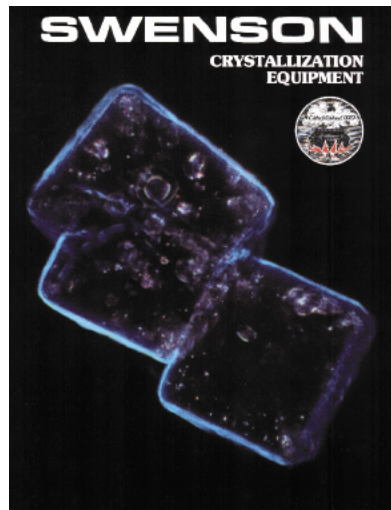




**Whiting Equipment Canada Inc.
Swenson Crystallization Equipment**





Crystallization: An industrial process for producing high-purity compounds from impure solutions with low energy input.

Crystallization is the process by which a chemical is separated from solution as a high-purity, definitively shaped solid.

A crystal may be defined as a solid composed of atoms arranged in an orderly, repetitive array. The inter-atomic distances in a crystal of any definite material are constant and characteristic of that material. Crystals are, in short, high-purity products with consistent shape and size, good appearance, high-bulk density and good handling characteristics.

Because the pattern or arrangement of the atoms is repeated in all directions, there are definite limitations on the shapes which crystals may assume. For each chemical compound, there are unique physical properties differentiating that material from others, so the formation of a crystalline material from its solution, or mother liquor, is accompanied by unique growth and nucleation characteristics.

While crystallization is a unit operation embracing well known concepts of heat and mass transfer, it is nevertheless strongly influenced by the individual characteristics of each material handled. Therefore, each crystallization plant requires many unique features based upon well established general principles. Each application must be evaluated on an individual basis to achieve optimum results.

The mechanical design of the crystallizer has a significant influence on the nucleation rate due to contact nucleation (that which is caused by contact of the crystals with each other and with the pump impeller, or propeller, when suspended in a supersaturated solution). This phenomenon yields varying rates of nucleation in scale up, and differences in the nucleation rates when the same equipment is used with different materials.

It is important in performing pilot plant tests that designs are used in which both the small- and large-scale characteristics are well known, and that the testing is performed by highly experienced personnel.

Swenson crystallization experience

The Swenson Evaporator Company, which later became the Swenson Division of Whiting Corporation, was formed in 1889 to build sugar evaporation and crystallization equipment. Since that time, Swenson has become a recognized force in the manufacture of a wide range of crystallization equipment, including evaporative, surface-, vacuum- and refrigerant-cooled crystallizers ranging in size from pilot plant equipment to units over 40' in diameter.

Swenson has long maintained a fully staffed, fully equipped laboratory for development and feasibility testing. The facilities include a rising film and falling film evaporator, a forced-circulation evaporative crystallizer, two draft tube baffle crystallizers, and a small glass unit capable of handling about three liters of slurry. Operation is typically "around the clock" and tests with centrifuges, filters and dryers can be made so that

22' diameter ammonium sulfate crystallizer designed for Shell Chemical Company to produce 600 tons of $(\text{NH}_4)_2\text{SO}_4$ per day

the laboratory product will resemble the commercial process and final product as closely as possible for customer evaluation.

In addition to having the outstanding capability to build almost any size of processing equipment anywhere in the world through its worldwide network of licensees, Swenson has an in-house staff and regionally located chemical sales engineers capable of engineering complete crystallization plants including steelwork, utilities, separation, drying and material handling equipment.

Choice of equipment

Swenson builds a wide range of general and special purpose crystallizers for the chemical process industries. For feeds where high rates of evaporation are required, where there are scaling compounds, where crystallization is achieved in inverted solubility solutions, or where the solution is of relatively high viscosity, the **forced-circulation** crystallizer is the best choice. Its most frequent use is in the continuous processing of such materials as sodium chloride, sodium sulfate, sodium carbonate monohydrate, citric acid, monosodium glutamate, urea and other similar crystalline materials.

