

Suspension Crystallization and Wash Column Separation

The W6 Chemical pilot plant is used to provide product and operation data for a specific application. It consists of a 30 liter scraped surface drum crystallizer and a 6 cm 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.

Product	Formula	CAS No.	Mol	Tmelt
			g/mol	[$^{\circ}\text{C}$]
CONFIDENTIAL			150.2	98.85
p-Nitrochlorobenzene	C6H4NO2Cl	100-00-5	157.5	82.5
Durene (tetramethylbenzene)	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
CONFIDENTIAL			88	36.4
Phosphoric acid		7764-38-2	107	29.3
Toluenediisocyanate (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 (NVP)	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

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

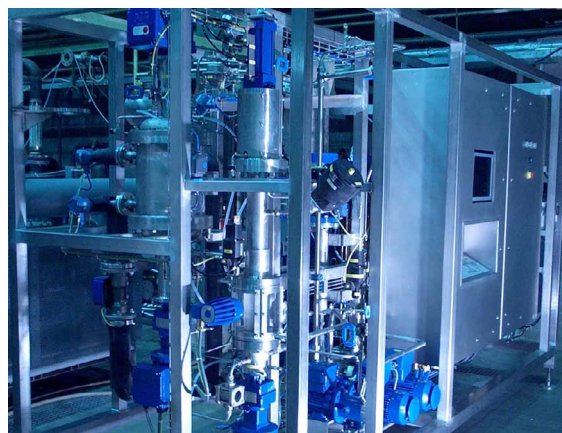
Process Engineering

GEA Messo PT

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Chemical Pilot Plants

Suspension Crystallization and Wash Column Separation



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

- ◆ Low process temperatures
- ◆ Ultra high purities
- ◆ Continuous operation

Suspension based crystallization and wash column separation may be applied to a wide variety of chemicals; some have been tested while many have not. The first step is to review the history of a specific chemical and evaluate its potential for purification by crystallization.

GEA Messo PT has experience and provides technical assistance in the initial review, test design, installation and execution of pilot plant tests.

Typical test run of an organic chemical

Required data for initial feasibility study:

Molecular weight	Mw
Specific heat capacity (liquid, solid)	Cp, sol
Heat of crystallization	Delta h, crys
Melting temperature of pure compound	Tm,p
Melting temperature of major impurity	Tm, imp

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.

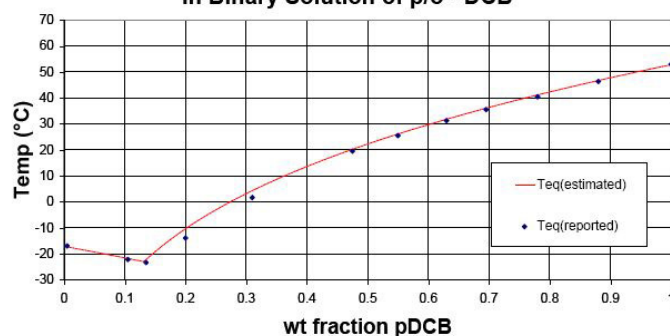
GEA Messo PT will be glad to assist you in all stages of a new process development.

For more information regarding this technology and your specific configuration requirements please contact us at: info.niropt.nl@geagroup.com or phone +31.736 390 390.

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

This form of the van 't Hoff gives a quick initial estimate

Solid/Liquid Equilibrium Data in Binary Solution of p/O - DCB



Typical test run data "Day-Test" W6-Chemical
Crystallizer Temperature and Impurity Concentration in Product

