(54) Titre : CONTROLE DU PROCESSUS DE DISTILLATION SOUS PRESSION OU SOUS VIDE SANS POMPES A VIDE; LES DISTILLERIES SONT DE DIVERS VOLUMES ET DISTILLEMENT UNE SUBSTANCE BRUTE A L'ETAT SEC OU LIQUIDE, PUIS EXTRAIENT ENSUITE DES HUILES ESSENTIELLES DE RESIDUS LIQUIDES APRES LA DISTILLATION

(54) Title: CONTROL THE PROCESS OF DISTILLATION UNDER PRESSURE OR VACUUM WITHOUT USE OF VACUUM PUMPS, DISTILLERIES ARE WITH DIFFERENT VOLUMES, THEY DISTILLING A RAW MATERIAL IN DRY OR LIQUID CONDITION, EXTRACTING FOR SECOND TIME ESSENTIAL OILS FROM LIQUID RESULTS OBTAINED AFTER DISTILATION

(57) Abrégé/Abstract:
This invention relates to control the distillation process for the production of essential oils using water or steam distillation in the processing of different materials in different sizes of the distillers under pressure and vacuum without use of vacuum pumps, in case of processing a sinking materials in distillers over 300 liters volume only in steam distillation the raw material is divided from grids diffusers, in case processing a raw material in liquid condition or processing on distillation leads from previous distillation is using cohabation attachment; precise control distillation process reduces the cost production of essential oils, produced distillate required for maximum extraction of essential oil percentage from the raw materials, is decreased by 2-2.5 times and the volume of extracted essential oil increased by 10-16%.
DIVISIONAL APPLICATION CA - 2,759,869

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ABSTRACT

This invention relates to control the distillation process for the production of essential oils using water or steam distillation in the processing of different materials in different sizes of the distillers under pressure and vacuum without use of vacuum pumps, in case of processing a sinking materials in distillers over 300 liters volume only in steam distillation the raw material is divided from grids diffusers, in case processing a raw material in liquid condition or processing on distillation leads from previous distillation is using cohabation attachment; precise control distillation process reduces the cost production of essential oils, produced distillate required for maximum extraction of essential oil percentage from the raw materials, is decreased by 2-2.5 times and the volume of extracted essential oil increased by 10-16%.
DESCRIPTION

The water distillation is the oldest way of distillation at which the material is immersed in water and boils together with it. It provides very good conditions for hydro-diffusion. At atmospheric pressure the temperature does not exceed 100 degrees C which is friendly to thermo labile components. It is irreplaceable for raw materials with high degree of sinking /rose blossoms, orange blossoms, ilang-ilang, etc./, for wild raw material for example such as – in France, Spain and Italy- lavender; Indonesia-kananga; China-cinnamon and for strongly ground plant parts since it ensures permanent mixing and contact with the water steam. It is technically and economical advantageous for raw materials with low degree of ethereal oil content. The water distillation disadvantages of the old methods are: the yield is less, the water-soluble and highly boiling components remain partially in the water remaining in the apparatus, the hydrolysis of the esters is accelerated and the quality of the oil is lowered, in cases of ineffective mixing the raw material or the extracted from it water-soluble substances could burn over the heating surface of the apparatus and to render a side odour of the ethereal oil, the duration of the distillation is restricted by the water present in the apparatus and in cases of a long process it is necessary an additional filling up, the apparatus capacity is not completely used, a considerable amount of water is heated which at the end of the process is thrown away which increases heat and time consumption.

On Figure 1 is shown Water generator of boiling water for water distillation soaking the raw material periodically, a process repeated many times with intense discharge of saturated steam with ethereal-oil fumes 1-Cylindrical reservoir; 2- Cone chamber; 3- Cylindrical chamber; 4- Circular curved pipe/ Fig. 2-elevation B/through which passes pre-heated water from pipe 6. In its circular profile it is exposed to peripheral heating from a gas burner 136/ Fig 14/ and enters reservoir 1; 5- Valve letting through the water coming from pipe 6 only in the direction of supply and not letting it through in the opposite direction; 6- Pipe supplying the already heated water to sponge 52 of cover 42 by means of a distributing cock 47- providing the hermetrical quality of reservoir 1; 7-Water generator for boiling water; 8- Boiler for processing the raw material; 9- Separating cone-shaped grid aiming at maintaining a short distance from cover 143 but at the same time providing enough air space for effective diffusion of the boiling water; 10-Cock for draining reservoir 1; 11- Cock for regulating water level in reservoir 1; 26- Legs which ensure fixed coupling between cone chamber 2 and the bottom of the generator, / the bottom shouldn’t be thinner than 2 mm in order to endure continuous direct fire because although for a very short span of time it remains dry without water and under vacuum yet deformations are undesirable/; 143 Hermetically welded /soldered/ upper lid of reservoir 1, concave on towards the reservoir with an opening in the middle with diameter equal to the diameter of cylindrical chamber 3. To 143 is welded the upper part of 3 and the lower part of 3 to 2 and by means of legs 26 is the fixed coupling with the bottom.