Where excess nucleation makes it difficult to achieve a crystal size in the range of 10 to 30 mesh, the **draft tube baffle** (DTB) crystallizer is preferred (U.S. Patents 3,873,275 & 3,961,904). This crystallizer, in both the adiabatic cooling and evaporative type, includes a baffle section surrounding a suspended magma of growing crystals from which a stream of mother liquor is removed containing excess fine crystals. These fines can be destroyed by adding heat (as in an evaporative crystallizer) or by adding water or unsaturated feed solution. The magma is suspended by means of a large, slow-moving propeller circulator which fluidizes the suspension and maintains relatively uniform growth zone conditions. This crystallizer design has proven very useful for producing such materials as ammonium sulfate, potassium chloride, diammonium phosphate, hypo, epsom salts, potassium sulfate, monosodium glutamate, borax, sodium carbonate decahydrate, trisodium phosphate, urea, XP soda, etc.

For special cases requiring very low operating temperatures only achieved by extraordinarily high vacuum, the **batch vacuum crystallizer** is still a good choice, costing less to operate than a continuous vacuum crystallizer. For operation at temperatures below which it is not economically possible to use vacuum equipment, or with solutions with very high boiling point elevations, the surface-cooled crystallizer utilizing a shell and tube exchanger is supplied. For cases at extraordinarily low temperatures where refrigerants must be used to achieve operating temperatures, a direct contact refrigeration crystallizer, wherein the refrigerant can be mixed with the slurry, would be ideal, utilizing either the forced-circulation or DTB principles.

The following pages go into further depth in describing the various types of SWENSON crystallizers and briefly illustrate Swenson's design expertise in the crystallizer field, an expertise that has made Swenson the name to consider for the most carefully engineered, quality-built crystallization equipment.

Mechanical recompression crystallizer installation at BASF plant in Antwerp, Belgium

Photograph from BASF-Antwerpen, N.V.



SWENSON DRAFT TUBE BAFFLE

For superior control over crystal size and characteristics

For superior control over particle size when excessive fine crystals are present, the SWENSON draft tube baffle (DTB) crystallizer has been proven highly effective. This type of crystallizer is used primarily in the production of a variety of large-size crystalline materials such as ammonium sulfate, potassium chloride and diammonium phosphate for the fertilizer industry.

The DTB crystallizer is built in both the adiabatic cooling and evaporative types and consists of a body in which growing crystals are circulated from the lower portion to the boiling surface by means of a large, slow-moving propeller circulator. Surrounding the suspended magma is an annular settling zone from which a stream of mother liquor bearing fine crystals can be removed. These fines are separated from the growing suspension of crystals by gravitational settling in the annular baffle zone.

Fines leaving the baffle zone are sent to a following stage, settler or heat exchanger in the case of an evaporative DTB crystallizer. The mother liquor is returned to the suction of the propeller circulator after the fines have been destroyed by heating or mixing with dilute feed or water, depending on the flowsheet.

Low temperature rise at low power input

In the case of adiabatic cooling or evaporative crystallizers, the temperature rise in the circulated magma caused by the mixing of the incoming feed or heated mother liquor at the eye of the propeller is approximately 1°F and thereby limits the supersaturation rate to very low values. The boiling action is concentrated in the center of the vessel and is well distributed across the surface by means of the vertical draft tube inlet.

Crystallizers of this type typically operate with a suspension of solids ranging from 25-50% apparent settled volume. The low temperature drop at the boiling surface and the uniform distribution of boiling created by the circulation pattern minimizes crystallization buildup on the walls of the unit and extends the operating cycle. There are no close clearances where salt buildup can produce a large reduction in the rate of circulation as in other crystallizer designs.

DTB crystallizer installation at Dow Badische's Freeport, Texas plant used for the production of ammonium sulfate



Baffling allows slurry density control

SWENSON DTB equipment is especially useful in multiple stage cooling crystallizer applications where cooling of the feed solution in each stage limits the natural slurry density to a few percent. By means of the baffled zone, the operating slurry density within the crystallizer can be raised to any convenient value by regulating the slurry underflow rate and removing the remaining mother liquor from the baffle section.

