Steam distillation of the superficial essential oils: Hypotheses from studies with lavenders and mints

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Essential oils used in cosmetics and perfumery are extracted from plant materials by hydrodistillation. Hopefully, improvements in the production of such base materials from self renewing resources will be of some interest to the user industries.

In Tasmania we have made large advances in both botanical and agricultural techniques aimed at producing more reliable materials at lower cost (see photo 1). But the value of such advances in the plantations would be much reduced if the recovery of the oil by distillation did not also make comparable progress. Here we have to break new ground because the orthodox explanations of the process provide no help. We cannot even ascertain the time that a new still will require to extract the oil from a given herb without actually building the equipment and testing it in the field. This is a serious impediment to the planning and cost estimation of new projects, because unless we know this extraction time we cannot determine the daily handling capacity of the distillery.

Accepted theories of essential oil distillation from the herb are wholly preoccupied with vapor pressures and for this reason are based on an assumption that is demonstrably untrue. But, if we add consideration of the latent heat, we reach a hypothesis which may explain those phenomena which to date have proven intractable. More importantly, especially in the case of oils secreted near the surface of the herb, the hypothesis relates the optional design features of a commercial distillery to its performance in the field. Thus, for any given scale of capital investment, the design of the distillery can be pre-calculated to give maximum through-put while minimizing damage to or losses from the oil.

Tables I and II cover the six test runs of peppermint and the 20 tests of five different lavenders carried out so far. The charge sizes range from 126 kg to 473 kg and the distillate flow rates from 67 to 192 litres per hour. The close relationship between the calculated extraction times and those actually observed suggests that these 26 tests conformed to the present hypothesis.

Field distilleries are relatively simple. The oil bearing herb at ambient day temperature is loaded into vat-like stills and a steam tight lid, often incorporating the outlet pipe, is clamped on top. A flow of steam is then introduced beneath the charge of plant material. Starting at the bottom, the steam condenses onto the herb surfaces and raises successive layers to boiling point. When the appropriate temperature reaches the top of the still any oil that is exposed on the herb surface will start to boil away. The oncoming steam will then drive a mixture of oil and water vapors off the top of the charge, at which time it is led to a condenser. This continues until the oil is virtually exhausted. In the liquid state the oil and water are immiscible and separate according to their densities (see fig. 1).

The ability of the steam to gather the herb's essential oil is usually explained like this: since equilibrium between a liquid surface and the contiguous vapor space occurs almost instantaneously, any oil that is exposed on the herb surface must immediately evaporate into the passing steam until oil saturation of the vapor space is reached or until the exposed surface oil becomes exhausted, whichever occurs first. Since the oil's proportion of the distillate is always much smaller than its saturation of the vapor space requires, it is assumed that only part of the herb's oil content can be exposed at the start of the distillation. The rest is taken to be overlaid by fibrous plant material through which it diffuses slowly to come in contact with the steam.

This proposition has been sustained for over 70 years by its apparent conformity with Dal-



Photo 1 Lavender fields at The Bridestowe Estate Nabowla, Tasmania

ton's Law when saturated steam is the medium of distillation. By this law the proportions of oil and water in the distillate should vary with the ratio of their vapor pressures inside the still. For example, if we distill with saturated steam under two atmospheres of absolute pressure instead of one, the higher temperature might triple the vapor pressure of the oil while it scarcely doubles that of water. The oil's proportion of the distillate will then increase in the ratio of 3:2 and only ⁴/₃ as much steam will have to pass to extract a given yield of oil. Although the oil's proportion of the distillate does not even approach the vapor saturation level at either operating pressure, it does, in fact, vary more or less as the accepted theory postulates.



Figure 1

If we now use superheated steam to increase the vapor pressure of the oil without increasing the partial pressure due to steam, we get an even more advantageous vapor pressure ratio of oil to water. According to the theory, this should further increase the oil's proportion of the distillate. But it does not. The resulting distillate is far less rich in oil than when saturated steam of equal temperature is used.

