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SODIUM CHLORIDE: CRYSTALLIZATION

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Introduction

Salt has been a part of human existence since time immemorial. It was used for cooking wheat and barley as early as 5000 BC. The first salt was gathered from shallow lagoons where seawater could evaporate. Later rock salt was mined, and in the Alps, for instance, rock salt is known to have been mined as early as 1400 BC. Because of its importance to human life, salt has had an influence on economy, history and culture. Many sayings and words are derived from the use of salt: e.g. ‘to be worth one’s salt’ is a compliment, the Bible speaks of ‘the salt of the earth’ and soldier is derived from the Latin ‘*sal dare*’ which means to give salt.

Indeed, in ancient times salt had a much greater value than it has nowadays. It was traded weight by weight with gold, and the salt trade was very profitable. The Hanseatic League started by trading in salt. Taxes on salt were very common, and in that sense salt has played a role in many important historic events. The French Revolution was partly in protest against salt taxes. Gandhi’s campaign of civil disobedience, which eventually led to the independence of India, started when he evaded the British salt monopoly by producing salt himself.

Uses of Salt

Before the industrial revolution and the discovery of the electrolysis process, the uses of salt were limited. The main uses were for the cooking, preserving and pickling of food and the tanning of hides. These uses are still very important, as salt is essential for the human body. With the development of chemical processes the uses of salt have diversified enormously. Apart from uses in the food industry, salt is, for instance, used in dyeing, paper production, highway de-icing, oil well drilling and the production of soda ash. Electrolysis of salt is the major source of chlorine and sodium hydroxide for the chemical industry. Chlorine is essential for the production of a number of plastics, insecticides and pharmaceutical compounds. Either directly, or in the form of derivatives, salt finds application in more than 14 000 ways. This multitude of applications can be divided into three major categories: chemical uses, highway de-icing, and food-related uses. In the industrialized nations the chemical industry accounts for approximately 50% of the salt consumption, and highway de-icing for about 30% while food applications make up the remainder. In developing countries most of the salt produced is used in food.

Production of Salt

As salt is the most abundant nonmetallic mineral, most countries have the ability to produce salt. It is so abundant that it is hard to estimate salt reserves. In the United States alone, reserves are estimated at 55 trillion tonnes. In 1996, 192 million tonnes of salt were produced, approximately 55% in the industrialized nations and about 45% in developing countries. Details are given **Table 1**.

Table 1 World production of salt in 1996

Country	*Amount produced (in millions of metric tons)*
United States	42.9
Peoples' Republic of China	28.9
Canada	12.3
Germany	10.9
India	9.5
Mexico	8.5
Australia	7.9
Other	71.8
Total	192.0

Salt is a cheap commodity. At a 1997 price level of US $60 per tonne for chemical grade, merchant-delivered salt and US $10 per tonne for captive use, salt is cheaper than any other refined chemical. Because of the low price of salt relative to the transportation costs, most salt production plants are in the vicinity of salt users. Producing an acceptable grade of purity at the lowest deliverable costs is the most important consideration for salt producers throughout the world.

How the salt is produced depends very much on the form in which the salt is available. In subtropic, arid regions salt is mostly produced by evaporating seawater. Large production facilities for so-called solar salt can be found in India, Australia and Mexico. In temperate regions, where the climate is less favourable for the evaporation of seawater, rock salt is mined. This can be done in two ways. Provided the salt deposit is close to the surface, it can be mined in the classical roof and pillar method. The rock salt is crushed, sorted and sold as a low grade quality. For more demanding applications further purification is needed. If the salt deposits are more deeply located, the solution mining technique is used. This involves pumping water down through a borehole to dissolve the salt, and recovering the resulting brine. The brine is then purified to remove foreign ions, and evaporated. The salt produced in this way is known as vacuum salt.

Fundamentals of Salt Crystallization

Salt or sodium chloride can occur in two forms. The first and best known form is the anhydrous form, NaCl. The second form is the dihydrate which is formed in a pure brine at temperatures below 0.1°C. The solubility of the dihydrate form is weakly temperature dependent, whereas the solubility of the anhydrous form is nearly temperature independent. The temperature dependence of the solubility of salt in water is given in **Figure 1**.

