

Parameter test distillations for the superficial essential oils

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In an earlier paper* this author suggested a method by which it appeared possible to calculate the through-put of a new distillery for essential oils during the planning phase if two relevant parameters were known. It was proposed that the oil from a single gland of an herb would form a circular surface patch when the gland bursts on contact with the steam in the distillery. This patch would have a basic area (a) and distillation would reduce its radius to nil at a rate proportional to the actual speed of the passing steam in the basic time (t) minutes; designated parameter (t).

During the initial heating period at any level of the plant charge, this basic area may be increased by an amount (da) by oil from each lower layer of herb having unit thickness. Steam of similar actual speed will then reduce the surface oil areas at the top of a lavender charge having height (H), from $(a + H \cdot da)$ to nil in (T) minutes, and this is the extraction time for the charge.

The factor of increase in extraction time due to raising charge height from H to H' is given by equation 1.

$$\frac{T}{t} = \left(\frac{a + H' \cdot da}{a + H \cdot da} \right)^{1/2}$$

From any two accurately timed charges of different height, the increment parameter (x) can be derived from equation 1 by expressing (a) in terms of (da) such that $a = x \cdot da$. Then the observed results of either charge will give the value of parameter (t) from the general expression for extraction time (equation 2).

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

The previous paper showed a table comparing calculated extraction times with those actually observed under various conditions in different seasons. The present paper gives the original distillery observations for five test charges of lavender genotype "MS."

The present calculations, also given in full, use a new set of entirely optional "standard conditions" and these are now the same for both the 1975 and 1976 seasons.

Parameter tests—general method

Three or more charges of similar herb, but having different heights, are distilled with steam of similar quality under virtually ambient atmospheric pressure. Precise two-minute fractions of the distillate are caught in separate buckets and then allowed to settle out in separatory funnels before the volumes of water and oil in each are measured and recorded beside the time each bucket was removed from under the condenser outlet. The mass of each charge and the dimensions of its top layer for cross-section area are also recorded.

The progressive total of the oil produced is then graphed against the clock time and a suitable systematic curve drawn as nearly as possible through the points observed. This curve will be extended by estimate to indicate approximately where it would become asymptotic with the time axis, and thereby, give a figure for the total oil content of the charge. Due to the presence of plant waxes and so forth other bodies will continue to be distilled long after all valuable essential oil has been exhausted. This makes it very difficult to get a usable "estimated virtual exhaustion" (E.V.E.) figure for the oil content of the charge by continuing the distillation well beyond any normal end-point.

Standard conditions

In practice it is neither possible nor necessary to ensure that all the test distillations are run under identical conditions. Oil yields, rates of flow, and even charge cross-section areas may have to vary between one run and another. Nonetheless, each distillation must be truly comparable with all the others. To effect this a number of "corrections" must be applied to convert the actual results observed to those that

*Perfumer & Flavorist 4(5), 14 (1979).

would have been recorded if it had been possible to conduct all the tests under identical standard conditions.

Any realistic set of conditions may be adopted as standard. In the present tests the following have been.

- Charge top cross-section area 1.026 m² for charge mass up to 310 kg.
- For larger charges a still extension is used with a top area of 1.1675 m².
- At normal density of packing a 300 kg charge is 95 cm high.
- The standard rate of distillate water flow is 3170 ml/minute.
- The standard oil content for this herb is taken at 9.09 ml/kg.
- The standard end-point is when oil production falls to 10 ml/minute.

Determining start time of average distillate flow

The total amount of water in all buckets except the first is divided by the time taken for it to pass. This gives the average flow rate for the distillation. The water in the first bucket is then divided by this average flow rate to get the time it should have taken to pass. The virtual start time of the distillation is then this amount of time earlier than that at which the first bucket was removed from under the condenser outlet.

