



Customs Laboratories
European Network

ILIADe 555:2022 | CLEN Method

**Gravimetric determination
of a fraction of tobacco with cut width of less than 1.5 mm**

Version 10 February 2022

Gravimetric determination of a fraction of tobacco with cut width of less than 1.5 mm

1. Scope

This method describes a procedure for assessing if a cut tobacco complies with Council Directive 2011/64/EU, article 5.2 on fine-cut tobacco intended for the rolling of cigarettes, for excise purposes. For cut tobacco to be suitable for HRT (Hand Rolling Tobacco) and classified as fine cut tobacco, at least 25% by weight of shreds must have a cut width of <1.5mm.

The method is applicable to cut tobacco.

For a tobacco mixture with cut tobacco and irregularly shaped particles (petioles and veins) or a mixture of cut tobacco and tobacco refuse, the cut tobacco fraction must be separated and the determination made separately with this fraction. The method cannot be applied to tobacco which does not contain cut tobacco, i.e. regular cut fibres.

Acknowledgement: *this method was established by Campden BRI, a laboratory based in the United Kingdom who kindly shared it with the CLEN community. It was tested during two CLEN proficiency tests on tobacco products, in 2015 and 2019. This 2021 version of the procedure is based on the Campden BRI October 2019 revised version, with their authorisation, and agreed within the CLEN following the 2019 inter-laboratory test.*

2. Principle

A portion of cut tobacco is checked for fibres/strands of cut width less than 1.5mm by separating and weighing them manually in order to calculate the percentage of these strands in the whole portion.

3. Reagents and materials

Not relevant

4. Apparatus

- 4.1 Stereo Microscope (if possible equipped with camera). Else, magnifying glasses
- 4.2 Tweezers – wide nib tweezers are beneficial (two pairs may be required)
- 4.3 Analytical balance with precision of 1mg
- 4.4 Petri dish with **1.5 millimetre-squared grid** 1,5 mm ± 0,1 mm (see Annex A)
If a new square grid is printed or otherwise made, its dimensions shall be checked before testing by calibrated image analysis.
- 4.5 2 x Sample pots (large enough to contain 5-6g shredded tobacco)
- 4.6 Suitable container for storing 5-6g subsample
- 4.7 Foil/lid to cover container in between use
- 4.8 Scalpel or spatula

5. Procedure

5.1. Sampling

For the classification of hand rolling tobacco, at least 5g of shredded tobacco is needed.

5.2. Shreds of tobacco and classification for HRT

5.2.1. Weigh out approximately 5-6g of tobacco (without conditioning, taking the sample as it is) into a container and temporarily cover with a lid/foil to help prevent the tobacco from drying out.

Note: As the tobacco can dry out and it is easy to lose shreds, you need over 5g to start with to ensure you have over 5g at the end. You do not need to record the weight at this stage.

5.2.2. Two sample pots are needed to place the shreds in after they have been sorted. Weigh each empty sample pot individually without its lid and note the weight on the pot to 2 decimal places.

Weighing the sample pots avoids the need to decant the tobacco into weigh boats after sorting and risking the loss of tobacco particles.

5.2.3. Label one pot $<1.5\text{mm}$ and the other $\geq 1.5\text{mm}$.

5.2.4. Using the 1.5 millimetre-squared petri dish and a stereo microscope, take a small portion of the subsample and place it onto one side of the Petri dish.

5.2.5. Using tweezers, pass the shreds across the grid square to gauge their cut widths and sort those measuring $<1.5\text{mm}$ and $\geq 1.5\text{mm}$ into their corresponding sample pots. There is no need to accurately measure each strand, see Annexes A, B and C. The fibres are laid flat between the individual grid lines. Fibres lying over the grid lines are added to the pot $\geq 1.5\text{mm}$.

Notes: Pieces of tobacco petiole and midrib (see Annex D), should also be sorted accordingly. Some of these pieces can show a thickness lower than 1.5mm but a width higher than 1.5mm (as the top piece shown in Annex D photos), in which case for the CLEN laboratories they are considered to have a cut width higher than 1.5mm.

Exclude from the analysis and document any erroneous material, such as silicate minerals, plastics and hairs. For samples of a particulate nature (i.e. contain cut shreds that are shorter than average), it may be more efficient to use a scalpel or spatula to 'spread' the tobacco particles across the 1.5 millimetre-squared Petri dish.

5.2.6. Repeat this process for the entire 5-6g subsample of tobacco ensuring all the shreds $<1.5\text{mm}$ and $\geq 1.5\text{mm}$ are sorted into their correctly labelled sample pots.

5.2.7. Once the entire subsample has been passed across the grid square, weigh each pot (without its lid) with its contents, recording the weight on the appropriate pot to 2 decimal places.

5.2.8. Calculate the percentage of shreds less than, and greater or equal to 1.5mm using the formula in section 6.

6. Calculation

For each pot calculate:

$$\begin{aligned}\text{Weight of pot and tobacco (g)} &= x \\ \text{Weight of pot (g)} &= y \\ \text{Weight of tobacco (g)} &= x - y\end{aligned}$$

This will give you the weight of tobacco for <1.5mm and ≥ 1.5 mm.