The anhydrous form, $NaCl \cdot 0H_2O$, crystallizes in the *Fm*3*m* space group in which each sodium ion is octahedrally coordinated by six chloride ions, and vice versa. The $\{1\ 0\ 0\}$ faces are the slowest growing faces, resulting in the typical cube shape of salt crystals. By adding additives the $\{1\ 1\ 1\}$ faces can be retarded, thus yielding octahedral shapes. Additives such as $Fe\ (CN)_6^{4-}$ or NTAA (nitrosyl triacetamide) poison the $\{1\ 0\ 0\}$ surfaces, resulting in preferential growth along edges and on corners. These shapes are given in **Figure 2**, together with a number of intermediate shapes where both cubic and octahedral faces are visible.

As mentioned before, salt is very soluble in water, so the growth rate of salt crystals is diffusion controlled. Like many other very soluble salts, the driving force needed to obtain acceptable growth rates (in the order of 10^{-8} m s^{-1}) is low, typically of the order of 0.1% or lower. Salt crystals produced in a forced circulation crystallizer (the most common type used for salt crystallization) typically have a mean size of 350–400 μm. In draft tube baffled (DTB) type crystallizers salt crystals can become larger, in the order of 500–1000 μm. Apart from the influence on the mean crystal size, the low driving force for crystallization also strongly reduces agglomeration. Agglomerated salt can only be obtained using techniques such as antisolvent crystallization where high driving forces are involved. There are three mechanisms for the incorporation of impurities in the final crystalline product. The first is direct incorporation of the impurity in the crystal lattice, the second is the formation of inclusions and the third is insufficient washing of the crystals.

In contrast to organic crystals, and to a lesser extent hydrated salt crystals, the ions are densely packed in the crystal lattice. This effectively prevents the incorporation of larger molecules in the crystal lattice as the lattice strain and the enthalpies involved are extremely unfavourable. This applies to many ions that have marked differences in ionic radius or charge from either the chlorine or sodium ion. The

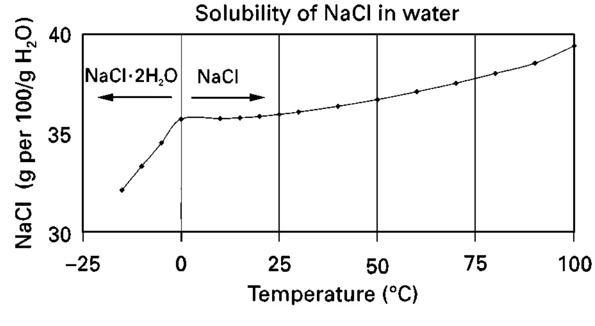

Figure 1 Solubility of NaCl in water.

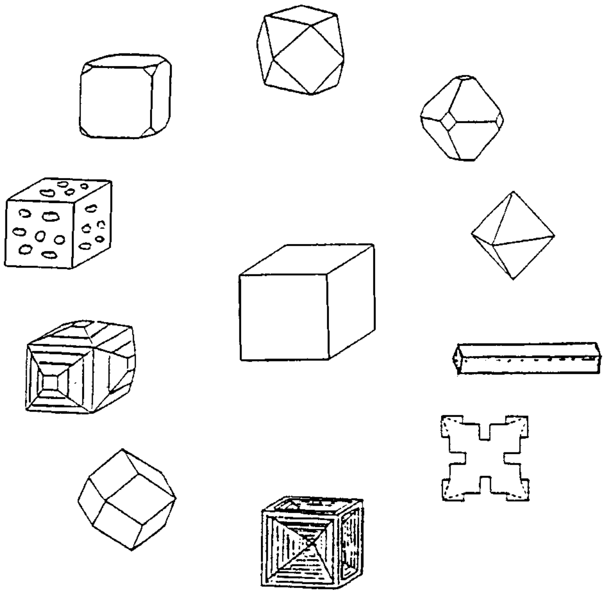

Figure 2 Different crystal forms observed during the crystallization of common salt. Reproduced with permission from Elsevier Science.

tendency of salt to form solid solutions is therefore very limited. The only notable exception is with the incorporation of bromide, which is therefore very hard to remove as an impurity, once incorporated.