Another difficulty is that in very many cases

the total oil content of the herb can be seen to be free and on the herb surface when distillation starts. According to the theory, the earliest distillate condensed should then contain oil and water in the vapor saturated ratio. Regrettably, it does not. Further, if oil is sprinkled all over the top of the charge in absurdly generous quantities before clamping down the lid, the early

Table I

Results of six test distillations of <u>Mentha piperita</u> carried out at The Bridestowe Estate's Nabowla distillery on February 27, 1973 to demonstrate the inverse relation between extraction time and speed of steam over the herb surfaces. Distillate flow rates are stated on the basis of litres condensed per min for each square metre of the cross section area of the top layer of the charge. Basic information is derived from charge No. 834/73 and used to predict the extraction times for all other charges by calculation.

non times for an other charges by calculation						
Charge No.	Mass kg	Flow	Extraction Times Calculated Observed			
854/73	282	2.86		18.8 min		
857/73	414	2.56	24.4 min	24.8 min		
859/73	288	1.78	32.4 min	31.5 min		
860/73	257	1.11	49.4 min	51.5 min		
861/73	187	1.96	25.0 min	25.5 min		
862/73	245	2.41	23.3 min	22.5 min		

Table II

Subsequent distillations of various genotypes of <u>Lavandula angustifolia</u> (Syn, <u>L. officinalis</u>) checking validity of the height and time formula. Parameters <u>1</u> and <u>x</u> are derived from the first two runs of each genotype, and used to predict the extraction time for the others by calculation.

Charge No.	Mass kg	Geno- type	<u>Extractio</u> Calculated	on Time Observed
80/75	166	от		
81/75	304	от		
83/75	423	от	19.05 min	19,20 min
25/75	126	SB		
26/75	274	SB		
28/75	400	SB	10 .96 min	10.90 min
928/77	344	JP		
927/77	429	JP		
938/77	192	JP	16.30 min	16.44 min
630/76	158	JP	14.54 min	14.90 min
629/76	453	JP	23.49 min	23.40 min
918/77	172	RB		
915/77	256	RB		
926/77	421	RB	20.44 min	20.46 min
726/74	Z38	RB	15.24 min	15.20 min
981/75	420	MS		
978/75	301	M5		
979/75	175	MS	13.83 min	13.83 min
614/76	473	MS	26.48 min	26.51 min
619/76	152	MS	14.56 min	14,57 min

distillate's proportions are no nearer saturation levels.

There are many practical objections of this sort to the common theory, but the fundamental considerations are even more intractable. For the oil to evaporate into the vapor stream it must receive latent heat, and inside a field still this can come only from condensing steam. Therefore, the temperature of the steam in the general vapor space must be higher than that at which oil is boiling in the presence of water on the surface of the herb. Otherwise there could be no temperature gradient to lead heat from the steam to the vaporizing oil. It follows that the oil's concentration in the general vapor space must be significantly less than saturation level. This ensures that the steam's temperature cannot fall below that at which it will exert the total ambient pressure, less only a meager contribution from the unsaturated oil. Obviously, this is higher than the temperature at the oil's point of evaporation on the leaf where, by definition, saturation conditions must prevail and the oil exert its full potential vapor pressure. Only then can we get the temperature gradient we require.

Conversely, if it were ever possible for the distillate mixture of oil and water to attain the vapor saturated composition, the temperature in the general vapor space would have to be the same as that at which oil and water boil together on the leaf. There could be no temperature gradient and distillation of the oil could not occur.

However, in present practice, for every oil and set of conditions there is a balance point for the maximum attainable oil concentration in the general vapor space. Any further enrichment of that vapor would lower its temperature and reduce the gradient. This would retard the rate that heat is delivered to the oil and reduce its rate of evaporation till the normal balance situation was restored. This is a perfect example of "feedback."

With this evidence we have plotted the oil to water ratio in the early distillates from five differently sized charges of lavender genotype MS. Although the largest charge contained over four times as much oil as the smallest, all start with about the same ratio of oil to water. The small differences are due to the greater reflux in taller charges (see fig. 2).

Since the common explanation of this distillation process is neither acceptable in theory nor useful in practice, it is worth searching for an alternative hypothesis.

With a low power magnifier, the oil glands on a ripe mint leaf are visible as small blisters approximately 50 microns in diameter. The outer membrane is very fragile and ruptures immediately when contacted by steam. This releases the oil to form a small and roughly circular oil wetted area on the leaf surface (see photos



Figure 2

2 and 3).

At the start of the distilling operation the steam will moisten all the leaves by condensing on them to raise the temperature. Scattered over these water moistened surfaces there will be numerous circular patches of homogeneous oil from the ruptured glands, and around each of these there has got to be an interface where oil and water are in contact.

