



## **Production Test Analysis of the VCLF Percolation System**

### *Urtica dioica* herba test percolation

This analysis of a test percolation was carried out between the 18<sup>th</sup> and 21<sup>st</sup> June 2009 using constant drop frequency control of the VCLF percolation apparatus set to 30gtt./min (mean deviation  $\pm$  15% calculated over entire process).

Standard reference values were provided by a quality market herbal manufacturer to be used for comparative purposes:

#### *Standard Reference Values used in analysis*

Preparation standard:	1:3, 25%
Percolation rate:	200gtt/min/kg
Solids (std.):	2.8 – 4.5%
S.G.:	0.975 – 0.995

*The preparation to be tested has been produced as an initial 1:6, 25% for comparative solids analysis purposes, allowing a quantitative evaluation of the VCLF Percolation System.*

#### **Production method:**

200g coarse comminuted *Urtica dioica* herba was pre-soaked for 24hrs with 200ml Menstrum.

The soaked herb was then packed in the percolation column and covered with Menstrum – Intermediate Maceration period 8 hrs.

The extractive was passed out of the herbal material substrate via a Whatman<sup>®</sup> 3 filter paper (11  $\mu$ m particle retention), which was placed at the bottom of the herb material packed in the column.

Column percolation was started at 30gtt/min for the 200g material (Equivalence of 150gtt/min/kg).

Four samples were collected during the percolation process. The total drop counter was recorded and then reset after sampling.

#### **Analysis procedure:**

The trial process was carried out to demonstrate process control and percolation product quality defined as the amount of total extracted solids.

Progressive sampling was taken at four intervals in order to be able to demonstrate the exhaustion pattern of the herbal material with regards to extraction time and from these figures interpolate the volume of menstrum that would be required to achieve total exhaustion of the herbal material. Four samples were taken throughout the process (A, B, C, & D).

Sedimentation estimation was carried out by agitating the total volume of each total sample and then subsequently placing 40ml of each of the sampled products in a 50ml sedimentation cylinder for 24 hrs. There was no visible sedimentation present in the four samples taken.

The submitted sample 'X' was taken following a complete blending of the entire percolation product.

The submitted sample 'Y' was the residue miscella recovered from the marc by pressing after the percolation process was completed. This sample is taken as a control to evaluate the remaining post percolation solids amount in the marc.



The 5 samples (A, B, C, D, X & Y) submitted for total extracted solids analysis were taken from agitated sampled product. Total Solids analysis was carried out *in-house* and the results have subsequently been verified by external laboratory analysis

**Process figures:**

<b>Urtica dioica 1:6, 25% Percolation</b>		
Process started (wetting): 18th June 2009		Estimated total process time: 72 hrs.
Process completed (end percolation): 21st June 2009		
Marc: 200g coarse comminuted herba		
Menstrum amounts		
Wetting (ml)		200
Intermediate Maceration & Percolation (ml)		1000
<i>Total menstrum used (ml)</i>		<u>1200</u>
Sample ID	Drop counter at sampling	Product amount (ml)
A	*11193	190
B	17400	290
C	16409	290
D	8313	135
	<i>Total product</i>	<u>905</u>
Calculated average gtt/ml:		58,91 **
Percentage retrieval of process menstrum volume (%):		75,42
NB: ** This figure appears high but has been verified correct by control		
* Interpolated (due to function error -data lost)		

**Total solids analysis**

Results of in-house tests carried out by Asclepius herbs:

Sample	Ev. Dish wt (g)	Pre Ev wt (incl. Dish) (g)	Sample wt (g)	Post Ev wt (incl. Dish) (g)	Solids wt (g)	% solids
A	26,294	30,577	4,283	26,494	0,200	<b>4,67</b>
B	22,079	27,074	4,995	22,340	0,261	<b>5,23</b>
C	21,077	25,068	3,991	21,345	0,268	<b>6,72</b>
D	24,274	29,254	4,980	24,341	0,067	<b>1,35</b>
X	21,808	26,518	4,711	21,972	0,165	<b>3,49</b>
Y	26,972	32,408	5,436	27,044	0,072	<b>1,32</b>

