

# Total FAT Quantitation using a new Microwave Assisted Extraction Workflow

## FAST FAT PROJECT



# Program

- **Introduction**
- **Total Fat Arbitration Methods**
- **Microwave M.A.E**
- **Advantages**
- **Figures of Merit**

- **History of more than 100 years in Analytical Chemistry**

[www.thermounicam.pt](http://www.thermounicam.pt)

**thermo**  
scientific

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Authorised Distributor

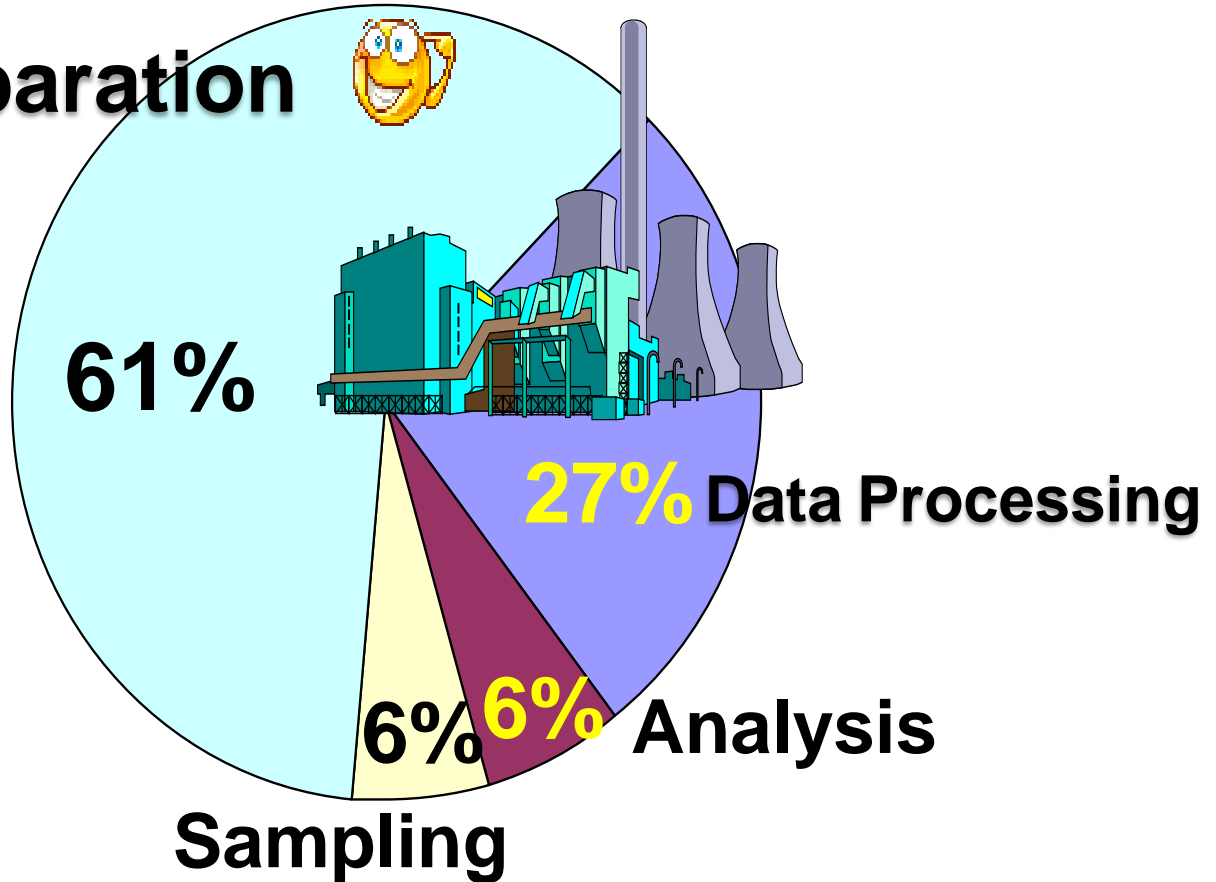
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**MILESTONE**  
H E L P I N G  
C H E M I S T S

