FIBER DETERMINATION

Sales Training Course

WHAT IS FIBER?

Fiber is a component of the vegetable cell walls and as a primary function of plant support.

From nutritional point of view it occupies more space in the digestive tract than any other substance because it is poorly digestible.

FIBER is the part of fruits and vegetables (including lignin, pectin, cellulose, hemicellulose) **that cannot be digested.**

WHAT IS FIBER?

The fiber is a mix of heterogeneous substances with rumen degradability and highly variable intestinal digestibility of the plant walls.

Naturally the vegetable walls are formed by a structural part (cell wall) and by the cellular content.

- Carbohydrates of the cell wall (Structural carbohydrates):

  Cellulose – hemicellulose – lignin – ash

- Carbohydrates of the cell (Non -Structural carbohydrates):

  Sugar – starch - pectin

WHAT IS FIBER?

From **the analytical point of view** fiber is divided in 2 macro areas:

RAW FIBER

The estimated value of fiber in **Animal Food** (feed). Defined as indigestible residue, after boiling with diluted solutions of strong mineral alkalies and acids (chemicals).

Official Methods: **ISO**, **AOAC** and others using **Wendee**, **Van Soest**, etc.

DIETARY FIBER

The estimated value of fiber in **Human Food**.
Defined as the residue after enzymatic digestions used to simulate the real digestion process occurring in living organisms.

Official Methods: **AOAC 985.29** however many other AOAC Methods exist

The enzymatic method is particularly interesting because it reproduces, more efficiently than the chemical methods, the natural phenomena occurring during digestion in humans.

WHY IS IT SO IMPORTANT?

NUTRITIONAL PURPOSE

Human beings and animals need an intake of **a certain amount of fiber for good functioning of the digestive tract**.

Its quantity has to be carefully controlled, however, because **a too high amount can cause indigestion problems**, while a shortage could cause certain diseases.

ECONOMICAL REASON

Food and Feed industries tend to use fiber as a raw material as much as they are allowed because it's a **cheap component of food stuff**.

LEGAL PURPOSE

Legal authorities of almost all Countries require that food and feed manufacturers **declare the fiber content** on the package as part of nutritional labelling.

FIBER ANALYZERS FOR THE FEED INDUSTRY

FIBER ANALYSIS

The demands for fibre content determination in feed are growing due to the importance of this parameter.

- **Monogastric animals**: a proper proportioning of fibre fractions increases the utilization of the compounded feed.
- **Ruminants**. fibre is an important part of the metabolism in the rumen.

METHOD OF ANALYSIS

“Because there is no guarantee of direct correspondence between chemical solubility and nutritional availability, in reality, fiber is defined by the method used to isolate it.
The actual definition of fiber becomes method dependent, which explains why there are so many different fiber analyses.”

(AFFCO - Critical Factors in Determining Fiber in Feeds and Forages)

1. CRUDE FIBER / RAW FIBER / WENDEE METHOD

2. DETERGENT FIBER / VAN SOEST METHOD

CRUDE FIBER – WENDEE METHOD

The universal used method is Weende.

However this method underestimates the real content of the fiber because the 50-90% of the lignin, 0-50% of cellulose and up to 85% of hemicellulose can be solubilized and therefore not measured as crude fiber.

CRUDE FIBER METHOD – WENDEE

SAMPLE PREPARATION

Finely grind the sample (**1 mm particle size**) in order to obtain a homogeneous compound following official guidelines.

BOILING & FILTERING

ACID TREATMENT
Using Sulphuric Acid

WASHING

BOILING & FILTERING

ALKALI TREATMENT
Using Sodium Hydroxide

DRYING, ASHING & WEIGHING

- Dry the sample in a dessicator
- Put the dried sample in a furnace at 550°C to produce ashes

CALCULATION

DETERGENT FIBER

Van Soest method was developed to reduce error of poor recovery of hemicellulose and lignin in CF determination.

The concept behind detergent fibre analysis is that plant cells can be divided into less digestible cell walls (hemicellulose, cellulose and lignin) and mostly digestible cell contents (comprising starch and sugars).

These two components can be separated by using two detergents: a neutral detergent and an acid detergent.