The second mechanism, the formation of inclusions, is much more common. In industrial crystallization the solid fraction in the slurry is high and therefore collisions between crystals are frequent. If the energy involved in a collision event is high enough the corners of the cubic salt crystals will be damaged and the crystals will be strained. Regrowth of these damaged corners is often imperfect and inclusions are formed. An effective method of reducing the impurity uptake through this mechanism, though at the cost of production capacity, is to lower the solids fraction of the slurry. It should be noted that this mechanism only occurs when large crystals are produced. If the crystals are small the kinetic energy involved in the collisions is not high enough to damage the crystals.

Good washing of the crystalline product is very important for the final purity. By evaporating water not only is salt produced, but the impurities are also concentrated. Any mother liquor that remains will therefore have a profound detrimental effect on the product purity. More impure mother liquors require better washing. This is more important for solar salt than for vacuum salt. Vacuum salt is produced from purified brine that contains, apart from NaCl, few very soluble salts, so despite incomplete washing the amount of impurities will still be relatively low. Concentrated seawater, in contrast, also contains high concentrations of very soluble salts, especially magnesium salts. Insufficient washing therefore has much more influence on the product purity. Another important aspect when washing solar salt is the agglomeration.

A striking example of the influence of agglomeration on the final product purity is provided by the antisolvent crystallization of NaCl. Though the crystals are crystallized from a mother liquor containing as much as 50% weight antisolvent (on a solvent basis) the uptake of the antisolvent is as low as 30 ppm. Diluting the antisolvent stream with water, which reduces the driving force for crystallization, should result in a decrease in the impurity concentration. However this effect is offset by the increased agglomeration of the system, so instead of the expected lowering of concentration the concentration of antisolvent is increased to 100 ppm.

Irrespective of the method by which salt is produced, seawater is the source of salt (salt deposits are the result of natural solar salt production). The impurities present in brine or rock salt are therefore the same. These impurities are mainly Ca^{2+}, Mg^{2+}, Sr^{2+}, Br^- and SO_4^{2-}. All these impurities have undesirable effects on the electrolysis of sodium chloride and need to be removed during the production process. During electrolysis calcium, strontium and magnesium are deposited as hydroxides on the electrodes, which is of course not desired. The presence of Br^- leads to the formation of ClBr, which is also unwanted. Finally the presence of sulfate increases the cell potential needed for electrolysis, thus increasing production costs. A further impurity is iron, which is not only present in the salt, but is also added in the form of ferrocyanide, an anticaking agent. Though needed for proper salt handling, the presence of iron interferes with the membrane electrolysis. The ferrocyanide must therefore be decomposed and the iron precipitated as the hydroxide salt prior to electrolysis of the sodium chloride solution.

The strategy for removing these impurities depends on the production method. For solar salt production fractional crystallization combined with careful washing is employed, whereas in vacuum salt production the brine is purified prior to crystallization of the sodium chloride. In the rock salt production various recrystallization methods are used.

Solar Salt

Crystallization sequence Seawater contains nearly all elements of the periodic system in varying amounts. The composition of seawater is given in **Table 2**. When seawater is evaporated many different salts will be formed, at different stages during the evaporation. In that sense production of pure sodium chloride from seawater somewhat resembles

Table 2 Composition of seawater

Component	*Amount present (g per 1000 g seawater)*
Ca	0.408
SO_4	2.643
Mg	1.265
Cl	18.95
K	0.380
Na	10.48
Br	0.065
Total	34.19

the distillation of crude oil, where one is interested in obtaining well defined fractions. When seawater is concentrated gradually iron oxide and calcium carbonate start to crystallize first, but the amount of iron oxide produced is negligible. Then calcium sulfate precipitates. It is important to note that when sodium chloride is subsequently crystallized, the mother liquor is concentrated with respect to both of the salts mentioned. After crystallizing about 75% of the available sodium chloride (at which stage 97% percent of the water has been evaporated), sodium bromide will start to crystallize as a solid solution with sodium chloride, and the so-called bitterns will also crystallize. The term bitterns is used for a collection of magnesium, potassium, sulfate and chloride salts, such as KCl, $MgCl_2$, $MgSO_4$ and double salts. It is derived from the bitter taste of these salts. The whole sequence is depicted graphically in **Figure 3**. This figure shows the relationship between the density of the brine expressed as degrees Baume (°Be = 145 − (145/specific density at 15.6°C)) and the crystallization of the various salts. The concentration factor can be deduced from the cumulative amount of water evaporated, which is also given in the figure.