To calculate the percentage:

$$\begin{aligned}\text{Weight of } <1.5\text{mm (g)} &= a \\ \text{Weight of } \geq 1.5\text{mm (g)} &= b \\ \text{Total weight (g)} &= a + b \\ \% <1.5\text{mm} &= \left[\frac{a}{a + b} \right] \times 100 \\ \\ \% \geq 1.5\text{mm} &= \left[\frac{b}{a + b} \right] \times 100\end{aligned}$$

If the sample contains more than 25% by weight of shreds with a cut width of <1.5mm it can be classified as fine cut tobacco for HRT.

7. Expression of the results

(refer to section 6. Calculation)

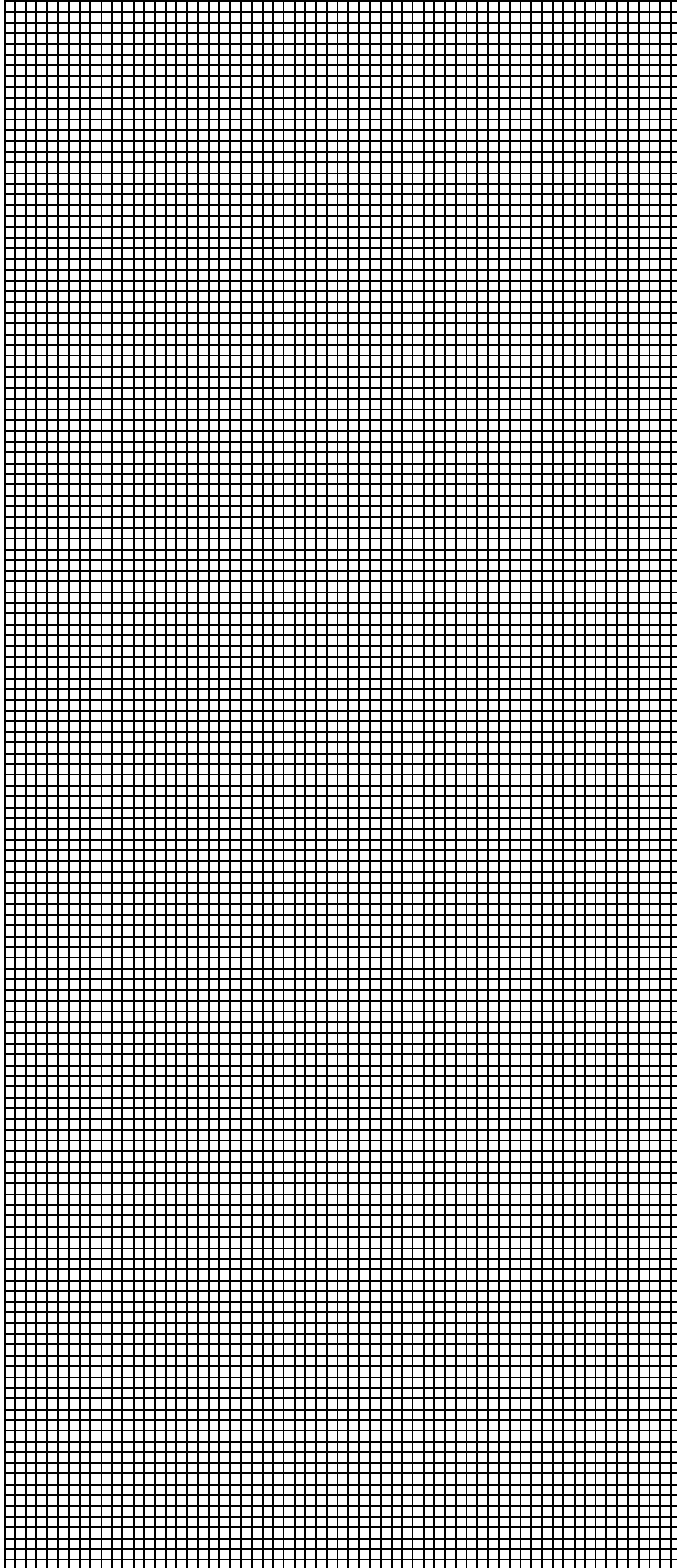
8. Precision

Precision data from obtained during the CLEN proficiency test on tobacco products in 2019 with this analytical method on fine cut tobacco: repeatability, $r=14\%$ and Reproducibility, $R=23\%$

However, several laboratories lacked of routine practice during this CLEN test. According to an experienced customs laboratory the precision expected on samples of regular fibre cut tobacco or mixtures of tobacco with cut tobacco would be $r=5\%$ w/w and $R=14\%$ w/w.