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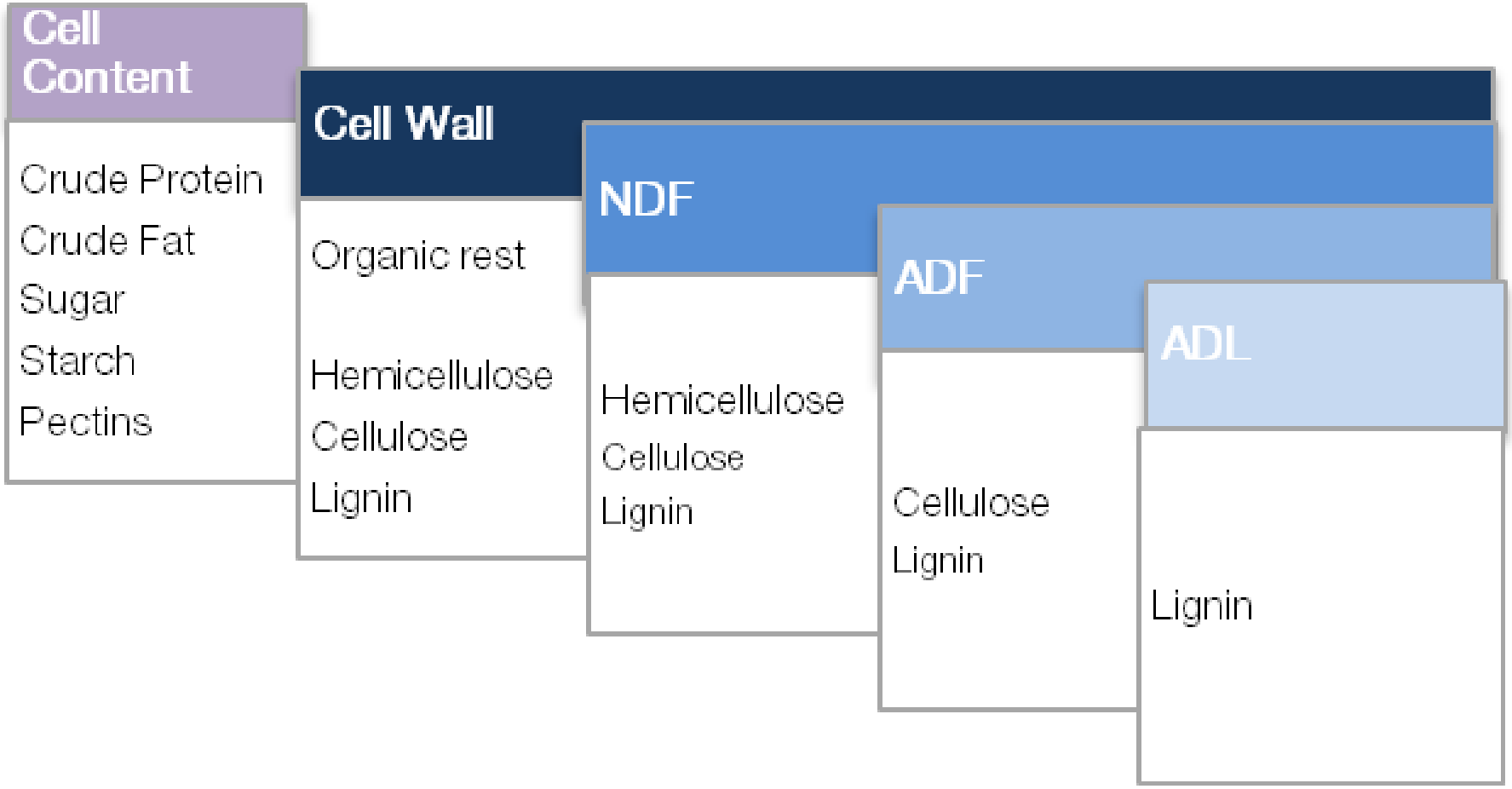
Neutral Detergent Fibre: a good indicator of bulk and thus feed intake.

Acid Detergent Fibre: a good indicator of digestibility and thus energy intake.

DETERGENT FIBER – VAN SOEST METHOD



DETERGENT FIBER

Neutral Detergent Fibre NDF: a good indicator of bulk and thus feed intake. As NDF percent increases, the dry matter intake generally decreases

Acid Detergent Fibre ADF : a good indicator of digestibility and thus energy intake. As ADF increases the ability to digest or the digestibility of the forage decreases.

Acid Detergent Lignin ADL : is good to determine the indigestible lignin content

VAN SOEST METHOD

PROCEDURE AND CALCULATION

In the first step the grinded sample is treated with a Neutral Detergent Solution (**NDS**) in order to make sugars, starches and pectin soluble, obtaining the less-digestible substances (**NDF**):

