

HISTORY OF DISTILLATION

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AN IMPARTIAL and dispassionate study of competent sources leads to the almost inevitable conclusion that distillation in the present sense of the word's meaning was not known to antiquity. The apparent references and even the mention of the word itself by Aristotle, Theophrastus, Pliny, Dioscorides, Galen, and others only indicate that the writers or their sources had made observations of certain conditions and events which had great significance to them but which, in fact, they did not fully comprehend and understand.

The Alexandrian chemists are frequently credited with the knowledge of distillation, but an examination of the utensils which they describe and which could have been at their disposal discloses that these were not equipped with efficient cooling devices for condensation of vapors from low-boiling liquids (Figure 1).

From time to time, fragmentary references concerning the culture and knowledge of old tribes and peoples appear and these are interpreted to mean that some of the early Asiatic people, especially the Chinese, knew alcohol and, therefore, distillation. The study of distillation is inseparably connected with the history of alcohol, which as a medicine and stimulant was known in very early times but only as a mysterious and unknown constituent of readily available natural products. Of these, wine was the most prominent.

Arabs and Syrians are mentioned by some investigators as having had the use of alcohol, but they certainly could not have known it as we define it. Some of this erroneous belief is due to the fact that the name is Arabic—*al Kohol*—but it refers to any finely-divided powder.

Therefore, if we choose to leave legend, hearsay, and speculation and confine ourselves to established and documented facts, it becomes almost certain that the discovery of alcohol—which indeed seems to have been the first product of any scientific distillation—was not

made in the Orient but actually in the Occident, most probably in Southern Italy in the eleventh or twelfth century.

ARISTOTLE AND THE DISTILLATION OF SEA WATER

Aristotle and some of his pupils, especially Theophrastus, observed that by pouring wine into sacrificial fires the flame is made to flicker and to become intensified, but there is no indication that they were aware of the constituent which produced this effect. However, the observations of Aristotle are not confined to wine. Some of the admirers of this early Macedonian philosopher and physician credit him with the knowledge of methodical distillation of sea water. It is, nevertheless, significant that about 500 years later his profound commentator, Alexander of Aphrodisias, can furnish no additional information on this certainly important subject.

Alexander in his own right, however, is deserving of attention. His place of birth, Aphrodisias, is situated not far from the glamorous Carian city of Ephesus which was then the commercial and cultural center of the Eastern Mediterranean. However, not content with living in Asia Minor, he belonged to a group of philosophers called "Peripatetics" and is known to have lectured in Athens in about the year 200 A.D.

His most enlightening comment quotes a passage from the master's "Meteorologica" (I, 2) in free translation as follows: "They boil the sea water and suspend large sponges from the mouth of a brass vessel to absorb what is evaporated, and after drawing the liquid from the sponges they find it to be sweet water."

This would be no more than a practical application of Aristotle's theory, as expounded in "Meteorologica" (Book I, Chapter 9), in which he attempts to describe and explain the rain cycle as some sort of natural distillation without, however, mentioning the word.

Now the earth remains but the moisture surrounding it is made to evaporate by the sun's rays and the other heat from above, and rises. But when the heat which was raising it leaves it, in part dispersing to the higher region, in part quenched through rising so far into the upper air, the vapour cools because its heat is gone and because the place is cold, and condenses again and turns from air into water. And after the water has formed it falls down again to the earth.

He even enlarges his observations on water and at least makes an attempt to apply it to other fluids.

Salt water when it turns into vapour becomes sweet, and the vapour does not form salt water when it condenses again. This I know by experiment. The same thing is true in every case of the kind: wine and all fluids that evaporate and condense back into a liquid state become water. They all are water modified by a certain admixture, the nature of which determines their flavor (Book II, Chapter 3).