Cone chamber 2 is in the form of a wide angular cone with low height in order to cover almost the whole bottom of the generator without touching it and at the same time to ensure a small quantity of water under it. In fact this solves the problem with the
heating of an enormous quantity of water. After 10-20 minutes of heating starts the boiling of the water with a rather characteristic noise. It is a result of the strong thermal whirl of the water in chamber 2 vertically upwards, through chamber 3, through grid 9 and the soaking of the raw material with boiling water. With the intensification of the fire under the influence of the weight of the water in reservoir 1 and the strong thermal whirl in chambers 2 and 3 in vertical direction upwards from chamber 1 enters new water from chamber 2. It is heated in chamber 2 and in chamber 3 a permanent flow of boiling water towards the raw material is acquired and that goes on until the water from reservoir 1 completely drains and soaks the overall raw material with boiling water /the quantity of water in reservoir 1 is calculated as to be enough for soaking of the overall working capacity of the boiler having in mind the quantity of the material /raw stuff/. After that occurs a momentary sucking back of the water from the raw material moving back through grid 9, chamber 3, chamber 2 in reservoir 1, and its duration is very short /momentary/. This is a result of the vacuum that has been created in reservoir 1, the lack of water in chamber 2 and the weight of water pushed out by the thermal whirl in the raw material. This unique process is repeated cyclically, many times and in a direct ratio to the intensification of the fire in burner 136 /Fig. 14/. From that follows that the duration of the cycle decreases with the increase of the fire. This manifold repetition of the cycle continues and in that way the temperature of the raw material levels with that of the water. With the gradual rising of the temperature of the raw material accumulating discharge and pushing out of saturated steam with ethereal oils occur. As a result of that gradual temperature rising and periodical absence of water all components of the ethereal oil released in the concrete temperature point are extracted. In boiling at slow fire the cycle may continue for about 5-10 minutes. This suggests that the process itself can very precisely be controlled and in that way the above mentioned disadvantages can avoided /whereas the maximum duration of 5-10 minutes depends on the working capacity, the smaller the working capacity the shorter the maximum duration is/. The speed of the hydrolysis of the esters is slowed down and by means of that disruption of the direct contact of water with the raw material are reduced the losses of the water-soluble and highly boiling components of the oil dissolving in water. This raises the quality and the volume of the extracted oil from the raw material. The intense whirl mixes the raw material and by means of the so formed construction there is no direct contact of the raw stuff with the heating bottom, this prevents burning of the material and formation of a side odour of the ethereal oil. With automated supply of the already heated water through pipe 4 the process becomes incessant and it is not necessary to dispose of the water in reservoir 1 at the end of each process. This eliminates thermal energy and time consumption. Naturally if different raw materials would be boiled for each of them there should be clear water which is supplied by pipe 6 already heated. Yet before the supply of clear water from crane is drained the previous water. The technological process constructed in that way with permanent movement of water through the heated surface does not allow any burning.

It is known that water has the highest degree of density at 3,98 degrees C and while heating up to 100 degrees C at zero above sea-level height its temperature expansion changes its volume by narrow margins almost equal to zero. Water is non-contractible. After 100 degrees C it transforms into steam expanding its volume approximately 1670 times. The uniqueness of the method can compared to pulsating
heart-as the water the heart muscle and the discharged saturated steam with ethereal oil is the blood pushed out by the heart. The raw material exposed at intervals to the influence of the temperature pressure and the cyclically repeated vacuum is a kind of a mechanic maceration which helps for the easier bursting of the oil depository in the processed plants /raw materials/. This helps for the maximum extraction of ethereal oil by means of the new method of water distillation.

Distillation method in which through a layer of material passes steam is called steam distillation which can be formed inside the very apparatus /water and steam or water-steam distillation/ or the steam is supplied by a special steam-generator. The steam distillation with a separate steam-generator is the most widespread method and it has the following technological and economical advantages: the speed can be precisely regulated; steam with different pressure is used i.e. with different temperature depending on the specification of the raw material; the water-soluble components are distilled too because they do not get into water environment; the continuity of the process is unlimited; the extraction of the water-soluble non-volatile components from the material is avoided as well as the overheating of the material and of the same components on the hot surfaces; the hydrolysis of the esters is reduced to minimum as well as the other destructive changes of the ethereal oils.

Up to now the constructed distilleries for steam distillation have the following disadvantages: they are not mobile/transportable/, they require steam boiler and respectively qualified personnel; they are not applicable in cases of materials with high degree of sinking or finely ground since the steam creates passages /channels-tunnels/ and it can not affect the material completely, in cases of material with high degree of sinking it is necessary to be mixed with inert non-sinking materials which makes the production more complicated and expensive, it is not convenient for distillation of materials with small quantity of ethereal oil in them due to the enormous consumption of steam; the warm water from the condensers is not used to the best advantage; intense formation of condensate which periodically must be drained which leads to a partial break of the process or to additional heating of the bottom aiming at the reduction of the condensate.

Variant of the steam distillation is the so called water-steam distillation. With it the steam is formed in the apparatus out of water which is located under the material and is separated from it by a perforated grid. The steam is saturated and is under atmospheric pressure. Due to the small quantity of water that can be filled one-time before the start of the boiling the distillation is short-termed and in case of a continued process it is necessary to stop the process in order to fill up water or to return the distillation water back into the apparatus which technically and economically is inexpedient and results in quantity and quality losses of the ethereal oil. The highly boiling components of the oils are distilled with difficulty and not incompletely due to the low temperature. Part of the non-volatile water-soluble substances of the raw material get along with the condensate in the water and cause the formation of carbon deposits on the heating surface of the apparatus which results in the formation of a side odours of the oil and disruption of the heat exchange with the water. The speed of distillation is difficult to be controlled.
On Fig. 3 is shown a steam generator for steam distillation which is compactly joined to the distillery and this makes it mobile and transportable. It is placed under the grid separating it from the processed material. It produces superheated steam under 100 degrees C in vacuum regime or over 100 degrees C dry saturated steam and up to or over 150 degrees C superheated steam in pressurized regime; 4-Circular curved pipe /Fig 2-elevation B/ through which passes a pre-heated water from pipe 6. In its circular profile it is exposed to peripheral heating from gas burner 136 /fig 14/ and it enters reservoir 139; 5- Valve letting through the water that passes through pipe 6 only in the direction of water supply and not letting through in the opposite direction; 6- Pipe supplying already heated water to sponge 52 of lid 42; 7- Steam generator; 8-Boiler for processing the raw material; 9-Separating grid which at its ends has a compact, not perforated band which purpose is to restrict the formation of steam flow along the walls of the boiler without a direct contact with the raw stuff /the so called tunnels/; 10- Cock for draining reservoir 139, 122- Cock for draining tar 14. 13-Manometer combined with thermometer; 14-Tar; 15- Nozzle of the steam generator; 16-Steam; 17- Visual and sound indicator for correcting the water level in the steam generator, 18- Steam diverting pipe welded hermetically to the bottom and in its upper part between the hermetrical weldings of the middle part of nozzle 15 and the upper lid of the generator 140 there are steam diverting outlets along the circumference of the pipe; 19- Steam diverting pipe passing through the middle part of nozzle 15 and welded hermetically to it, suspended in pipe 18 and in its lower part ending with a cone; 20- cone chamber; 21- Cylindrical chamber; 22- Cone-shaped platform; 23- Pipe diverting the steam towards a separating and steam-diffusing grid 31; 24-Cock controlling the steam supply; 26- Legs ensuring fixed coupling between the cone chamber 20 and the bottom of the bottom of the generator; / the bottom shouldn’t be thinner than 2 mm in order to endure continuous direct fire/; 138 Pressure-relief valve; 139-Reservoir of the steam generator, 140- Upper lid of the steam generator.