Determining clock time of end-point

For the extraction times of all charges to be comparable they must have a common end-point when all oil patches on the top of the charge have been reduced to the same nearly negligible but none the less equal terminal radius, designated (q). Herbs of similar origin will have oil glands of similar average size so that variations in oil content may be taken as accurately reflecting the variations in the number of glands present on the top layer of the charge per unit area. To ensure that a variable number of oil patches have been reduced to the same radius (q):

$$\text{True comparable terminal oil flow} = \text{Standard flow} \times \frac{\text{Actual oil content in ml/kg}}{\text{Standard oil content}}$$

Similarly, variation in the rate of distillate water-flow affects the amount of oil delivered per minute. If this were faster than the standard rate it would result in the oil patches on the top layer of the charge being reduced below radius (q) if the appropriate correction is not made such that:

$$\text{True comparable terminal oil flow} = \text{Standard flow} \times \frac{\text{Distillate water's actual flow at end-point}}{\text{Standard distillate water flow}}$$

This true comparable oil flow can be depicted by a straight line on the graph of oil produced against clock time. The point where it makes a tangent to the curve shows the clock time of the end-point and the oil produced at that time. The time interval from the

virtual start to this end-point is the extraction time under actual conditions.

Standardising extraction time

Assuming equal density of packaging in all charges, the rate of steam displacement will be proportional to the rate of distillate flow. Theoretical considerations have suggested that the rate at which the oil patch radii recede is proportional to the steam's rate of displacement. The actual extraction time will therefore vary inversely as the rate of distillate flow, such that:

$$\text{Comparable extraction time} = \text{Actual time} \times \frac{\text{Actual distillate water flow}}{\text{Standard water flow}}$$

Also, at any given rate of distillate flow the steam's rate of displacement inside the still is inversely proportional to the cross-section area of the charge. In the case of lavender the top layer of herb receives the greatest build-up of oil during the initial heating period and the surface oil patches at that level take the most time to shrink to radius (q). It follows that it is only necessary to consider the effect of varied cross-section area at the top of the charge such that:

$$\text{Comparable extraction time} = \text{Actual time} \times \frac{\text{Standard cross-section area}}{\text{Actual cross-section area}}$$

These two corrections convert the extraction time under actual conditions to the time that would be observed under the standard conditions.

It should be noted that, other conditions being unchanged, variations in charge cross-section area do not affect the terminal oil flow for determining end-point. The variation in the number of oil glands present on the top layer of the charge will be exactly compensated by the change in the amount of steam traversing each in unit time.

Standardising charge height

The build-up of oil on the top layer of the charge during the heating period, before distillate starts to flow, depends on the concentration of the oil vapour condensing onto it. In the case of lavender and other herbs of similar behaviour, each lower layer of equal oil content and specific heat may be regarded as making an equal contribution to this condensing oil. As far as charge height is concerned, the effects of variations in the herb's oil content and the variations in still cross-section area are adequately compensated if a virtual charge height is used such that:

$$\text{Comparable charge height} = \frac{\text{Estimated virtual exhaustion oil content (EVE)}}{\text{Standard oil content per unit height}}$$

It will usually be convenient to adopt one of the test charges as having a normal oil content per unit of charge height and then use this figure as the standard.

Estimated virtual exhaustion (E.V.E.) oil content

It is quite impossible to determine accurately the exact time that a distillation ceases to produce valu-

able essential oil. It is, therefore, both experimentally and operationally convenient to treat the extraction as coming to an end at a point where the rate of oil production ceases to defray the cost of operation; the so called "commercial end-point." Nonetheless, we require a working estimate of the total oil content of the charge and this can only be obtained by extending the graph of oil produced against time, and testing the various estimates till compatible figures are obtained. As both a guide to and a check on this estimation, the oil not recovered at the commercial end-point will be proportional to the herb's oil content per kilo, since the number of glands remaining at radius (q) is also proportional to this oil content.

