Distillation Water Treatment

written by :Mustafa M Hathal Alfukaiki

AL_nahrain university – chem..engin.department

Distillation is one of the oldest methods of water treatment and is still in use today though not commonly as a home treatment method. It can effectively remove many contaminants from drinking water, including bacteria, inorganic and many organic compounds.

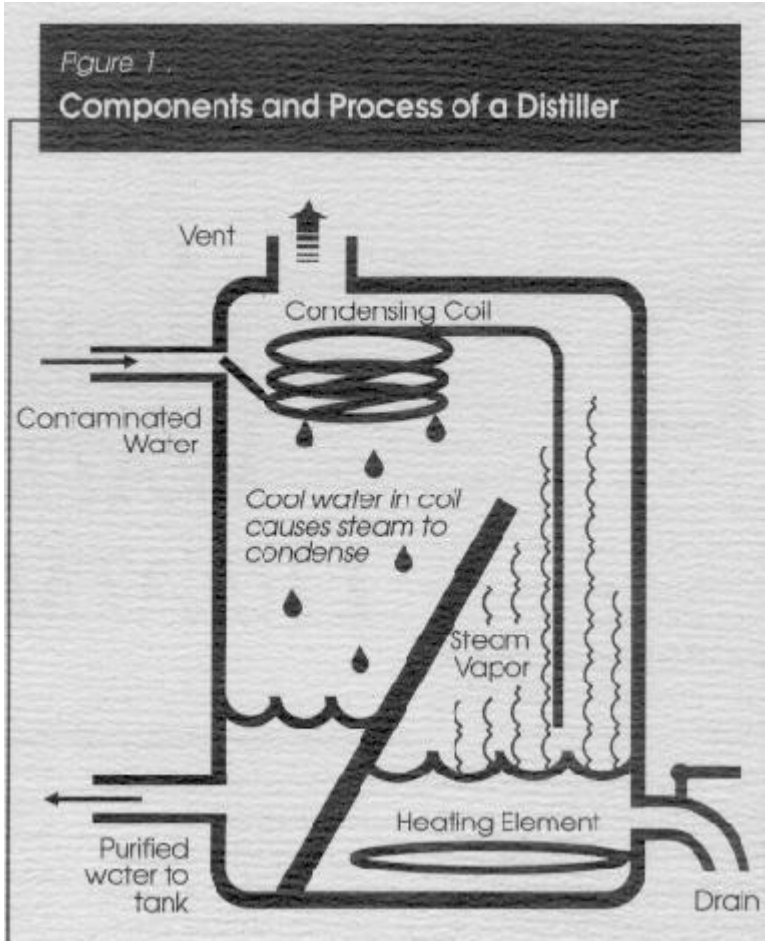
Note that home water treatment is considered only a temporary solution. The best solutions to a contaminated drinking water problem are to either end the practices causing the contamination or change water sources. Distillation is a process that relies on evaporation to purify water. Contaminated water is heated to form steam. Inorganic compounds and large non-volatile organic molecules do not evaporate with the water and are left behind. The steam then cools and condenses to form purified water.

Distillation is most effective in removing inorganic compounds such as metals (iron and lead) and nitrate; hardness (calcium and magnesium); and particulates from a contaminated water supply. The boiling process also kills microorganisms such as bacteria and some viruses. The effectiveness of distillation in removing organic compounds varies, depending on such chemical characteristics of the organic compound as solubility and boiling point. Organic compounds that boil at temperatures greater than the boiling point of water (some pesticides) can be effectively removed from the water. Organic compounds that boil at temperatures lower than the boiling point of water (ex., benzene and toluene) will be vaporized along with the water. If these harmful compounds are not removed prior to condensation, they will recontaminate the purified product.

Distillation Units

Distillation units or stills generally consist of a boiling chamber, where the water enters, is heated and vaporized; condensing coils or chamber, where the water is cooled and converted back to liquid water; and a storage tank for

purified water



Distillation units are usually installed as point- of-use (POU) systems. They are generally placed at the kitchen faucet

and used to purify water intended for (drinking and cooking purposes only. Stills vary in size, depending on the amount of purified water they produce The production rate varies from 3 to 11 gallons per day. Home stills can be located on the counter or floor, or attached to the wall.

Models can be fully or partially automated, or manual. Some stills have columns or volatile gas vents to eliminate organic chemicals with boiling points lower than water, thus ensuring uncontaminated water.

Operation, Maintenance and Cost

As with all home water treatment systems, stills require some level of regular maintenance to keep the unit operating properly. Unevaporated pollutants remaining in the boiling chamber need to be regularly flushed to the septic or sewer system. Even with regular removal of the residual water that contains unevaporated pollutants, a calcium and magnesium, scale will collect at the bottom of the boiling chamber. This scale eventually needs to be removed, usually by hand scrubbing or by an application of acid.

Heating water to form steam requires energy. This means that operating costs for distillation units are generally higher than those of other forms of home water treatment. The distillation process also removes oxygen and some trace metals from water. Some people claim this leaves the water tasting flat.

References

This information comes from Michigan State University Extension bulletin WQ 22, Distillation for Home Water

Treatment.

This information.was reviewed as of June 2008. For more information about the contents please contact *costner@msu.edu* for webpage problems *strausc@msu.edu* .

Distillation units are usually installed as point-of-use (POU) systems. They are generally placed at the kitchen faucet and used to purify water intended for drinking and cooking purposes only.

Distilled water is also called steam-distilled water. Distilled water is water which has been heated to the boiling point so that impurities are separated from the water, which becomes vapour or steam at 100 degree Centigrade or 212 degrees Fahrenheit. Steam is then cooled and condensed back into pure liquid form. The impurities remain as residue in the steam kettle.

Distillation is effective at removing:

- hardness; calcium and magnesium
- inorganics such as metals - aluminum, arsenic, barium, cadmium, chloride, chromium, copper, fluoride, magnesium, iron, lead, manganese, mercury, nitrate, selenium, silver, sulfate, and zinc
- nitrate, sulfate
- bacteria - giardia and cryptosporidium cysts, parasites and viruses
- particulates from a contaminated water supply.