Plant layout To produce pure salt, the crystallization of iron oxide, calcium carbonate and calcium sulfate must be physically separated from the sodium chloride crystallization. This is achieved in solar salt works by having two kinds of ponds: concentration ponds and crystallizer ponds. Approximately 90% of the water must be evaporated before salt starts to crystallize, so the concentration ponds are much larger than the crystallizer ponds. Though the water is not completely evaporated in the crystallization section (the brine is discharged before the bitterns start to crystallize), the concentration pond/crystallizing pond area ratio is usually around 10 to 1. Evaporation is a slow process, so solar plants must occupy a large area. The solar salt plants in Australia and Mexico, which supply the chemical industries in Japan and the United States, are tens of square kilometres in size. The seawater needs to be concentrated in stages so most solar salt plants have 5–10

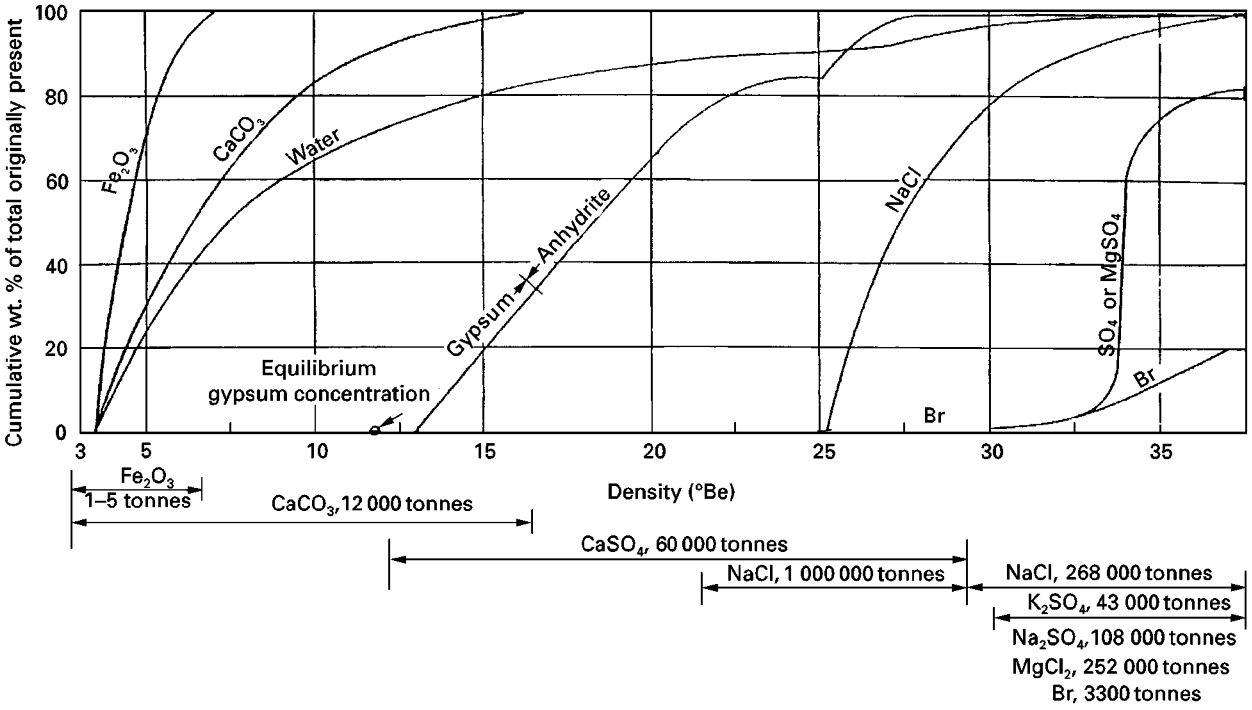

Figure 3 Deposition of salts during the evaporation of sea water at 25°C.

concentration ponds in series. These ponds are shallow to obtain the best surface area/volume ratio, with the depth usually between 50 and 80 cm. Small, low levees separate the different ponds. The crystallizer ponds are smaller, ranging from several hundred square metres in the case of manual harvesting to several acres in the case of mechanical harvesting. After harvesting the salt is transported to the washing plant. An example of the layout of a solar salt plant is given in **Figure 4**.