Parameter test procedure

- Prepare and weigh three or more differently sized charges of similar test herb. Carry out distillations catching each two-minute fraction of the distillates in a separate bucket. Tabulate times the buckets were changed and the volumes of water and oil in each (see Tables I and II). Draw graph of progres-

sive oil produced against clock time, making a systematic curve as nearly as possible through the points observed. In cases of very uneven distillate flow it may be necessary to graph the oil produced against the water passed and adopt virtual clock times derived from the standard rate of water flow (see figs. 1 and 2). This will not normally be necessary. In the present tests, the curves nowhere depart from the observed points by more than 0.1%.

- Adopting the oil content per kilo of the mid-sized charge as standard for the herb under test, and using the standard distillate water flow rate to modify the actual flow around the end point, determine the comparable terminal oil flow. Draw tangent to graph and find the clock time of the end point and oil recovered as described in "Determining Clock Time of End-point," above.
- Determine average distillate water flow and virtual start time of distillation. Ascertain extraction time under actual conditions using equation 1.
- Apply corrections in "Standardising Extraction Time," above, to the actual extraction time to ob-

Table I. Parameter test distillations 1975 - lavender genotype "MS"

Distillery observations February 3, 1975				
Charge Details	CLOCK time bucket removed	WATER in fraction ml	OIL in fraction ml	PROGRESS total oil produced ml
No. 978/75	1503	2300	475	
	1505	5805	1005	1480
Mass 301 kg	1507	5900	545	2025
Cross-section area	1509	5950	253	2278
1.026m ²	1511	6045	144	2422
	1513	5520	79	2501
Oil content at "Estimated Virtual Exhaustion"	1515	5485	63	2564
E.V.E. = 2736 ml oil	1517	5485	46	2610
	1519	6130	36	2646
Adopted as standard charge for oil content in these tests	1521	6140	27	2673
	1523	6150	17	2690
	1525	6150	12	2702
No. 979/75	1601	3990	575	
	1603	6140	430	1005
Mass 175 kg	1605	6275	180	1185
	1607	6350	105	1290
Cross-section area	1609	6360	66	1356
1.026m ²	1611	6285	48	1404
E.V.E. = 1506 ml oil	1613	6355	32	1436
	1615	6370	22	1458
	1617	6310	18	1476
	1619	6370	14	1490
No. 981/75	1914	2660	600	
	1916	5060	1000	1600
Mass 420 kg	1918	5280	795	2395
Cross-section area	1920	5440	550	2945
1.1675 m ²	1922	5550	420	3365
	1924	5660	305	3670
E.V.E. = 4518 ml oil	1926	5715	220	3890
	1928	5760	155	4045
	1930	5330	115	4160
	1932	5875	94	4254
	1934	5890	61	4315
	1936	5945	52	4367
	1938	5975	39	4406
	1940	6010	33	4439
	1942	6000	20	4459
	1944	5990	19	4478

Note on Seasonal Conditions—Table I

1975 was a year of above average rainfall and exceptionally lush growth, which led to more and heavier stalk being cut with the flowers than is normally the case. The oil content per kilo of harvested material is scarcely more than 80% of normal for this genotype.

Test distillations

Table II. Parameter test distillations 1976 - lavender genotype "MS"

Distillery observations January 22, 1976				
Charge Details	CLOCK time bucket removed	WATER in fraction ml	OIL in fraction ml	PROGRESS total oil produced ml
No. 619/76	1752	3730	620	
	1754	6160	550	1170
Mass 156 kg	1756	6470	220	1390
Cross-section area 1.026m ²	1758	6550	115	1505
	1800	6630	75	1580
	1802	6620	52	1632
E.V.E. = 1760 ml oil	1804	6650	36	1668
	1806	6640	25	1693
	1808	6640	18	1711
	1810	6630	14	1725
<hr/>				
No. 614/76	1555	5900	1380	
	1557	6105	1190	2570
Mass 473 kg	1559	6840	1030	3600
Cross-section area 1.1675m ²	1601	6630	800	4400
	1603	6740	610	5010
	1605	6690	460	5470
E.V.E. = 6435 ml oil	1607	6780	287	5757
	1609	6790	190	5947
	1611	6790	125	6072
	1613	6770	91	6163
	1615	6870	69	6232
	1617	6750	50	6282
	1619	6820	42	6324
	1621	6810	33	6357
	1623	6830	27	6384
	1625	6830	17	6401