Organic and inorganic chemicals produced by the SWENSON draft tube baffle crystallizer include: hypo, epsom salts, potassium sulfate, monosodium glutamate, borax, sodium carbonate decahydrate, trisodium phosphate, sodium chlorate, boric acid, MAP, urea, YP soda, etc.

Basic Principles of the SWENSON DTB Crystallizer:

- A. Growing crystals are brought to the boiling surface where supersaturation is most intense and growth is most rapid.
- B. The baffle permits separation of unwanted fine crystals from the suspension of growing crystals, thereby affecting control of the product size.
- C. Sufficient seed surface is maintained at the boiling surface to minimize harmful salt deposits on the equipment surfaces.
- D. Low head loss in the internal circulation paths make large flows at low power requirements feasible.

Advantages of the DTB Crystallizer

- A. Capable of producing large singular crystals.
- B. Longer operating cycles.
- C. Lower operating costs.
- D. Minimum space requirements, single support elevation.
- E. Adaptable to most corrosion resistant materials of construction.
- F. Can be easily instrument-controlled.
- G. Simplicity of operation, start-up and shutdown.
- H. Produces a narrow crystal size distribution for easier drying and less caking.
- I. The product size varies only slightly with large changes in production rate.

DTB crystallizer at CF Chemical in Plant City, Florida produces 70 tons of ammonium sulfate daily from acid-scrubbing waste as an antipollution measure



CRYSTALLIZERS

Direct contact refrigeration crystallization

When crystallization occurs at such a low temperature that it is impractical to use surface cooling or when the rapid crystallization of solids on the tube walls would foul a conventional surface-cooled crystallizer, a draft tube baffle crystallizer (or a forced-circulation unit) utilizing the direct contact refrigeration technique can be used,

In this operation, a refrigerant is mixed with the circulating magma within the crystallizer body where it absorbs heat and is vaporized. Refrigerant vapor leaves the surface of the crystallizer similar to water vapor in a conventional evaporative crystallizer. It then must be compressed, condensed and recirculated to the crystallizer to maintain continuous operating conditions. Refrigerants must be relatively insoluble in the solutions processed and have the thermodynamic characteristics to minimize compressor horsepower.

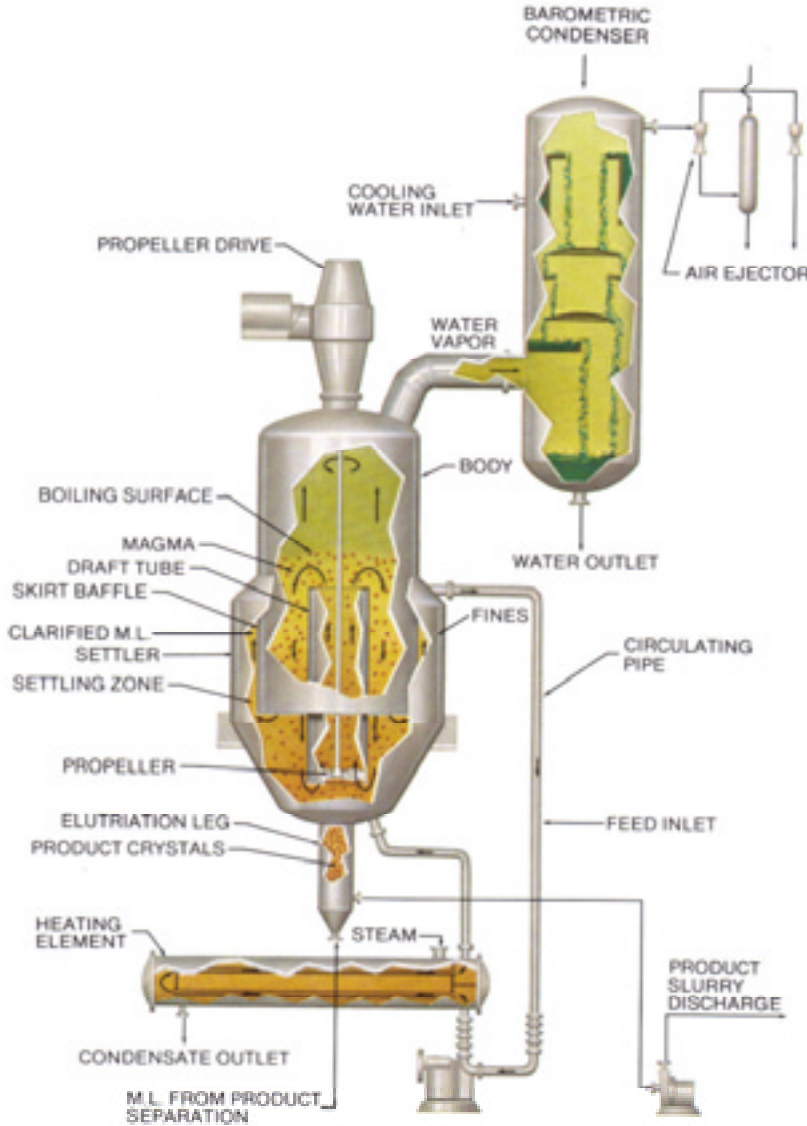
Examples are the crystallization of caustic dihydrate with freon or propane, and that of paraxylene with liquid propane refrigerant.