Solar salt production The need for large shallow ponds in the vicinity of the sea determines where solar salt plants can be operated. Small plants can be found in all coastal areas near the tropics, large plants only in India, Australia and Mexico. Such plants are operated all year round. Further north than the tropics, in arid areas as far north as western France, the operation is seasonal; the salt is harvested before the winter rains dissolve the salt produced.

Production of solar salt is started by taking in seawater. The seawater is concentrated by evaporation and the brine is reduced to about 60% of its original volume. After the first concentration stage the brine is transferred to another area where calcium carbonate starts to precipitate. Here a further 15% of the original volume is evaporated and the brine is transferred to a third type of pond, the gypsum precipitation pond. The brine is concentrated to the point at which sodium chloride nearly starts to crystallize before it is transferred to the pickling pond where the brine, now saturated with sodium chloride, is kept before being transferred to the crystallizer pond. This pond is needed to reduce the gypsum supersaturation in the brine. At this point about 90% of the water has been evaporated. In the crystallizers, depending on the required purity, 70–75% of the available sodium chloride is crystallized before the remaining bitterns are discharged. Production of high quality solar salt is very much a question of knowing when to start producing halite and when to stop.

A major concern is the sealing of the ponds to prevent losses. It is impossible to treat the bottom of the ponds because of their large size, so the ponds must have a base such as clay, which is (fairly) impervious to water. The precipitated calcium carbonate (and also gypsum) will improve the sealing during operation so the losses will go down with time. By regularly changing the brine flow through the plant, all concentration ponds will be used as calcium carbonate and gypsum precipitation ponds, thus ensuring minimum brine losses (provided of course that the ponds are equal in size). Note that this is only possible in small plants.