Note on Seasonal Conditions—Table II

Rainfall was only just adequate in 1976 to produce a good crop of flowers. Green growth was somewhat restricted and very little stalk was cut with the flowering heads. This led to the highest yields of oil per kilo of cut material that we have ever recorded. At the same time very warm weather helped the stills to run at a much faster rate of distillate flow than was used in 1975. This gives a good opportunity to demonstrate the adaptability of the parameters for varied yields and rates of steam displacement.

Figure 1

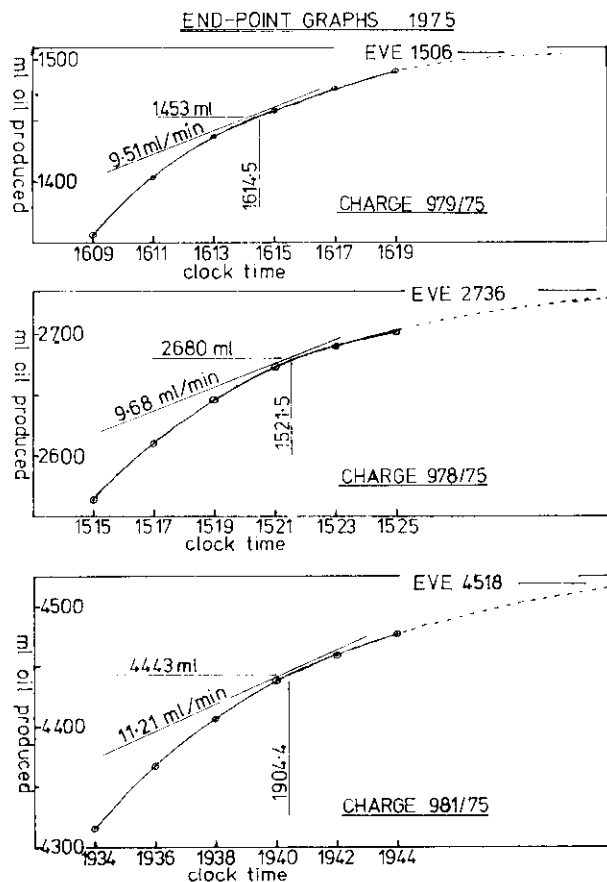


Figure 2

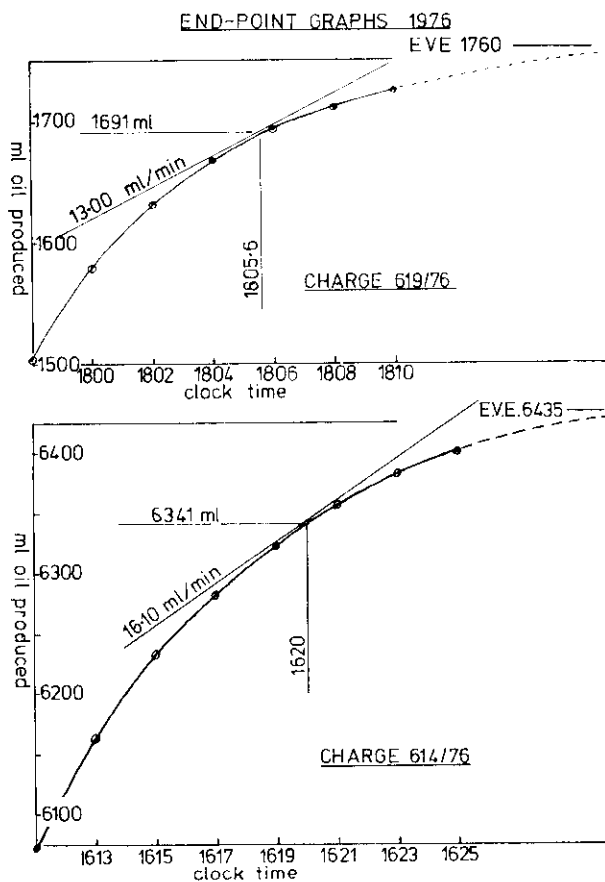


Table III. Results under standard conditions

Charge No.	Time	Height
979/75	14.68 min = T1	52.47 cm = H1
978/75	17.73 min = T2	95.32 cm = H2
981/75	21.43 min = T3	157.42 cm = H3
619/76	15.19 min = T4	61.32 cm = H4
614/76	24.93 min = T5	224.22 cm = H5

tain the extraction time under standard conditions. Tabulate the times under standard conditions at T1, T2, T3, and so forth (see Table III).

- Using the oil content of the midsized charge per unit of its actual height derive virtual heights for the smaller and larger charges as set out in "Standardising Charge Height," above. Tabulate these as H1, H2, H3, and so forth (see Table III).
- Using these figures for T and H from any two charges, equation 1 will allow parameter (x) to be determined by expressing (a) in terms of (da) such that $a=x.da$, for herb of this oil content per kilo.
- With a value for (x), and T and H taken from either of the charges used in (f), the value of parameter (t) can be found from equation 2. This will be the basic time for the test herb under the standard rate of displacement of the steam.
- Parameters (t) and (x) may now be used with equation 2 to calculate extraction times for all other charges for which virtual heights have been derived. These may be compared with the times that would have been observed under standard conditions.