With the steam generator are used the same methods of thermal whirl of the water heated in the cone chamber 20 whirling in an upward direction through the cylindrical chamber 21. It disperses on the cone platform 22 which stops the boiling water. The so designed construction helps for saving time and thermal energy, heating a small quantity of water and for an intense discharge of steam. The overflow of the boiling water from the cone platform 22 ensures great discharge of intensely saturated steam. As the water overflows it goes back, as a result of which a circular heat exchange occurs with the basic water in the reservoir 139. The released steam from the cone platform 22 passes through the holes made on the lid 140 around pipe 18, and the holes made along the circumference of pipe 18 in the lower part of nozzle 15. It enters pipe 18 and directs downwards to the heated bottom in the lower part of pipe 18. There the steam superheats and is drawn by the cone in the lower part of pipe 19, which ensures a larger heating surface for the steam. Through pipe 19 vertically upward it enters in a diffuser with circularly located outlets along the circumference of the nozzle 15. In that way the equally distributed superheated steam is supercharged and through grid 9 it enters in the processed raw material. The holes along the circumference of pipe 18 are located in the highest point of the lower part of nozzle 15. The water level in reservoir 139 is calculated so that without additional filling up the water to be enough for the boiling of 3 boilers of raw material. At the same time the volume of the water should be smaller than the
volume of half of the chamber of reservoir 139. The aim is when emptying the boiler from the material, after boiling. It is in a biased position that is more than 90 degrees/ and if the water is more than the have volume of reservoir 139 there is a risk of entering into the cylindrical pipe 18. The water in the process of boiling will be pushed out through nozzle 15 in the tar that will disrupt their quality. After the experiments that have been made with an empty boiler without any material and under the maximum thermal traction of burner 136/fig.14/ steam temperature has been reached of 145 degrees C with fully open cocks 56, 57 and 58 between lid 42 and balancing the pressure sponge 52/fig. 8 and 9/. This proves the good quality of the steam generator because depending on how compact the material is arranged, the maximum temperature increases in a direct ratio to the increase of the density. The cocks 56, 57, and 58 help very precisely to control with what pressure and respectively temperature the concrete raw material to be processed. The temperature that the steam can reach exceed 150 degrees C with closed cocks 56, 57, and 58 under pressure of 0.7 atmospheres. With seeds, in some cases with blossoms and finely ground materials, it is necessary for the stuff to be separated by grid-diffusers/fig. 4, 5, and 6/. This is necessary with the large working quantities. For example for 500 liters is needed one separating grid, with 750 and 1000 liters two separating grids. The grids are used only when the system is constructed for steam distillation. In case of water distillation separating grids are not used. The grids are located towards the perpendicular of the boiler 8/fig.4/ under 3 degree bias aiming at producing a better quality of steam-lining without stopping the released steam saturated with ethereal oils. The grids 31/fig. 5 and 6/ are constructed with a specific form to facilitate their assembly and disassembly. Technologically they are made by a compact imperious plane located below. It ensures the hermetrical quality of separation between the discharged saturated vapour with ethereal oil from the remaining below processed material and the supplied steam for processing the material above. Between base 32/fig. 7/ a moistened paper gasket is placed over which is placed a grid in order to increase the hermetrical quality of separation. The upper plane of the grid is perforated. The perforation as distanced from the periphery in order to prevent the formation of tunnels that may be formed towards the walls of the boiler. The two planes are separated by rings located so as to ensure an even distribution of the steam and not to allow any bending of the grid under the weight of the material and its pressing during loading. The separating grids solve the problem with the mixing of the raw material with the inert materials. In that way the complications and raising the cost of the process are avoided. On fig. 4 is shown the working part of boiler 8, without the steam generator/fig.3/ or the generator for boiling water/fig. 1/. It is wrapped with glass wool 33 which is pressed by wooden laths 34 tightened by braces 35. This thermo isolation has the purpose of ensuring better heat exchange between steam and material or steam-water and for economy of heat and time. The wooden laths also play the role of reinforcement which strengthens the construction. They prevent the possibility mechanical damage while loading or emptying the raw material. By steam diverting pipe 23 steam is supplied to the next levels, which is regulated by cocks 24 and 25. Pipes 29 and 30 divert the steam saturated with ethereal oils to the highest part of lid 42. Cocks 27 and 28 and 56, 57 and 58 control the even processing of the material. With different combinations of opening and closing of cocks 24, 25, 27, 28, 56, 57, and 58 it is possible very precisely to control the vacuum and pressure. Respectively the temperature for
processing the material totally for the whole boiler or for a particular separating level of raw material. In case of any possible error or closing of cocks 24 and 27 a safety valve 138 is activated which opens and prevents the system from excessive pressure. The angular profile is circular-curved metal shaped iron around which is plated the upper border of boiler 8 of flat copper. To the inner periphery of the upper part of the boiler is welded a circular-curved copper rim 36, and along the outside periphery is welded a circular-curved copper rim 38. Thus between the rims 36 and 38 a furrow is formed in which there is a silicone gasket and lid 42 is placed. It tightens and hermetically seals with clamps 133/fig. 14/. On fig.8 and 9 is shown lid 42 balancing the pressure by sponge 52. It has the form of a hemisphere and half of its surface has a water jacket 53. The water jacket is intended to have a maximum small volume with the purpose of quick heat exchange and cooling of the steam within very small temperature limits in order to prevent intense cooling and curdling of the steam. It is fed with water from the cooler by a hose 51 and is regulated by cock 50. The water jacket 53 can be replaced by a spiral-curved pipes the outlet towards cock 47 is the initial point with the narrowest diameter of the spiral and the entrance from cock 50 is the end of the spiral with the widest diameter in it. The distribution cock 47 changes the direction of the outgoing water from the water jacket. In direction by hose 6 towards the generator for boiling water (fig.1) if the distillery is designed for water distillation or steam generator (fig.3) if it is a system for steam distillation or by hose 48 towards radiator 129/fig.14/ for cooling and gathering in reservoir 128/fig.14/. This water jacket 53 has two main tasks in order to acquire a continuous and gradual cooling of the steam, which results in a good aggregation of the distillate. The purpose is to increase the diameter of the oil drops released in the water of the Florentine/an oil-separating vessel/. The other task when it is necessary to feed new water in the steam generator/fig.3/ or in the generator for boiling water /fig.1/, the water to be already heated to the possible maximum of the concrete situation. The angular profile 43 is a circular-curved shaped iron around which is plated lid 42/made of flat copper/. Compensating the steam-diversion pipes 44. The regulation of steam-diversion through pipes 44 is controlled by cocks 56, 57, and 58. The main steam-diverting pipe is 45. The manometer 46 for measuring the pressure. Straight rim in the form of semicircle functions as a reinforcement of sponge 52 to which rim 59 is welded with an opening for suspending the sponge on arm 61 (fig.10 and 11). Cone chamber 54 to which hermetically is welded a Hollander fitting with a nut 55 for creating a fixed coupling with the cooler 77 (fig.12). Base 56.