PLINY AND HIS "NATURAL HISTORY"

Even before the appearance of Alexander's commentaries, but more than 300 years after Aristotle, the great historian and philosopher Pliny the Elder (Plinius Secundus) who died in the destruction of Pompei, in 79 A.D., describes on his own account a process to obtain fresh water at sea and on shore. Since, however, it is known that his knowledge was not all original but derived partly from the great Greek philosophers before him, it is not impossible that he was to some extent copying from Aristotle's "Meteorologica." Nevertheless, it must be conceded that Pliny, basically a historian, was primarily concerned with the recording of known facts; whereas Aristotle was more of a theorist and philosopher.

In any event, Pliny writes in his "Natural History" (3):

As persons out at sea often suffer great inconvenience from the want of fresh water, we will here describe some methods of obviating it. Fleeces are spread round the ship, and on becoming moistened with the exhalations arising from the sea, the water is wrung from them, and found to be quite fresh.

Although not quite germane to this discussion, it is certainly of interest to also quote the sentence which follows immediately:

Hollow balls of wax, also, or empty vessels sealed at the mouth, upon being let down into the sea in a net, become filled with water that is fresh and potable.

A member of the Byzantine group which took up the work of the dispersed Alexandrians also mentions and considerably expands on the experiment described by Aristotle. He uses—perhaps for the first time—the word "distillare" or "destillare" which, however, means simply "to drop" or "drip off." The word is employed by several contemporaries. It is also used by Pliny in connection with snow and further by Seneca and Celsus, the medical writer of the first century A.D. While, therefore, we find the word "distillare" in a number of places, there can be no doubt that it was never used in our sense of the word.

In this respect, it is important to emphasize that the interpretation of "distillation" as used not only by the

Greeks and Romans, but perhaps even before them by the Arabians, is confused with what later was recognized and what certainly we now understand as sublimation.

The preparation of mercury and calamine, for instance, is described by Dioscorides who lived in the same century as Pliny, and later by Zosimus.

Pliny says in his "Natural History:"

Putting an iron Spoon having Cinnabaris in an earthen pot, they cover the Cup dawning it about with clay, then they make a fire under with coals: and ye soot that sticks to ye pot, being scraped off and cooled, becomes Hydrargyrum. It is found also in ye place where silver is melted standing together by drops on ye roofs.

The "Greek Herbal" of Dioscorides (4) contains this enlightening passage:

And ye servant that is under with all doth do ye same, and casteth on more coals, until all ye Cadmia that was laid on be consumed, so that by the burning, the thin and light part is carried into the upper room, and sticks to the walls and to ye roof thereof.

THE ALEXANDRIANS

Zosimus of Panopolis (5) who lived about 300 years after Pliny is credited with having written 28 books on alchemy, although only a few have been preserved. It is of particular interest to note that he describes some apparatus which he claims to have seen in the Temple of Serapis at Memphis where apparently some then elaborate laboratories were maintained.

There can be hardly any doubt that the distillation, or rather sublimation, of mercury was known to the old Egyptians at least one or two thousand years before Zosimus, because they combined the mercury which they separated from cinnabar with other metals and thereby obtained the magnificent pigments with which they adorned the funeral chambers and vestments of their kings. They also applied the pigments to the hieroglyphics in the halls of their temples, some of

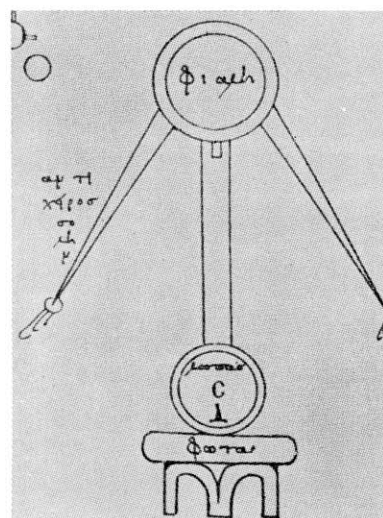


Figure 1. Dibicos of Cleopatra, First Century A.D.

which as in Karnak have retained their vivid colorings although exposed to the air for several milleniums.