Considerations:

Distillation is the process of boiling and condensing water. The amount of electrical energy you use to operate the unit depends on it's size and whether you have been diligent in it's maintenance. Efficient distillers use about 3 KWH of electricity for each gallon of water that is distilled. You can calculate the approximate cost per gallon by multiplying 3 times the KWH rate in your area. *Consider energy consumption.*

The heat generated by the unit spills into your home. A medium size distillation unit is like having a kettle boiling in your kitchen, ...all the time. When sizing an air conditioner for your home the HVAC experts use tables to determine heat load. A 10 cup coffee maker requires an additional 1/2 ton of air conditioning. *Consider heat load*.

The process of distilling water will leave a residue of inorganic compounds such as calcium and magnesium which will coat the area where the water was boiled-off. This scale eventually needs to be removed, usually by hand scrubbing or by an application of acid. Regular maintenance by scrubbing and chemicals or vinegar is required. Un-evaporated pollutants remaining in the boiling chamber need to be regularly flushed. *Consider maintenance.*

Distillation does not remove all harmful organic compounds and may need to be supplemented with carbon filtration for this purpose. Some contaminants carry over with the steam in the distillation process. Some contaminants have lower boiling points than that of water which is why distillers are equipped with a volatile gas vent, a design feature which removes most of these contaminants. For even

further protection, each distiller is also equipped with a carbon post-filter to provide you with the highest quality distilled water with a 99% or higher removal rate of contaminants.

The process of distillation is not fast. Water is first brought to a boil then the vapour is condensed back to water. Distillation is slow. Typically, 2 gallons of input water yield about 1 gallon of distilled water after 4 to 6 hours. Large counter top distillers can distill up to a gallon of water per hour. Small units produce less than 1 quart of water per hour. You do not want to store Distilled water longer than a day as the tank of processed water is vented and open to the air. Distilled water like RO water is very soft and relatively pure. Pure water is frequently termed as the universal solvent because it "wants" to pick up or dissolve many of the things it comes in contact with. *Consider the amount of purified water you need.*

Efficiency, many smaller, slower units and less expensive counter top units are more effective at removing organic matter from the water because they heat the water more slowly. Quickly heating water can allow compounds to be tossed from the water into the steam allowing these compounds to either coat the walls of the distillation coils or be transported into the distilled water. *Consider efficiency.*

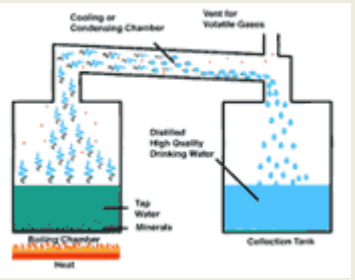
Both Distilled Water RO water have been implicated in the leaching of nutrients from the body. Our investigation of this while definitely not as thorough as the paper done by the WQA on this topic has not found scientific proof of this.

Introduction

Distillers can effectively remove most or all contaminants, including minerals, metals, organic chemicals, and microorganisms from water. Since

distilled water has no minerals, some people claim distilled water tastes flat or slightly sweet. Distillation also kills or removes microorganisms, including most pathogens. Distillation can also remove organic contaminants, but its efficiency depends on the chemical characteristics of the contaminant. Distillation efficiency also decreases as the TDS of the water increases. Volatile organic chemicals (VOCs) like benzene and TCE vaporize along with the water and recontaminate the distilled water if not removed prior to distillation. Some distillation units may initially purge some steam and volatile chemicals. These units should be properly exhausted to prevent indoor air contamination. Some

home distillation units have activated carbon filters to remove VOCs during distillation.



Distillation process

click to enlarge

Principles of Distillation

The principle for operation of a distiller is simple. Water is heated to boiling in an enclosed container. As the water evaporates, inorganic chemicals, large non-volatile organic chemicals, and microorganisms are left behind or killed off in the boiling chamber. The steam then enters condensing coils or a chamber where the steam is cooled by air or water and condenses back to a liquid. The distilled water then goes into a storage container, usually 1.5 to 3 gallons in capacity.

Distillation Units

Also called stills, distillation units generally consist of a

boiling chamber (where the water enters, is heated, and vaporized), condensing coils or chamber (where the water is cooled and converted back to liquid), and a storage tank for treated water. Distillation units are usually installed as point-of-use (POU) systems that are placed near the kitchen faucet and used to purify water for drinking and cooking only. Home stills can be located on the counter or floor, or attached to the wall, depending on size. Models can be manual, partially automated, or fully automated.

Distillers vary from small, round units that distill less than one quart of water per hour to rectangular carts that distill about one-half gallon of water per hour.

Operation and Maintenance

As with all home water treatment systems, distillation units require some level of regular maintenance to keep the unit operating properly. Contaminants left in the boiling chamber need to be regularly flushed out. Even with regular removal of the brackish (saline) residues, calcium and magnesium scalewill quickly collect at the bottom of the boiling chamber. Over time, this scale reduces heat transfer and should regularly be removed either by hand scrubbing or by soaking with acetic acid. Vinegar is commonly used to clean home distillers. Although minerals that can cause corrosion and scaling are removed during distillation, distilled (and RO) water

is very corrosive (aggressive). It should not be stored or transferred in metal pipes.

Costs

Small still units (capacity: 1.5 gallons, 6 liters) cost $250 or more. Large units (capacity: 15 gallons, 57 liters) vary from $450 to $1,450 in purchase price.

Distillation is the most effective, but also the most expensive (energy-intensive) form of water purification. The power rating of the still and the local electricity rates determine the cost of operation of these units. To calculate the cost to produce one gallon of water, multiply the price of a kilowatt hour times the rated kilowatt hour use of your model, times the number of hours it takes to

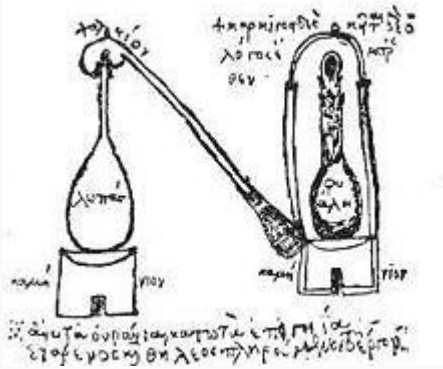
produce one gallon of water. For example, if local electricity costs 0.10 cents per kilo watt hour and your unit is rated at 800 watts (or 0.8 kilowatt hour) and it takes 4 hours to produce one gallon of water, your operating cost is 0.32 cents per gallon, excluding purchase and maintenance costs.

(Portions of this text has been adapted from Plowman, F.T., "Distillation," Durham: University of New Hampshire Cooperative Extension, Water Quality Fact Sheet 21, 1989.)