Total solids analysis carried out with drying at 70 °C until total evaporation was reached. Cooling carried out in dry environment to avoid moisture absorption

**Solids Results discussion**

The extraction was produced on the basis of an initial 1:6 ratio of herb material/menstrum using 200g herbal material and 1200ml menstrum. The process produced 905ml product. The industry definition of the product ratio can vary; it can be applied to the initial ratio prior to extraction, or to the ratio of retrieved product to herbal material used. In this test percolation the preparation produced could be defined as being:

An initial 1:6 preparation

A retrieved 2:9 (1:4.5) preparation



From the standard figures stated at the beginning of this report, it can be calculated that the total extracted solids should be within the range:

1.4 – 2,25% for a 1:6 defined preparation

1.86 – 3.0% for a 2:9 defined preparation

The in-house results demonstrated a percentage total extracted solids result for the entire collected extract (sample X) of 3.49%. This is considerably higher than the equivalent expected arithmetic mean results calculated from the 1:3 standard reference values (calculated at 1.82% and 2.43% respectively for a 1:6 and a 2:9 defined preparation), which therefore clearly demonstrates the VCLF Percolation System's ability to effectively extract constituents from the herbal drug material undergoing percolation.

Based on the standard reference values provided, the VCLF produced preparation shows a solids equivalence of a 1:3 preparation (which has a calculated percentage solids range of 2.8 – 4.5%).

### **Solids Results Interpolation regards extraction**

By generating a graph relating total extracted solids to sampling period and interpolating from the sample results and it is possible to calculate when exhaustion of the herbal material would theoretically occur and from this evaluate the extraction efficiency for the 1:6 preparation produced using the VCLF Percolation System at the defined drop frequency of (30gtt).

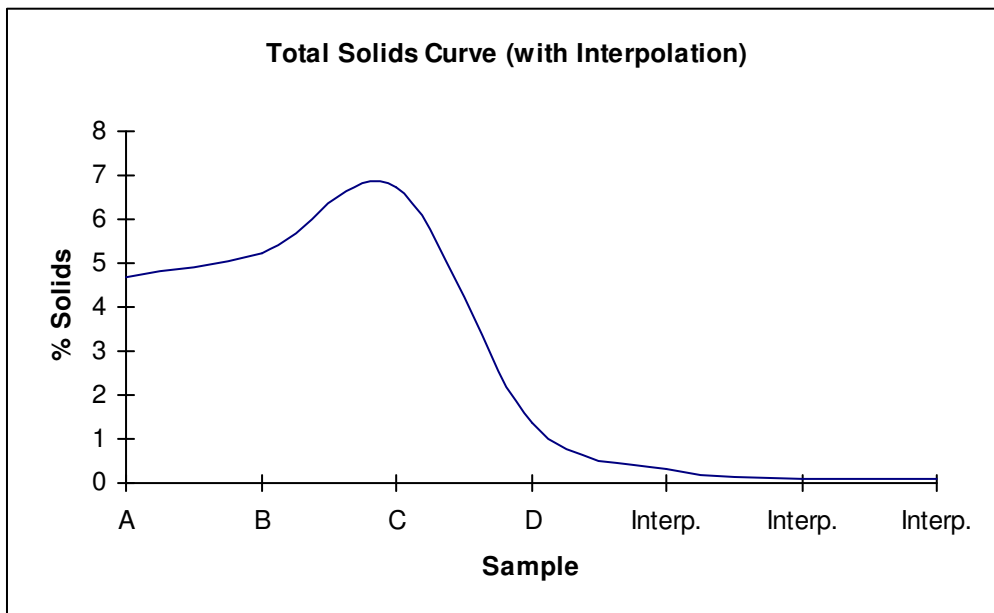
Figure 1 shows the extracted solids graph with interpolation to exhaustion. The initial form of the curve demonstrates what would be expected in the percolation system; an increase in solids immediately following intermediate maceration, which then falls exponentially throughout the percolation process (this also verifies the series of extracted solids analysis results). It can be estimated that total exhaustion of the herb material would effectively occur at 106630gtt with a drop rate of 30gtt/min. The amount of menstrum needed to achieve this would therefore be: 1,81L. The estimated extracted solids amount interpolated from the graph data would suggest that this would result in an estimated total soluble solids level of approximately 4.9% in the entire 1.81L of the hypothetically produced 1:12 preparation.