Reactive crystallization

Reactive crystallization, where a solid phase crystalline material results from the reaction of two components, can often be performed more profitably in a crystallizer than in a separate reactor. An example of reactive crystallization is the production of ammonium sulfate from liquid or gaseous ammonia and concentrated sulfuric acid.

The draft tube baffle crystallizer is particularly suited for reactive crystallization. The reactants are mixed in the draft tube of the DTB unit where a large volume of slurry is mixed continuously with the materials to minimize the driving force (supersaturation) created by the reaction. Removal of the heat produced by the reaction is accomplished by vaporizing water or other solvents as in a conventional evaporative type crystallizer.

Reactive crystallization can also be performed in a forced-circulation type crystallizer where the reactants are mixed in the circulation piping.



SWENSON DTB Crystallizer

Eight SWENSON DTB units produce potassium chloride from Dead Sea brine at a rate of 58 tons per hour



Two DTB Crystallizers used for the production of potash at Cleveland Potash, England



SWENSON FORCED-

For applications where high evaporation rates are required

For feeds where high rates of evaporation are required, where there are scaling components, or when crystallization must be achieved in solutions with inverted solubility or relatively high viscosity, the SWENSON forced-circulation crystallizer is the best choice.

This type of unit - also known as the circulating magma crystallizer or the mixed suspension-mixed product removal (MSMPR) crystallizer - consists of a body sized for vapor release with a liquid level high enough to enclose the growing crystals. Suction from the lower portion of the body passes through a circulation pump and a heat exchanger and returns to the body through a tangent or vertical inlet. The heat exchanger is omitted when adiabatic cooling is sufficient to produce a yield of crystals.

The most common use of this crystallizer is as an evaporative crystallizer with materials having relatively flat or inverted solubility. It is also useful with compounds crystallized from solutions with scaling components.

Controlled supersaturation

When the heat exchanger is used, it is normally one- or two-pass and is designed for relatively low temperature rises in the solution. This limits the supersaturation of scaling components when heating materials of inverted solubility. In most applications, the steam-to-liquid Δt is also limited to prevent mass boiling within the tubes or vaporization at the tube wall.

40'6" diameter forced-circulation mechanical recompression evaporative crystallizer at the Montedison plant in Ciro Marina, Italy

SWENSON forced-circulation crystallizer used for producing sodium sulfate at American Cyanamids Fort Worth, Texas facilities



CIRCULATION CRYSTALLIZERS

Most widely used crystallization method

Forced circulation is the most widely practiced method of crystallization. PC crystallizers are found in sizes ranging from 2' diameter laboratory models to over 40' diameter units for continuous processing. Per pound of product, it is ordinarily the most inexpensive type of equipment available, particularly when substantial amounts of evaporation are required.

Although SWENSON forced-circulation crystallizers can be operated on a batch basis, their most frequent use is for continuous processing of such materials as sodium chloride, sodium sulfate, sodium carbonate monohydrate, citric acid, monosodium glutamate, urea and similar crystalline materials.