# The Lab : Time is money

**Sample Preparation**



**Sampling**

From: Ronald Majors "Overview on sample prep." LG\_GC. VOL. 9 . 1991

# The Modern Lab

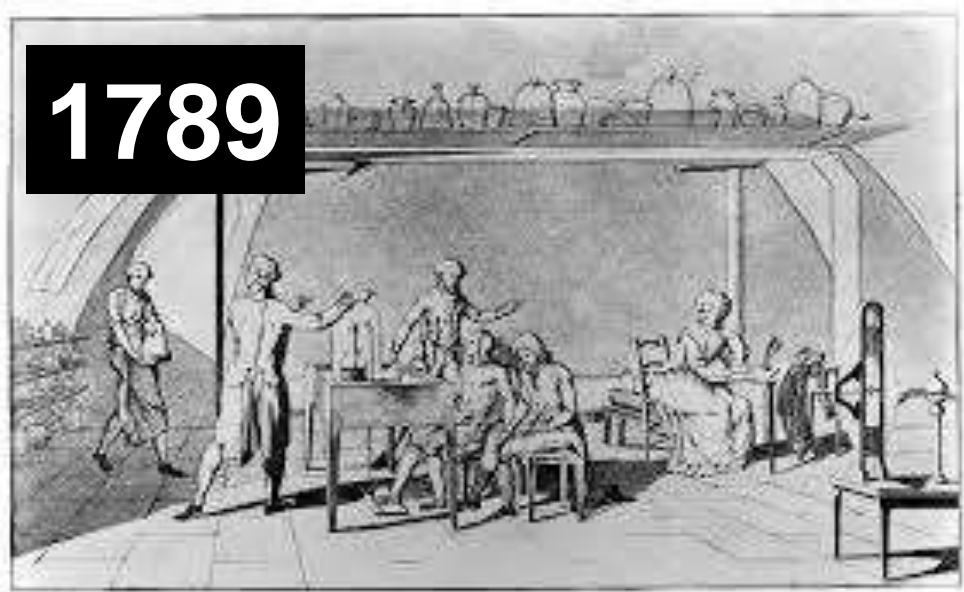
- HPLC – HPLCMS – ICP- GCMS
- Modern technologies in ANALYSIS



1.5M€

# Sample Preparation

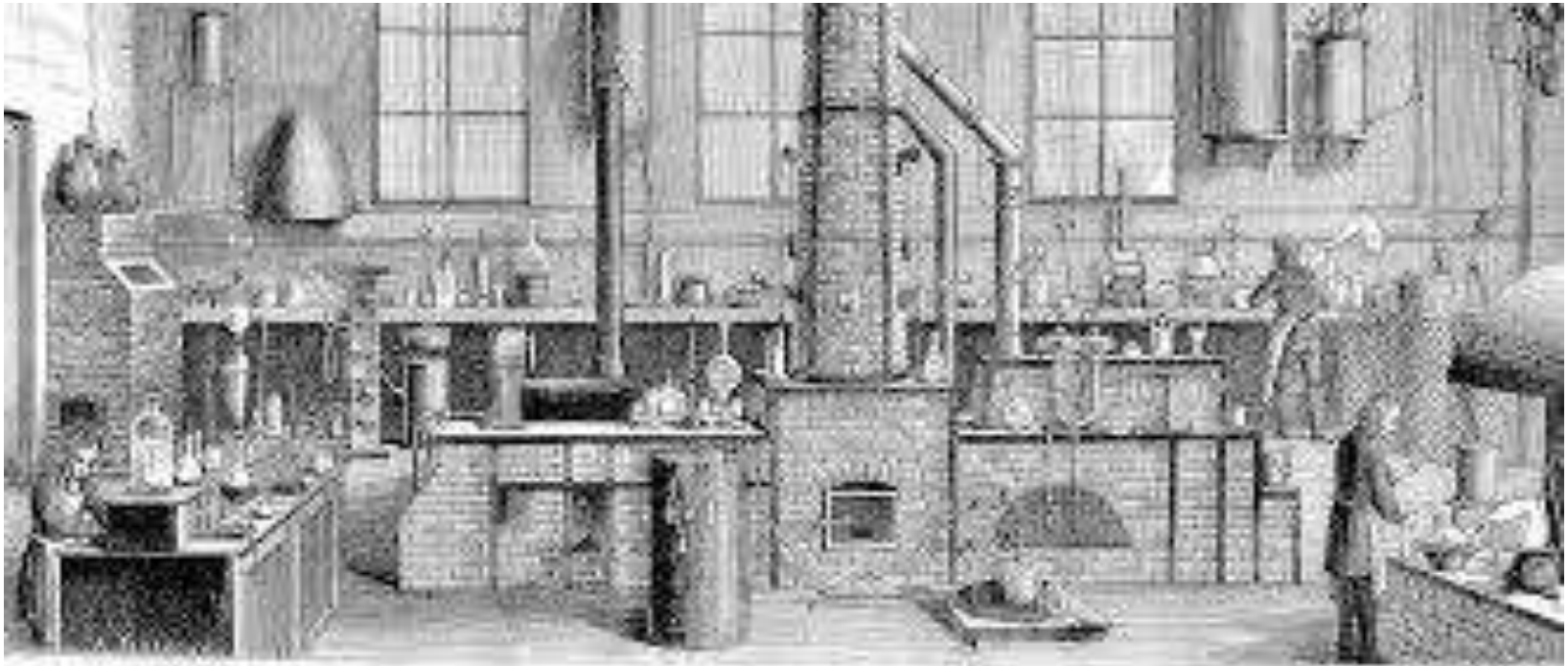
# Lavoisier : law of conservation of mass