As a control, a solids analysis of the residual miscella held in the remaining marc was carried out following pressing of the marc material upon completion of the percolation process. The post percolation solids amount in the resulting miscella (sample Y) was shown to be 1.32%. This amount would confirm the *Total Solid Curve* interpolation, as it corresponds roughly with the average solids percentage of the area under the curve following sample D.

From these analysis results it can be calculated that the VCLF percolation process extraction affectivity is:

72% (including residual miscella wastage in the calculation)

97% (excluding residual miscella wastage in the calculation)





### Interpolated extraction curve

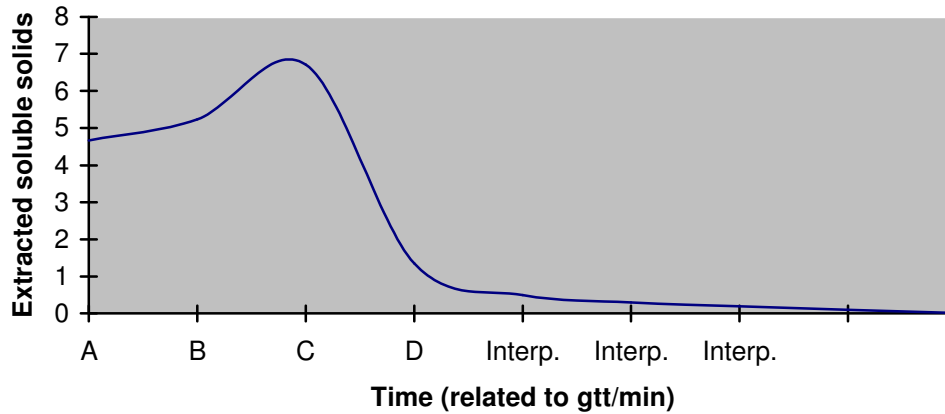


Figure 1

#### Discussion

The standard reference expression for drop frequency for a given herb is given as gtt/min/kg. However, because the percolation process relies on two major factors for extraction; rinsing and diffusion, it could be argued that the expression is an incorrect model for the diffusion phase of extraction as the exposure time between herb material and the menstrum would be the important factor for extraction.

The drop factor in the reference expression is the variable that relates to herb material exposure time to menstrum and the formula can be re written: as vol/min/kg. If the exposure time factor is to be taken into account, then the column diameter is the factor which dictates exposure time between the menstrum and the herbal substrate. 30gtt/min is equivalent to 30,6ml/hr or 30,6cm<sup>3</sup>/hr for the 1:6 *Urtica dioica* preparation produced (at 58,91gtt/ml). The 6cm diameter column used in this analysis has a cross sectional area of: 28,26cm<sup>2</sup>, from which a passage rate through the herbal substrate can be calculated as 1.083cm/hr. In a larger column of 10cm diameter this would be 0,39cm/hr, with a drop frequency of 30gtt/min. Therefore, it can be seen that the standard reference expression does not account for percolation column diameter.

#### Conclusion

In order to be able to optimise production and therewith have an economically efficient extraction process it is necessary to re-visit the reference standards used in the industry and re-analyse the process using progressive sampling and interpolation of the generated solid/extraction time curve to analyse extraction efficiency and relate this to percolation column dimension. This would allow percolation system specific reference parameters to be produced for each herb that is to be percolated.

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**Document revision notes:**

- Rev. 2.0 Data sheet updated and corrected with latest solids analysis data for sample X  
Data and notes provided for solids analysis Y
- Rev. 2.1 Text layout changes