Recompression crystallization

For applications where the solution's boiling point elevation is low, such as for sodium carbonate, it is possible to significantly reduce the cost of the operation by vapor recompression.

In this technique the vapor, which is removed to precipitate the crystalline product, is compressed by a positive displacement or centrifugal compressor to a pressure high enough so that it may be used as steam in the heat exchanger.

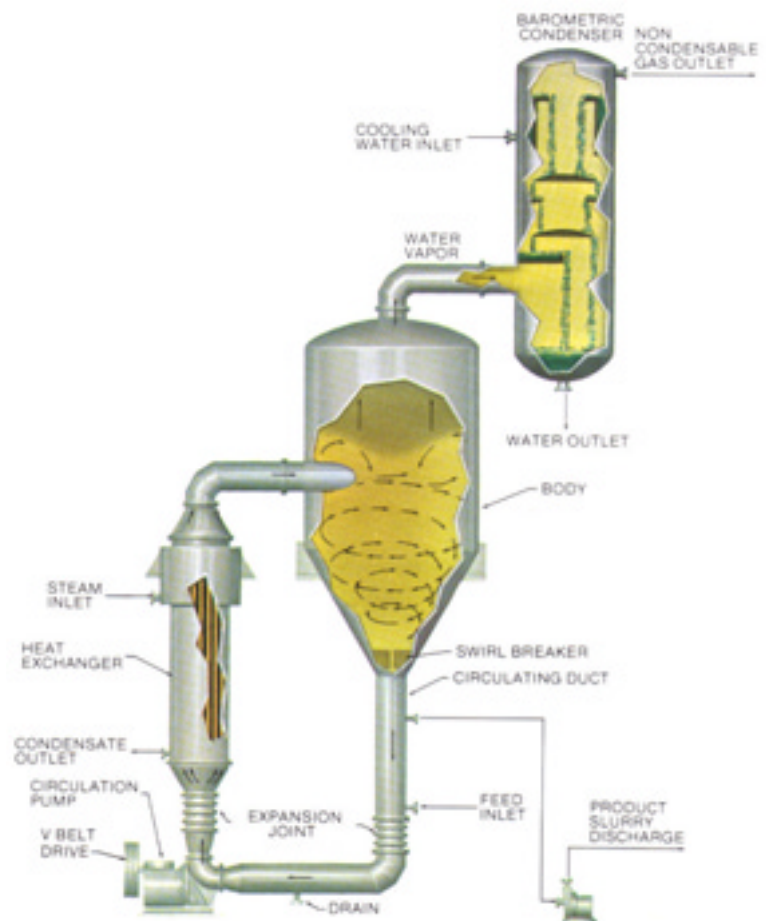
By proper heat exchange, many mechanical recompression applications can operate efficiently without any external source of heat, except for start-up purposes. For other applications, a small quantity of steam is required at the heating element to maintain the system on a continuous basis.

The drive for the compressor may be either a steam turbine or an electric motor depending on the availability and cost

of power. To minimize power requirements, the heat exchanger is designed for the lowest possible Δt , thereby minimizing salting and scaling.

A steam booster can be used in lieu of a mechanical compressor when steam costs are relatively inexpensive compared to electrical power costs. In this case, part of the vapor is compressed by the high pressure steam in the booster and used as the first-effect heating media. Vapor not compressed goes to the second effect. The recompression crystallization technique can also be applied to the DTB or SWENSON OSLO (growth type) crystallizers.

Forced-circulation thermal recompression crystallizer at the FMC Corporations Middleport, New York facility