Perhaps most interesting with regard to the history of distillation is that part in the Encyclopaedia of Zosimus describing the work of two female alchemists who lived about the beginning of the Christian Era—one Cleopatra (not to be confused with the temptress of Caesar's time and the heroine of Shakespeare's and Shaw's dramatic creations) and the other, Mary, the Jewess. The French chemist and historian Berthelot (6, 7) has given a detailed study of these two early female alchemists and outstanding members of the Alexandrian group. Cleopatra wrote a treatise, "Chrysopoea," on gold making, of which unfortunately only a single page has survived. It describes what is almost without any doubt the earliest distilling apparatus. It consisted of a sort of heating device under a circular vessel with a vertical tube leading into an alembic onto which were attached two condensers, which probably also served as receivers and in their twin capacity were called "Dibicos" (Figure 1).

Mary, the Jewess, wrote an extensive treatise on heating devices, which mostly were plates on which metals were heated and exposed to the action of mercury or arsenic vapor. She also described a double-walled heating vessel filled with water, which vessel

survived, almost unchanged, into our times as the indispensable water bath, the "balneum Mariae." The French designation for this universal instrument has remained to this day, "bain Marie," after its original creator.

It is therefore, evident that this group of Greek alchemists were acquainted with some sort of distillation, but the processes they employed did not differentiate sharply between sublimation and genuine distillation. In any event, they produced such remarkable results that the Romans became fearful of their influence and under a decree by the emperor Diocletian most of the records were unfortunately destroyed. However, some papyri survived and served Berthelot as basis for his enlightening studies.

There can be very little doubt that some of this primitive knowledge was not confined to the Greeks and Romans. It undoubtedly spread to other parts of the world and there similar discoveries and further developments took place.

ALCOHOL

All sorts of references can be found which indicate that peoples in many parts of the world prepared numerous types of fermented beverages from the grape and other fruits or from grains and vegetables. They recognized the presence of a powerful ingredient and attempted in many ways to extract it, especially because they suspected that it would have great healing power. There appeared some Arabic writings attributed to a group of alchemists called "True Brothers" which probably were written in the ninth or tenth century, but refer back to much older sources. These refer to the distillation of water without, however, presenting any practical procedure. Mentioned more prominently is the preparation of "rose water." Whether this "rose water," which appears frequently in many parts of the world, was to be used more for pharmaceutical or cosmetic purposes is not evident. It is worthy of note that these writings do not speak of wine or the recovery of anything which could be likened to alcohol.

Similarly, some sort of Persian pharmacopoeia which was written in the year 950 by a Persian physician, Abu Mansur, recommends distilled water for pharmaceutical purposes and indicates this application as a new discovery. It also mentions the art of distilling sea water as something entirely new so that marooned mariners would no longer have to die of thirst. Some investigators ascribe to a well known Arabic physician, Razi, the knowledge of alcohol and its use as medicine. However Razi uses the word "fermentari" only in connection with sugar, honey, and rice and describes the result as "vina falsa," or "imitation wine" (Latin words as translated from Arabic by Berthelot). Even better known is a later Arabic physician who lived about 100 years later. His name was Ibin Sina, but he became famous under the name of "Avicenna," the form his name took in Latin translation. He wrote more than 100 medical treatises, creating new doctrines enlarging on those of Galen and Hippocrates and modifying the



Figure 2. Title Page of Brunschwygk's "Liber de Arte Distillandi," 1500

concepts of Aristotle. Some sources ascribe a knowledge of alcohol to him without, however, any convincing evidence. Careful examination of the works of another and even later physician of Spanish-Arabic origin, Abul-Kasim, who died about 1107, discloses his knowledge of implements which in many ways resemble those of the Alexandrians but were used chiefly or exclusively for the production of "rose water." Significantly enough, he does not refer back to the comparatively recent works of the other Arabic physicians but rather to the much earlier writings of Aristotle, stating almost in passing that "Likewise it is possible, if so desired, to also distill wine," without, however, stating that this actually had been done.

Consequently, a dispassionate examination of these and other writings leads to the almost inescapable conclusion that the discovery of alcohol, which would obviously be connected with the knowledge of distillation, cannot be ascribed to the Arabians.

