality management system for caprolactam and its intermediates some ten years ago. We are proud to be certified according to the internationally recognized DIN EN ISO 9001 standard since 1992.

But the caprolactam quality management system is not limited to testing the quality of the product: it also includes all activities that might affect quality, from the acquisition of raw materials to the complete production process all the way to order processing and shipping.

We pose extremely rigorous demands on the purity of our raw materials. Regular tests of incoming goods ensure that our detailed specifications are satisfied, whether the intermediates come from our own company or from outside suppliers. State-of-the-art measurement and control systems make sure that processing parameters are maintained during the manufacturing process. Prior to delivery every shipment is inspected again in a special lactam quality laboratory. Unless otherwise agreed upon, reference samples are retained for six months. On request we will even present our customers with appropriate quality certificates of analysis. The cleanliness of the shipping vehicles is also conscientiously controlled.

To augment our quality control program for caprolactam we have instituted a comprehensive training program for our employees. Should complaints or queries arise despite all our precautions, we will look conscientiously into the root of the cause.
BASF’s innovation for smooth processing: solid caprolactam in the form of tablets.
Caprolactam: what it is and how it reacts

At room temperature caprolactam is a white, hygroscopic, crystalline solid with high solubility in water and an extremely low vapor pressure of 0.002 hPa (mbar) at 25 °C.

Its essential feature is its capacity to form polyamides through polymerization. This can happen in either of two ways:

The usual way is polymerization in the presence of water. The caprolactam ring opens at 260 to 270 °C, and 99% of the caprolactam is converted into linear polyamides.

Another possibility is „anionic polymerization“, i.e. in the absence of water. Here strong bases act as catalysts. This form of processing requires a special caprolactam with a low water content of no more than 100 mg/kg.

Being a cyclic amide, caprolactam can be oxidized, hydrolyzed, N-acylated and nitrated.

In the total absence of oxygen, caprolactam is thermally stable. Molten caprolactam absorbs oxygen from the air. At temperatures above 75 °C it forms a hydroperoxide with atmospheric oxygen and reacts further to yield adipimide. Trace metals accelerate this process. In the presence of aqueous acids or alkalis, caprolactam is hydrolyzed quantitatively to aminocaproic acid.

In the gaseous phase and in the presence of dehydrating catalysts, caprolactam reacts with methanol to form methyl-caprolactam, a versatile solvent and extraction agent.

With acid anhydrides or acid chlorides, caprolactam reacts to yield N-acylcaprolactam, and with ethyne it forms N-vinylcaprolactam. Nitro-caprolactam is formed by nitration, and may be converted to aminocaprolactam by hydrogenation. L-lysine is produced from this by hydrolysis and separation of the racemic mixture.
Crystalline caprolactam: solidification point at 69 °C

Information on the relevant physical data can be found in the Safety Data Sheet under item 9.

### Caprolactam: the key physical properties at a glance

<table>
<thead>
<tr>
<th>Physical property</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density at 80 °C</td>
<td>kg/l</td>
<td>1.014</td>
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<tr>
<td>Bulk density</td>
<td>kg/m³</td>
<td>500 - 550</td>
</tr>
<tr>
<td>Boiling point at 1.013 bar</td>
<td>°C</td>
<td>270.8</td>
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<tr>
<td>Flash point to DIN 51758</td>
<td>°C</td>
<td>152</td>
</tr>
<tr>
<td>Melting/solidification point</td>
<td>°C</td>
<td>69.2</td>
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<tr>
<td>Solubility in water at 25 °C</td>
<td>kg/l</td>
<td>5.25</td>
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<tr>
<td>Heat of polymerization</td>
<td>kJ/kg</td>
<td>140</td>
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<tr>
<td>Heat of fusion</td>
<td>kJ/kg</td>
<td>142.6</td>
</tr>
<tr>
<td>Heat of combustion</td>
<td>kJ/kg</td>
<td>31,900</td>
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</table>
Caprolactam from BASF: liquid in the tank, or as free flowing tablets

Caprolactam from BASF: the only caprolactam available not only in the classical liquid form but as small tablets, making it easier and safer to process.

Liquid caprolactam is shipped in vehicles with heatable, insulated tanks, tank containers or bulk containers made of stainless steel (e.g. AISI 304 1/V2A) or aluminum.

Transport options:

- Tank truck: approx. 22 t
- Tank container (road/rail): approx. 25 - 30 t
- Bulk container (road/rail/ship): approx. 20 t
- Railroad tankcar: approx. 50 t

Caprolactam tablets are available in various forms of packaging:

- Export bag (polyethylene inner bag with outer bag) for shipment in containers
- Polyethylene bag on pallets with shrink-wrapped cover for shipment by truck, rail or container
- Polyethylene bag with inner bag lined with aluminum for anionic polymerization

The bigbag: practical for shipping and handling

Caprolactam tablets in bigbags are the front-runner in handling: the bigbag is simply lowered over a special spike and the tablets pour through the opening – virtually dust-free and with no danger of contamination.
The special spike ensures that big-bags can be emptied smoothly and quickly.
... during shipping

Liquid caprolactam is shipped at a temperature of 75 to 90 °C. Excess nitrogen pressure of 0.5 bar prevents air from entering.

The following standard shipping containers may be used:

- Movable tanks of stainless steel (e.g. AISI 304 L or V2A) or aluminum, equipped with coil/jacket heating with heatable drain valve. Heating media include hot water, unconfined steam or electrical energy.

- Road tank trucks equipped as described above.

To empty the contents, the jacket of the tank or container must be heated with hot water or steam to a temperature that enables all the caprolactam to drain.

Once emptied, the tank must be refilled with nitrogen to an excess pressure of 0.5 bar.

... during storage and processing

Caprolactam tablets should not be exposed to moisture or direct sunlight. The storage temperature may not exceed 45 °C. It is advisable to process the tablets within six months.

Bigbags and other bags should only be moved on their pallets using forklifts. A safety data sheet contains appropriate recommendations for their disposal.

Storage tanks and containers for liquid caprolactam should be made of stainless steel, e.g. AISI 304 L or V2A, or aluminum.

The storage areas should be protected against outside influence. Good ventilation is important, as are appropriate fire extinguishing mechanisms.

Hose connections on all ungrounded and moveable facilities must be earthed during connection and prior to activation. Hoses with metal braids may be used, but we recommend installing metal piping with appropriate couplings. To ensure that you choose suitable connection options, please contact our sales department.
Moreover, all tanks, pumps and pipes used for storing caprolactam must be inspected for leaks prior to filling, namely by testing them with pressurized water or nitrogen. It is very important that the systems be carefully drained afterwards and dried with nitrogen. Ideally, they should then be rinsed with caprolactam, which ensures that all water has been removed. The rinse caprolactam should not, however, be re-used, but disposed of in accordance with statutory regulations.

Squeaky clean in any packaging: caprolactam tablets can also be delivered in stackable bags on easily shipped pallets.

When handling caprolactam, always keep two of its properties in mind:
- its relatively high solidification point,
- its high solubility in water and its related high hygroscopicity.
Stages in an exciting product: from shimmering caprolactam crystals ...

... via high-value polymers ...

... to ultra-fine polyamide 6 fibers, the basis for modern products of every shape and size.
Get in contact with us!