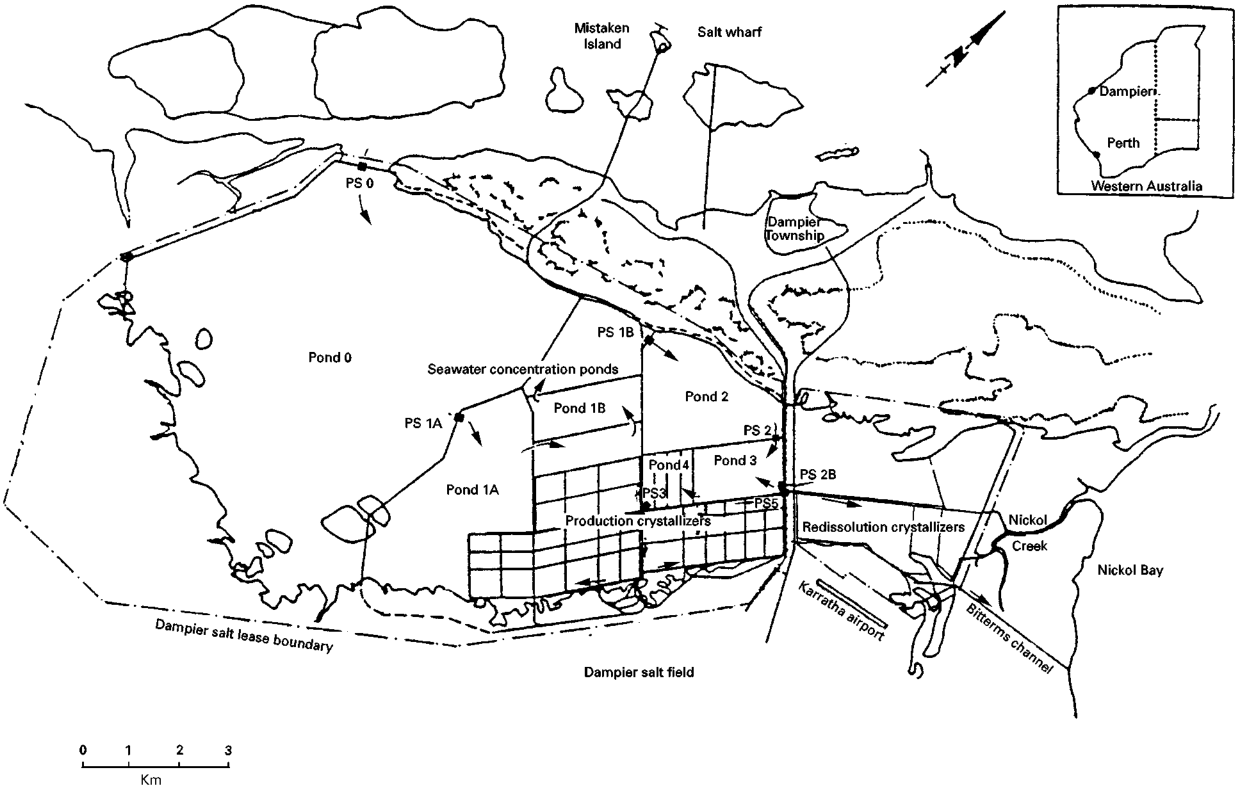

Figure 4 Schematic diagram of Dampier salt field layout and location map of Western Australia. Reprinted from Garrett DE, with kind permission from Elsevier Science Ltd.

The transmission of solar light in the brine is high, which reduces the evaporation rate. To enhance sunlight absorption dyes are added. The most common practice nowadays is to add algae which reduce the light transmission from 96% to 55–70%. An additional advantage is that the algae mats will also plug the pond bottom. Care must be taken not to increase the viscosity too much through abundant algae growth, or other organisms have to be introduced to keep the algae concentration within limits. Red halophilic bacteria are added to the crystallizing ponds for the same purposes.

The rate at which water evaporates depends on the climate. The amount of sunshine, the mean temperature, the relative humidity, the wind velocity and the average amount of precipitation determine the net evaporation rate. Accurate figures for the net evaporation are needed both for design purposes and for process control (read brine management). For design purposes climate data (incorporating amount of sunshine, air temperature and humidity and wind velocity) and brine data are taken into account. The gross evaporation rate can then be calculated by multiplying the difference in vapour pressure between air and brine by a mass transfer coefficient. In this mass transfer coefficient factors such as temperature, net gain of radiant energy and the heat transfer coefficient are taken into account.

For production purposes another model is generally used. The evaporation of water from a fresh water pan, situated on the site, is measured. This figure is then corrected for the salinity, the size of the pan and the rainfall.

$$\begin{aligned}\text{Evaporation (pond)} = {} & (\text{evaporation (pan)} \\ & \times k_{\text{scale}} \times k_{\text{salinity}} - \text{rainfall}) \\ & \times \text{area} \times \rho_w\end{aligned}$$

Here k_{scale} and k_{salinity} the scale factors and ρ_w is the density of water. Using this method it is possible to estimate how much has been evaporated from each pond, to decide when to pump brine from one pond to another, to take in sea water, etc.

In the crystallizer ponds a salt floor is formed during the crystallization of sodium chloride. The salt is harvested by completely removing the salt floor (in seasonal operations) or scraping the top layer (in year round operations). When the salt is mechanically harvested great care should be taken that the floor is strong enough to accommodate heavy equipment. Usually salt floors for mechanical harvesting are at least half a metre thick.

After harvesting the salt is washed by mixing the crystals with fresh saturated brine, and transferred to a mesh conveyor where the brine is drained off. Generally sea water sprays are then used to finish the washing process. Provided the washing is carried out very thoroughly, and the crystals are crushed to remove the mother liquor contained in cavities, solar salt can reasonably pure, though not as pure as vacuum salt. In most cases solar salt contains significantly more magnesium (300–500 ppm), calcium (200–300 ppm) and sulfate (1000–1500 ppm) than vacuum salt.