- Finally, it is desirable to check that the quantity of oil abandoned by stopping distillation at the commercial end-point is proportional to the herb's oil content per kilo. If there is a wide discrepancy in this, it may be necessary to reconsider the figures for E.V.E. and recalculate both (t) and (x).
- Once the reliability of the parameters has been confirmed they can be adapted to obtain extraction times for any other stills processing herb of the same type to the same end-point even though yields and rates of steam displacement may be different. The methods of adaptation are given at the end of the worked examples.

The value of this procedure is obviously a question of cost-benefit. With two assistants, a competent operator could carry out the tests done on lavender "MS" described in this paper in one day's work in the distillery, and about three days in the office. Normally, this would be less costly than building the distillery and then finding it did not have the required handling capacity.

Conversion of distillery observations to standard conditions

Charge No. 978/75 (see fig. 1) was adopted as having the oil content per kilo and per cm of charge height to be used as standard for this herb.

Test distillations

Standard still, area 1.026 m²
Mass: 301 kg
Actual charge height: $(301/300) \times 95 = 95.32$ cm
Total oil content on basis of E.V.E.: 2736 ml
Standard oil contents for this herb in all tests:
 $2736/301 = 9.09$ ml/kg and
 $2736/95.32 = 28.7$ ml/cm layer
Distillate water flow in end-point region: 3069 ml/min
Terminal oil flow at end-point: $10 \times (3069/3170) = 9.68$ ml/min
From the graph this occurs at 1521.5 hrs
Output is 2680 ml oil
Average water flow from 1503 to 1521 is
2914 ml/min
Virtual time to pass first bucket at this flow:
 $2300/2914 = 0.789$ min
Plus time from 1503 to 1521.5 = 18.500 min
Equals extraction time (actual conditions) = 19.289 min
Extraction time (standard conditions):
 $19.289 (2914/3170) = 17.73$ min
Virtual height=actual height (this charge only): 92.32 cm