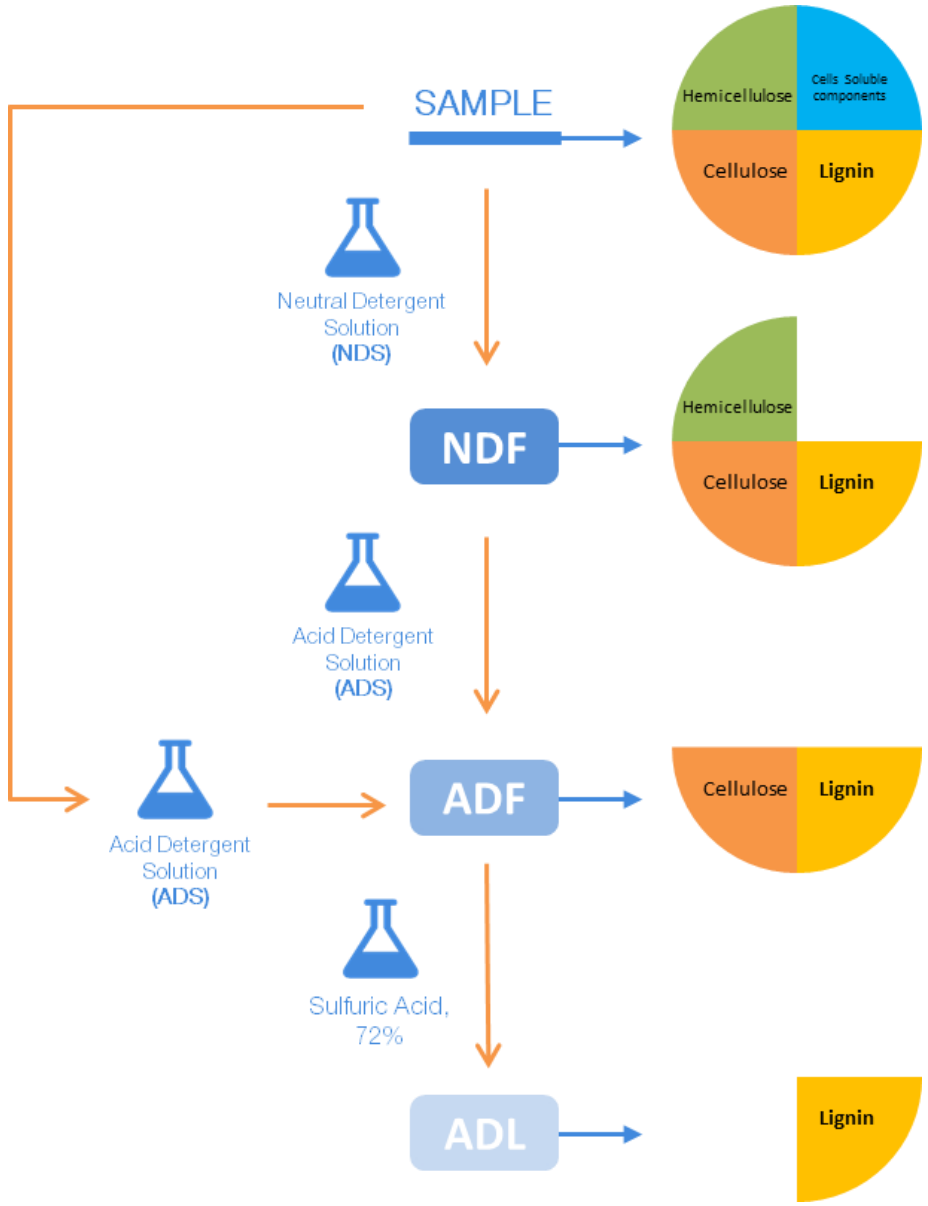
- Hemicellulose
- Cellulose
- Lignin

In the second step, another grinded sample is treated with an Acid Detergent Solution (**ADS**) in order to make hemicellulose soluble, leaving the cellulose and lignin in the residues (**ADF**).

NDFw – ADFw= Hemicellulose

The residue is treated with concentrated sulfuric acid (72%) dissolving the cellulose and leaving the lignin in the residue (**ADL**)

ADFw – ADLw= Cellulose



DETERGENT FIBER METHOD – VAN SOEST

SAMPLE PREPARATION

Finely grind the sample (**1 mm particle size**) in order to obtain a homogeneous compound following official guidelines.

BOILING & FILTERING

DRYING, ASHING & WEIGHING

- Dry the sample in a dessicator
- Put the dried sample in a furnace at 550°C to produce ashes

CALCULATION

CALCULATION

During the analysis we have weighed the crucibles in different stages, and now we have 3 weights:

- **F0**: initial weight
- **F1**: crucible weight after drying
- **F2**: crucible weight with ashes

With these weights it is now possible to calculate the % of raw fiber:

$$\% \, \boldsymbol{Fiber} = \frac{\boldsymbol{F1 - F2}}{\boldsymbol{F0}} \, \mathbf{x} \, \mathbf{100}$$