The so build construction of lid 42 with sponge 52 comes to be a basic and important junction for the good work of the system. The large volume of sponge 52 and at the same time the small diameter of pipe 45 helps for the balancing of the pressure with which the steam is supplied. Thus under the cone of lid 42 the system is under pressure, and in sponge 52 under the impact of the vacuum formed in the cooler 77, the pressure in sponge 52 is almost invariably equal to zero. This doesn’t load cooler 77 by cocks 56, 57 and 58 it is possible very precisely to control the pressure hence the temperature needed for processing of the concrete raw material. Based on the so formed construction of work of cooler 77 at full power of cooling in sponge 52 is formed a strong vacuum and at a minimum supply of steam by the precise opening of cock 56, 57 and 58 the system starts to work under vacuum. During the time of tests, when the system works under vacuum with remove clamps 133/fig.14/ it is practically impossible the lid
42 to open until the vacuum doesn't fall down. When the water in water jacket 53 is approximately equal to 40 degrees C then under the influence of the vacuum formed by the cooler in the material are extracted the thermo-labile components from the ethereal oil materials with temperature around 60 degrees C. At the experiments made by supplying the necessary volume of steam at a slow fire, the necessary degree of cooling and the precise opening and closing of cocks 56, 57, and 58 the system working under vacuum can process the raw material from 60 to 100 degrees C. And vice versa when the system works under pressure, the water in water jacket 53 under the thermal influence of the steam supplied by pipes 45 and 44 almost boils. If necessary it is possible to be supplied by hose 6 to the steam generator /fig. 3/ or to the generator for boiling water /fig.1/. In that way is saved a lot of heat and time.

On fig. 10 and elevation D /fig.11/ is shown the basic idea during a working process of loading and emptying of the raw material. The attention is drawn on the mechanical construction.

60-double-row bearing, ensuring the rotation around bar 68 of arm 61; 61-arm on which is hung lid 42; 62-rod which ensures a rectilinear movement of bar 68 vertically upward with the purpose of taking the lid 42 out of the furrow of boiler 8; 63 fixture; 64-rod which is firmly welded to a metal frame 72 which by means of a bearing is located in pipe 75; 65-arms reinforcing the triangular construction; 66-boltscrewed up in a nut 73 and fixing the boiler 8 in a vertical position; 67-muff, ensuring hermetical quality of the grease and of the normal lubrication and movement of bar 68 in pipe 74; 68-bar; 69-step; 70-triangle construction, base; 71-vertical reinforced rim; 72-metal frame, ensuring monolithic connection between the boiler and the base; 73-nut in which a bolt is screwed, fixing the boiler in a vertical position; 74-pipe, 75-pipe with friction bearing; 76-base welded on one side to pipe 75 and on the other to arms 65;

On fig. 12 is shown a cooler 77. This is a heat exchanging apparatus /condenser/ which is intended to condense the vapour of the water and the ethereal oil and to cool them to a certain temperature. The most popular in industry have become coolers of the type pipe bundle and serpentine (worm-pipe). The cooler of the type pipe bundle is compact, with a large cooling surface, it is easy to be disassembled and cleaned. It allows higher speed of distillation due to the lower resistance of the steam. As a result of the quick trickling of the distillate and its falling down in the collector the oil drops become smaller, which is not favorable for the extraction of the oil in the oil-separating vessel /Florentine/. The cooling surface is not used completely because the distillate trickles down in streams. That's why it is recommended the theoretically calculated surface of cooling to be doubled. The advantages of biased coolers of the type pipe bundle in comparison with the vertical pipe-cased coolers is the stable temperature regime of work which once settled preserves for the rest of the process. Practically it is found out that 1 square meter cooling surface cools 20-25 liters distillate for an hour.

