APPLICATION NOTE

F&F-S-001-2013/A1

Total Fat Determination in Pasta

according to the Randall method

Reference: **AOAC 2003.05** Crude Fat in Feeds, Cereal Grains, and Forages; **AOAC 920.39.B**

Tested with **VELP Scientifica SER 148/6 Solvent Extractor** (Code F30300242) and **HU6 Hydrolysis Unit** (Code F30300110)

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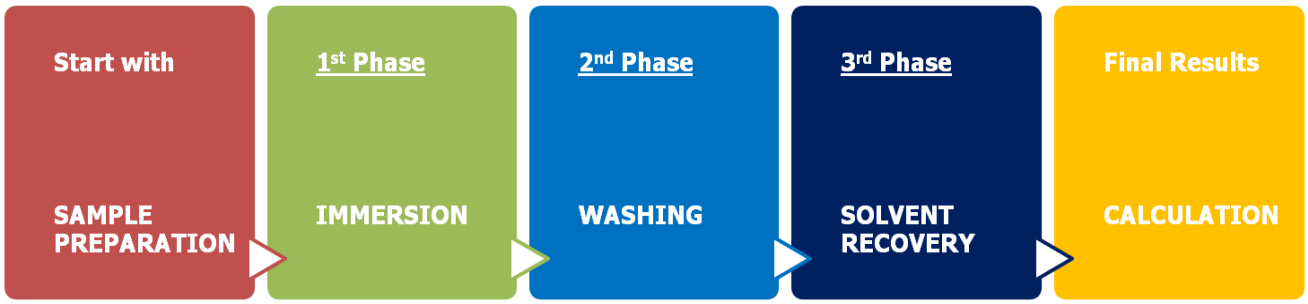
TOTAL FAT DETERMINATION IN PASTA
RANDALL METHOD

Introduction

Dried and fresh pasta are traditional Italian products, known all over the world in a number of shapes and varieties, as shown by the 310 specific forms and by over 1300 names having been recently documented. Basic pasta dough has always been made mostly of durum wheat flour or semolina; other grains can be used, including those from barley, buckwheat, rye, rice and maize, as well as chestnut and chickpea flours. It's always important knowing the ingredients and the amount of all the components, as fat, for different reasons, including legal and nutritional purposes (labeling).

Fat Determination in Pasta

Randall method is a modification of the standard Soxhlet extraction: in Randall, the test portion is in contact with pure hot solvent, ensuring a fast solubilization with a considerable reduction of the extraction time (approx. 90 minutes).
Three extraction phases are necessary with SER 148 (see the following picture):

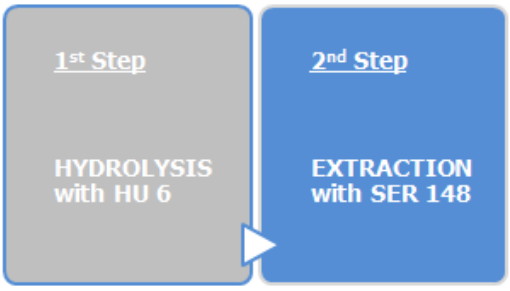


First, the extraction is made by immersion of the sample in the boiling solvent. This step is followed by a rinsing phase with hot solvent. Then, all the solvent evaporates and it is recovered in a condenser. The calculation of the total fat content follows gravimetrically after drying the extract.

Sample

Durum Wheat Dried Pasta Fat labeled value: 1.5 - 2 g / 100 g

In dried pasta a direct extraction with solvents is not efficient because lipids are chemically bonded with other components. The determination of the total fat content of these samples, requires a *preliminary hydrolysis* using hydrochloric acid 4 N, followed by filtration and washing, in order to free completely the fat molecules. Then the hydrolyzed sample is ready for the extraction and it can be easily transferred to SER 148 Solvent Extraction Unit avoiding any possible sample loss and ensuring improved accuracy of results (see the following picture).



Chemicals and Equipment Required

- Grinder
- Glass sand, 0.4-0.8 mm (Code A00000089)
- Celite 545 (Code A00000097)
- Glass crucibles P1 (Code A00000086)
- Hydrochloric acid 4N
- Test tube 250 ml (Code A000000144)
- Glass bottle for waste collector (Code A0000088)
- Glass extraction cups (Code A00000142)
- Vaflon seals (Code A00000061)
- Ethyl ether as solvent

All the accessories required for the hydrolysis are included in the kit code A00000085.

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Sample Preparation

- Hydrolisis Crucibles Preparation

Put 25 g of glass sand in glass crucibles P1 and 3g of Celite 545: the two layers resulting have not be mixed together otherwise the phase in Celite may bypass the crucible negatively affecting the end result.