- Ovens, Heating baths, ...

# 150 years ago Mendeleev

- Ovens, Heating baths, ...



Слева направо: Демидовский завод (1790-е годы), 1890-е. — Дворец Исаакитов.





# Soxhlet

German agricultural chemist Franz Ritter von Soxhlet first introduced its Soxhlet laboratory extractor in **1879** which deals in the determination of milk fat.



# Standard Methods for Total Fat

## Arbitration method

### gravimetric test

- **„Weibull-Stoldt“** (universal method)
- „Schmidt-Bondzynski“ (cheese)
- „Röse-Gottlieb“ (milk, cream)
- *normal cheese must be analysed accord. to „Schmidt-Bondzynski“*
- *cheese with herbs/pepper must be analysed accord. to „Weibull-Stoldt“*
- *AOAC Methods – NP similar to W-S and are the common reference*

### Other fast-method

butyrometric test,

or volumetric determination

- for milk: „Gerber“
- for cheese „Gulik“
- for cream: „Roeder“
- for skim milk: „Konrad“
- NIR/NMR

depending on user performance,  
not very precise

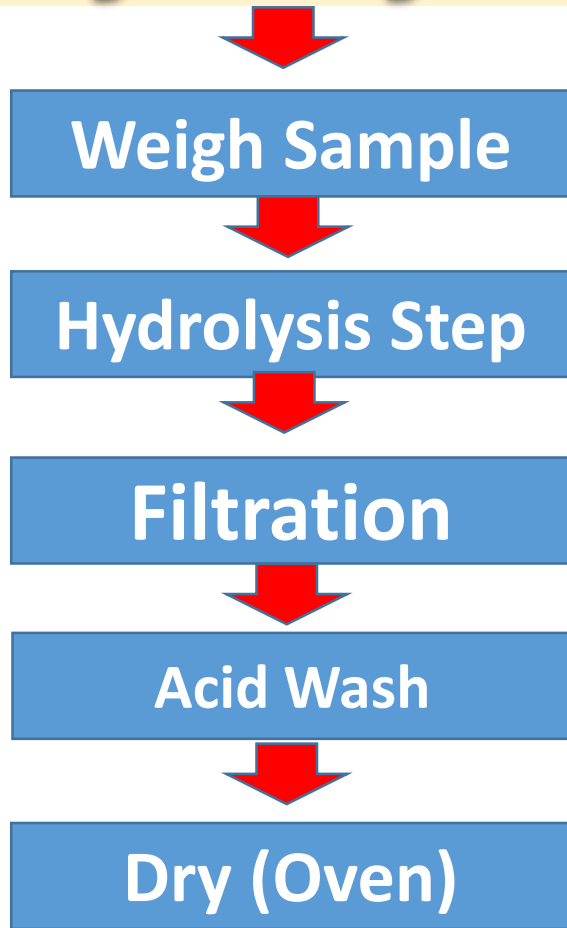


# Weibull-Stoldt Method Workflow

## Hydrolysis

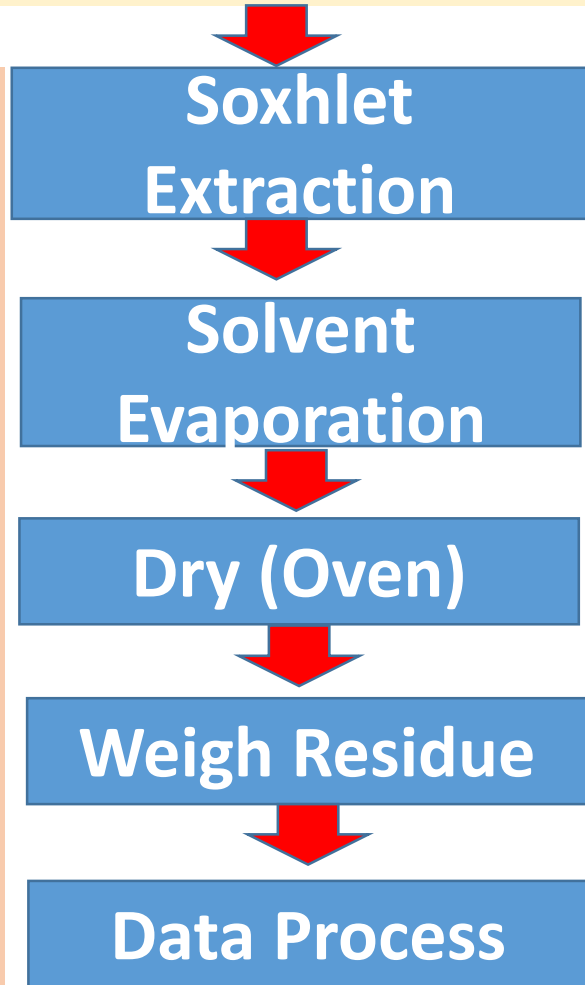


## Extraction



8 HOURS

8 HOURS



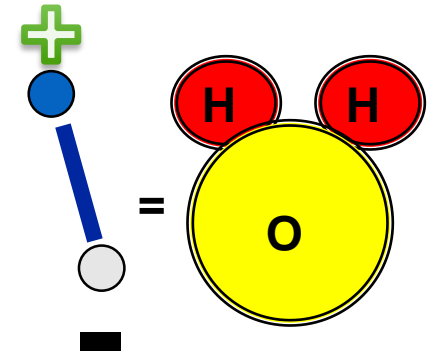
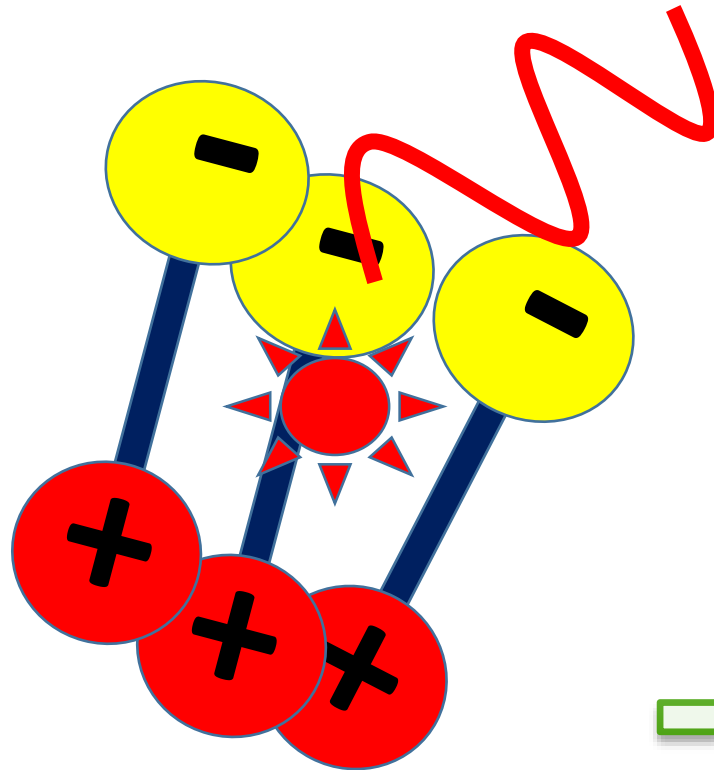
# The Problem