The worm-pipe cooler is simple in construction and easy for repairing. The condensation in it is facilitated by the constant change of direction of the vapour. The aggregation of the oil drops is better and improves the discharge of ethereal oil in the oil-separating vessel /Florentine/. The disadvantages are the practical impossibility for tinning of the inner pipe, larger volume than that of the pipe bundle, little possibility to increase the speed of distillation due to the shown resistance, only part of the cooling surface is used in the cooling zone that is why it is recommended to triple the
theoretically calculated surface for cooling. To improve the exploitation parameters very often it is resorted to a gradual narrowing of the section of the pipe in the lower part which results in a more efficient use of the cooling surface. At normal work 1 square meter cooling surface cools for 1 hour 25-35 liters distillate with consumption of cooling water with an outgoing temperature of 70-80 degrees C. The worm-pipe coolers so far have been applied predominantly in the extraction installations and vacuum apparatuses, where it is worked with vapour that is cooled with difficulty.

51-hose supplying the already heated water coming out of the cooler towards sponge 52, 78-cock, regulating the supply of the outgoing water from the cooler 77; 79-cock, regulating the cold water towards the cooler; 80-hose, supplying the already cooled water from reservoir 128; 81-chamber with cooling water; 82 chambers I, II, III, IV, and V through which passes the steam and a process of condensation takes place; 83 and 84-pipes diverting the already condensed vapour /distillate/ containing ethereal oil; 85-Hollander part on the side of the cooler 77, ensuring hermetrical coupling with sponge 52; 86-thermometer by means of which is controlled the distillation process; 87-filter gathering the deposition particles from the oil sacks, which is cleaned periodically; 122-a drip pipe diverting the distillate; 123-a tip pipe supplying distillate to the oil-separating vessel /Florentine/; 148 and 149 technological outlets with caps for cleaning pipes 84 and 83.

The so designed construction of cooler 77 is consistent with above mentioned advantages and disadvantages. With the two types of coolers- pipe bundle and serpentine/worm-pipe/ the advantages are preserved and the disadvantages eliminated of each of the types. Chamber I is the primary calmer of the incoming vapour. The acquired condensate drains down through pipe 84 in chamber V and directs towards the outgoing pipe 122. The vapour passes through chamber II, which has the form of a frustum of a cone where it is further calmed down and the process of condensation goes on. In chamber III the vapour changes its direction vertically upward and at the same time meets the abrupt narrowing in the passage towards chamber IV. Pipe 83 diverts the condensed vapour /distillate/ into chamber V and so on. Pipes 83 and 84 function as a feedback ensuring no remaining distillate in the cooler. Pipe 84 diverts the distillate already formed in sponge 52 and that is the reason for the construction of the Hollander coupling in sponge 52. It has a cone-shaped outlet aiming at no retaining of distillate in sponge 52. Chambers IV and V are formed on the outer side with a cylinder in which is inserted a frustum of a cone with its narrow ending upward to the chamber I. Thus in the upper part between them is formed a larger volume whereas downwards a narrowing occurs with the aim of conge sting and increasing of the cooling surface. Practically chambers IV and V come to be a type of a generalized pipe bundle in which over the diameter of the cylinder the steam moving back upwards constantly changes its direction from chamber IV to chamber V resulting in an intense process of condensation. At the experiments made, with one and the same measurements of the cooler with pipe bundle and this new type of cooler we acquired 5 liters distillate more for 1 hour at one and the same working regime of working of the system. We approached the results with the coolers of the type serpentine/worm-pipe/. The cooler is not vertical but biased and once set on a definite working regime it does not change. IT can work at high speed of distillation and in that relation it is facilitated mainly by chambers IV and V. There is a constant change of direction of the vapour which does not result in the decrease of the size of the condensed
oil drops. After the construction changes that have been made in comparison with the cooler of the type pipe bundle we made the way of the steam longer 3 times and increased the heat exchange surface almost 50%. This is so because in order to increase the surface with the pipe bundle it is the number of pipes that is increased per one surface measure but this is done within certain limits because when the pipes become too many it is technically impossible to achieve a high quality welding of the pipes which results in many defects and the cooler becomes unreliable. Another thing that can be made is to make the pipes longer. This can be done within certain limits, too because the overall size of the cooler shouldn’t be too big. Its mobility/transportability/ is affected whereas the aim is to increase its efficiency.

On fig. 12-1 is shown a cooler 77 with the same measurements as those shown on fig. 12. It is with much greater efficiency, can work with a higher speed of distillation without decreasing its quality parameters. With it a new method is applied—that of the impulse cooling at which by cock 152 and pipe 155 is supplied cold water to the installed in serpentine 156. In the chamber formed by sections VI and VII, so that the cold water passing through the serpentine causes an intense process of condensation. It is the finalizing part of the cooling so that gradualness of the general cooling is acquired so that there is no curdling /abrupt cooling/. This gradualness helps the aggregation of the distillate with large oil drops. Through opening 157 at the end of serpentine 156 the heated water directs to the outgoing water from the cooler. The supply of the cooling water is regulated by cock 79 and when there are minimal changes in the process of cooling cock 152 is opened till the filling up of serpentine 156 with new cold water. In that way periodically the supply is provide at a regular intervals of time. At a high-speed distillation or at distillation under vacuum, cock 152 can be permanently open and regulate the capacity of the supplied cold water. The designed construction allows very precisely and permanently to control the process of cooling. The steam from chamber I through pipe 159 enters the hemispheric distributing chamber II. There it changes its direction to 180 degrees and then enters chambers III, IV, and V which are the space between the cylinder within a cylinder and which like terrace-like change their size. Consecutively passing from one to the another chamber the direction of the steam is changed. This helps the good quality of condensation of the steam and provides the necessary time for heat exchange. The cylindrical form of the chambers is a kind of a pipe bundle along which the condensed distillate drops from the upper part of the chamber and in its lowest part each of the chambers has a pipe diverting the distillate to chamber VII, the pipes 83, 153 and 154. Each of the pipes 83, 84, 153 and 154 has technological outlets for periodical cleaning with caps 148, 149, 150 and 151. The outside wall of chamber VIII is a cone which with the horizontal line joins a 3 degree bias. The aim is to ensure an unimpeded streaming of the condensed steam in it. In chamber VIII the steam once again changes its direction and enters the chamber from the sections VI and VII which are form with an outer wall cylinder and on the inner side there is a built in cylinder with a frustum of a cone. The chamber formed between the cylinder and frustum of the cone are the pointed on fig. 12-1 upper section VI and lower section VII. The outlets 157 serve for air freeing of the water chamber formed between the terrace-like chambers III, IV, V and chamber VI and together with outlets 158 they ensure normal heat exchange and diversion of the heated water towards the outgoing opening of the cooler. The construction designed in that way lengthens the steam path 5 times and
increases the surface of the heat exchange from 3 to 4 times / with the increase of the number of windings of serpentine 156. / The temperature of the outgoing water from the cooler reach 80-90 degrees C.