When the temperature reaches, for example,

98°C under normal atmospheric pressure, the additive properties of the two mutually insoluble liquids' vapor pressures will cause the oil and water mixture at the interface to boil. Oil saturated vapors must now rise from the entire perimeter of the surface oil patch so that the homogeneous oil area will be overlaid by oil saturated vapors in equilibrium with its surface. Net evaporation from this oil surface cannot now occur, so it cannot receive latent heat directly from condensing steam. But steam particles will readily condense into water surfaces in immediate contact with the surface oil's perimeter, and this provides the latent heat to vaporize contiguous oil.

It must appear that all the latent heat to vaporize the oil is delivered via water surfaces adjacent to the interface. Boiling of the oil can occur only at the edges of the numerous surface patches and these perimeters recede during distillation until the radii are nil and the oil has been exhausted. Clearly, the time to reduce these radii is the extraction time for the oil. To calculate this time we need to understand both the factors which vary the length of the radii and those which govern the speed with which the perimeters recede.

The rate at which oil will vaporize depends, first, on the speed with which oil saturated vapors are removed from over the interface and, second, on the rate at which fresh steam can be delivered to the point of contact to replace that which has already surrendered latent heat to the vaporizing oil. Evidently, both these factors are proportional to the speed of the steam inside the still. In other words, the time required to extract a surface born oil from its parent plant material by reducing the radii of the oil patches to nil is inversely proportional to the rate of displacement of the steam over the herb surfaces inside the still.

All of this implies the existence of an area around the interface from which the vaporizing oil attracts its latent heat. While the factors which influence the size of this heat transferring area are of supreme importance to efficient distillation, they are not germane to the present discussion, and it must suffice to say that the magnitude of the heat transfer area is predetermined by conditions on the surface of the herb which do not alter with changes in operating pressure taken on its own.

It can be shown from first principles that the mass of steam continuously in contact with a given heat transfer area, and hence the mass of oil evaporating at any time, must follow the value of the cube root of the steam's density squared which varies with changes in operating pressure.

where M = mass of steam in contact with a surface
D = density of steam
S = area of contact surface
K =
$$\left(\frac{9N.\pi}{16W}\right)^{1/3}$$

N = molecules per mole steam

W = molecular weight of water Returning to our earlier example, we operate with saturated steam and raise the absolute pressure from one atmosphere to two. The cube root of the steam's density squared rises by a factor close to 1.5 so that, under the higher pressure, 1.5 times as much steam is in contact with the heat transfer area. Heat is delivered to the oil at 1.5 times the previous rate and it boils away proportionately faster. Therefore, only 3/3 as much steam is required to pass to exhaust a given yield of oil. However, if we merely superheat the steam at atmospheric pressure, there is no increase in the density nor in the rate that the oil receives heat and boils away. Although other adverse factors also operate, this is the fundamental reason that superheated steam fails as a medium of distillation compared to saturated steam of equal temperature.

In addition to the optional factors like speed of steam and working pressure which influence

$$M = \frac{S \cdot D^{2/3}}{K}$$

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the extraction of all surface born oils in much the same way, there are also the non-optional factors characteristic of each oil and plant material. The oil's volatility and latent heat are obviously important and so is the condition of the herb surface for the transference of heat. Less obvious, but just as important, is the arrangement of the oil glands on the plant material.

The glands on mint are widely separated and their oil should not spread enough to run together in a well conducted distillation (see photo 2). Those on lavender, however, are crowded together so that shortly after distilling temperature is reached the oil from several glands will join together to form one large surface patch out of numerous smaller ones. This causes systematic changes in the relation between the total length of active oil-water interface and the amount of oil present (see photo 4). The influence of these factors upon extraction time is largely determined during the period when the charge is being raised to boiling temperature.



Photo 4. Lavender oil glands crowded together. Scanning electron microscopy courtesy of John Innes Institute, Norwich, U.K.