SWENSON Forced-Circulation Crystallizer

SWENSON BATCH VACUUM CRYSTALLIZERS

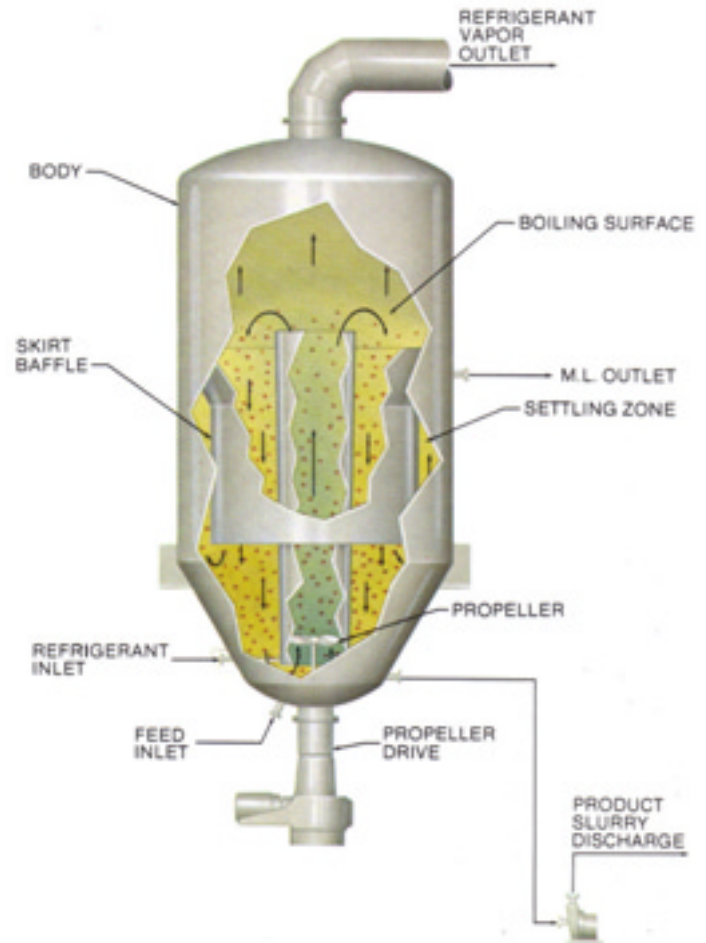
For cooling through a wide range of temperatures or when batch sizes are most economical

For special cases requiring very low operating temperatures achieved only by very high vacuum, and for those applications involving relatively small amounts of material— or when the material being processed must be handled on less than a continuous basis— it is often both convenient and economical to use a SWENSON batch vacuum crystallizer.

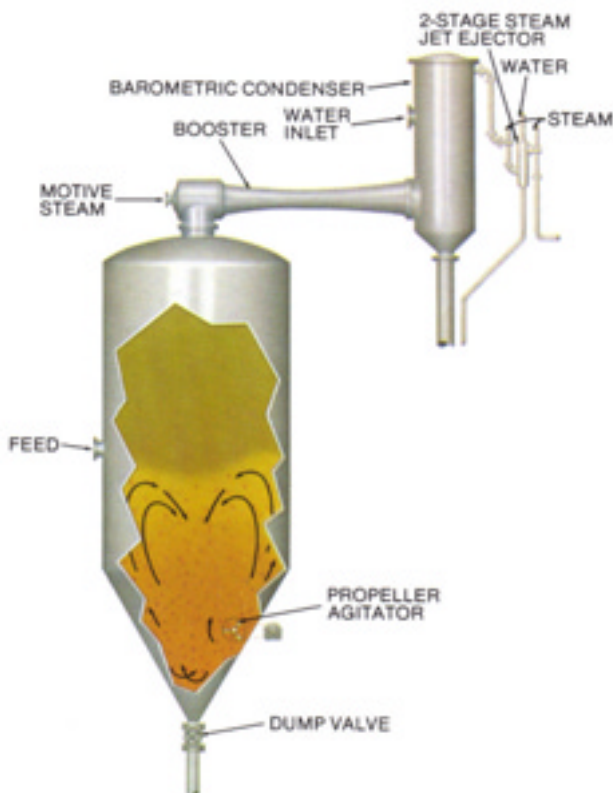
Cycles on batch equipment range from two to eight hours, not including loading and unloading time. At the conclusion of the cycle, the material is deposited in an agitated tank from which it is removed on either a batch or continuous basis for separation and drying. The entire cycle for such equipment may be automated.

Where the material is cooled through a very wide range and! or to a final temperature which requires a very high vacuum, a large ejector or booster is utilized to compress the vapor to a pressure high enough for condensation with available cooling water. In such cases, the batch vacuum crystallizer's economies of steam usage are approached only by multiple stage continuous equipment of five or more stages.

