

Crude Fiber Determination in Tea according to ISO 15598:1999

Reference: ISO 15598:1999 Tea - Determination of crude fibre content

Tested with VELP Scientifica FIWE 3 Fiber Analyzer (Code F30520201).



Introduction

Fiber is the part of fruits and vegetables (lignin, cellulose, hemicellulose, pectin) that cannot be digested and assimilated by the human gastrointestinal tract. However, fiber analysis is important for:

- Nutritional reason: the quantity of fiber is important to keep the digestive system healthy and functional. Too much fiber can cause digestive problems; a low fiber intake irregularities in the functioning of the digestive tract.
- Legal reason: authorities of almost all countries require manufacturers of food & feed products to declare the fiber content as part of the nutritional table.

Chemically, fiber is defined as indigestible residue, after boiling with diluted solutions of strong mineral alkalies and acids.

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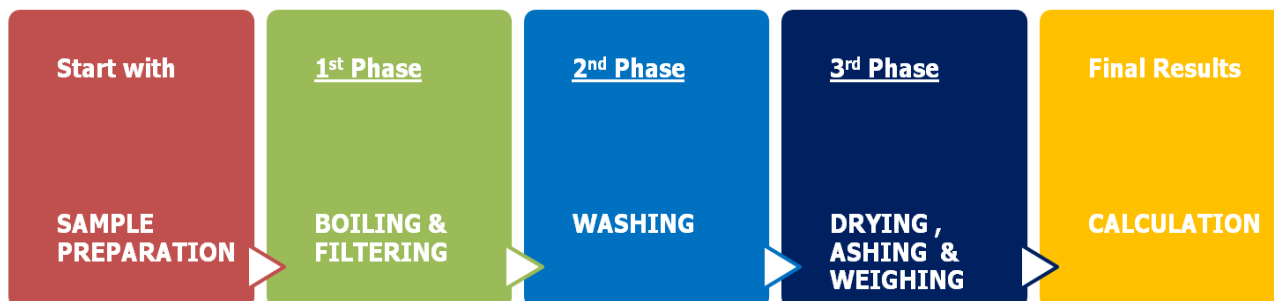
The suitably grinded sample is treated with boiling sulfuric acid solution and sodium hydroxide solution. The residue is separated by filtration, washed, dried, weighed and then ashed. The loss in mass resulting from ashing is the crude fiber content.

Reagents

- | | |
|---------------------------------------------------------------------------------------------|------------------|
| - Celite 545 | - HCl 1 % |
| - H ₂ SO ₄ 0.255 M: 125 ml H ₂ SO ₄ 20 % in 2 L | - Ethanol > 95 % |
| - NaOH 0.313 M: 1.5 g NaOH in pellets dissolved in 1 L | - Acetone |
| - Octanol | |

Analysis Procedure

The diagram below shows the steps involved in the procedure:



Grind the sample using a mill (particle size 1mm).

Weigh into the crucible 1 g of celite and 1 g of homogeneous grinded sample (W1) with an accuracy of ±1 mg and position the crucible (P1 type - Code A0000139) into the FIWE unit.

The sample must be homogeneous.

Add 150 ml of pre-heated reagent H₂SO₄ 0.255 M and 3-4 drops of octanol: boil 30 minutes exactly from the beginning of the boiling. At the end, connect to vacuum for acid draining.

Use the water spray device (A00001135) in order to remove the residues of sample that could have been remained on the glass columns. Repeat the same operations for the first reagent, using as second reagent NaOH 0.313 M.

Wash the residues using hydrochloric acid solution and boiling water again.

Then twice with ethanol >95 % and three times with acetone.

After draining, remove the crucibles and determine the dry weight after drying into an oven at 103 °C for two hours.

Let cool the crucibles in a desiccator up to constant weight.

This weight represents the crucible containing crude fiber and ashes (**W2**).

Place the crucibles in a muffle at 550 °C, starting from room temperature, for three hours and weigh again after cooling in a desiccator. This weight represents the crucible containing ashes (**W3**).

Calculation

$$\% \text{ Fiber} = ((W2 - W3) / W1) * 100$$

W1 = sample weight (1g)

W2 = crucible weight with fiber and ashes, after drying in an oven at 103 °C for 2 h

W3 = crucible weight with ashes, after muffle at 550 °C for three hours

Typical Results on Classic Tea (repeated twice)

W1 (g)	W2 (g)	W3 (g)	Fiber % (m/m)
1.000	31.470	31.337	14.013
0.996	31.372	31.241	13.886
1.009	31.399	31.267	13.827
0.992	31.546	31.417	13.731
1.006	31.437	31.302	14.149
1.001	31.590	31.459	13.835
		Average ± SD%	13.907 ± 0.137
		RSD% *	0.985

* RSD% = (Standard Deviation * 100) / Average

The crude fibre content is expressed as a mass fraction, in percent, of the product on a dry basis.

Conclusion

The use of an extraction apparatus purposely devised for this method as FIWE unit makes very easy the standardization of analytical conditions. The FIWE Series is suitable for fiber determination in tea.

Benefits of FIWE are:

- 3 or 6 positions simultaneously: FIWE units can support up to 3 (FIWE 3) or 6 (FIWE 6) crucibles. Samples can also be processed individually
- Time saving: fast analysis (2 hours with FIWE vs. 6 hours manually)
- Easy to use: convenient filtration, with pump and air pressure
- Precision: high reproducibility of the results: ±1% relative or better

In order to avoid losses of fiber, it's important to remember that crucibles life is around 20-30 analyses, because the fritted filter could be damaged from basic and acid solutions. Hence it's suggested to change them after 20-30 analyses.