Consider the right hand diagram in figure 3a. Picture the plant charge as a number of layers each 1 cm thick. Let steam be admitted to the still at a rate sufficient both to raise one layer to boiling point in seven seconds and also evaporate oil from one layer for seven seconds. When the lowest layer attains boiling temperature, a mixture of steam and oil vapor will rise off it and condense onto the second layer for a period of seven seconds. When the second layer boils, the vapors, which will rise and condense onto layer three, will comprise the usual seven seconds worth of steam plus the first seven seconds of oil from layer one and the first seven seconds of oil from layer two. This will go on up the still with every heating layer receiving an extra quantity of oil equal to the first seven seconds of oil distilled from every layer below it.

This crude model shows that, for the time it





takes to rise from starting temperature to boiling point, every piece of herb must be receiving condensing oil as well as steam. Moreover, it suggests that the amount of oil received will increase with the distance up the charge.

A proportion of this condensing oil must lodge into the natural oil areas formed by bursting glands. This increases their radii and extraction time. Those on the highest layer affected by these increments will receive the most oil and will take the longest to distill. If they also happen to be the top layer of the charge, their extraction time will be that for the exhaustion of the oil.

We can now measure accurately the rate at which both oil and water are condensing at all stages of the distillation of two charges having different heights but both composed of similar test material. This is best done by collecting each two minute fraction of the distillate in a separate bucket and measuring the amount of oil and water in each.

From these readings we can derive two very important figures: the basic time required to extract a natural oil patch formed from bursting glands as though no increment in its area related to charge height had occurred and a parameter for the increment in these natural oil areas due to each unit of charge height.

Both in theory and in practice it can be shown that, for superficial oils, the numerical value of the basic time varies inversely with the speed of steam and that of the increment parameter inversely with the herb's oil content per kilo. As the yield of oil and the steam speed will be known for the test charges, these parameters can be adapted to any other set of conditions in which it is proposed to distill commercial quantities of the tested herb.

Thus, if the adapted basic time is t and the adapted increment parameter is x, the extraction time for any charge of the tested material having height H will be given by T:

$$T = t \left(\frac{x + H}{x}\right)^{\frac{1}{2}}$$

This formula is valid for cases like lavender where temperature gradient and vapor composition stability are not attained during the heating of practical sized charges. However, it is achieved very quickly afterwards by the temperature gradient.

Now look at the right hand diagram in figure 3b. The problem is even simpler with other superficial oils like mint where the gradient equilibrium is attained at height H during the heating period, and H is less than the normal charge height. Subject only to simple modification for optional changes of conditions, the value of T becomes characteristic of all charges, and the relation between extra extraction time and additional charge height above the vapor equilibrium level at H will be linear under a steady flow of steam.



This is clear from the distillation curves of two differing sized charges of mint, where the curved part of the graph represents the extraction of the oil from below the level at which equilibrium was attained during heating and both are similar in shape. The straight part of the graphs presents the linear relation between the amount of steam required to pass and differing amounts of oil which start distillation above the heating period's equilibrium level (see fig. 4).

We can compare the curves for mint and lavender to show the effect of equilibrium occur-



ring at a level below the top of the charge during the heating period. The early part of the mint graph is straight, but that for lavender is curved throughout its length (see fig. 5).



All this basic information can be obtained from a few test runs with any commercial still that is roughly cylindrical in shape. The optimum design of the distillery for any other set of given conditions can then be calculated very simply before turning to the drawing board. This is preferable to building the equipment first and then hoping for the best when it is tested in the field.

Appendix 1

Derivation of the formula relating charge height and extraction time

Let the basic natural oil patches from the glands have radius r and area a. A given steady flow of steam will reduce this radius to nil in the basic time, t minutes, where $t = E^{-1}$. $r = E^{-1}(a/\pi)^{\frac{1}{2}}$ if E^{-1} is the constant of proportionality.

During heating, a mean increment da is added to the basic area a for each unit height of the charge, so that for a charge of any height H the magnitude of the homogeneous oil areas on the top layer of the charge will become (a + H.da) by the time distilling temperature attains that level. The extraction time T, under the same steady flow of steam, will then be given by

$$T = E^{-1} \left(\frac{a + H \cdot da}{\pi} \right)^{\frac{1}{2}}$$

The factor of increase in extraction time from T to T' due to raising the charge height from H to H' is given by

 $\frac{\mathrm{T}}{\mathrm{T}}^{\prime} = \left(\frac{\mathrm{a} + \mathrm{H}^{\prime} \mathrm{.da}}{\mathrm{a} + \mathrm{H} \mathrm{.da}}\right)^{\frac{1}{2}}$

From any two accurately timed charges having different heights but composed of similar test material, we can now evaluate the basic area ain terms of the increment for unit height of charge da. Then, in the expression a = x.da, the ratio x becomes the increment parameter for the plant material under test.