VELP SOLUTIONS

COEX

FIWE Series
3 or 6 positions
Semi automatic units

ermes enabled

FIWE Advance
6 positions
Fully automatic unit

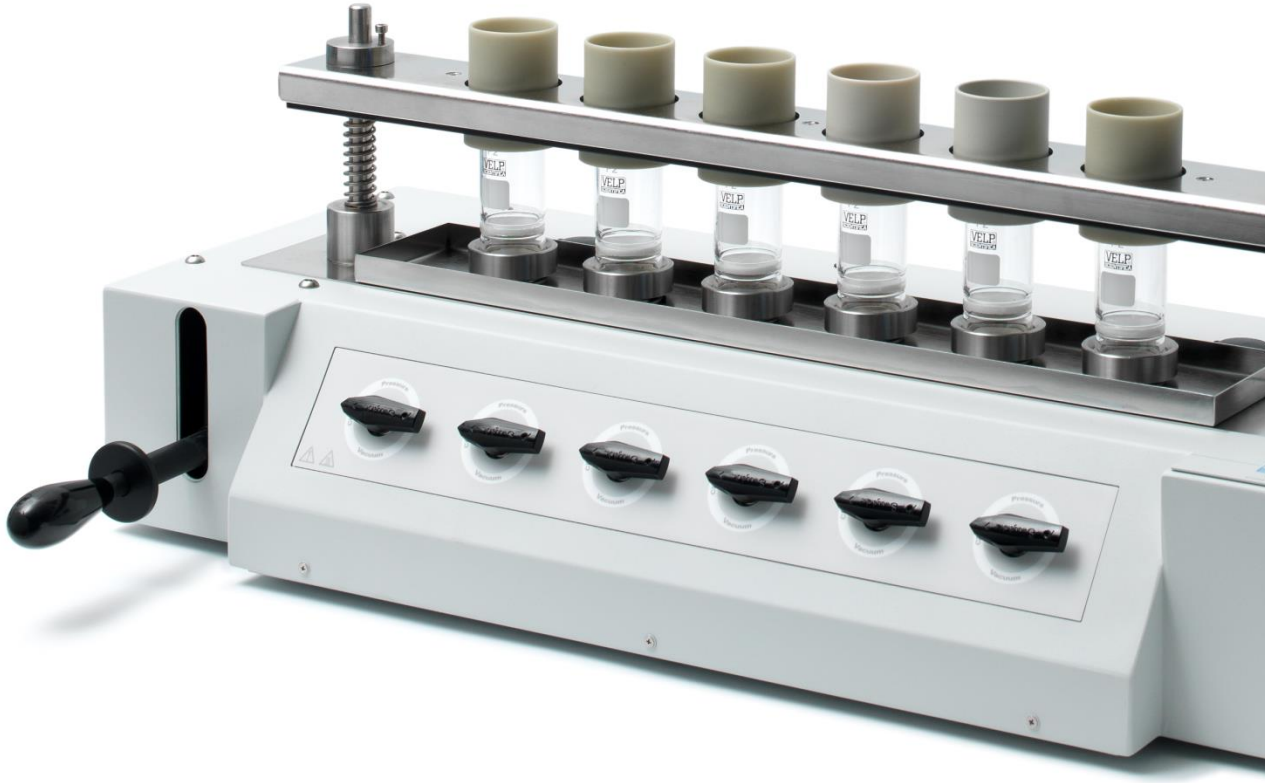
PRELIMINARY FAT EXTRACTION - CF

If the **fatty matter content** is between 5 and 10 % extraction is recommended, but if it is more than 10% is mandatory.

PROCEDURE

- Place the 6 crucibles containing the samples to be defatted into COEX and pull down the lever until automatically blocked.
- Use 25 ml of acetone, hexane or petroleum ether 40-60 °C for each gram of sample, by introducing the solvent into the tubes located over each crucible.

Perform 3 times, with the help of the vacuum in order to speed up the sample washing.



OFFICIAL STANDARDS

- **Weende or crude fiber: AOAC 978.10 (Fritted Glass Crucible Method) and ISO 6865:2000** *(Ankom standard AOCS Ba6a-05)*
- **Acid Detergent Fiber: AOAC 973.18 and ISO 13906.2008**
- **Neutral Detergent fiber: AOAC 2002:04 and ISO 16472:2006**

FIWE ADVANCE AUTOMATIC FIBER ANALYZER

FIWE ADVANCE RAW FIBER ANALYZER

FIWE Advance is the VELP fully automatic system for the determination of crude fibre, detergent fibre and related parameters according to international standard reference methods such as **Weende** and **van Soest**

- Unattended measurement of up to 6 samples simultaneously ensuring the lowest operator time, accuracy and precision in raw materials and finished products in Feed and Agriculture sectors.
- FIWE Advance takes care of all the analysis steps requiring **less than 2 minutes of the operator time.**