Go to other treatment methods:

History[edit]

See also: Distilled beverage



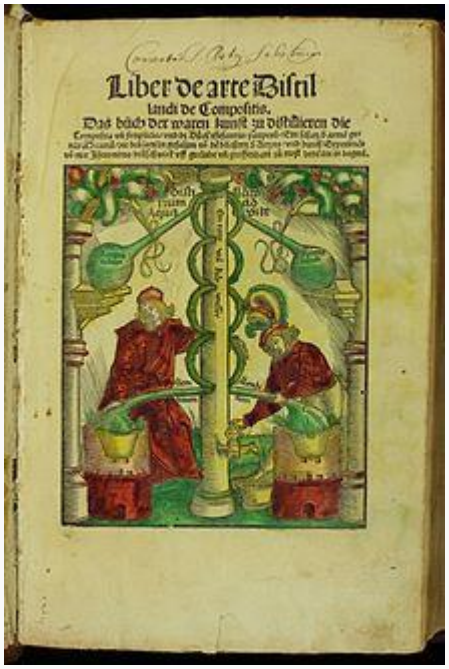
Distillation apparatus of Zosimos of Panopolis, from Marcelin Berthelot, *Collection des anciens alchimistes grecs* (3 vol., Paris, 1887–1888).

The first evidence of distillation comes from Greek alchemists working in Alexandria in the 1st century AD.[2] Distilled water has been known since at least c. 200, when Alexander of Aphrodisias described the process.[3] Distillation in China could have begun during the Eastern Han Dynasty (1st–2nd centuries), but archaeological evidence indicates that actual distillation of beverages began in the Jin and Southern Songdynasties.[4] A still was found in an archaeological site in Qinglong, Hebei province dating to the 12th century. Distilled beverages were more common during the Yuan dynasty.[4] Arabs learned the process from the Alexandrians and used it extensively in their chemical experiments[*citation needed*].

Clear evidence of the distillation of alcohol comes from the School of Salerno in the 12th century.[2][5] Fractional distillation was developed by Tadeo Alderotti in the 13th century.[6]

In 1500, German alchemist Hieronymus Braunschweig published *Liber de arte destillandi* (The Book of the Art of Distillation)[7] the first book solely dedicated to the subject of distillation, followed in 1512 by a much expanded version. In 1651, John French published The Art of Distillation the first major English

compendium of practice, though it has been claimed[8] that much of it derives from Braunschweig's work. This includes diagrams with people in them showing the industrial rather than bench scale of the operation.



Hieronymus Brunschwig's *Liber de arte Distillandi de Compositis* (Strassburg, 1512)Chemical Heritage Foundation

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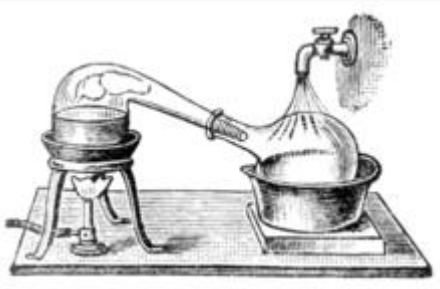
A retort



Distillation

Old Ukrainian vodka still

Simple liqueur distillation in East Timor

As alchemy evolved into the science of chemistry, vessels called retorts became used for distillations. Both alembics and retorts are forms ofglassware with long necks pointing to the side at a downward angle which acted as air-cooled condensers to condense the distillate and let it drip downward for collection. Later, copper alembics were invented. Riveted joints were often kept tight by using various mixtures, for instance a dough made of rye flour.[9] These alembics often featured a cooling system around the beak, using cold water for instance, which made the condensation of alcohol more efficient. These were called pot stills. Today, the retorts and pot stills have been largely supplanted by more efficient distillation methods in most industrial processes. However, the pot still is still widely used for the elaboration of some fine alcohols such as cognac, Scotch whisky, tequila and some vodkas. Pot stills made of various materials (wood, clay, stainless steel) are also used bybootleggers in various countries. Small pot stills are also sold for the domestic production[10] of flower water or essential oils.

Early forms of distillation were batch processes using one vaporization and one condensation. Purity was improved by further distillation of the condensate. Greater volumes were processed by simply repeating the distillation. Chemists were reported to carry out as many as 500 to 600 distillations in order to obtain a pure compound.[11]

In the early 19th century the basics of modern techniques including pre-heating and reflux were developed, particularly by the French,[11] then in 1830 a British Patent was issued to Aeneas Coffey for a whiskey distillation column,[12] which worked continuously and may be regarded as the archetype of modern petrochemical units. In 1877, Ernest Solvay was granted a U.S. Patent for a tray column for ammonia distillation[13] and the same and subsequent years saw developments of this theme for oil and spirits.

With the emergence of chemical engineering as a discipline at the end of the 19th century, scientific rather than empirical methods could be applied. The developing petroleum industry in the early 20th century provided the impetus for the development of accurate design methods such as the McCabe-Thiele method and the Fenske equation. The availability of powerful computers has also allowed direct computer simulation of distillation columns.

Applications of distillation[edit]

The application of distillation can roughly be divided in four groups: laboratory scale, industrial distillation, distillation of herbs for perfumery and medicinals (herbal distillate), and food processing. The latter two are distinctively different from the former two in that in the processing of beverages, the distillation is not used as a true purification method but more to transfer all volatiles from the source materials to the distillate.

The main difference between laboratory scale distillation and industrial distillation is that laboratory scale distillation is often performed batch-wise, whereas industrial distillation often occurs continuously. In batch distillation, the composition of the source material, the vapors of the distilling compounds and the distillate change during the distillation. In batch distillation, a still is charged (supplied) with a batch of feed mixture, which is then separated into its component fractions which are collected sequentially from most volatile to less volatile, with the bottoms (remaining least or non-volatile fraction) removed at the end. The still can then be recharged and the process repeated.

In continuous distillation, the source materials, vapors, and distillate are kept at a constant composition by carefully replenishing the source material and removing fractions from both vapor and liquid in the system. This results in a better control of the separation process.

Idealized distillation model[edit]

The boiling point of a liquid is the temperature at which the vapor pressure of the liquid equals the pressure in the liquid, enabling bubbles to form without being crushed. A special case is the normal boiling point, where the vapor pressure of the liquid equals the ambient atmospheric pressure.