This development, therefore, moves almost definitely to the Occident, as already stated. It is not certain whether it took place first in Southern Italy or, as some believe, in Germany or possibly almost simultaneously in both places. Actual priority would be difficult to establish.

The fact is that there has been found in the South German monastery, Weissenau (8), a parchment document originating in the twelfth century. It is covered with medicinal rules and recipes, some of them in code in several different handwritings. One of these recipes mentions "aqua ardens," or in German "Brennendes Wasser," or therefore alcohol. It describes the process, freely translated as follows: "Place in the cucumber ('Gurke'—the distilling vessel) one pint wine and one pound red sulphur salt, or also four ounces living sulphur, which has been roasted in an ordinary pot and four ounces tartar, which you add all together and then close with a cover (ventosa), the watery part (aquositas) which flows down through the nose (nasum) of the cover you collect."

It so happens that this recipe is almost identical with a codex, also originating in the twelfth century, which was discovered in the Tuscan cloister of San Gimignano.

These two parchments indicate independent directions for the carrying out of an operation which certainly must be accepted as some sort of distillation. Nevertheless, there is a possibility, however remote, that both refer to a common source which at least one investigator claims to have traced back to the eighth century without being able to furnish any convincing proof of this supposition. How closely the two parchments can be associated with the work and the discovery of the group in Salerno, in particular the work of their leader, Magister Salernus (9), is difficult to appraise.

ALBERTUS MAGNUS AND VILLANOVA

Out of all this somewhat conflicting mass of evidence if such it can be called, there emerges clearly the fact that the first actual distillation and with it the first

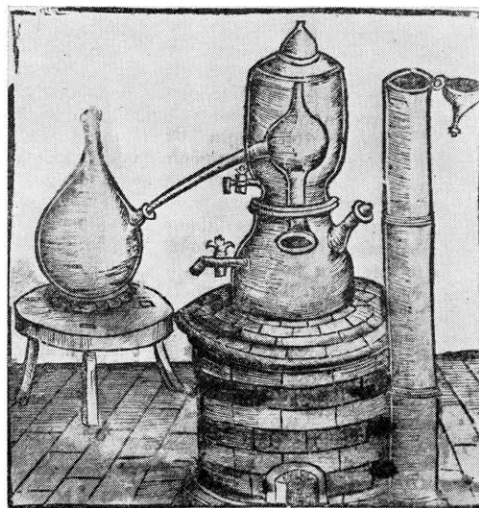


Figure 3. Brunschwygk: Still with Water-cooled Head

production of "aqua vitae" (alcohol)—two events which must be closely associated—took place almost definitely during the twelfth century. This supposition is further supported in the writings of one of the most reliable authors and scientists of this period, namely, Albert the Great (10, 11), a very versatile physician, philosopher, and naturalist born about 1193 in a small Suabian village near Ulm. He traveled, lived, and worked in a great many cities in Germany, Holland, Italy, and France, first as a student, then as lecturer, and even as a preacher for the Crusades and as Bishop of the Church. Albertus not only examined closely the writings of Aristotle, Pliny, and others pointing out their errors and misleading statements, but he also developed a classification of plants which led him very close to a system which Linné perfected several centuries later. In some of his writings, we again meet the confusion existing between distillation and sublimation. He states at one point that by "sublimating"—he does not use the word "distilling"—wine, also rose water, there is obtained a light liquid which swims on the surface and is inflammable or in his words, "liquor inflammabilis supernatans."

Albertus died in 1280, about halfway through the lifetime of another scientist to whom is ascribed, perhaps more generally than to Albertus, the first description of alcohol and its production by distillation. He was born as Arnald de Villanova (12) about 1235 in Spain but reputedly studied, presumably medicine, at Naples, and from then on traveled in many European countries as physician and lecturer.