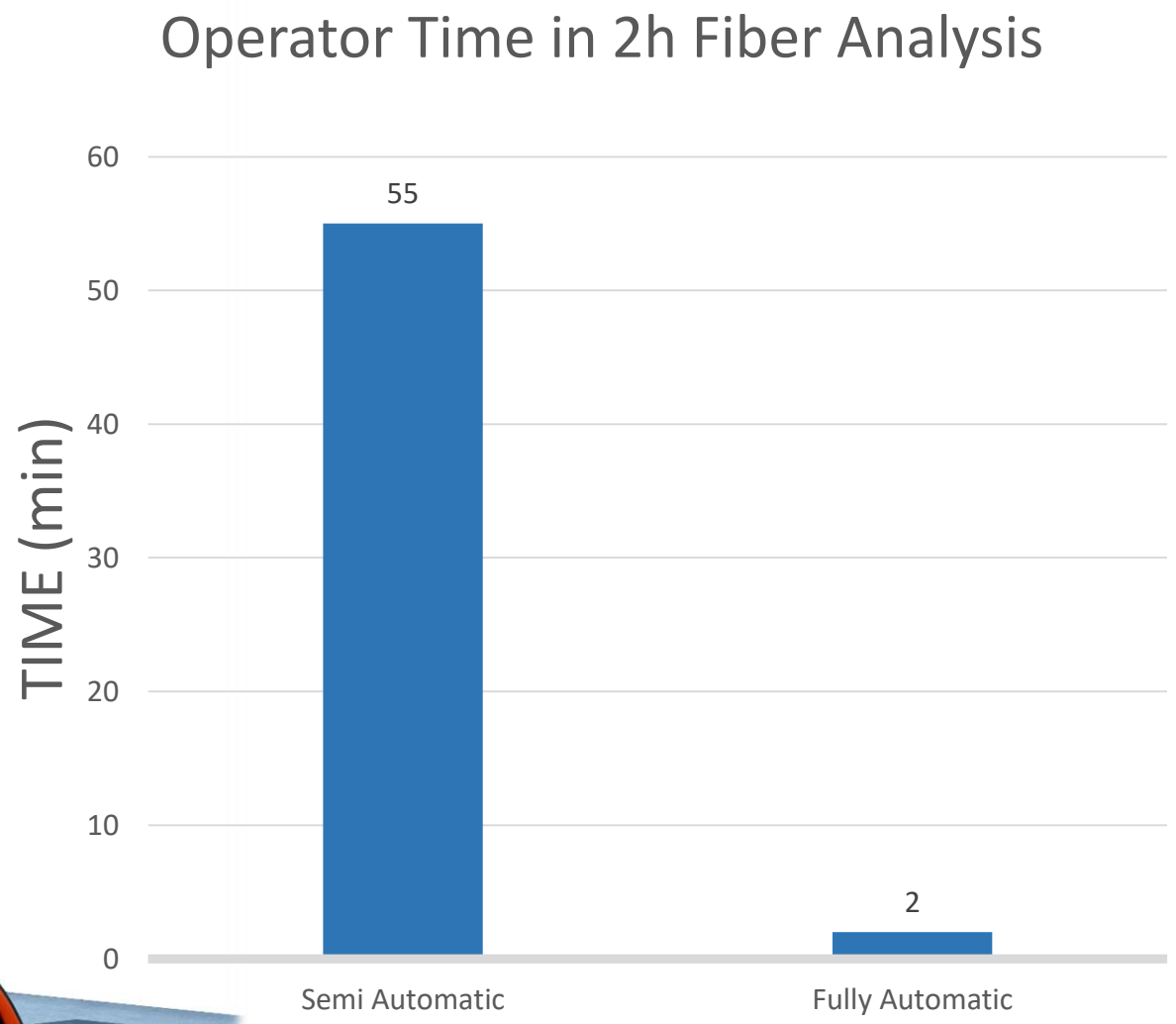
FIWE ADVANCE RAW FIBER ANALYZER

- Automatic **heating** and **dispensing** of the reagents ensuring no contact with chemicals and fumes
- The **VELP Dispensing Nozzle** precisely dispenses the chemicals into each column avoiding contact with dangerous hot chemicals and their fumes.
- **Boiling**, **washing** with water and **filtration** are carried out without supervision

FIWE ADVANCE SAFETY

- Safety sensors will ensure complete protection during all the stages of the analysis
- The FIWE Advance is able to adjust the heating power to ensure gentle boiling, automatically
- State-of-the-art filtration control guarantees proper filtration in all conditions
- All residues are automatically driven to external tank.

LOAD AND GO OPERATION SAVES OPERARATOR TIME



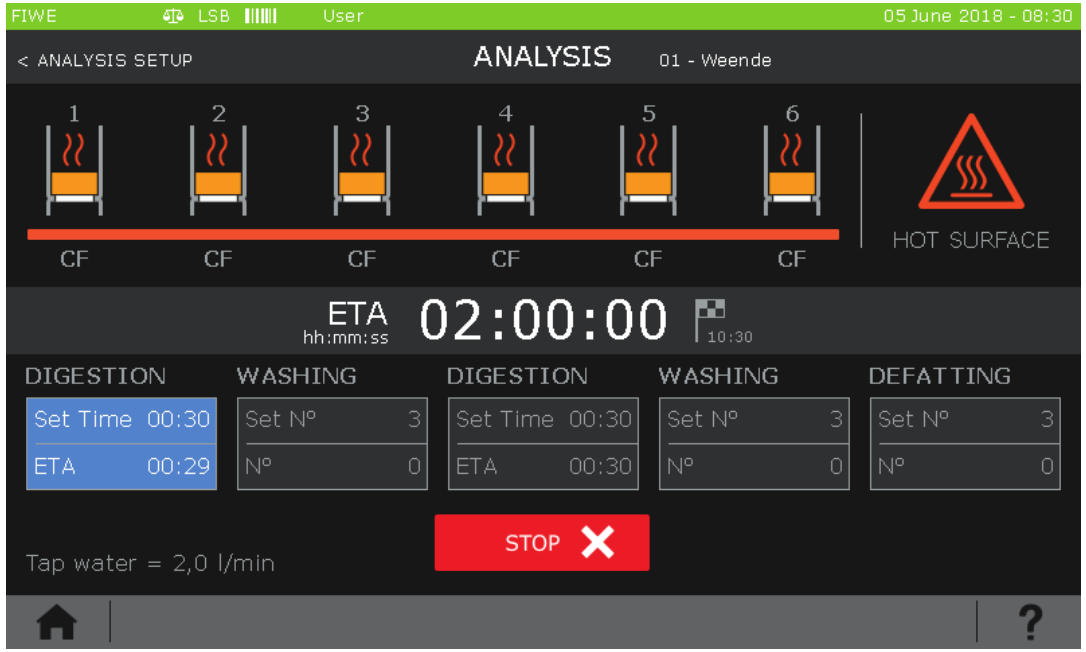
AUTOMATIC vs SEMI AUTOMATIC – TIME COMPARISON

Semi Automatic Extractor	CRUDE FIBER DETERMINATION	FIWE Advance
1 min	Crucible loading and instrument start	1 min
5 min	Preheating and reagent loading (Acid + Antifoam)	-
10 min	Heating up until reagent boiling	-
10 min	Draining and washing (hot water)	-
5 min	Preheating and reagent loading (Alkali + Antifoam)	-
9 min	Heating up until reagent boiling	-
15 min	Draining and washing (hot water, cold water)	-
1 min	Crucibles removing	1 min
57 min	**TOTAL OPERATOR TIME**	**2 min**

AUTOMATIC vs SEMI AUTOMATIC – TIME COMPARISON (CF)

	FIWE	FIWE Advance
		
Analysis per day	36 samples	36 samples
Average Operator Time in front of the instrument	6h	0,12 h

FIWE ADVANCE SMART AND EASY



- Intuitive **7” color touch screen** showing all the information at a glance
- **Load&Go** operations. One-click start of the analysis
- Balance connection, automatic results calculation and storage

BARCODE READERS & CRUCIBLES

To improve the productivity of laboratories VELP offers a wide range of accessories.

The VELP crucibles (P0, P2, P3 porosity) are now provided with an **embedded barcode** that can be scanned in order to **transfer the data automatically to the FIWE Advance** by either USB or wireless connection.