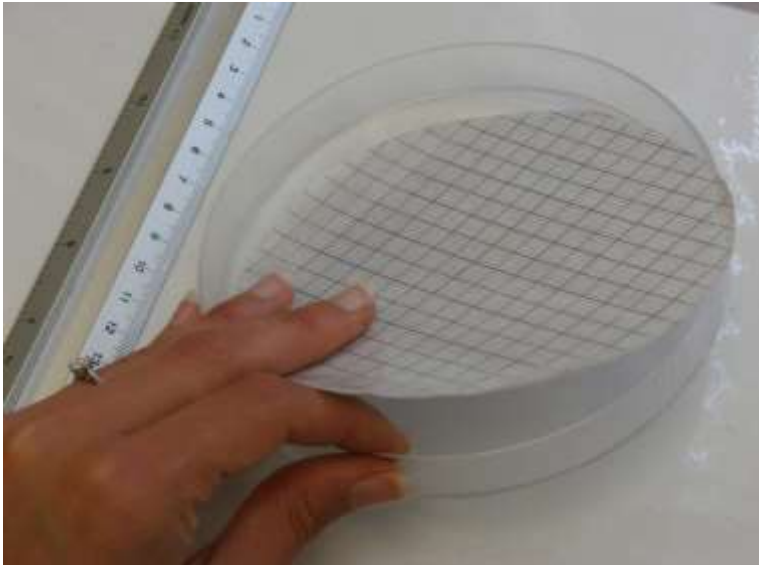
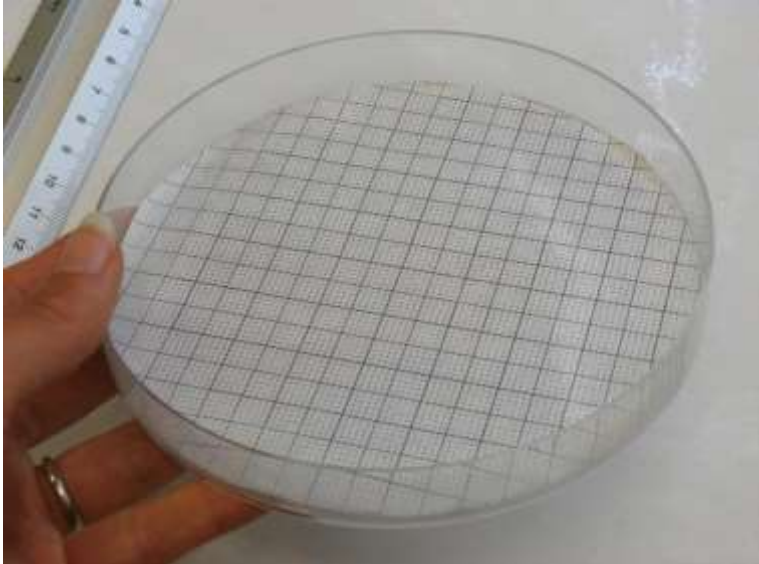
Annexes

ANNEX A. Examples of grids

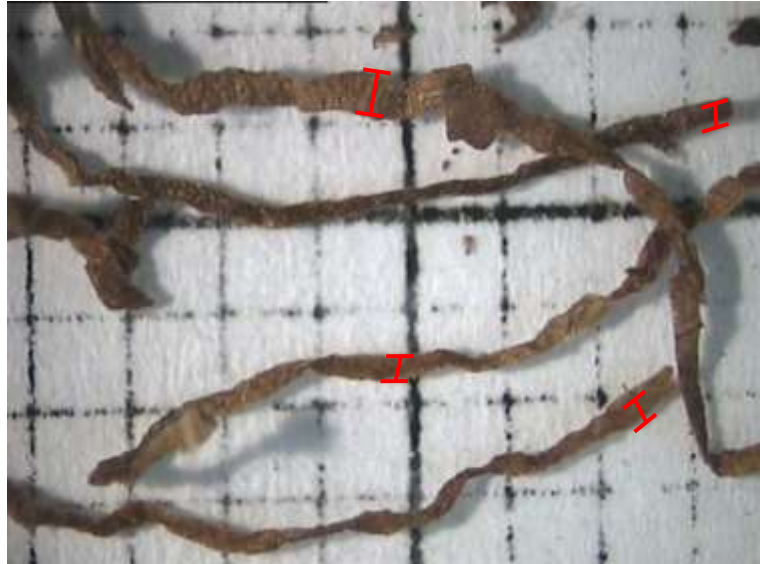


Grid pattern: 1,5 ($\pm 0,1$) mm \times 1,5 ($\pm 0,1$) mm
Line thickness 0,23 ($\pm 0,04$) mm
Dimensions must always be checked after printing

Example images of a 1.5 millimetre-squared Petri dish



ANNEX B. Image to illustrate the cut-width of tobacco shreds (highlighted in red), photographed against a 1.5 millimetre-squared grid



ANNEX C. Image to illustrate how tweezers are used to guide the tobacco shreds across the 1.5 millimetre-squared grid. The direction of shreds is from left to right



ANNEX D. Examples of pieces of tobacco petiole/midrib material (photographed against a 1.5 millimetre-squared grid).

The top piece is quite sizeable when laid flat (top image, part of the piece with a width larger than 1.5mm), even though the top piece thickness is less than 1.5mm (highlighted in red, bottom image).

The top strand is thus considered, for the EU Customs Laboratories, to have a cut width higher than 1.5 mm.

