

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**

UNICAM

Sistemas Analíticos, Lda.

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

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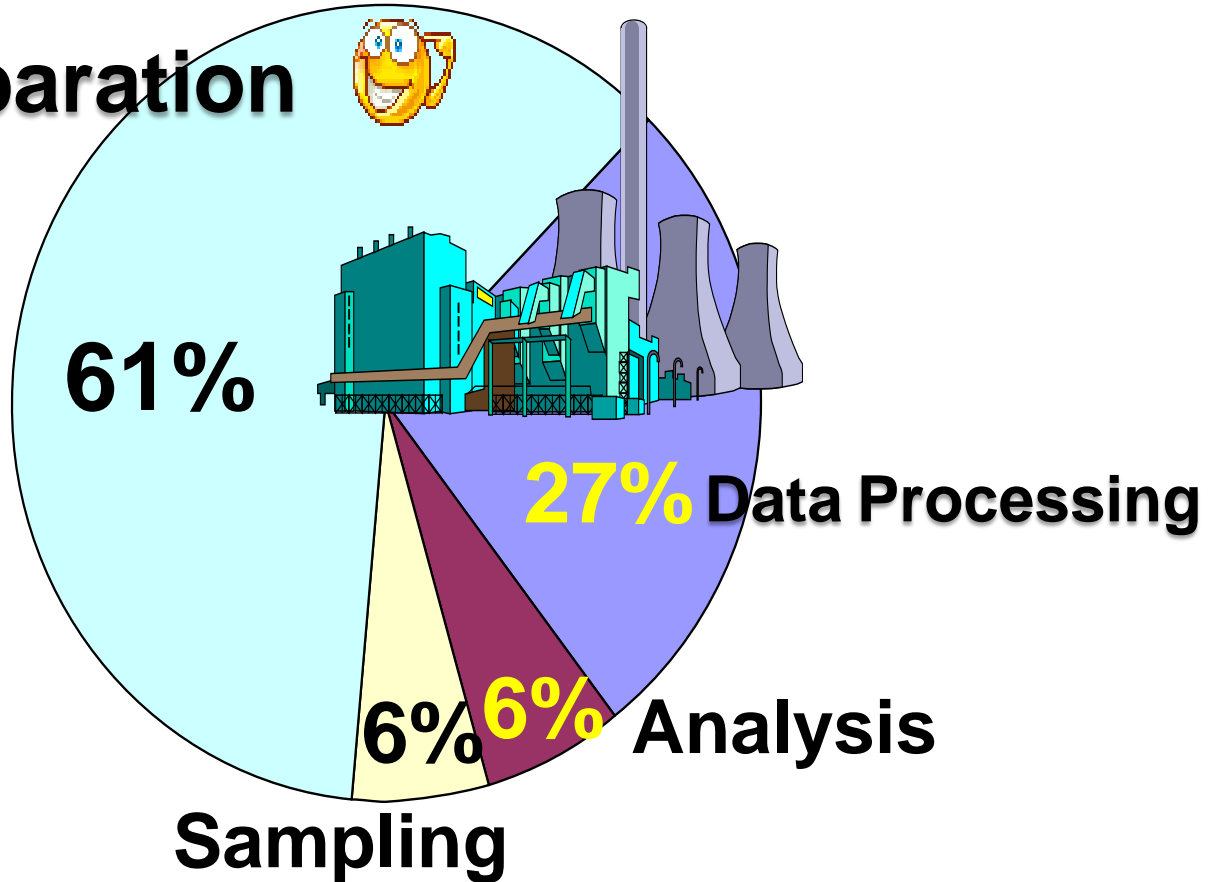


MILESTONE

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

The Lab : Time is money

Sample Preparation



From: Ronald Majors "Overview on sample prep." LG_GC. VOL. 9 . 1991

The Modern Lab

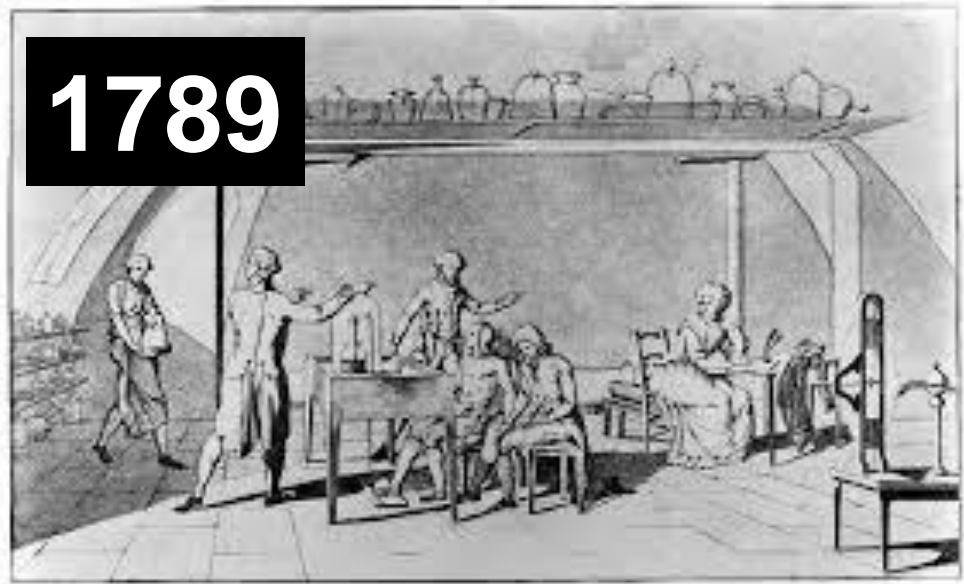
- HPLC – HPLCMS – ICP- GCMS
- Modern technologies in ANALYSIS



1.5M€

Sample Preparation

Lavoisier : law of conservation of mass



- Ovens, Heating baths, ...