The benefits of using bar code technology:

- **Reduce Risk of errors**
- **Time Saving**
- **Improved automation**

A00000364

Barcode scanner with USB

A00000365

Wireless barcode scanner

	A00000138	A00000139	A00000140	A00000137
DESCRIPTION	Glass crucible P0 6pcs/box	Glass crucible P1 6pcs/box	Glass crucible P2 6pcs/box	Glass crucibles P3 6pcs/box
FILTER	P0	P1	P2	P3
NOMINAL PORE	Size 160-250 micron	Size 100-160 micron	Size 40-100 micron	Size 16-40 micron
EXAMPLE OF APPLICATIONS FIELDS	Special crucible; Filtration of very coarse precipitates	Special crucible; Filtration of coarse precipitates	Standard crucible; preparatory work with crystalline precipitates	Special crucible; analytical work with medium-fine precipitates

FIWE ADVANCE CONNECTIVITY

- The FIWE advance works unattended to allow overnight work benefiting of **cloud connectivity** for real time control and immediate notifications.
- Connect your FIWE Advance to the VELP Ermes platform to access your instrument anytime, anywhere.
- Get access to your database from PC, smartphone and tablet and benefit of enhanced service.
- **The instrument is designed to improve over time with regular software updates**, introducing new features, functionality and performance.

FIWE SERIES - BENEFITS

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.

REDUCED TIME FOR ANALYSIS

2 hours with FIWE vs. 6 hours manually.

EASY TO USE

Convenient filtration - with pump and air pressure

PRECISION and ACCURACY

High reproducibility of the results: ±1% relative or better

FIWE SERIES - BENEFITS

HIGH PERFORMANCE

Back-pressure pump prevents clogging of crucibles.

VERSATYLE

Able to perform:

- Total raw fiber extraction using Weende technique, ISO, AOAC
- Neutral & acid detergent fiber with NDF, ADF, Van Soest extractions
- Acid detergent lignin extraction with ADL & Van Soest methods
- Fiber fraction analysis (cellulose, hemicellulose & pectin)

COEX - BENEFITS

6-POSITION

Degrease up to 6 samples simultaneously

SHARED CRUCIBLES

COEX and FIWE support the same kind of glass crucibles

RESISTANCE

COEX ensures high resistance against reagents and solvents used during the analysis

GLASS CRUCIBLES

VELP Glass crucibles are made to perfectly fit the COEX and FIWE station.

In order to prevent sample loosing during all the phases of the analysis, VELP Scientifica recommends, as the official methods do, using **P2 porosity crucibles**,

After **≈20 analysis** (depends on the analysis) the crucibles could be clogged and this could compromise the analysis, for this reason it is necessary to replace them with new ones.

Durability is strictly tied to:

- correct use of crucibles for ash analysis (muffle oven)
- correct cleaning procedure: chemical and mechanical cleaning

GLASS CRUCIBLES

VELP Scientifica produces different types of crucibles depending on the type of sample to be analyzed:

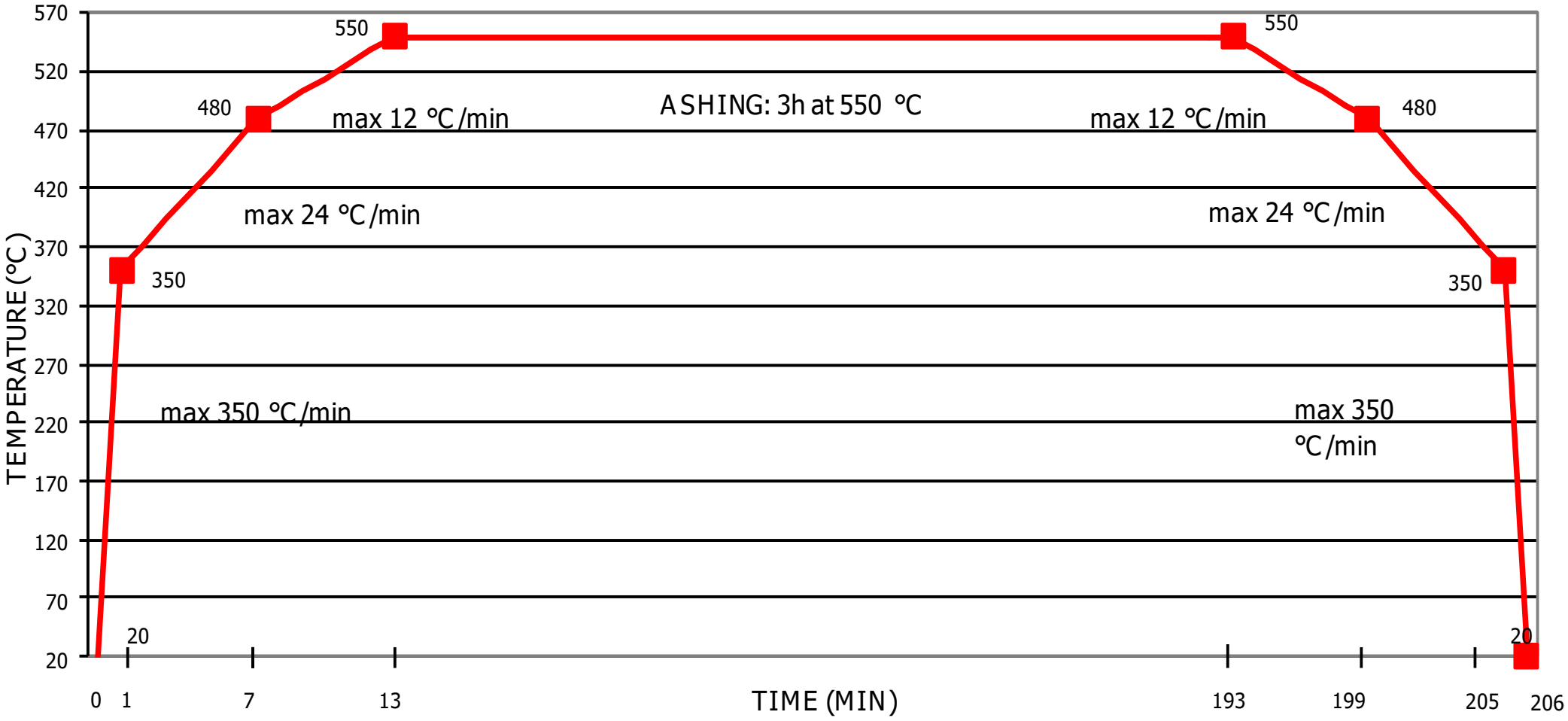
STANDARD CRUCIBLE

- For FIWE, COEX and CSF6
- Preparatory work with crystalline precipitates
- **P2** Porosity: 40-100 µm

SPECIAL CRUCIBLE

- For FIWE, COEX and CSF6
- Filtration of very coarse precipitates
- 3 different porosities:
  - **P0**: 160-250 µm
  - **P1**: 100-160 µm
  - **P3**: 16-40 µm

MAXIMUM RATES FOR HEATING/COOLING GLASS CRUCIBLES



FIBER ANALYZERS FOR THE FOOD INDUSTRY

DIETARY FIBER DETERMINATION

Dietary fiber is not digested by the human small intestine and represents a crucial nutrient in the human diet for its ability to ensure **satiety**, to **regulate intestinal functions** and to **modulate** the **nutrients absorption**.

Thanks to its benefits, the **fiber is increasingly used** in functional foods, and food manufacturers declare the fiber content on the package as part of nutritional labeling.

Dietary Fiber can be described as the indigestible portion of food derivate from plants.

DIETARY FIBER DETERMINATION

The general method for dietary fiber determination is the **enzymatic method**.

After defatting, a food sample is treated with enzymes that mimic the digestive process in the human small intestine.

Digestible carbohydrates are removed from the sample by **precipitation** and **filtration**.

The **non-digestible precipitate contains** not only dietary fiber but also **proteins** and **inorganic materials**, which must be measured separately and subtracted from the weight.

DIETARY FIBER DETERMINATION

The **AOAC methods** is the most widely used for the total Dietary Fiber determination.

The **AOAC method 985.29** is advantageous since it is simple and well known for routine analysis.

AOAC METHOD: 985.29 - PROCEDURE

Duplicate of samples of dried, fat-free foods are gelatinized with heat-stable alpha-amylase and digested using amyloglucosidase to remove starch, and protease to remove proteins.

The soluble dietary fiber is precipitated with ethanol.

The residue is then filtered, washed with ethanol and acetone, dried and weighed.

One of the duplicate samples is assayed for indigested proteins and the other is ashed.

The total dietary fiber is the weight of the enzymatic digestion residue less the weight of the undigested protein and ash.

This procedure requires lots of time and it is important to follow every single step, in order to have precise results.

VELP SOLUTIONS

GDE

CSF6

GDE – ENZYMATIC DIGESTOR

The VELP incubator for Dietary Fiber Analysis.

A complete package of:

- immersion heating head
- transparent tank
- MULTISTIRRER with 6 positions

VELP RACCOMENDATION

In order to reduce the temperature and possibility of losing sample, caused by the evaporation, during the analysis it is recommended to use **VELP hollow balls**.

A00000241
Hallow Balls
200pcs/box

GDE – ENZYMATIC DIGESTOR

CONSTRUCTION MATERIAL

- Transparent tank in polycarbonate. Enhanced resistance and able to reach high **temperature up to 105 °C**
- The design of the thermostating group improve the **protection of electric** and **electronic components**

EXCELLENT THERMOREGULATION

- **Excellent thermoregulation**: ± 0.2 °C
- Precise temperature control during critical enzymatic digestions

INTERNAL COMPONENTS

- **Stainless steel level sensor** allows the use of deionized water avoiding the limestone formation

MAIN BOARD

- Integrated Digital **Timer** from 00:00 to 24:59 (hh:mm)

CFS6 FILTRATION UNIT

- Indipendent 6 positions
- Less than 20 minutes for filtering
- Pumps to speed up filtration process and washing
- Same crucibles of FIWE and COEX

CFS6 FILTRATION UNIT

The VELP Solution for **filter** and **wash** phases. It is recommended to filter and wash one by one sample.

The high efficient pump allows to speed-up the filtration step and the final washing.

Compressed air can be also injected from the bottom to remove any sample accumulated in the filter of the crucible, which affects the quality of filtration.

