

## Purification of Acetonitrile

The growth in consumption of acetonitrile as HPLC solvent and reagent in pharmaceutical synthesis has not only increased the demand for this chemical but also the need for disposal of the spent solvent.

The smallest amount of impurities can significantly affect the quality of the final products. Therefore, ultra-pure starting materials are essential. Most of the acetonitrile produced today comes as a by-product from the production of acrylonitrile. Another significant source of this product can be found in the waste of universities, research labs and pharmaceutical production facilities. These streams, although generally quite pure, are still considered hazardous waste and are bound for incineration in licensed disposal centers. Crystallization can provide these locations with a cost efficient method to recover this product as ultra-pure acetonitrile.

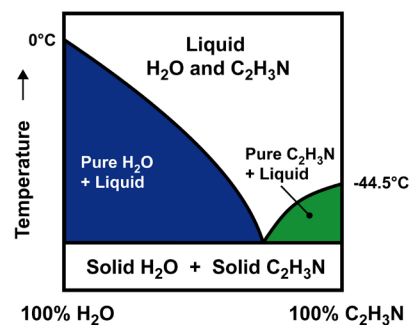
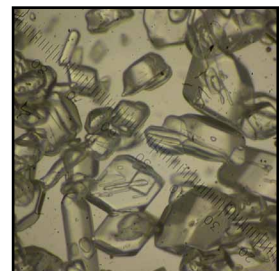
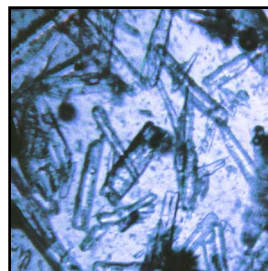
A waste stream of relatively pure feed stock with between 98wt% and 99wt% acetonitrile can easily be upgraded to product around 99.99wt% (the highest purity available) with suspension crystallization. This method offers an efficient means to reduce waste disposal costs and provides a valuable product.

The solid-liquid phase diagram for a solution of acetonitrile and water is shown below. Unlike distillation techniques, suspension crystallization provides the possibility to separate ultra-pure acetonitrile crystals in a single step.

Crystallization is a proven industrial purification technique and GEA Messo PT has been active in its application on a variety of products for over 30 years. Acetonitrile purification is just one new applications for this technology. Please see reverse page for a general description of our crystallization process.

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## End Purification with Suspension Crystallization



Process Engineering

**GEA Messo PT**

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## Key Benefits

### High product purity

The highest purity HPLC grade acetonitrile available on the market today.

### Waste product recovery

Waste disposal volumes are reduced and valuable product is recovered.

### Feedstock

Operates on a range of feed stocks. When relatively pure feed is available the specific impurity composition is irrelevant.

Simplified design and control – The system is supplied as a skid mounted crystallization and separation unit including refrigeration.

### Economics

The operating costs are significantly lower than other separation techniques.

The production costs (including capital, utilities and maintenance) for this crystallization unit are less than Euro 1 per liter purified product.

This example case is based on:

- continuous operation
- unit capacity of  $\pm 40$  kg/h purified product
- a feed of 98-99wt% acetonitrile

Other feed compositions, recoveries and production capacities are possible; GEA Messo PT can provide assistance in determining the optimum configuration and cost information for your specific circumstances.

## Process Description

The melt crystallization process is carried out in an industrially proven scraped surface crystallizer where final purification is completed in the GEA Messo PT Purifier. The feed product is cooled inside the crystallizer where part of the product is converted into pure crystal solids leaving the impurities concentrated in the residual mother liquor.

The crystallizer consists of a jacketed vessel with a rotating scraper assembly. Evaporating refrigerant or a secondary coolant in the outer jacket cools the inner wall of the vessel.

The scraper sweeps the wall surface and prevents buildup of crystals to maintain a clean heat transfer surface and continuous supply of product crystals.

Each individual crystal provides growth surface that can absorb the supersaturation caused by cooling the product at the swept surface. With billions of individual crystals present, this will provide near ideal growth conditions and ensure the production of ultra-pure crystals.

The Messo™ Purifier completes the separation of this mixture of pure product crystals and residual mother liquor. The purifier is

based on GEA Messo PT's patented wash column technology and is an essential component in this purification process.

The crystal slurry is compressed within the GEA Messo PT Purifier to remove most of the mother liquor and form a packed crystal bed. This bed consists of the pure product crystals surrounded by some residual mother liquor. The new crystals entering the purifier will force the bed toward the scraper assembly at the opposite end. The scraper disintegrates the crystal bed and a circulation pump provides melted product to reslurry the crystals. The circulation flow carries the crystals to a heat exchanger where e.g. steam or hot gas refrigerant from the refrigerant compressor provides the heat necessary to melt the crystals. The melted product can then be discharged to product storage.

The melted product in the recirculation stream countercurrently washes the residual mother liquor from the packed crystal bed as it moves through the purifier. The length of the crystal bed provides an extremely efficient wash zone for removal of any remaining impurities in the mother liquor ensuring ultra-pure product. Unlike other solid/liquid separation devices operating with a much shorter bed depth, the wash liquid forms an internal reflux loop and is therefore recovered as recrystallized product in the melt circuit instead of leaving with the mother liquor.

## Experience

GEA Messo PT is a technology oriented engineering company with over 30 years experience in the design and execution of crystallization plants worldwide. We started by crystallizing water from liquid food products in 1973 and since then have built and installed over 100 purification units around the world including applications for water, PX, PDCB, phosphoric acid as well as many other classified organic chemicals.

New developments in process technologies pose new challenges every day which require innovative and low-cost solutions. Practical orientation and customer requirement play a vital role in this connection. GEA Messo PT consistently orientates itself towards the needs of the customer. GEA Messo PT has been certified according to DIN ISO 9001 standard since 1996 and since June 2003 the ISO 9001:2000 standards were implemented following an audit by the international bureau Lloyd's Register Quality Assurance.

## Next Steps...

On-site demonstration of this technology is possible in various configurations from 3 kg/h up to 300 kg/h using one of GEA Messo PT's pilot plants. For more information regarding this technology and your specific configuration requirements please contact us at: [info.niropt.nl@geagroup.com](mailto:info.niropt.nl@geagroup.com) or phone +31.736 390 390.