Charge No. 979/75 (see fig. 1)
Area 1.026 m²
Mass: 175 kg
E.V.E.: 1506 ml
Oil content: 8.61 ml/kg
Water flow about end-point: 3183 ml/min
Terminal oil flow: $10 \times (8.61/9.09) (3183/3170) = 9.51$ ml/min
This occurs at 1614.5 hrs
Output is 1453 ml oil

Average water flow from 1601 to 1615 is
3152 ml/min
Virtual time for first bucket:
 $3990/3152$ ml/min = 1.266 min
Plus time from 1601 to 1614.5 = 13.5 min
Equals extraction time (actual conditions) = 14.766 min
Extraction time (standard conditions):
 $14.766 (3152/3170) = 14.68$ min
Virtual height at standard oil content:
 $1506/28.7 = 52.47$ cm

Charge No. 981/75 (see fig. 1)
Still extension, area 1.1675 m²
Mass: 420 kg
E.V.E.: 4518 ml
Oil content: 10.76 ml/kg
Water flow about end-point: 3003 ml/min
Terminal oil flow: $10 \times (10.76/9.09) (3003/3170) = 11.21$ ml/min
This occurs at 1940.4 hrs

Output is 4443 ml oil
Average water flow from 1914 to 1940 is
2827 ml/min
Virtual time for first bucket: $2660/2827 = 0.940$ min
Plus time from 1914 to 1940.4 = 26.4 min
Equals extraction time (actual conditions) = 27.340 min
Extraction time (standard conditions):
 $27.34 (2827/3170) (1.026/1.1675) = 21.43$ min
Virtual height at standard oil content:
 $4518/28.7 = 157.42$ cm

Charge No. 619/76 (see fig. 2)
Area 1.026 m²
Mass: 156 kg*
E.V.E.: 1760 ml
Oil content: 11.28 ml/kg
Water flow about end-point: 3320 ml/min
Terminal oil flow: $10 \times (11.28/9.09) (3320/3170) = 13.00$ ml/min
This occurs at 1805.6 hrs
Output is 1691 ml oil
Average water flow from 1752 to 1806 is
3266 ml/min
Virtual time for first bucket: $3730/3266 = 1.142$ min
Plus time from 1752 to 1805.6 = 13.6 min
Equals extraction time (actual conditions) = 14.742 min
Extraction time (standard conditions):
 $14.742 (3266/3170) = 15.19$ min
Virtual height at standard oil content:
 $1760/28.7 = 61.32$ cm

Charge No. 614/76 (see fig. 2)
Still extension, area 1.1675 m²
Mass: 473 kg
E.V.E.: 6435 ml
Oil content: 13.60 ml/kg
Water flow about end-point: 3410 ml/min
Terminal oil flow: $10 \times (13.6/9.09) (3410/3170) = 16.09$ ml/min
This occurs at 1620.0 hrs
Output is 6341 ml oil
Average water flow from 1555 to 1620 is
3361 ml/min
Virtual time for first bucket: $5900/3361 = 1.755$ min
Plus time from 1555 to 1620 = 25.0 min
Equals extraction time (actual conditions) = 26.755 min
Extraction time (standard conditions):
 $26.755 (3361/3170) (1.026/1.1675) = 24.93$ min

*In the earlier paper the mass of this charge was incorrectly shown as 152 kg.

Virtual height at standard oil content:
 $6435/28.7 = 224.22 \text{ cm}$

Deriving parameters (x) and (t)

Use formula $\left(\frac{T_2}{T_1}\right)^2 = \frac{a + H_2 \cdot da}{a + H_1 \cdot da}$

to derive (x) from $a = x \cdot da$:

$$\left(\frac{17.73}{14.68}\right)^2 = 1.4587 = \frac{a + 95.32 \cdot da}{a + 52.47 \cdot da}$$

$$x = 40.946$$

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

to derive (t) from results of Charge No. 978/75

$$t = 17.73 \left(\frac{40.946}{40.946 + 95.32}\right)^{\frac{1}{2}} = 9.719 \text{ mins.}$$