KJELDAHL PROTEIN DETERMINATION – AOAC 960.52

Test Tubes Ø26x300mm, 6 pcs/box

Code No A00000146

DK 20/26

DK 42/26

DKL 42/26

KJELDAHL PROTEIN DETERMINATION – AOAC 960.52

JP PUMP
for fumes aspiration

SCRUBBER SMS
for fumes neutralization

JP + SMS + DK/DKL
Standard configuration

KJELDAHL PROTEIN DETERMINATION – AOAC 960.52

UDK 129

Distillation Unit
(Entry Level)

UDK 139

Semi-Automatic
Distillation Unit

UDK 149

Fully Automatic
Distillation Unit
(Connectable with
External Titrator)

UDK 159/169

Automatic, Interactive
Distillation & Titration System
(Top of the Range)

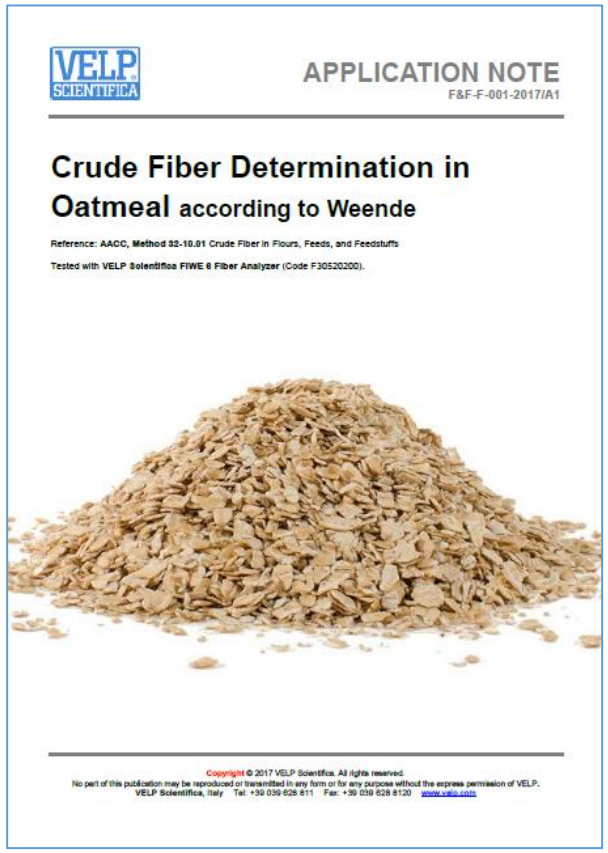
UDK 169 & Autokjel

Fully Automatic, Interactive
Distillation & Titration System
(Top of the Range)

APPLICATION NOTE

Application notes are available on our website.

Register @ www.velp.it to grant access to our wide and Free Application Notes Database


VELP SCIENTIFICA

APPLICATION NOTE
F&F-F-001-2017/A1

Crude Fiber Determination in Oatmeal according to Weende

Reference: AACC, Method 32-10.01 Crude Fiber in Flours, Feeds, and Feedstuffs

Tested with VELP Scientifica FIWE 6 Fiber Analyzer (Code F30520200).


VELP SCIENTIFICA

APPLICATION NOTE
F&F-F-001-2013/A1

Total Dietary Fiber in Corn Flakes according to AOAC 985.29

Reference: AOAC 985.29 Total Dietary Fiber in Foods

Tested with VELP Scientifica GDE Enzymatic Digester (Code F30400209) and CSF6 Filtration Unit (Code F30420210). Protein determination was performed with DK 6 Kjeldahl Digestion Unit (Code F30100182) and UDK 169 Automatic Kjeldahl Distillation & Titration System (Code F30200150)

OATMEAL

Oats are a whole-grain cereal and a good source od fiber, vitamins, mineral and antioxidants.

Oats contain more soluble fiber than other grains, leading to slower digestion, increased satiety and suppression of appetite.

Sample preparation

Dry the sample in oven and cool in desiccator and grind test sample to 1 mm particle size. Put 1 g of the sample in the crucible and start the analysis.

OATMEAL - RESULTS

W1 (g)	W2 (g)	W3 (g)	Fiber %
0,985	30,5253	30,4533	7,31%
1,010	30,4192	30,3444	7,41%
0,968	30,3726	30,3001	7,49%
0,970	30,3752	30,3043	7,31%
1,024	29,9166	29,8414	7,35%
0,970	30,4862	30,4146	7,38%
		Average ± SD%	**7.37 ± 0.07**
		RSD% *	**0.9**

Fiber Labeled Value: 7.0±0.6%

CORN FLAKES

Dietary fiber is not digested from human small intestine and it represent a crucial nutrient in human diet for its ability to enhance satiety, to regulate intestinal functions and to modulate the nutrients absorption.

Fiber labeled value: **3 %**

Sample preparation

Homogenize test sample and dry over night in oven at 105 °C. Cool in desiccator and grind test sample to 0.3–0.5 mm mesh.

CORN FLAKES - RESULTS

Crucible	Sample ID	Crucible Tare (g)	Sample Weight (g)	Crucibles Weight (g)	Total Residue (g)	CSR (g)	TDF % d.m.	TDF %
1	BLANK a	31.0831	-	31.1275	0.0444			
2	BLANK p	31.0481	-	31.0915	0.0434			
3	1p	30.6026	1.0000	30.7151	0.1125	0.0279	2.8	**2,7**
4	2p	31.1503	1.0003	31.2641	0.1138	0.0292	2.9	**2.8**
5	1a	31.0548	0.9999	31.1701	0.1153	0.0307	3.1	**2.9**
6	2a	30.8120	0.9999	30.9241	0.1121	0.0275	2.8	**2.6**
						Average ± SD%	2.9 ± 0.1	**2.7 ± 0.1**

Thank You

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