



Chemical Pilot Plants

Suspension Crystallization and Wash Column Separation

Application

The W6 Chemical pilot plant is used to provide product and operation data for a specific application. It consists of a scraped surface drum crystallizer and a piston type wash column as separation unit. A secondary refrigerant circulating in the outer jacket of the drum crystallizer provides the cooling for crystallization. Two primary refrigeration systems are available providing an operational range between -60°C and $+130^{\circ}\text{C}$.

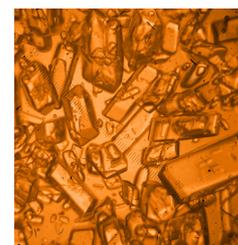
The unit is provided as a self-contained skid including all process, control and refrigeration components. Designed according to EC directive 94/9/EC (ATEX) and built based on industrial components, the W6 can be operated in practically any location. Initial feasibility tests can be completed in a day. Extended trials provide sufficient information to scale up to industrial capacities and at the same time can be used to demonstrate the robustness and range of the of the crystallization process on any specific application.

Listing of some of the products that have been successfully tested using GEA pilot plants

| Product | Formula | CAS No. | Mol [g/mol] | Tmelt [$^{\circ}\text{C}$] |
|-----------------------|-----------|-----------|----------------|---------------------------------|
| CONFIDENTIAL | | | 150.2 | 98.85 |
| p-Nitrochlorobenzene | C6H4NO2Cl | 100-00-5 | 157.5 | 82.5 |
| Durene | C10H14 | 95-93-2 | 134.2 | 79.24 |
| Caprolactam | C6H11NO | 105-60-2 | 113.6 | 69.2 |
| o-Phenylphenol | C12H10O | 90-43-7 | 170.2 | 57.45 |
| p-dichlorobenzene | C6H4Cl2 | 106-46-7 | 147 | 53.1 |
| CONFIDENTIAL | - | - | 134.1 | 49.5 |
| 4,4 MDI | - | 101-68-8 | 250.25 | 40.5 |
| CONFIDENTIAL | - | - | 88 | 36.4 |
| Phosphoric acid | - | 7764-38-2 | 107 | 29.3 |
| 2,4 TDI | C9H6N2O2 | 584-84-9 | 174.1 | 21.8 |
| Methacrylic acid | C4H6O2 | 79-41-4 | 86 | 14.35 |
| Acrylic Acid | C3H4O2 | 79-10-7 | 72 | 13.5 |
| N-vinyl-2-pyrrolidone | C6H9NO | 88-12-0 | 111.1 | 13.5 |
| p-Xylene | C8H10 | 106-42-3 | 106.1 | 13.26 |
| p-Chlorotoluene | C7H7Cl | 106-43-4 | 126.5 | 7.55 |
| Water | H2O | 7732-18-5 | 18 | 0 |
| Hydrogen Peroxide | H2O2 | 7722-84-1 | 34 | -0.4 |
| Chiral component | - | - | 118.1 | -2.7 |
| Acetonitrile | C2H3N | 75-05-8 | 41 | -44.5 |
| m-Xylene | C8H10 | 108-38-3 | 106.1 | -47.87 |

Chemical Pilot Plants

Suspension Crystallization and Wash Column Separation



Typical test run on an organic chemical

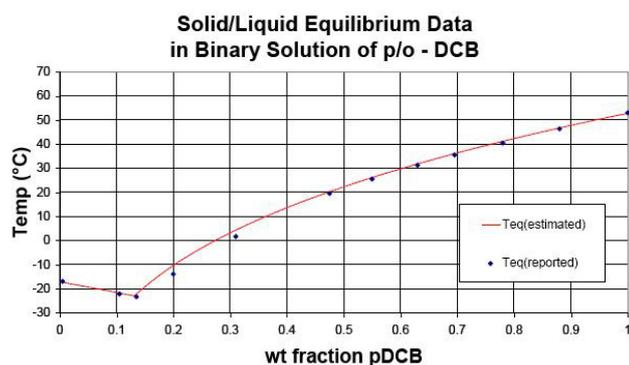
| | |
|--|-----------|
| Molecular weight | Mw |
| Specific heat capacity (liquid, solid) | Cp, sol |
| Heat of crystallization | ΔHm, crys |
| Melting temperature of pure compound | Tm (prod) |
| Melting temperature of major impurity | Tm, imp |

$$\ln x_p = \frac{\Delta H_m \cdot Mw}{R \cdot T} \cdot \left(\frac{T}{T_{m,p}} - 1 \right)$$

When other data is not available, binary mixtures of isomers can be quickly estimated using the van 't Hoff equation and many times can give an accurate estimate over the total range. However, in most cases we are only concerned with the upper (pure) region of the curve.

With this basic information we can then plan the initial test run. Our engineering staff will review all areas of the test planning with the client.

The W6 pilot plant requires at least 50 liters to fill the crystallizer and wash column. Depending on the goals of the test, the unit can be operated on total recycle so this initial charge may be sufficient to run a number of trials.



A normal test run requires at least one day for charging and making the first batch of crystals for separation. This is sufficient to demonstrate the feasibility and purity of the final product. Additional runs are required to determine the limits of the process and completely demonstrate the crystallization process.

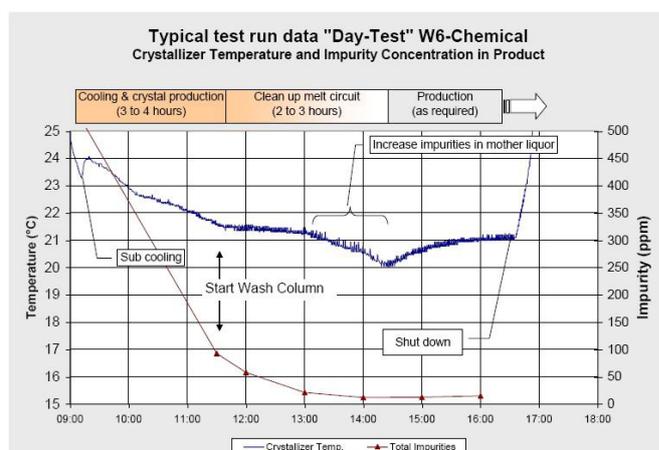
Suspension based crystallization offers the possibility to reduce the overall cost of the purification required by many organic chemicals.

Features

- Low process temperatures
- Ultra high purities
- Continuous operation

Suspension based crystallization and wash column separation may be applied to a wide variety of chemicals. The first step is to review the history of a specific chemical and evaluate its potential for purification by crystallization.

GEA provides technical assistance in the initial product review, test design, installation and execution of pilot plant tests.



On-site demonstration of this technology is possible in various configurations using GEA pilot plants. For more information regarding this technology and your specific configuration requirements, please contact us or get in touch with your local GEA contact on gea.com via the Application Chemical, Specialty & Fine Chemicals.

GEA Netherlands

GEA Niro PT B.V.

De Beverspijken 7-b

5221 EE 's-Hertogenbosch, Netherlands

Tel +31 73 6390390

Fax +31 73 6312349

info.niropt.nl@gea.com

gea.com