- **Total Fat is a mandatory analysis used for Food/Feed labelling**
- **It takes 2 days to get to results**
- **Solvent Waste**

**Can we make it faster?  
And Greener?**

# MW HEATING

Can we use Microwaves?




2.45GHz  
12.2cm

ROTAÇÃO DIPOLAR E MIGRAÇÃO IÓNICA

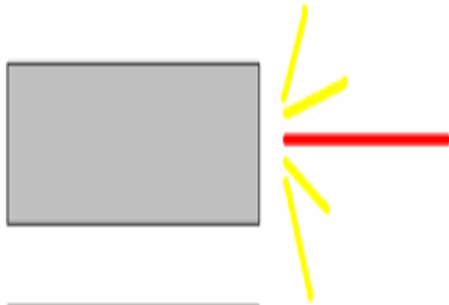
# Can we use MW M. A.E for TOTAL FAT Extraction ?

- The solvent of extraction is superheated by microwave (above boiling point )
- Extractions Kinetics

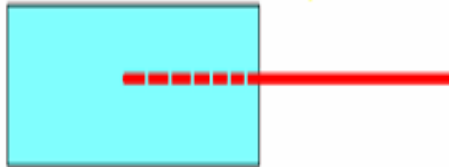
$$k = Ae^{-\left(\frac{Ea}{RT}\right)}$$


# Microwaves and Heating

- The material can be :



- Reflective (metals)



- Absorbent (Water)



- Transparent (Hexane)