It is a common misconception that in a liquid mixture at a given pressure, each component boils at the boiling point corresponding to the given pressure and the vapors of each component will collect separately and purely. This, however, does not occur even in an idealized system. Idealized models of distillation are essentially governed by Raoult's law and Dalton's law, and assume that vapor-liquid equilibria are attained.

Raoult's law assumes that a component contributes to the total vapor pressure of the mixture in proportion to its percentage of the mixture and its vapor pressure when pure, or succinctly: partial pressure equals mole fraction multiplied by vapor pressure when pure. If one component changes another component's vapor pressure, or if the volatility of a component is dependent on its percentage in the mixture, the law will fail.

Dalton's law states that the total vapor pressure is the sum of the vapor pressures of each individual component in the mixture. When a multi-component liquid is heated, the vapor pressure of each component will rise, thus causing the total vapor pressure to rise. When the total vapor pressure reaches the pressure surrounding the liquid, boiling occurs and liquid turns to gas throughout the bulk of the liquid. Note that a mixture with a given composition has one boiling point at a given pressure, when the components are mutually soluble.

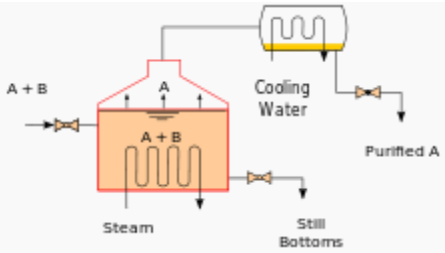
An implication of one boiling point is that lighter components never cleanly "boil first". At boiling point, all volatile components boil, but for a component, its percentage in the vapor is the same as its percentage of the total vapor pressure. Lighter components have a higher partial pressure and thus are concentrated in the vapor, but heavier volatile components also have a (smaller) partial pressure and necessarily evaporate also, albeit being less concentrated in the vapor. Indeed, batch distillation and fractionation succeed by varying the composition of the mixture. In batch distillation, the batch evaporates, which changes its composition; in fractionation, liquid higher in the fractionation column contains more lights and boils at lower temperatures.

The idealized model is accurate in the case of chemically similar liquids, such as benzene and toluene. In other cases, severe deviations from Raoult's law and Dalton's law are observed, most famously in the mixture of ethanol and water. These compounds, when heated together, form an azeotrope, which is a composition with a boiling point higher or lower than the boiling point of each separate liquid. Virtually all liquids, when mixed and heated, will display azeotropic behaviour. Although there are computational methods that can be used to estimate the behavior of a mixture of arbitrary components, the only way to obtain accurate vapor-liquid equilibrium data is by measurement.

It is not possible to *completely* purify a mixture of components by distillation, as this would require each component in the mixture to have a zero partial pressure. If ultra-pure products are the goal, then further chemical separation must be applied. When a binary mixture is evaporated and the other component, e.g. a salt, has zero partial pressure for practical purposes, the process is simpler and is called evaporation in engineering.

Batch distillation[edit]

Main article: Batch distillation



A batch still showing the separation of A and B.

Heating an ideal mixture of two volatile substances A and B (with A having the higher volatility, or lower boiling point) in a batch distillation setup (such as in an apparatus depicted in the opening figure) until the mixture is boiling results in a vapor above the liquid which contains a mixture of A and B. The ratio between A and B in the vapor will be different from the ratio in the liquid: the ratio in the liquid will be determined by how the original mixture was prepared, while the ratio in the vapor will be enriched in the more volatile compound, A (due to Raoult's Law, see above). The vapor goes through the condenser and is removed from the system. This in turn means that the ratio of compounds in the remaining liquid is now different from the initial ratio (i.e. more enriched in B than the starting liquid).

The result is that the ratio in the liquid mixture is changing, becoming richer in component B. This causes the boiling point of the mixture to rise, which in turn results in a rise in the temperature in the vapor, which results in a changing ratio of A : B in the gas phase (as distillation continues, there is an increasing proportion of B in the gas phase). This results in a slowly changing ratio A : B in the distillate.

If the difference in vapor pressure between the two components A and B is large (generally expressed as the difference in boiling points), the mixture in the beginning of the distillation is highly enriched in component A, and when component A has distilled off, the boiling liquid is enriched in component B.

Continuous distillation[edit]

Main article: Continuous distillation

Continuous distillation is an ongoing distillation in which a liquid mixture is continuously (without interruption) fed into the process and separated fractions are removed continuously as output streams as time passes during the operation. Continuous distillation produces at least two output fractions, including at least

one volatile distillate fraction, which has boiled and been separately captured as a vapor condensed to a liquid. There is always a bottoms (or residue) fraction, which is the least volatile residue that has not been separately captured as a condensed vapor.

Continuous distillation differs from batch distillation in the respect that concentrations should not change over time. Continuous distillation can be run at a steady state for an arbitrary amount of time. For any source material of specific composition, the main variables that affect the purity of products in continuous distillation are the reflux ratio and the number of theoretical equilibrium stages (practically, the number of trays or the height of packing). Reflux is a flow from the condenser back to the column, which generates a recycle that allows a better separation with a given number of trays. Equilibrium stages are ideal steps where compositions achieve vapor-liquid equilibrium, repeating the separation process and allowing better separation given a reflux ratio. A column with a high reflux ratio may have fewer stages, but it refluxes a large amount of liquid, giving a wide column with a large holdup. Conversely, a column with a low reflux ratio must have a large number of stages, thus requiring a taller column.