With similar reasoning and substituting x.da for a, we can now derive the other necessary parameter, basic time t, from the results of either of the test charges, such that

$$t = T\left(\frac{x}{x + H}\right)^{\frac{1}{2}}$$

Then, if the numerical value of t is adjusted inversely for any change in the rate of displacement of the steam over the herb surface and that of x inversely for any expected change in the yield of oil per kilo of herb, the extraction time Tfor any charge of the tested material having height H will be given by

$$T = t\left(\frac{x + H}{x}\right)^{\frac{1}{2}}$$

End points

It is quite impossible to determine the exact time that distillation of oil ceases from any charge. Of more importance is the point at which the rate of oil production no longer defrays the cost of operation. In most of this work, end points based on terminal oil flows of 15 ml or 10 ml per minute have been adopted. In carrying out parameter test distillations these figures must be adjusted to ensure that the oil patches on the top layer of all subject charges are reduced to the same nearly negligible but, none the less, equal radius.

Comment

A more penetrating exposition than is possible within a single 20 minute paper must reveal that oil distillation from the herb is not a true equilibrium or steady state process even though it has been convenient to use such terms in the text.

Therefore, although the reasoning offered relating extra charge height to additional extraction time appears tenable as far as the principal factor is concerned, it hardly needs to be stated that other factors operate as well. One of the most obvious is the increasing tendency with height for oil from several ruptured glands to run together and form a single larger surface oil patch from numerous smaller ones, thereby increasing the amount of oil present relative to the length of active oil-water interface at the perimeter from which oil can be distilled. Another factor is the geometric reduction of the steam's moisture content with increasing charge height; there are several others of which the individual effects are understood but of which the net result of their interaction becomes imponderable.

However, being derived by experimental means, the mean value for parameter x becomes a very serviceable statement for the resultant of the principal factor as it is modified in practice by the other effects. In view of this, although the

parameter method is not rigorously sound in calculating extraction times for those distillations like mint, where vapor composition stability is attained during heating at some level below the top of the charge, its use will not normally lead to design errors of any commercial significance because the empirically derived mean value for x will automatically contain some compensation for the departure in method.

Appendix 2

Application to elementary design problems– Parameter method (For lavender and oils of similar behavior)

Problem no. 1: A client has a one ton electric hoist and a 25 horsepower boiler. He requires a cylindrical still to handle one ton of fresh lavender MS per hour of which the oil content is expected to be ten ml per kg in an average season. Recommend still dimensions, distillate flow rate, and terminal oil flow to obtain this through-put with the equipment in hand.

Basic data: Parameters for fresh lavender MS have been determined, t = 7.0887 minutes and x = 24.396 for the following conditions:

Still cross section 1.026m² Terminal oil flow 15 ml/min Herb oil content 9.066 ml/kg Distillate water flow 3.17 litres/min Charge density of packing 308 kg/cu.m Operating pressure—atmospheric

Method: The material will be harvested into a steel container ready for distillation and general considerations suggest the charge weight may not exceed 700 kg. A still 1.6 m tall and 1.3 m diameter will have cross section 1.327 sq.m and capacity 2.123 cu.m, with charge mass 654 kg.

To duplicate the test rate of displacement of steam on the larger cross section area charge we require $3.17 \times (1.327/1.026) = 4.10$ litres/min. The numerical value of *t* will then remain unchanged.

The value of the increment parameter must vary inversely as the oil content per kilo so that x becomes 24.396(9.066/10) = 22.1174 (for charge heights measured in cm). Extraction time is now given by

$$T = t \left(\frac{x + H}{x}\right)^{\frac{1}{2}} = 7.0887 \left(\frac{22.1174 + 160}{22.1174}\right)^{\frac{1}{2}} = 20.34 \text{ min}$$

The heating time is calculated at 17.81 minutes bringing the total time that is inversely proportional to the rate of steam supply to 38.15 minutes. In addition, five minutes are required to replace an exhausted charge so the total time each charge must occupy the still is 43.15 minutes. (Handling capacity is then $654 \times 60/$ 43.15 = 909 kg per hour.)