First check on reliability of parameters derived

Use equation 2 to calculate extraction times for the other charges under standard conditions, and compare these times with those actually observed under the same conditions. Find extraction time (T3) if (H3) is given as 157.42 cm as in Charge No. 981/75. Time observed was 21.43 mins.

$$T_3 = 9.719 \left(1 + \frac{157.42}{40.946}\right)^{\frac{1}{2}} = 21.39 \text{ min.}$$

Find (T4) if (H4) is given as 61.32 cm as in Charge No. 619/76. Time observed was 15.19 mins.

$$T_4 = 9.719 \left(1 + \frac{61.320}{40.946}\right)^{\frac{1}{2}} = 15.36 \text{ min.}$$

Find (T5) if (H5) is given as 224.22 cm as in Charge No. 614/76. Time observed was 24.93 mins.

$$T_5 = 9.719 \left(1 + \frac{224.22}{40.946}\right)^{\frac{1}{2}} = 24.73 \text{ min.}$$

Second check on reliability of parameters

Check that the oil abandoned in each charge (i.e. E.V.E. less oil recovered), per sq.m of top layer area, is proportional to oil content/kg (see Table IV).

Adaptability of parameters

The extraction time (T) is inversely proportional to the rate of distillate flow and $T = t \cdot F(H/x)$. If the steam speed over herb surfaces is altered by a factor (c), the extraction time becomes $T/c \equiv (t/c) \cdot F(H/x)$. It follows that extraction times for differing rates of flow may be calculated if the numerical value of parameter (t) is varied inversely as the change in the rate of displacement of the steam inside the still.

Using a virtual height to allow for variations in the herb's oil content per kilo treats charge height as being proportional to the number of layers of herb each containing the same quantity of oil as did the

Table IV. Second check on reliability of parameters

	979/75 A	978/75 B	981/75 C	619/76 D	614/76 E
Mass kg	175	301	420	156	473
E.V.E. ml oil	1506	2736	4518	1760	6435
Oil recovered ml	1453	2680	4443	1691	6341
Oil abandoned ml - gross	53	56	75	69	94
Oil abandoned ml/m ² of area	51.75	54.58	64.24	67.25	80.51
Oil content ml/kg	8.61	9.09	10.76	11.28	13.60
Charges compared					
	A:B	B:C	A:C	D:C	C:E
Ratio oil loss per sq.m	1.055	1.177	1.241	1.047	1.253
Ratio oil contents per kg	1.056	1.184	1.250	1.048	1.264
	A:D	A:E	B:D	B:E	D:E
Ratio oil loss per sq.m	1.300	1.556	1.232	1.475	1.197
Ratio oil contents per kg	1.310	1.580	1.241	1.496	1.206

standard charge per unit of charge height. This is convenient in parameter tests where the value of (x) is yet to be found. In calculating extraction times for charges of known actual height, differing oil contents per kilo of herb may be allowed for by varying the numerical value of (x) inversely as the factor of change in potential yield. If the herb's oil content is expected to alter by a factor (b), the following obvious identity for extraction time will apply:

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

The parameters cannot be adapted to terminal rates of oil flow which differ from that used when they were determined. Parameters for other chosen commercial end points can always be found by reworking from the original distillery observations using the appropriate terminal oil flow.

The parameters cannot be readily adapted to the use of steam with properties greatly different from that used in the test distillations. The earlier paper discussed very briefly the effect of saturated and super-heated steam under differing pressure, but this was from a purely academic viewpoint. The variations in the steam's moisture content, which may or may not be associated with changes in operating pressure, are very important to efficient distillation, but their effects are more or less imponderable.

Anomalous yields

Every effort was made to ensure that the herb used in the three 1975 distillations was as similar as possible. The same applied to the 1976 season's tests. Nonetheless, the smaller charges consistently return smaller yields of oil per kilo. This is due to an orderly phenomenon that could be discussed on another occasion.