- Sample Preparation in Test Tube

Grind the sample, reducing particle size of pasta to 0.75 -1 mm. Weigh 8 g of the crushed sample (M_{sample}) and 2 g of Celite 545 in the test tube. For each test tube add 50 ml of 4N hydrochloric acid, shake gently and carefully, and finally add a further 50 ml hydrochloric acid in order to rinse the sample residue that may remain on the walls of the test tube.

- Glass Extraction Cups Preparation

Keep the empty glass extraction cups in oven at 105 °C for 1 hour. Cool them in a dessicator and record the accurate weight of the tare (M_{tare}).

Hydrolysis Procedure with HU 6

Place 6 crucibles P1 on the HU6 and connect the aspirating tubes: one side needs to be placed on the sealing ring of the crucible and the other one has to be in contact with the corresponding test tube.
Set 170 °C for 60 minutes.
Place the 6 test tubes in the heating block, lower the glassware and activate the vacuum pump.

➢ *In case of foam, add 4 N hydrochloric acid drop by drop inside the test tubes.*

At the end of the procedure, switch off and allow aspiration of the content of the test tubes in the crucible.
Raise the glassware to its maximum point and secure it, by tightening the knob.
Then, add hot water (40-50 °C, about 250 ml) slowly, in order to aspirate all the residues of hydrolysis in the test tubes.

Remove the aspirating tubes and the crucibles containing the sample after the washing: mix the layer of hydrolyzed sample with the layer of Celite by using a spatula in order to break any clot.

➢ *Take care not damage the layer of glass sand.*

This operation helps the sample drying, which must take place in an oven at 105 °C for 6 hours.

Extraction Procedure with SER 148

If dried test portions will not be immediately extracted, put them in a desiccator, or let the crucibles cool to room temperature and then, mix the layer of hydrolyzed sample and Celite with a spatula to obtain a powdery hydrolyzed.

➢ *Take care not damage the layer of glass sand.*

At this point, before starting the extraction, add a new layer of glass sand (20 g) to avoid the Celite buoyancy. Select one of the programs (1-29) and set the following parameters:

- Temperature: 110 °C
- Immersion Time: 90 minutes
- Washing Time: 60 minutes
- Recovery Time 25 minutes

By using the dedicated magnetic adaptors (included in the kit code A00000085), it is possible to put in the SER 148 the same crucible used with HU 6
Introduce the glass cups containing the solvent (ethyl ether, 60 ml) in the extraction equipment.
Close the extraction unit, and press START button to activate the cooling water flow and the heating.

Immerse the glass crucibles into the boiling solvent by placing the slider into *"Immersion"* position.

After 90 minutes press ENTER and extract the crucibles out of solvent, by placing the slider into *"Washing"*.

After 60 minutes of reflux washing press ENTER and close the knob, let the solvent evaporate and recover it by condensation. In this step the air pump was turned on by pressing AIR to speed up the solvent recovery.

➢ *Prevent the complete solvent evaporation (few ml are required).*

Dry the cups in an oven (1 hour at 105 °C). Then, let them cool to room temperature in a dessiccator and record the accurate weight (M_{tot}).

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Typical Results on Dried Pasta

The results are gravimetrically determined, by using the formula:

$$Fat\ \% = (M_{tot} - M_{tare}) \times 100 / (M_{sample})$$

Where:
M_{sample} = *sample weight (g)*
M_{tare} = *weight of the empty cup (g)*
M_{tot} = *weight of the cup containing the fat residues (g)*

Sample quantity (g)	Fat %
7.743	1.54
7.893	1.55
7.901	1.54
7.996	1.55
8.003	1.55
8.014	1.56
Average ± SD%	**1.55 ± 0.01**
RSD% *	**0.49**
Fat Labeled Value: 1.5-2 g/ 100 g	

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

Conclusion

The obtained results are reliable and reproducible in accordance with the expected values, with a low relative standard deviation (RSD < 1%), that means high repeatability of the results.
The combination of VELP HU 6 Hydrolysis Unit with the SER 148 Solvent Extractor is ideal for the fat content determination in pasta.

Benefits of HU 6 Hydrolysis Unit are:
- safety with performance, reducing manual handling to the minimum
- no sample transfer required when passing from HU 6 to SER 148
- suitable for both acid and basic hydrolysis

Benefits of Randall method by using SER 148 are:
- up to 5 times faster than Soxhlet (hot solvent vs. cold solvent)
- low solvent consumption (high solvent recovery)
- limited cost per analysis
- full operator safety in compliance with IP55
- worldwide official method

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