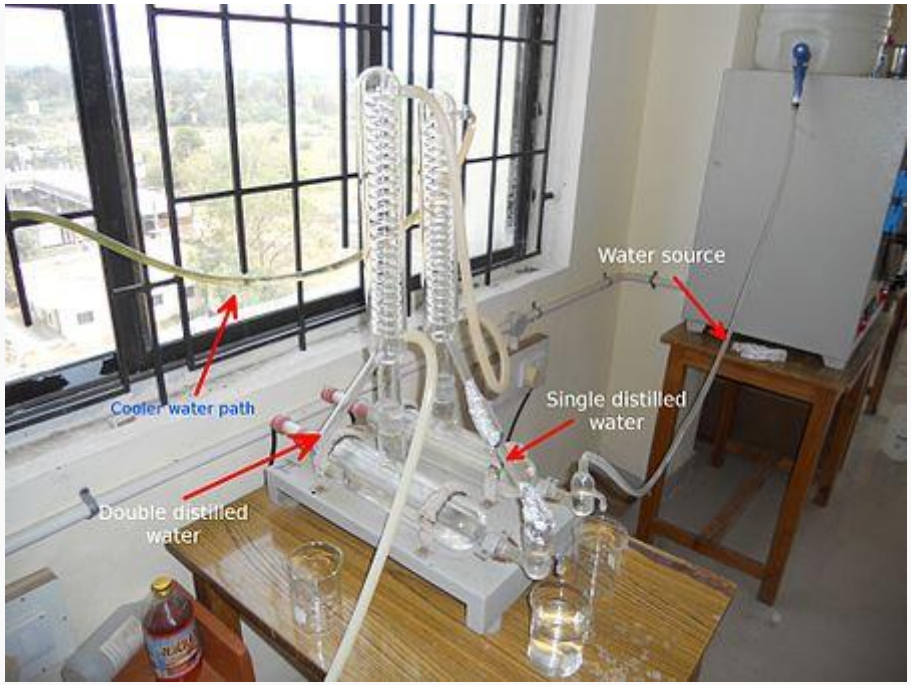
General improvements[edit]

Both batch and continuous distillations can be improved by making use of a fractionating column on top of the distillation flask. The column improves separation by providing a larger surface area for the vapor and condensate to come into contact. This helps it remain at equilibrium for as long as possible. The column can even consist of small subsystems ('trays' or 'dishes') which all contain an enriched, boiling liquid mixture, all with their own vapor-liquid equilibrium.

There are differences between laboratory-scale and industrial-scale fractionating columns, but the principles are the same. Examples of laboratory-scale fractionating columns (in increasing efficiency) include:

- Air condenser
- Vigreux column (usually laboratory scale only)
- Packed column (packed with glass beads, metal pieces, or other chemically inert material)
- Spinning band distillation system.

Laboratory scale distillation[edit]



Typical laboratory distillation unit

Laboratory scale distillations are almost exclusively run as batch distillations. The device used in distillation, sometimes referred to as a *still*, consists at a minimum of a **reboiler** or *pot* in which the source material is heated, a **condenser** in which the heated vapour is cooled back to the liquid state, and a **receiver** in which the concentrated or purified liquid, called the **distillate**, is collected. Several laboratory scale techniques for distillation exist (see also distillation types).

Simple distillation[edit]

In **simple distillation**, the vapor is immediately channeled into a condenser. Consequently, the distillate is not pure but rather its composition is identical to the composition of the vapors at the given temperature and pressure. That concentration follows Raoult's law.

As a result, simple distillation is effective only when the liquid boiling points differ greatly (rule of thumb is 25 °C)[14] or when separating liquids from non-volatile solids or oils. For these cases, the vapor pressures of the components are usually sufficiently different that the distillate may be sufficiently pure for its intended purpose.

Fractional distillation[edit]

Main article: Fractional distillation

For many cases, the boiling points of the components in the mixture will be sufficiently close that Raoult's law must be taken into consideration. Therefore, **fractional distillation** must be used in order to separate the components by repeated vaporization-condensation cycles within a packed fractionating column. This separation, by successive distillations, is also referred to as **rectification**.[15]

As the solution to be purified is heated, its vapors rise to the fractionating column. As it rises, it cools, condensing on the condenser walls and the surfaces of the packing material. Here, the condensate continues to be heated by the rising hot vapors; it vaporizes once more. However, the composition of the fresh vapors are determined once again by Raoult's law. Each vaporization-condensation cycle (called a *theoretical plate*) will yield a purer solution of the more volatile component.[16] In reality, each cycle at a given temperature does not occur at exactly the same position in the fractionating column; *theoretical plate* is thus a concept rather than an accurate description.

More theoretical plates lead to better separations. A spinning band distillation system uses a spinning band of Teflon or metal to force the rising vapors into close contact with the descending condensate, increasing the number of theoretical plates.[17]

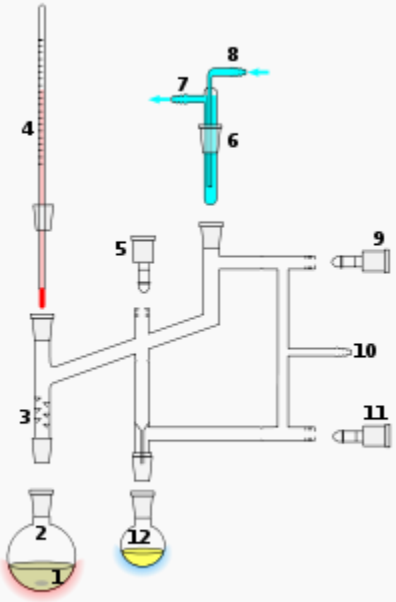
Steam distillation[edit]

Main article: Steam distillation

Like vacuum distillation, **steam distillation** is a method for distilling compounds which are heat-sensitive.[18] The temperature of the steam is easier to control than the surface of a heating element, and allows a high rate of heat transfer without heating at a very high temperature. This process involves bubbling steam through a heated mixture of the raw material. By Raoult's law, some of the target compound will vaporize (in accordance with its partial pressure). The vapor mixture is cooled and condensed, usually yielding a layer of oil and a layer of water.

Steam distillation of various aromatic herbs and flowers can result in two products; an essential oil as well as a watery herbal distillate. The essential oils are often used in perfumery andaromatherapy while the watery distillates have many applications in aromatherapy, food processing and skin care.

Dimethyl sulfoxide usually boils at 189 °C. Under a vacuum, it distills off into the receiver at only 70 °C.



Perkin triangle distillation setup

1: Stirrer bar/anti-bumping granules **2:** Still pot **3:** Fractionating column **4:**Thermometer/Boiling point temperature **5:**Teflon tap 1 **6:** Cold finger **7:** Cooling water out **8:** Cooling water in **9:** Teflon tap 2 **10:**Vacuum/gas inlet **11:** Teflon tap 3 **12:** Still receiver

Vacuum distillation[edit]

Main article: Vacuum distillation

Some compounds have very high boiling points. To boil such compounds, it is often better to lower the pressure at which such compounds are boiled instead of increasing the temperature. Once the pressure is lowered to the vapor pressure of the compound (at the given temperature), boiling and the rest of the

distillation process can commence. This technique is referred to as **vacuum distillation**and it is commonly found in the laboratory in the form of the rotary evaporator.