He treated several of the Popes in Rome and at Avignon, and also James II in Barcelona. Undoubtedly, he spent much time in France and lectured at the then well known University of Montpellier about 1305. He was a very prolific writer and is considered by many as one of the most remarkable medical personalities of the Middle Ages. We find his books on many subjects published under a number of titles. In one of them, "Rosarius," he expounds completely

distillation principally for the production of the healing waters and elixirs which were then in general use. From primitive beginnings by the early Egyptians and the Alexandrian alchemists to the alchemists and healing monks of the Middle Ages, distillation was recognized as the principal tool for the making of healing draughts and scented preparations to cure all sorts of ills. It is, therefore, readily understandable that such a well-read and versatile scientist as Brunschwygk should have undertaken to write the first compendium on distillation. It is also natural that his book shows the predominating influence of the physician, but it gives, nevertheless, a clear and understandable picture of the distillation process as it was then known and of the apparatus which was then available (Figure 3).

In addition, however, it was a sort of compendium for various pharmacological preparations that could be obtained by distillation, enumerating the principal herbs to be used. The very interesting title page of the book shows not only an assortment of distilling apparatus but also some sort of botanical garden whose plants could furnish medicines, essential oils, and perfumes. The book must have found ready acceptance and quick distribution because it was followed rapidly by several revised editions. The first issue, being shorter and of smaller format, is now known as "Das Kleine Distillierbuch."

In later editions, the book was gradually enlarged and emerged finally in 1512 as "Das Grosse Distillierbuch" (17) (Figure 4), in a larger format and about twice the volume. This book constitutes an outstandingly fine example of early printing and shows an almost incredible wealth of beautifully-executed and highly-instructive woodcuts. Although Brunschwygk's book was preceded by several smaller printed works, as for instance one by Schrick (18) under the title of "List of Burned Waters" printed in 1483, these earlier works were not much more than short treatises.

As printing became more popular and as the art of distillation was developed and became better known, books on the subject began to appear more frequently in many cities of Europe and later elsewhere. The rapid progress which was being made is exemplified in a book printed in Frankfurt, in 1556, under the title "Neu Gross Destillierbuch," written by Walter Ryff (19) and distinguished by its wealth of fine illustrations with numerous woodcuts. Of particular interest are some that show the progress in heating and condensation devices (Figure 5).

PARACELSUS

It is perhaps no coincidence that during the time of Brunschwygk and Ryff there emerged a physician and student of nature who had a most profound influence not only on the scientific thinking of his time, but also most likely on the development of all science thereafter. He was born in a small village in Switzerland near the famous cloister and place of pilgrimage, Einsiedeln, where his father had also been a physician, and he bore

the impressive name of Aureolus Philippus Theophrastus Bombast Von Hohenheim. Some time in the course of his travels he decided to assume another name, either because he considered his own too much of a hardship or perhaps he already estimated himself at least as good, if not better, than the famous Celsus of the first century. In any event, he now became known as "Equal-to-Celsus," or Paracelsus (20, 21).



Figure 5. Ryff: Worm Condenser

Not willing to accept old and antiquated concepts, he not only became rebellious but also in somewhat antagonistic fashion proclaimed as obsolete the almost sacrosanct medical doctrines and concepts of Galen and Avicenna. He voiced his conviction that experience and observation should be the most essential parts of the curriculum of the physician. He urged alchemists and physicians to stop making gold and seeking mysterious elixirs and instead to study the fundamentals of science and find true medicines. In what is probably his best known work, "De natura rerum," he says:

I praise the chemical physicians . . . they busy themselves at learning the steps of alchemy—distillation, solution, putrefaction, extraction, reduction, coagulation, tincture.

Thus he really introduced chemical thinking into medicine.