On fig. 13 is shown the oil-separating vessel-container /Florentine/ combined for light and heavy oils. Two-sectional glass module for primary check of presence of ethereal oil in the entering in Florentine distillate and a glass chamber for edging the already released ethereal oil.

The separating vessels /Florentine/ work on the principle of the interconnected vessels. They ensure a constant release of water and in some cases of oil. The separation of the components of the mixture in it becomes possible due to their practical inability to mix and the difference concerning their density. Depending on the relative density of the ethereal oil its layer is above the water or under it and respectively in a separating vessel for light oils, heavy oils or combined - both for light and heavy oils.

The separating ability of the receptacles for light oil is determined by the ratio K = \( \frac{V_1}{V_2} \), where

- \( K \) - the separating ability;
- \( V_1 \) – the velocity with which the oil comes to the surface, m/s;
- \( V_2 \) - the velocity of sweeping along of the oil drops by the distillate downward, m/s.

The speed of coming to the surface of the oil drops is determined by the of Stoke's law:

\[ V_1 = \frac{d^2 (p_2 - p_1) g}{18 \mu} \]

where
- \( d \) – is the equivalent diameter of the oil drops, m;
- \( p_1 \) - the density of the oil, kg/m\(^3\);
- \( p_2 \) - the density of the water, kg/m\(^3\);
- \( g \) - the acceleration of gravity, m/s\(^2\);
- \( \mu \) - the dynamic viscosity of the water, kg/(m.s).

At \( p_2 > p_1 \) (light oil) \( V_1 \) is a positive, i.e. the oil comes to the water surface.
At \( p_1 > p_2 \) \( V_1 \) is negative, i.e. the oil sinks to the bottom of receptacle.

The velocity of movement of the distillate downwards depends on its capacity and on the working section of the receptacle, i.e. on the surface through which the distillate drains down:

\[ V_2 = \frac{Q}{S} \]

Where \( Q \) - is the capacity of the incoming distillate, m\(^3\)/s;
- \( S \) - is the surface of the section of the working part of the vessel, m\(^2\).

It is accepted that for a normal part of the receptacle \( V_2 \) shouldn't be greater than 0,001 m/s at an unidirectional movement of the distillate and the ethereal oil, while at a polydirectional 0,0005 m/s.

From the above pointed formulas it is seen that the better separation of the oil is ensure at a lower velocity of distillation, larger drops of the ethereal oil, greater difference in the
density of the oil and water and small viscosity of the water. The last two factors depend on the temperature of the distillate. The more the temperature rises the more the difference between the density grows and the viscosity of the water decreases, which facilitates the separation of the oil from the water. Knowing that water at 3.98 degrees C, two molecules water unite in one molecule (H₂O); then the water has the greatest density. The size of the drops of the ethereal oil depends on the speed of condensation. The gradual condensation favours the formation of large drops of ethereal oil. The high temperature of the outgoing from the coolers cooling steam allows the gradual condensation.

It is accepted that the capacity of the separating vessel of the periodical functioning apparatuses at average speed of distillation is enough to be 2-3 % of the capacity of the distillation apparatus. For the good quality of separation it is necessary the distillate to stay in the vessel at least 15-20 minutes.

The separating vessels are usually located one per distillation apparatus but in case of expensive oils two vessels are located consecutively. The second has a controlling function. When working with expensive oils it is usually resorted to gathering the material from all the apparatuses in one bigger receptacle, which results in less smearing, losses due to evaporations and pollution.