This technique is also very useful for compounds which boil beyond their decomposition temperatureat atmospheric pressure and which would therefore be decomposed by any attempt to boil them under atmospheric pressure.

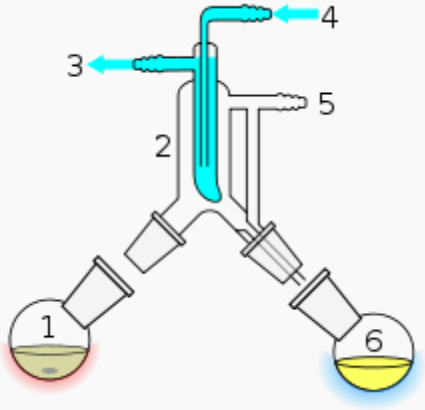
Molecular distillation is vacuum distillation below the pressure of 0.01 torr.[19] 0.01 torr is one order of magnitude above high vacuum, where fluids are in the free molecular flow regime, i.e. the mean free path of molecules is comparable to the size of the equipment. The gaseous phase no longer exerts significant pressure on the substance to be evaporated, and consequently, rate of evaporation no longer depends on pressure. That is, because the continuum assumptions of fluid dynamics no longer apply, mass transport is governed by molecular dynamics rather than fluid dynamics. Thus, a short path between the hot surface and the cold surface is necessary, typically by suspending a hot plate covered with a film of feed next to a cold plate with a line of sight in between. Molecular distillation is used industrially for purification of oils.

Air-sensitive vacuum distillation[edit]

Some compounds have high boiling points as well as being air sensitive. A simple vacuum distillation system as exemplified above can be used, whereby the vacuum is replaced with an inert gas after the distillation is complete. However, this is a less satisfactory system if one desires to collect fractions under a reduced pressure. To do this a "cow" or "pig" adaptor can be added to the end of the condenser, or for better results or for very air sensitive compounds a Perkin triangle apparatus can be used.

The Perkin triangle, has means via a series of glass or Teflon taps to allows fractions to be isolated from the rest of the still, without the main body of the distillation being removed from either the vacuum or heat source, and thus can remain in a state of reflux. To do this, the sample is first isolated from the vacuum by means of the taps, the vacuum over the sample is then replaced with an inert gas (such as nitrogen or argon) and can then be stoppered and removed. A fresh collection vessel can then be added to the system, evacuated and linked back into the distillation system via the taps to collect a second fraction, and so on, until all fractions have been collected.

Short path distillation[edit]



Short path vacuum distillation apparatus with vertical condenser (cold finger), to minimize the distillation path; **1:** Still pot with stirrer bar/anti-bumping granules **2:** Cold finger – bent to direct condensate **3:** Cooling water out **4:** cooling water in **5:** Vacuum/gas inlet **6:** Distillate flask/distillate.

Short path distillation is a distillation technique that involves the distillate travelling a short distance, often only a few centimeters, and is normally done at reduced pressure.[20] A classic example would be a distillation involving the distillate travelling from one glass bulb to another, without the need for a condenser separating the two chambers. This technique is often used for compounds which are unstable at high temperatures or to purify small amounts of compound. The advantage is that the heating temperature can be considerably lower (at reduced pressure) than the boiling point of the liquid at standard pressure, and the distillate only has to travel a short distance before condensing. A short path ensures that little compound is lost on the sides of the apparatus. The Kugelrohr is a kind of a short path distillation apparatus which often contain multiple chambers to collect distillate fractions.

Zone distillation[edit]

Zone distillation is a distillation process in long container with partial melting of refined matter in moving liquid zone and condensation of vapor in the solid phase at condensate pulling in cold area. The process is worked in theory. When zone heater is moving from the top to the bottom of the container then solid condensate with irregular impurity distribution is forming. Then most pure part of the condensate may be extracted as product. The process may be iterated many times by moving (without turnover) the received condensate to the bottom part of the container on the place of refined matter. The irregular impurity

distribution in the condensate (that is efficiency of purification) increases with number of repetitions of the process. Zone distillation is a distillation analog of zone recrystallization. Impurity distribution in the condensate is described by known equations of zone recrystallization with various numbers of iteration of process – with replacement distribution efficient k of crystallization on separation factor α of distillation.[21]

Other types[edit]

- The process of reactive distillation involves using the reaction vessel as the still. In this process, the product is usually significantly lower-boiling than its reactants. As the product is formed from the reactants, it is vaporized and removed from the reaction mixture. This technique is an example of a continuous vs. a batch process; advantages include less downtime to charge the reaction vessel with starting material, and less workup.
- Catalytic distillation is the process by which the reactants are catalyzed while being distilled to continuously separate the products from the reactants. This method is used to assist equilibrium reactions reach completion.
- Pervaporation is a method for the separation of mixtures of liquids by partial vaporization through a non-porous membrane.
- Extractive distillation is defined as distillation in the presence of a miscible, high boiling, relatively non-volatile component, the solvent, that forms no azeotrope with the other components in the mixture.
- Flash evaporation (or partial evaporation) is the partial vaporization that occurs when a saturated liquid stream undergoes a reduction in pressure by passing through a throttling valve or other throttling device. This process is one of the simplest unit operations, being equivalent to a distillation with only one equilibrium stage.
- Codistillation is distillation which is performed on mixtures in which the two compounds are not miscible.

The unit process of evaporation may also be called "distillation":

- In rotary evaporation a vacuum distillation apparatus is used to remove bulk solvents from a sample. Typically the vacuum is generated by a water aspirator or a membrane pump.
- In a kugelrohr a short path distillation apparatus is typically used (generally in combination with a (high) vacuum) to distill high boiling (> 300 °C) compounds. The apparatus consists of an oven in which the compound to be distilled is placed, a receiving portion which is outside of the oven, and a means of rotating the sample. The vacuum is normally generated by using a high vacuum pump.

Other uses:

- Dry distillation or destructive distillation, despite the name, is not truly distillation, but rather a chemical reaction known as pyrolysis in which solid substances are heated in an inert orreducing atmosphere and any volatile fractions, containing high-boiling liquids and products of pyrolysis, are collected. The destructive distillation of wood to give methanol is the root of its common name – *wood alcohol*.
- Freeze distillation is an analogous method of purification using freezing instead of evaporation. It is not truly distillation, but a recrystallization where the product is the mother liquor, and does not produce products equivalent to distillation. This process is used in the production of ice beer and ice wine to increase ethanol and sugar content, respectively. It is also used to produce applejack. Unlike distillation, freeze distillation concentrates poisonous congeners rather than removing them; As a result, many countries prohibit such applejack as a health measure. However, reducing methanol with the absorption of 4A molecular sieve is a practical method for production.[22] Also, distillation by evaporation can separate these since they have different boiling points.