An added advantage of the batch vacuum type of crystallizer is its capacity for self-cleaning, which is particularly helpful when dealing with materials prone to grow on the walls of continuous crystallization equipment.



SWENSON Direct Contact Refrigeration DTB Crystallizer



SWENSON Batch Vacuum Crystallizer With Booster

Top view showing booster of SWENSON batch vacuum crystallizer for producing Glaubers Salt at Foote Mineral, Kings Mountain, N.C.



SWENSON SURFACE-COOLED CRYSTALLIZERS

For low temperature or high boiling point elevation jobs

For operation at temperatures below which it is not economically feasible to use vacuum equipment, or for solutions with very high boiling point elevations, the SWENSON surface-cooled crystallizer is generally specified.

The surface-cooled unit is a type of forced-circulation crystallizer consisting of a shell and tube heat exchanger through which is pumped the slurry of growing crystals, a crystallizer body to provide retention time, and a recirculation pump and piping. Within the crystallizer body is a baffle designed to keep excessively fine crystals separated from the growing magma for size and slurry density control purposes.

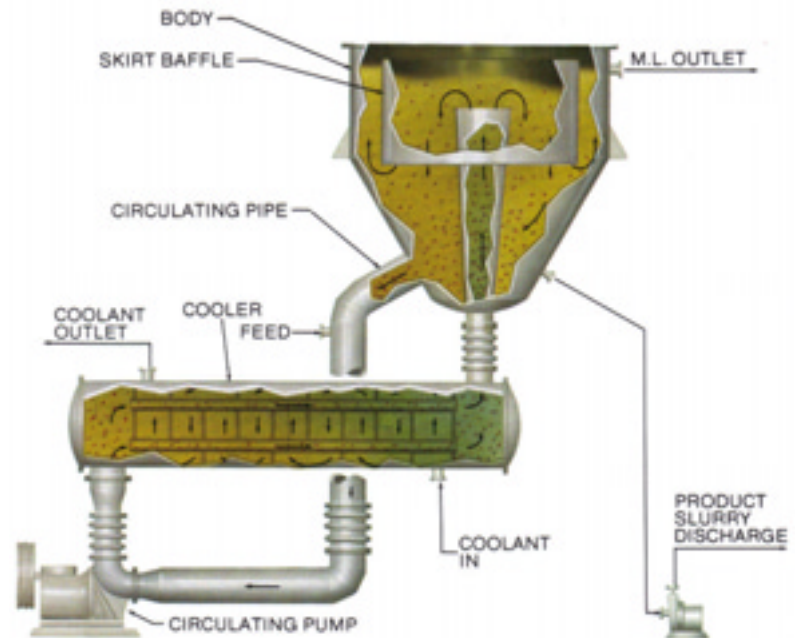
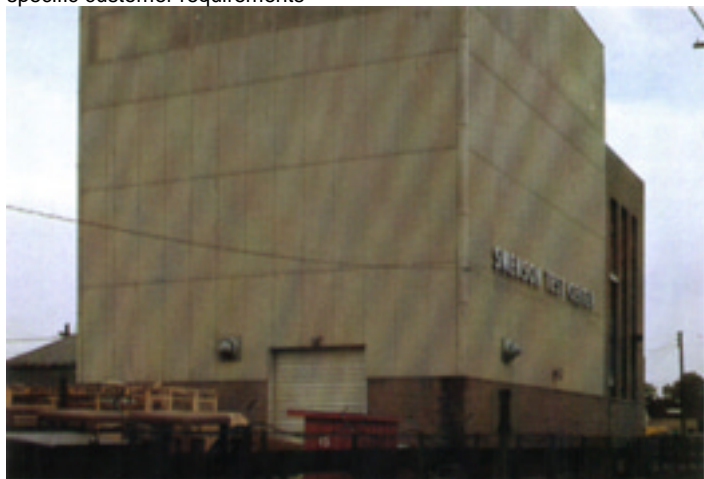
The circulation rate through the heat exchanger is normally high enough so the temperature drop is in the range of 1-2°F. Surrounding the tubes is the cooling media- either a tempered water circulated through segmented baffles or a vaporized refrigerant.