150 years ago Mendeleev

- Ovens, Heating baths, ...

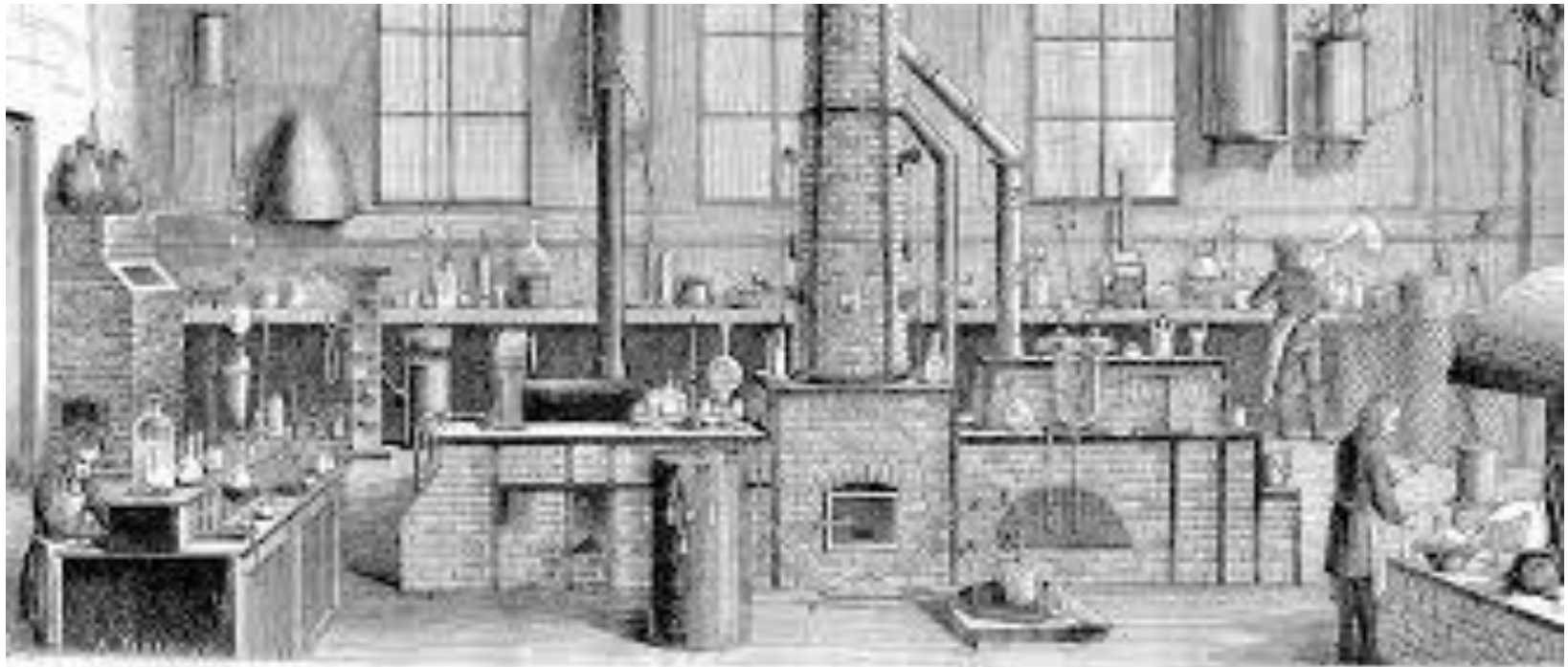
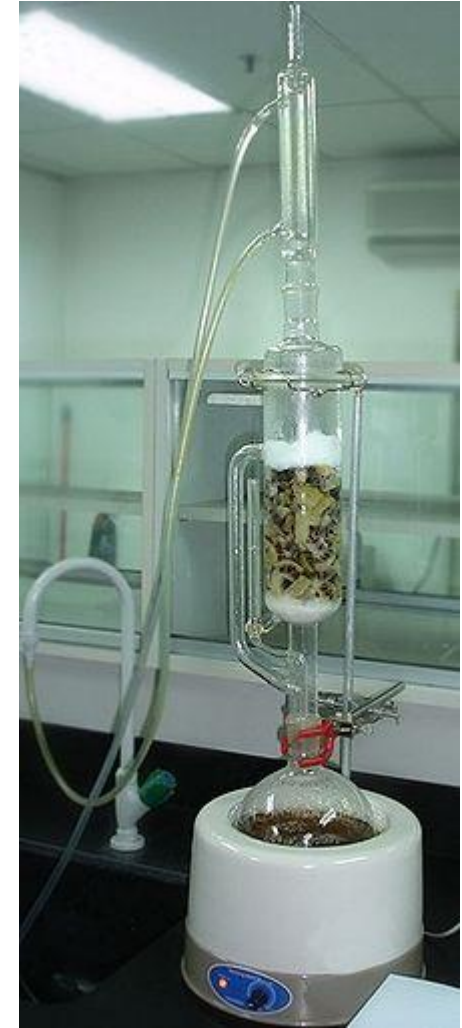


Схема кабинета химии в Цюрихе, 1869 г. — Д.И. Менделеев.