Azeotropic distillation[edit]

Main article: Azeotropic distillation

Interactions between the components of the solution create properties unique to the solution, as most processes entail nonideal mixtures, where Raoult's law does not hold. Such interactions can result in a constant-boiling **azeotrope** which behaves as if it were a pure compound (i.e., boils at a single temperature instead of a range). At an azeotrope, the solution contains the given component in the same proportion as the vapor, so that evaporation does not change the purity, and distillation does not effect separation. For example, ethyl alcoholand water form an azeotrope of 95.6% at 78.1 °C.

If the azeotrope is not considered sufficiently pure for use, there exist some techniques to break the azeotrope to give a pure distillate. This set of techniques are known as **azeotropic distillation**. Some techniques achieve this by "jumping" over the azeotropic composition (by adding an additional component to create a new azeotrope, or by varying the pressure). Others work by chemically or physically removing or sequestering the impurity. For example, to purify ethanol beyond 95%, a drying agent or a (desiccant such as potassium carbonate) can be added to convert the soluble water into insoluble water of crystallization. Molecular sieves are often used for this purpose as well.

Immiscible liquids, such as water and toluene, easily form azeotropes. Commonly, these azeotropes are referred to as a low boiling azeotrope because the boiling point of the azeotrope is lower than the boiling point of either pure component. The temperature and composition of the azeotrope is easily predicted from the vapor pressure of the pure components, without use of Raoult's law. The azeotrope is easily broken in a distillation set-up by using a liquid-liquid separator (a decanter) to separate the two liquid layers that are condensed overhead. Only one of the two liquid layers is refluxed to the distillation set-up.

High boiling azeotropes, such as a 20 weight percent mixture of hydrochloric acid in water, also exist. As implied by the name, the boiling point of the azeotrope is greater than the boiling point of either pure component.

To break azeotropic distillations and cross distillation boundaries, such as in the DeRosier Problem, it is necessary to increase the composition of the light key in the distillate.

Breaking an azeotrope with unidirectional pressure manipulation[edit]

The boiling points of components in an azeotrope overlap to form a band. By exposing an azeotrope to a vacuum or positive pressure, it's possible to bias the boiling point of one component away from the other by exploiting the differing vapour pressure curves of each; the curves may overlap at the azeotropic point, but are unlikely to be remain identical further along the pressure axis either side of the azeotropic point. When the bias is great enough, the two boiling points no longer overlap and so the azeotropic band disappears.

This method can remove the need to add other chemicals to a distillation, but it has two potential drawbacks.

Under negative pressure, power for a vacuum source is needed and the reduced boiling points of the distillates requires that the condenser be run cooler to prevent distillate vapours being lost to the vacuum source. Increased cooling demands will often require additional energy and possibly new equipment or a change of coolant.

Alternatively, if positive pressures are required, standard glassware can not be used, energy must be used for pressurization and there is a higher chance of side reactions occurring in the distillation, such as decomposition, due to the higher temperatures required to effect boiling.

A unidirectional distillation will rely on a pressure change in one direction, either positive or negative.

Pressure-swing distillation[edit]

Further information: Pressure-Swing Distillation (section on the main Azeotrope page)

This section **may be confusing or unclear to readers**. *(May 2009)*

Pressure-swing distillation is essentially the same as the unidirectional distillation used to break azeotropic mixtures, but here both positive and negative pressures may be employed.[*clarification needed*]

This has an important impact on the selectivity of the distillation and allows a chemist[citation needed] to optimize a process such that fewer extremes of pressure and temperature are required and less energy is consumed. This is particularly important in commercial applications.

Pressure-swing distillation is employed during the industrial purification of ethyl acetate after its catalytic synthesis from ethanol.

Industrial distillation[edit]

Typical industrial distillation towers

Main article: Continuous distillation

Large scale **industrial distillation** applications include both batch and continuous fractional, vacuum, azeotropic, extractive, and steam distillation. The most widely used industrial applications of continuous, steady-state fractional distillation are in petroleum refineries,petrochemical and chemical plants and natural gas processing plants.

Industrial distillation[15][23] is typically performed in large, vertical cylindrical columns known as **distillation towers** or **distillation columns** with diameters ranging from about 65 centimeters to 16 meters and heights ranging from about 6 meters to 90 meters or more. When the process feed has a diverse composition, as in distilling crude oil, liquid outlets at intervals up the column allow for the withdrawal of different *fractions* or products having different boiling points or boiling ranges. The "lightest" products (those with the lowest boiling point) exit from the top of the columns and the "heaviest" products (those with the highest boiling point) exit from the bottom of the column and are often called the **bottoms**.

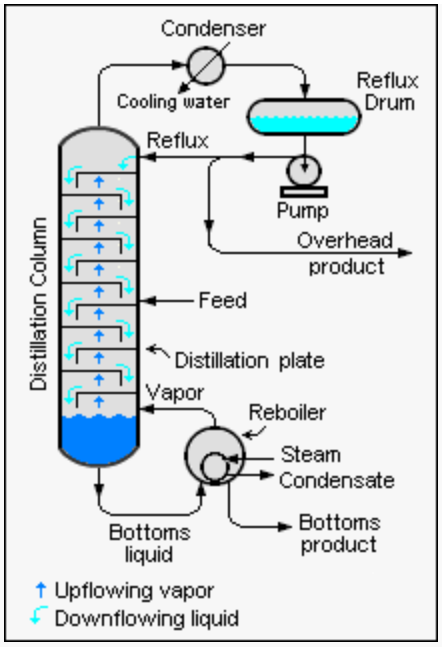
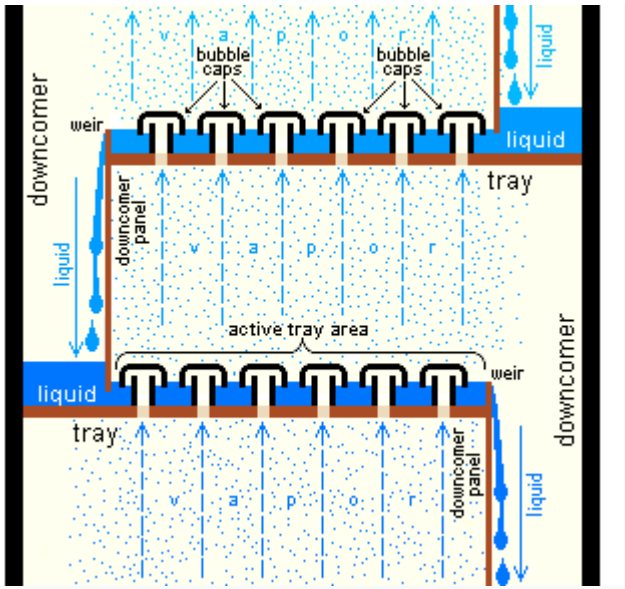