Florentine fig. 13: 87-periodically cleaned filter gathering the depositions and particles from the oil sacks, 88-cover hermetically sealing glass chamber 89, in which the primary edging is made and is determined the thickness of the oil ring of the entering distillate in the oil-separating vessel. By closing of cock 90 periodically a test is made for the oil content in the distillate. Thus by means of testing samples is controlled the duration of the process of boiling: 91-module for periodical edging; 92, 114 and 115 silicone, ensuring the hermetical sealing of the connections of the glass modules with the respective modules of the Florentine; 93-pipe through the distillate is supplied to the working cone vessel 107, ending in its lower part with a perpendicular spoon 94, by which the direction of movement of the distillate is changed vertically upward. In that way the trajectory of movement of the distillate is prolonged and the time for oil discharge, 95-a cock which under normal working conditions is closed and when necessary can be used for transferring the distillate into a second separating vessel or for direct use of the ethereal-oil water; 96-a cock which is constantly open and is closed only when cock 95 is open or when is made a high-speed draining of the already separated light oil. Then both cocks-95 and 96 are closed so that the entering distillate in 107 raises the level only in chamber 107, whereas the level of the liquid in chamber 121 does not change. In the process of permanent incessant draining of the light oil by half-closing of cock 96 the capacity of the entering distillate is regulated through cock 96 in chamber 121 to smaller than the capacity of the distillate entering from pipe 93. In chamber 107 this leads to a gradual raising of the level only in chamber 107 and a continued draining occurs by edging in glass chamber 113 of constantly releasing ethereal oil; 97-hollander technological coupling which provides easy assembly and disassembly if cock 96 becomes defective; 98-a cock for draining the already separated secondary light oils; 99-a cock with pipe 108 for diverting the water separated from the distillate / at work it is always open/, 100-cock for emptying or draining of the separated secondary heavy oils,101-a cock for draining of 107 or for heavy oils, 102-air-vent with stopper 103 for
hermetrical sealing. The stopper is removed during a process of non-high-speed draining of the released ethereal oil as cock 99 is closed resulting in raising the level in chamber 118, that gradually starts to fill up. If the stopper 103 is not removed it may cause babbling with bubbles in the already separated light oil so this may disrupt its clarity and quality, 104-oil-diverting pipe of the already edged oil in chamber 125; 105- heavy oil; 106- light oil; 107- a primary working chamber for oil-separation. It is comformable with the formula of decreasing sweeping in downward direction and that is why chamber 107 has the form of an asymmetric frustum of a cone. The lower base of the cone is bent toward cock 101 with the purpose of normal gathering and draining of the heavy oils when such are processed, 108- a pipe intended to ensure the constant process of overflow of water sucking from the middle part of the Florentine 121 in order to be at an equal distance from the secondary light and heavy oils and not to affect the normal heat exchange sucking up the coldest water from below, 109- a cone pipe, an overflow drain of the water from the primary chamber 107 to the secondary chamber 121. The construction of overflows by means of the cone pipe 109 is made so as to get a difference between the levels of the liquids so that the level in chamber 107 to be higher than the level in chamber 121 thus to preserve one of the basic ideas referring the normal heat exchange between the liquids in chambers 107 and 121. Not to disrupt the interconnected vessels but at the same time to ensure a greater difference between the temperatures of the releasing light oil 106 and the temperature of the water in the lower part of chamber 121 must approach the temperature with which the water has the greatest density-3.98 degrees C. In that way is preserved the formula of the speed of coming to the surface of the oil drops, defined by Stoke’s law \( V_1 = \frac{d^2 (p_2 - p_1) g}{18 \mu} \), so that the water to be with the greatest density, but at the same time the temperature in the upper part of chamber 107 where the light oil is, to be the highest ensuring a process of maximum oil-release. If we accept that during a polydirectional movement of the distillate and the ethereal oil \( V_2 = 0.0005 \text{ m/s} \text{ for 20 min in a vessel with cylindrical form, as the section is one and the same along the section of the column in the process of release of the oil the oil drops will pass a 60 centimeters column.} \) That is why chamber 107 has the form of a cone, along the vertical plane the section is variable, towards the base the section enlarges from which it follows that \( V_2 \) decreases. At \( V_2 = 0.0004 \) for 20 min is necessary 48 cm column with \( V_2 = 0.0003 \) for 20 min it is 36 cm column, etc. The narrowing of the section of the cone along the vertical plane causes the released oil drops to accelerate in their movement upwards, whereas the distillate moving downwards decreases its velocity. The idea of the so designed construction is to achieve a maximum high parameter for \( V_1 \) and maximum low parameter for \( V_2 \). Thus we achieve a maximum ability of separation K and the Florentine is multifunctional, applicable to raw materials with small content of ethereal oil as well to materials with greater content of ethereal oil. Its quality parameters are not changed after the connection of more than one distillery. After the experiments that have been made with the Florentine with capacity 120 liters three distilleries can be connected with working capacity of 1000 liters. 110-thermometer, 111-hermetically sealing cover, 112- bolts with nuts screwed tight and ensuring the hermetrical quality between the modules and the glass chamber 113; 113-glass chamber which serves to exert visual control over separating oil from water during the draining of the oil in the dehydrator 124; 116- module with a small volume of
chamber 125 with the purpose of making a direct edging of the oil. That is also convenient for processing of materials both with small content of ethereal oil and with greater content of ethereal oil: 117- a horizontal base for tightening between modules 116 and chamber 107 of the glass chamber 113; 119- vertical base; 120-secondary light oil.

On fig. 14 is shown the principle diagram of distillery: 126-water pump, the supply of cold water can be provided by interconnected vessels without a pump; 127-a pipe-sucker, 128- a tank for collecting the cooled water; 129-a radiator cooling the water with a vane 130; 131-a hose supplying the cooled water from the radiator to the reservoir 128, 132- a silicone gasket between boiler 8 and lid 42; 133-U-shaped clamps ensuring a hermetrical fixed coupling between the boiler and the lid. Depending on the diameter of the boiler the number of the clamps 133 increases as the diameter increases, too. They are located at 20-25 cm from each other. In the process of work the furrow formed between the periphery of the boiler and the lid is poured water. If there are outgoing bubbles periodically the concrete clamps 133 are tightened; 134-gas bottle /propane-butane/, 135-a hose supplying the gas to burner 136. 137-sheet iron cylinder perforated and keeping the heat to the bottom of the boiler and at the same time preventing the fire of burner 136 from draughts; 138-safety valve.

At the testing samples made for example with boiling SUMAC OIL-OLEUM COTINI (OLEUM COGGYGIARIAE)

Normally in Bulgaria it is derived from 0.13-0.21 % ethereal oil, whereas we achieved 0.24-0.26 %. Our research at Bulgarian rose-Kazanlak after the processing 1/one/ ton dry material of sumac for the maximum extraction of ethereal oil from 21 % are used up 400 liters distillate. With new method and the new technical solutions with the distillery was extracted 0.26 % ethereal oil and were used up 175 liters distillate which is 2.29 times less of the produced distillate for extraction of a greater quantity of oil compared to the distilleries used up to now.

After the processing of SALVIA OIL in Bulgaria from OLEUM SALVIAE SCLAREA from the young plants reaching the height of 0.90-1.10 meters the extraction of ethereal oil that has been reached amounts to 0.22-0.25 %, and from the second 1.30-1.70 meters up to 0.27-0.30 %. With OLEUM SALVIAE OFICINALIS in Bulgaria has been reached extraction of ethereal oil up to 0.18-25 % as the upper limit refers to plants reaching the height of 1.50 meters. With the distillery has been extracted ethereal oil amounting to 0.30 % young material with height of 0.90 meters.