The salt can be upgraded using the Salex® process. This process utilizes the difference in density and morphology between sodium chloride and other minerals. The salt crystals containing the impurity crystals are countercurrently washed with a saturated brine. The salt crystals settle, and the impurity (mainly gypsum) crystals are carried away with the brine and left to settle in a separate tank. The clarified brine can then be reused. By leaving the produced salt piles exposed to rain, impurities will preferentially dissolve, thus improving the salt quality. This is called the rain wash method.

Rock Salt

Provided the rock salt deposits are close to the surface, rock salt can be mined in the classical way. First a vertical shaft is dug until the salt bearing deposit has been reached. Then horizontal shafts are blasted and the salt thus produced is transported to the surface. For de-icing use the salt is only crushed and sorted by size. For chemical applications the rock salt must be upgraded. This can be achieved in several ways. First of all, the rock salt can be completely dissolved. The resulting brine is then treated in the same manner as brine obtained by solution mining. As this process is more expensive than solution mining it is hardly ever used. The second option is to recrystallize the brine by making use of the fact that sodium chloride also has a hydrated solid phase.

This process works as follows. First small, crushed salt crystals are suspended in a brine at a temperature of 5°C. The brine is then cooled to a temperature lower than 0°C. At this temperature NaCl is more soluble than the hydrated phase, $NaCl \cdot 2H_2O$. The anhydrous sodium chloride crystals will dissolve and sodium chloride dihydrate crystals will be formed. When the slurry now containing the dihydrate crystals is heated, the process is reversed. The dihydrate crystals will dissolve and anhydrous crystals will be formed. During the two consecutive crystallization steps the impurities present in the dissolving crystals will remain to a large extent in the brine, instead of ending up in the new crystals formed. Using this method, the purity of the salt can be markedly improved without having to evaporate the brine. After the recrystallization steps the now purified crystals are separated from the brine and the brine is reused after treatment.

Vacuum Salt

Brine purification Vacuum salt is the name given to salt produced by evaporative crystallization. Water is injected into a salt deposit through a borehole, and dissolves the salt so the resulting brine can be recovered. This brine is then purified before salt is produced by evaporative crystallization. The deposits used for this kind of salt production lie at depths between a few hundred and three thousand metres. Two pipes are used, one bearing fresh water to the cavern, the other transporting brine to the surface. Because of surface subsidence and ground movements, the caverns cannot get too large. Normally the size is smaller than 100 m in width. Furthermore, above the cavern a layer of a few metres of salt must be maintained to protect the overlying strata from brine penetration. Once the cavern has reached its maximum allowable size, solution mining is stopped and a new borehole is drilled.

It is interesting to note that the salt mined in such a way has already been crystallized once. All salt deposits are remains from earlier lakes and seas, which have been evaporated. Thus the salt is already separated from the calcium sulfate and bitterns originally present in the seawater.

Though already purer than the saturatd brine produced by seawater evaporation, the brine produced still contains significant amounts of calcium, magnesium, sulfate and bromide ions. Most of these ions are removed in the brine purification process. First the sulfate is removed by adding calcium oxide, which leads to the precipitation of gypsum. The oxide increases the pH of the brine, which induces the precipitation of magnesium hydroxide. Thus in the first stage sulfate and magnesium are removed. To reduce production costs this process is carried out using very simple equipment. Calcium oxide is simply mixed with the brine in a very large tank. To decrease the supersaturation further tanks are used. The solids are removed by draining the tanks and emptying them.

A drawback to this procedure is the increased calcium level in the brine. To reduce this level gases from an on-site power plant are usually used. The carbon dioxide yields carbonate, due to the high pH of the brine, which together with calcium forms the almost insoluble calcium carbonate. Because of the high salt concentration and high temperature, vaterite is formed rather than the usual calcite. This is an advantage because strontium, which is also present in the brine, is also effectively removed as strontium carbonate forms a solid solution with vaterite.

Crystallization After purification the water is evaporated and salt is crystallized. To conserve energy this is done in several stages. Each stage is operated under a lower pressure so the brine boils at a lower temperature. The vapour produced by the previous stage condenses in a heat exchanger and as this steam was formed at a higher temperature, the brine starts to boil. In this way the steam used in the primary stage can be reused several times. This principle is shown in **Figure 5**. Thus the total amount of energy involved in the production of vacuum salt is greatly reduced. In a typical four-effect installation (see **Table 3**) the first effect operates at slightly elevated pressures while the other effects operate under reduced pressure, hence the name vacuum salt.