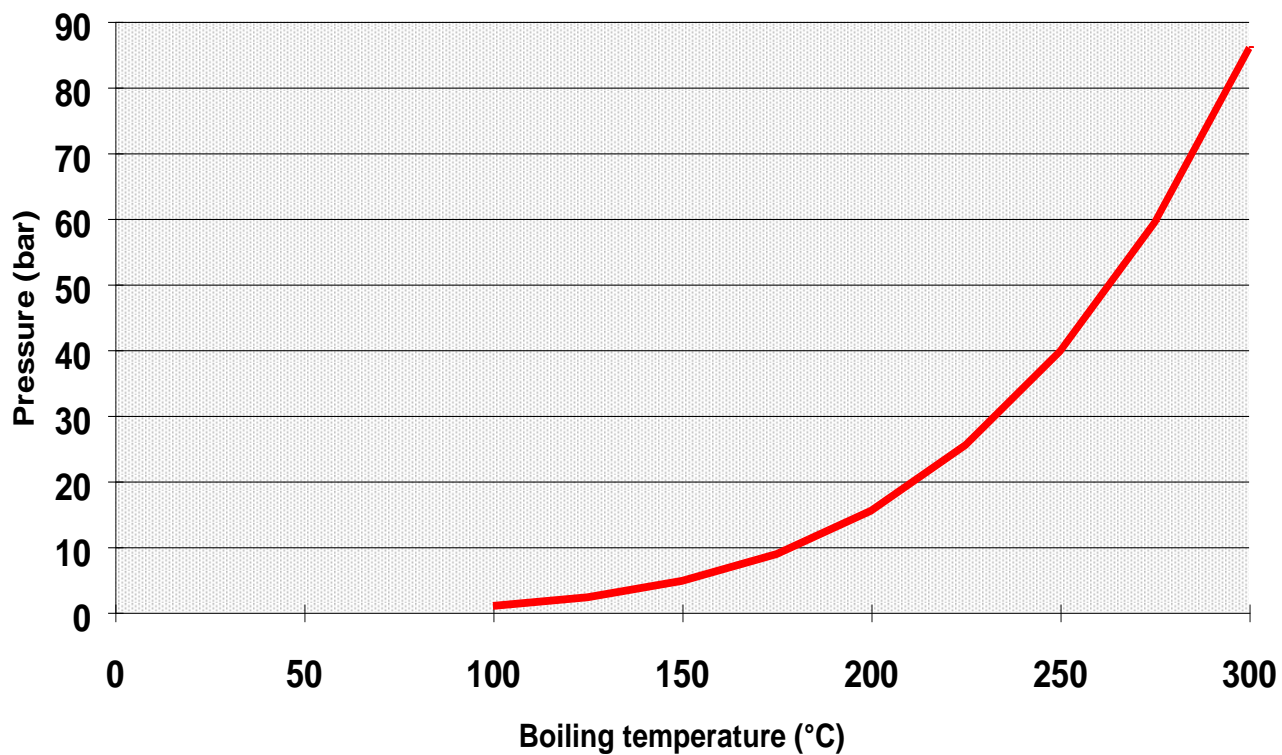
# Microwaves in the Lab

- **CLOSED VESSEL Sample Digestion**
- **CLOSED VESSEL Sample Hydrolysis**
- **CLOSED VESSEL Extraction with Organic solvents and stirring**





# **BOILING TEMPERATURE OF WATER vs. PRESSURE VAPOUR**



# Total Fat Determination

- *Hypothesis: A fast and precise method for food and feed samples*



# Proposed MAE Method Workflow

**Hydrolysis** | **Extraction**

**Weigh Sample**

**Hydrolysis Step**

**Soxhlet Extraction**

**Aliquot Pipet**

**Filtration**

**Solvent  
Evaporation**

**Dry (Oven)**

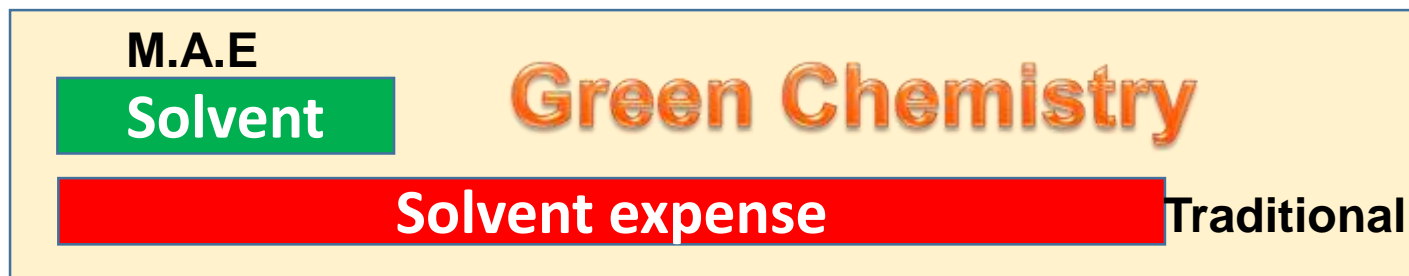
**Weigh Residue**

**Data Process**

**Stirring**

# Advantages

One Method for all



# Proposed Hardware for Total Fat



Professional MW



Balance 12 Position



Evaporation



\* Vacuum pump with pressure condensation for improved solvent recovery



**FAST**  
Fast Fat Analysis

# Figures of Merit

## Comparison of a New Total Fat Quantification Method in Cheese using Microwave Assisted Extraction (MAE) and Soxhlet

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Conclusion:

M.A.E proved to be easier, simpler and equivalent to Soxhlet Methods for Cheese samples

# Already tested and future

- **12 Samples in the same instrument**
- **Or ...different samples in same run**
  - **Meat**
  - **Sausages**
  - **Yogurth** (High and low fat)
  - **Milk**
  - **Cheese**

# Acknowledgements

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