Because the tube wall is the coldest part of the crystallizing system, the temperature differences between the wall and the slurry being pumped through the tube must be as small as practical. Practical values depend on the operating cycles and the properties and characteristics of the materials. Temperature differences ranging from 5 to 15°F are required to achieve reasonable operating cycles.

Surface-cooled crystallizers are used where the solution's boiling point elevation is extremely high, as in the case of caustic solutions, or when the temperature level is so low evaporation by vacuum is impossible.

Typical applications for the SWENSON surface-cooled crystallizer are in processing sodium chloride from caustic solutions, sodium carbonate decahydrate from waste solutions, and sodium chlorate from solutions saturated with sodium chloride.

Swenson test Center contains fully equipped laboratory for determining specific customer requirements



SWENSON Surface-Cooled Crystallizer

Surface-cooled forced-circulation crystallizer at Domtar Chemicals, Ltd., Toronto, Ontario, Canada





Shop view of Swenson vessel in fabrication

INFORMATION REQUIRED FOR EVALUATION

Before a potential crystallizer application can be properly evaluated, it is necessary to have certain basic information regarding the material to be crystallized and its mother liquor.

Is the material hydrated or anhydrous? What is the solubility of the compound in water or any other solvents under consideration and how does this change with temperature? Are there other compounds in the solution which co-precipitate or remain in solution? If so, how does their presence affect the solubility of the main component? What will be the influence of the impurities on the crystal habit and the growth and nucleation rates? What is the production rate and on what basis is the production rate computed? What materials of construction can be used in contact with the solutions at various temperatures? What utilities will be used at the crystallizer location and what are the costs associated with these utilities?

These and other questions should be considered in completing a data sheet which should accompany an inquiry for crystallization equipment. In preparing the inquiry data sheet, use one of the enclosed forms or, if none of these are available, prepare a new sheet following the basic format shown on the next page.

When your inquiry is received, Swenson engineers will welcome the opportunity to survey your crystallization needs and quote on your requirements.



SWENSON two-stage forced-circulation evaporative crystallizer at Armour Agricultural Chemical Company

Model of proposed potash plant illustrates scope of Swenson project engineering



SWENSON[®] CRYSTALLIZER CHECKLIST

CHECKLIST OF DATA REQUESTED ON CRYSTALLIZER INQUIRIES

Fill in or attach as much information as is available so the most economic design can be prepared.

1. Product being crystallized _____	Production Rate _____
2. Liquid Properties	Feed Mother Liquor Purge
Rate	_____
Temperature	_____
Composition _____	_____
_____	_____
_____	_____
Density	_____
Viscosity	_____
K (thermal conductivity)	_____
Cp (specific heat)	_____
Boiling point elevation	_____
3. Evaporation Rate _____	
For organic vapors only give latent heat of evaporation/specific volume/vapor viscosity _____	

4. Solubility of Solute in solvent at various temperatures (solubility curve) or phase diagram if several solutes are present.	
5. Product	Heat of Reaction if Reaction in Crystallizer Produces Crystals
Heat of Crystallization	Specific Gravity
_____	_____
_____	_____
_____	_____
_____	_____
6. Crystal shape if known _____	
7. Product size required _____	
8. Suitable materials of construction for operating temperature range: Bodies _____ Tubes _____ Pumps _____ Vacuum Equipment _____	
9. Type of seals required in agitators or pumps and their materials of construction _____	

10. Gasket material required if known _____	
11. Utilities Available	Steam Pressure Cooling Water Temperature Current Characteristics
_____	_____
_____	_____
12. Describe flowsheet required or used and whether one or several bodies are required (or the economic factors which are to be used in determining the number of stages or effects. These factors include steam cost, water cost, electricity cost, amortization period, etc.)	
13. Any other data which the customer feels would be of help such as heat transfer data: present conditions under which the product is produced and type of equipment used; present crystal size; and effects of agitation, additives and/or temperature on crystal size, shape, recovery, or purity.	
14. If the solution is organic and test work is desired, give the flash point of the solution _____	