Generally salt crystallizers are of the forced circulation type with external heat exchangers. For a production of 1 million tonnes per year, a total crystallizer capacity of 800–1000 m^3 is needed. The separate crystallizers can be operated in different modes. The salt produced can be separated from the mother liquor separately for each crystallizer, or the slurry can be transported from one effect to the other, thus increasing the solids content of the slurry in each successive stage. This has important implications for the purity. In the first case the salt produced in the first crystallizer is the purest. Little water has (yet)

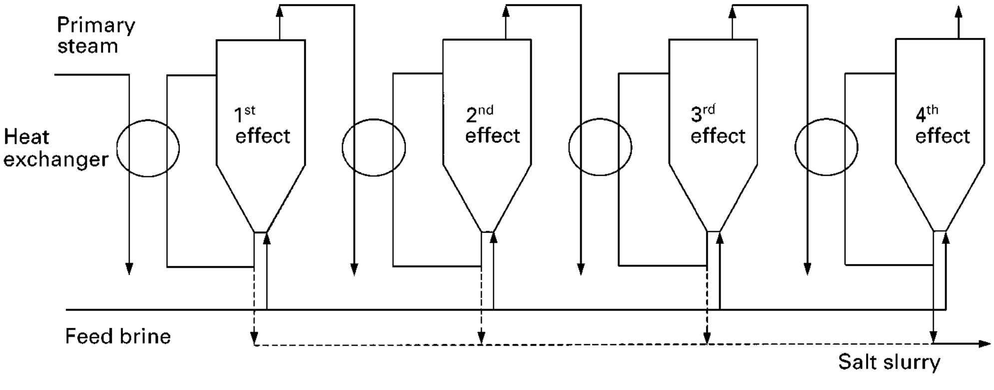

Figure 5 Scheme of a four effect evaporative crystallization plant.

Table 3 Temperatures and pressures in a four-effect evaporative crystallization plant

Effect	*1*	*2*	*3*	*4*
Pressure (bar)	2.0	0.89	0.34	0.18
Temperature (°C)	120	96	72	58

been evaporated and the impurity concentration is low. In the successive crystallizers the impurities are gradually concentrated, which has a detrimental effect on the purity. Thus different grades of salt are produced.

When the slurry is transported from one effect to the other, only one grade of salt is produced, which represents the mean purity when compared with the other method. There will be local differences in purity, the centres of the 'oldest' crystals being the purest, whereas the newer crystals and the collision prone corners of the larger crystals will contain more impurities.

Brine recovery As with the production of solar salt, the amount of impurities determines when the crystallization is stopped. Again bromide is important in that respect, as it is very difficult to remove. One is then left with a brine containing valuable salt that unfortunately it is difficult to recover because of the high impurity content. Several procedures have been devised to cope with this problem. The first process, used by Akzo Nobel, is the Bromin process. In this process the remaining sulfate- and bromine-rich mother liquor is further evaporated in a separate crystallizer, where sodium sulfate is crystallized in addition to sodium chloride. The remaining, greatly reduced amount of mother liquor, now very rich in bromine, is discharged in a nonproductive borehole. Sodium chloride and sodium sulfate are added to the raw brine entering the brine purification. Sodium sulfate dissolves and the sulfate precipitates as calcium sulfate.

At the Salinen Austria GmbH production facility nearly the same procedure is used, but instead of crystallization of anhydrous sodium sulfate and sodium chloride, the company claims that sodium chloride and glaserite ($Na_2SO_4 \cdot 3K_2SO_4$) are formed. After washing with raw brine, the glaserite is dissolved and the sulfate serves to precipitate calcium while the sodium chloride remains behind.

Another method involves cooling the remaining brine, thus producing Glauber salt, $Na_2SO_4 \cdot 10H_2O$. Subsequent evaporation of the mother liquor yields sodium chloride. The remaining brine is then discarded.

See Colour Plate 117.

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SOLID-PHASE EXTRACTION OF DRUGS

See **III / BIOANALYTICAL APPLICATIONS: SOLID-PHASE EXTRACTION**