Actual time that can be allowed each charge minus time not influenced by flow rate

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$$.654 \times 60 = 39 \min \frac{5}{34} \min \frac{34}{34} \min \frac{5}{34} \lim_{n \to \infty} \frac{5}{34}$$

Answer: Flow rate must be increased in ratio 38.15/34 and becomes 4.6 litres per min. This does not exceed 75% of the boiler's capacity.

The base terminal oil flow must be adjusted for changed yield and water flow rate, becoming 15(10/9.066) (4.6/3.17) = 24 ml oil/min. (No adjustment should be made for altered cross section area.)

Application to elementary design problems– Vapor equilibrium method (For mint and oils of similar behavior)

Problem no. 2: When the lavender season is over, the still proposed as a solution to problem no. I will be used to process peppermint herb. Advise the probable handling capacity per hour and terminal oil flow rate. The herb is expected to be chopped to lengths that will pack into the still at the rate of 275 kg per cubic metre and to contain 6.4 ml oil per kg.

Basic data: Basic information for mint distillations was derived from *Mentha piperita* herb in average clover dry condition on the basis of a terminal oil production rate of 15 ml per minute under a standard distillate flow of 3.17 litres of distillate water per minute through a charge of 1.026 square metres of top cross section area and an oil content of 7.52 ml oil per kg. The basic data are as follows:

- Recoverable oil below temperature gradient equilibrium level 768 ml/m²
- (2) Oil not recovered at equivalent terminal oil flow 31 ml/m^2
- (3) Distillate water required to extract these 768 ml oil 33.7 l/m²
- (4) All oil starting distillation above the gradient equilibrium will be extracted at the rate of 63.34 ml/l water.

Method: The proposed still has cross section area $1.327m^2$ and capacity 2.123 cu.m. A typical charge will be $275 \times 2.123 = 584$ kg and will contain 584×6.4 ml oil = 3738 ml of which $31 \times 1.327 = 41$ are not recoverable, leaving 3738 - 41 = 3697 to be recovered. Of these, $768 \times 1.327 = 1019$ will start below the gradient equilibrium level and for their extraction will require to pass $33.7 \times 1.327 = 44.7$ l.H₂O. The remaining oil will start distillation above the gradient equilibrium level. Whence 3697 - 1019 = 2678 ml oil will be extracted at the vapor equilibrium rate of 63.34 ml/litre of distillate water passed and will therefore require passage of 42.3 l.H₂O.

At the proposed rate of distillate flow, 4.60 litres of water per minute, the 87 litres of water required to pass as distillate will flow in 19 minutes and this will be the extraction time.

Answer: Heating and change time can be calculated at 18 minutes, so the total time each charge must occupy the still is 37 minutes. Therefore, the hourly handling capacity is

$$584 \times \frac{60}{37} = 947 \text{ kg}$$

The base 15 ml/minute terminal oil flow must be adjusted for the yield per kilo of herb and the different flow rate which is

$$15 \times \frac{6.4}{7.5} \times \frac{4.60}{3.17} = 18.6$$
 ml oil per min

Design problems-General notes

(1) Still dimensions: Both the parameters and the vapor equilibrium propositions give mathematical expression to the general experience that stills for processing relatively coarse materials which yield their oil fairly rapidly should be as tall in relation to diameter as convenient handling will allow. However, the rule is not quite universal and must be accepted with a little caution. It is less likely to apply to the processing of finely comminuted materials which yield oil slowly. Indeed, it is open to question whether steam is the best method of hydrodistillation for some of these materials.

Distillery handling capacity: An essential oil

distillery will only give the designed throughput in terms of tons of herb per hour if the yield of the material per kilo is the same as that postulated in the design conditions. Lower yielding herb will increase the tonnage handled per hour but will reduce the oil produced in the same period below the design rate. Conversely, higher yielding herb will reduce the tons of herb put through per hour but will raise the oil produced above the designed amount. From any single set of parameters or basic data any likely range of handling capacity or daily production can be calculated.

Caution: The example problems and solutions are given to illustrate the methods of calculation used only when test steam and herb are typical of the quality to be employed in commercial operation. Factors, other than operating pressure, which affect heat transference on the herb surface have not yet been discussed and these have a large bearing on extraction times.

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