Before Paracelsus, alchemy had been some sort of

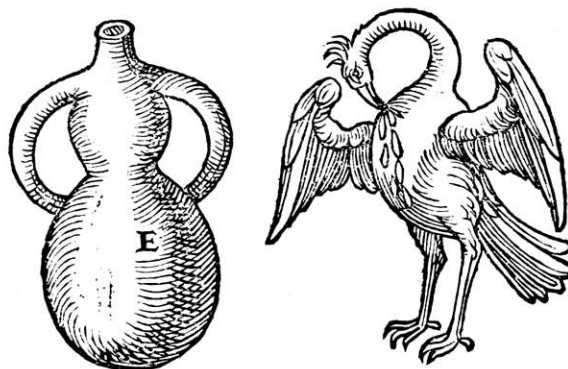


Figure 6. Porta: Pelican

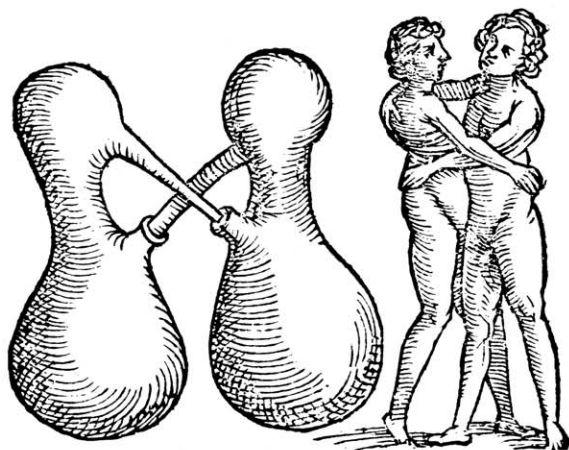


Figure 7. Porta: Twins

mysterious technique. Under his influence emerged the science of chemistry, and he was actually the first to use the word. It is significant that in many later books and by many writers chemistry became known as "the art of distillation."

Whether or not due to the appearance of books on distillation by Brunschwygk and Ryff and others, the knowledge of the art seems to have spread rapidly and considerable progress was achieved in methods and apparatus. Additional books on the subject appeared. One of outstanding importance was by a Neopolitan, Johannes Baptista Porta, sometimes called the "Italian Brunschwygk." He was born in 1545 and became a professor at the Academy of Naples in 1603 after having traveled extensively in his own country, Spain, and France.

Porta was a very learned man who lectured and wrote on many subjects such as philosophy, mathematics, astrology, and even a treatise on teeth. Outstanding is his work on distillation in nine volumes under the title, "de Distillationibus" (22). In one chapter, he points

out that distillation has been so named because it "is running drop by drop."

Porta also differentiates two classes of distillation, according to whether the vapors rise or are forced down. In accordance with "destillatio per ascendum" or "per descendum" the condensers and receivers are constructed and placed. He used various forms of digesting vessels which he likened to animals and sometimes even human beings. His favorite comparison is with a pelican. This name is used not only by Porta, but also by many later writers. Two connected vessels in human shape were known as "twins" or "brothers" (Figures 6 and 7).

Of particular interest is a multiple pelican showing six connected retorts or chambers with receiving vessels attached to the five upper ones (Figure 8). This arrangement can really be considered a primitive forerunner of our modern column still. However, it must be emphasized that Brunschwygk, Ryff, and Porta seemed to rely for condensing solely on air cooling and only rarely mentioned more efficient methods.

The important improvement represented by water cooling seems to be reserved to a Swiss scientist, Conrad Gessner, (or Gesner) born in Zürich in 1516. Despite his origin he is sometimes called the "Pliny of Germany," perhaps because he taught for a considerable length of time at Strassburg. However, he studied in France and after graduating as a physician in Basel settled again in his native city of Zürich. He wrote and published an astonishing number of works on different subjects, especially on medicine, of which the most important is "De Remediis secretis" (23) in which he devotes extensive chapters to the methods of distillation and various types of equipment. Besides explaining the difference between rising and descending distillation, he points out comprehensively the use of the water bath in preparing distillates and in a number of illustrations shows an interesting assortment of water coolers (Figure 9).