On fig. 15 and fig. 16 is presented a nylon profiled cloth with cylindrical chambers 141 which are filled with water, then are sealed and rolled in order to be frozen in a freezer. It is used at work in the open field where the raw material is in case of necessity. For example as shown on fig. 16, the already frozen cloth is wrapped around the Florentine 121 and is tightened with belts 142. This saves time and consumption of cold water and the process of oil-separation is stable.

On fig. 17 is shown a triple unifying module for periodical edging. Analogous to module 91 to which three distilleries can be joined as the processes of each of the distilleries can be controlled and check by cocks 143, 146 and 147.

On fig. 18,19, and 20 is shown a device for boiling cohabation of secondary water or a billet transformed into pulp. Fig 20 is barrel 144, which is placed in the boiler over the grid and a cylinder is inserted in it with a cone lid along the outer periphery of the
upper part of cylinder 145. It is oriented as to be in the center of the boiler and its lid to enter in the furrow of the boiler pressed by lid 42 and tightened by clamps 133. The diameter of barrel 144 is a little bit bigger than that of 145 so that the space between the barrel and the cylinder is small. It ensures an unimpeded passing through of the superheated steam through it reaching the bottom of the barrel 144 from there sweeping vertically upward and transforming the oil drops into vapour. The construction is designed so that the superheated steam wraps the overall surface of barrel 144. It heats the liquid in it and the formed pressure makes the steam to pass through the passage between the barrel 144 and cylinder 145, it sweeps the oil drops before the entire liquid has started to boil with a very characteristic babbling. The quantity of the liquid can be to the maximum of 2/3 of the capacity of the barrel. This is so because the pressure of the steam pushes out the liquid which is in the space between the barrel and the cylinder in cylinder 145. The maximum pressure is up to 0.7 atmospheres, knowing that 0.1 atmosphere displaces 1 meter of the water column. This device makes the system multifunctional and convenient for work and in that way the ethereal oil production cycle closes.
CLAIMS

1. In an apparatus for producing ethereal oils by distillation, a lid unit located above a water or steam distillation unit and comprising:

   a lid (42) in the shape of an inverted cone, seated between two concentric rims located at the top of distillation unit and engaging sealing means disposed between the two concentric rims, the lid further comprising:

   main steam-diverting pipe (45) extending from the apex of the lid and fluidly connecting the water or steam distillation unit to a space above the lid

   secondary steam-diversion pipes (44) passing through the lid and fluidly connecting the water or steam distillation unit to a space above the lid, the steam-diversion pipes being equipped with cocks (56, 57, 58)

   clamping means (133) to hold the lid in place,

   a hemisphere (52) located above the lid, such that when the hemisphere is in a closed position, the outer rim of hemisphere engages an outer portion of the lid, the hemisphere further comprising:

   cooling means to cool the steam entering the space formed between the lid and the hemisphere such that the steam is cooled within very small temperature limits,

   lifting means to allow the hemisphere to be raised from its position above the lid with slope on the horizontal to drain condensed steam into chamber (I) from the cooler,

   cone chamber (54) connected to the hemisphere at a lower position thereof, comprising a union connection fitting to provide connection to a cooler (77) for the steam exiting the space formed between the lid and the hemisphere,
the volume of the hemisphere and the diameter of the main steam-diverting pipe are chosen such that, in operation, when the system is operating under vacuum, a large enough vacuum is generated to keep the hemisphere in connection with the lid.

2. The lid unit of claim 1, for distillation with boiling water or steam is using lid with hemisphere for control the processing in dry or liquid condition of raw material into boiler (8), under pressure or under vacuum without using pumps.

3. The lid unit of claims 1 and 2, wherein the cooling means is water jacket (53), located inside on top curve of hemisphere.

4. The lid unit of claim 3, water jacket can be replaced with bent pipe with form of spiral, wherein the largest diameter of the spiral is connected to a cock (50) through which enters the water coming from cooler and the small diameter of the spiral is connected to a cock (47) from which water comes out, thereby facilitating construction of the unit.

5. The lid unit of claims 3 and 4, wherein the water jacket is fed by hose (51) with the cooling water coming from a cooler already heated, thereby resulting in condensation of the steam into the cooler.

6. The lid unit of claim 5, wherein use of the water jacket in the hemisphere extends the path for cooling steam.

7. The lid unit of claims 5 and 6, whereby the dimensions of the water jacket are such that they contribute to the gradual cooling of the steam within small temperature limits, thereby increasing aggregation of oil droplets and improving oil separation of oil from the oil-water mixture.
8. **The lid unit of claim 3**, wherein a hose (6) is provided to supply near-boiling water from the water jacket to a reservoir of the water or steam distillation unit.

9. **The lid unit of claims 1 and 2**, wherein the cocks provided on the secondary steam-diversion pipes help control the amount of steam passed into the hemisphere, thereby maintaining the pressure in a boiler.

10. **The lid unit of claim 9**, wherein, in operation, when working at full power vacuum in the hemisphere, the opening of the cocks of secondary steam-diversion pipes helps create a vacuum in a boiler.

11. **The lid unit of claims 9 and 10**, wherein, in order to prevent the creation of steam tunnels in a raw material being treated, the raw material is divided and supplied on different levels within a boiler using a diffuser grille (31).

12. **The lid unit of claim 11**, further comprising a pipe (23) to supply steam to the levels and pipes (29, 30) for discharging steam saturated with essential oil from the levels, said pipes to supply steam and said pipes for discharging steam being equipped with cocks (24, 25, 27, 28).

13. **The lid unit of claim 10**, further comprising cohabitation device (144, 145) installed in boiler for processing raw material in liquid condition, thus creating a liquefied raw material.

14. **The lid unit of claim 13**, in operation, steam entrains oil droplets separated from the liquefied raw material at high temperature.