Diagram of a typical industrial distillation tower

Industrial towers use reflux to achieve a more complete separation of products. Reflux refers to the portion of the condensed overhead liquid product from a distillation or fractionation tower that is returned to the upper part of the tower as shown in the schematic diagram of a typical, large-scale industrial distillation tower. Inside the tower, the downflowing reflux liquid provides cooling and condensation of the upflowing vapors thereby increasing the efficiency of the distillation tower. The more reflux that is provided for a given number of theoretical plates, the better the tower's separation of lower boiling materials from higher boiling materials. Alternatively, the more reflux that is provided for a given desired separation, the fewer the number of theoretical plates required.

Such industrial fractionating towers are also used in air separation, producing liquid oxygen, liquid nitrogen, and high purity argon. Distillation ofchlorosilanes also enables the production of high-purity silicon for use as a semiconductor.



Section of an industrial distillation tower showing detail of trays with bubble caps

Design and operation of a distillation tower depends on the feed and desired products. Given a simple, binary component feed, analytical methods such as theMcCabe-Thiele method[15][24] or the Fenske equation[15] can be used. For a multi-component feed, simulation models are used both for design and operation. Moreover, the efficiencies of the vapor-liquid contact devices (referred to as "plates" or "trays") used in distillation towers are typically lower than that of a theoretical 100% efficient equilibrium stage. Hence, a distillation tower needs more trays than the number of theoretical vapor-liquid equilibrium stages.

In modern industrial uses, a packing material is used in the column instead of trays when low pressure drops across the column are required. Other factors that favor packing are: vacuum systems, smaller diameter columns, corrosive systems, systems prone to foaming, systems requiring low liquid holdup and batch distillation. Conversely, factors that favor plate columns are: presence of solids in feed, high liquid rates, large column diameters, complex columns, columns with wide feed composition variation, columns with a chemical reaction, absorption columns, columns limited by foundation weight tolerance, low liquid rate, large turn-down ratio and those processes subject to process surges.

Large-scale, industrial vacuum distillation column[25]

This packing material can either be random dumped packing (1–3" wide) such as Raschig rings or structured sheet metal. Liquids tend to wet the surface of the packing and the vapors pass across this wetted surface, where mass transfer takes place. Unlike conventional tray distillation in which every tray represents a separate point of vapor-liquid equilibrium, the vapor-liquid equilibrium curve in a packed column is continuous. However, when modeling packed columns, it is useful to compute a number of "theoretical stages" to denote the separation efficiency of the packed column with respect to more traditional trays. Differently shaped packings have different surface areas and void space between packings. Both of these factors affect packing performance.

Another factor in addition to the packing shape and surface area that affects the performance of random or structured packing is the liquid and vapor distribution entering the packed bed. The number of theoretical stages required to make a given separation is calculated using a specific vapor to liquid ratio. If the liquid and vapor are not evenly distributed across the superficial tower area as it enters the packed bed, the liquid to vapor ratio will not be correct in the packed bed and the required separation will not be achieved. The packing will appear to not be working properly. The height equivalent of a theoretical plate (HETP) will be greater than expected. The problem is not the packing itself but the mal-distribution of the fluids entering the packed bed. Liquid mal-distribution is more frequently the problem than vapor. The design of the liquid distributors used to introduce the feed and reflux to a packed bed is critical to making the packing perform to it maximum efficiency. Methods of evaluating the effectiveness of a liquid distributor to evenly distribute the liquid entering a packed bed can be found in references.[26][27] Considerable work as been done on this topic by Fractionation Research, Inc. (commonly known as FRI).[28]

Multi-effect distillation[edit]

The goal of multi-effect distillation is to increase the energy efficiency of the process, for use in desalination, or in some cases one stage in the production of ultrapure water. The number of effects is proportional to the $kW \cdot h/m^3$ of water recovered figure, and refers to the volume of water recovered per unit of energy compared with single-effect distillation. One effect is roughly 636 $kW \cdot h/m^3$.

- Multi-stage flash distillation Can achieve more than 20 effects with thermal energy input, as mentioned in the article.
- Vapor compression evaporation Commercial large-scale units can achieve around 72 effects with electrical energy input, according to manufacturers.

There are many other types of multi-effect distillation processes, including one referred to as simply multi-effect distillation (MED), in which multiple chambers, with intervening heat exchangers, are employed.

Distillation in food processing[edit]

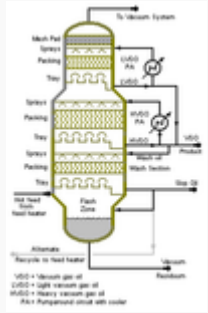
Distilled beverages[edit]

Main article: Distilled beverage

Carbohydrate-containing plant materials are allowed to ferment, producing a dilute solution of ethanol in the process. Spirits such as whiskey and rum are prepared by distilling these dilute solutions of ethanol. Components other than ethanol, including water, esters, and other alcohols, are collected in the condensate, which account for the flavor of the beverage.

Gallery[edit]

	Chemistry in its beginnings used retorts as laboratory equipment exclusively for distillation processes.
	A simple set-up to distill dry and oxygen-free toluene.

	Diagram of an industrial-scale vacuum distillation column as commonly used in oil refineries
	A rotary evaporator is able to distill solvents more quickly at lower temperatures through the use of a vacuum.

	Distillation using semi-microscale apparatus. The jointless design eliminates the need to fit pieces together. The pear-shaped flask allows the last drop of residue to be removed, compared with a similarly-sized round-bottom flask The small holdup volume prevents losses. A pig is used to channel the various distillates into three receiving flasks. If necessary the distillation can be carried out under vacuum using the vacuum adapter at the pig.