After Gessner's books, the most important ones are probably those by Libavius, already mentioned briefly, whose book "Alchymia" was printed in Frankfurt in 1606. The books by this author begin to show a number of modern tendencies. For example, a multiple head already indicated in Porta's book in a much more primitive form appears to be the forerunner of the column still. These gradual technical developments and improvements have been described in an admirably comprehensive and condensed article by Egloff and Lowry (24), "Distillation methods, ancient and modern." It follows the development of the process and the apparatus up to modern oil-distillation units, which are probably the last word in the development of this art unless the very intricate fractionating devices used in the modern laboratories can be considered a further refinement. As Egloff convincingly points out, more impressive than the reading of any books is the fabulous exhibition of early alchemical distillation and its growth

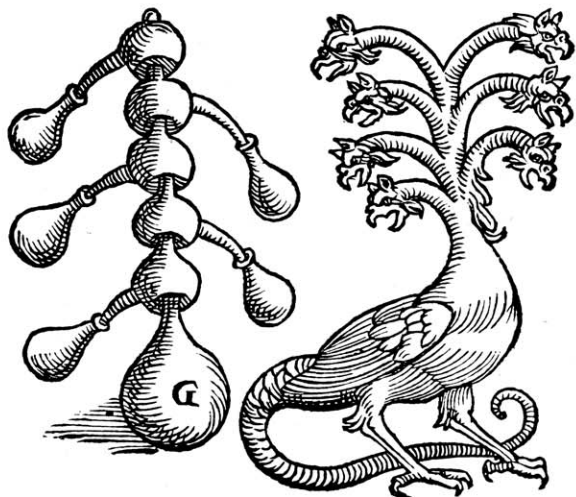


Figure 8. Porta: Hydra

and development into modern distillation in the Deutsches Museum in Munich, a wonderland of art and science. There is in existence, of course, a wealth of books, most of them highly specialized and devoted to every phase of the now highly diversified distilling industry.

In England, the first original books on distillation appeared some time in the seventeenth century. Notable is one by John French, "Dr. of Physick," printed in London in 1650 (25). His introduction "To the reader" begins with a sentence which certainly is true of a very large part of the writings on distillation of his and earlier times: "There is a glut of chymical books, but a scarcity of chymical truthes."

The first fairly comprehensive book was by Y. Worth, "The Compleat Distiller" published in 1705 (26), followed by some others of which perhaps the most interesting is "The Complete Distiller" by Cooper, first published in London in 1757 (27).

American books on the subject did not appear until the early part of the nineteenth century. The first one was by Michael Krafft, "The American Distiller," published in Philadelphia in 1804 (28). Interestingly enough, it carried a two-page dedication to President Jefferson. From then on books on distillation appeared with increasing frequency as the field for this art increased and broadened, the equipment expanded and improved, and the theoretical knowledge progressed under systematic scientific research and development. Notable among more recent reviews are those by Forbes (29), Underwood (30), and Lippmann (31).

The student of the art of distillation of today has at his beck and call a comprehensive array of literature covering its every principle, essential part, and aspect. He is the fortunate heir to all that has gone before in thought, experimentation, and discovery.

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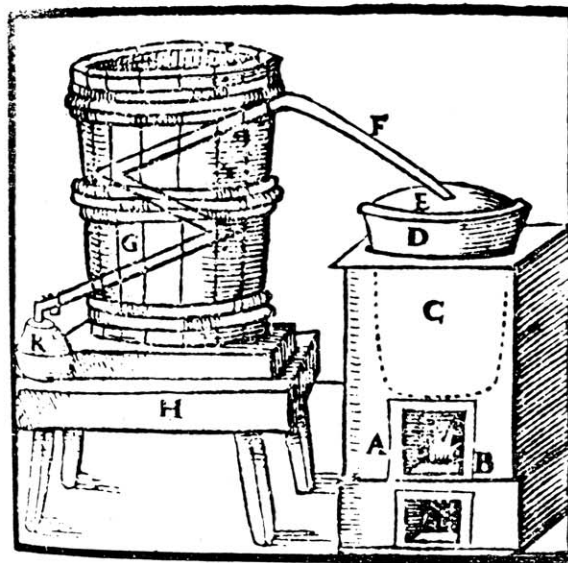


Figure 9. Gessner: Still with Water-cooled Condenser