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**

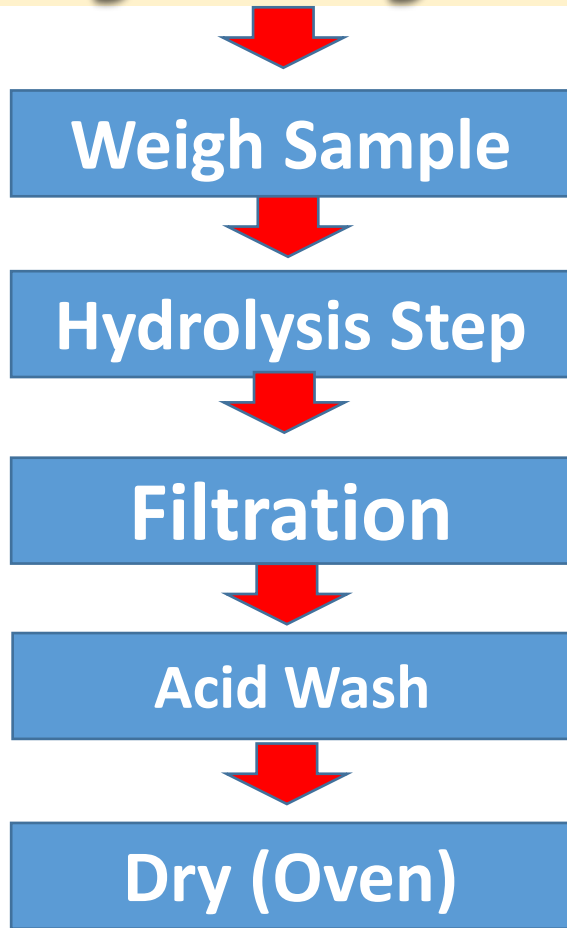


Weibull-Stoldt Method Workflow

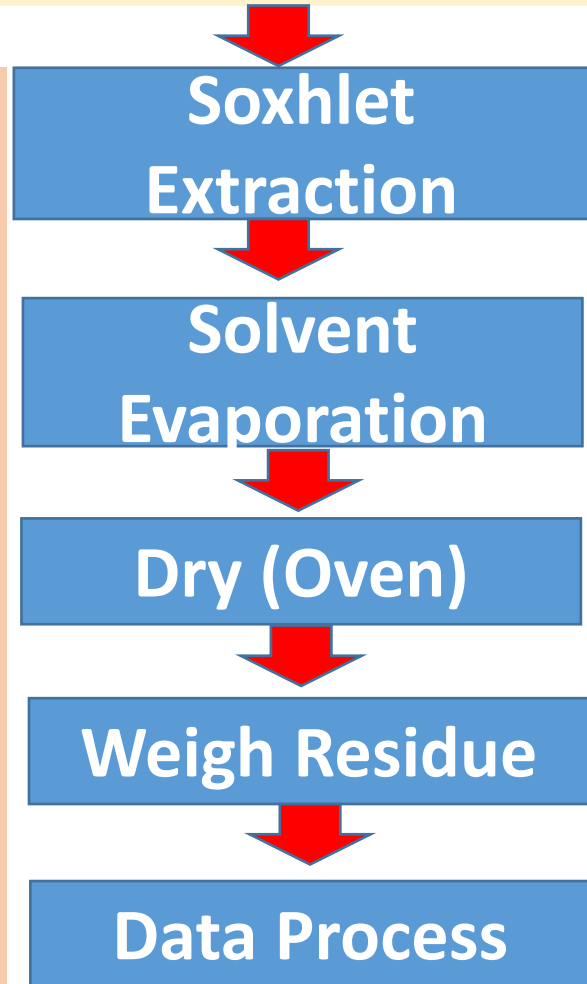
Hydrolysis



Extraction



8 HOURS



8 HOURS

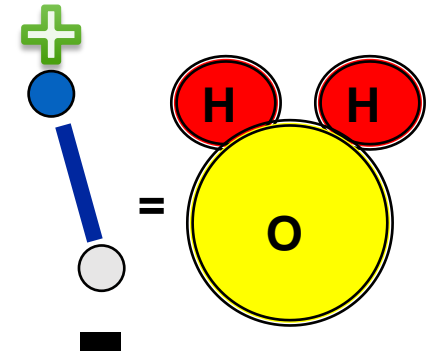
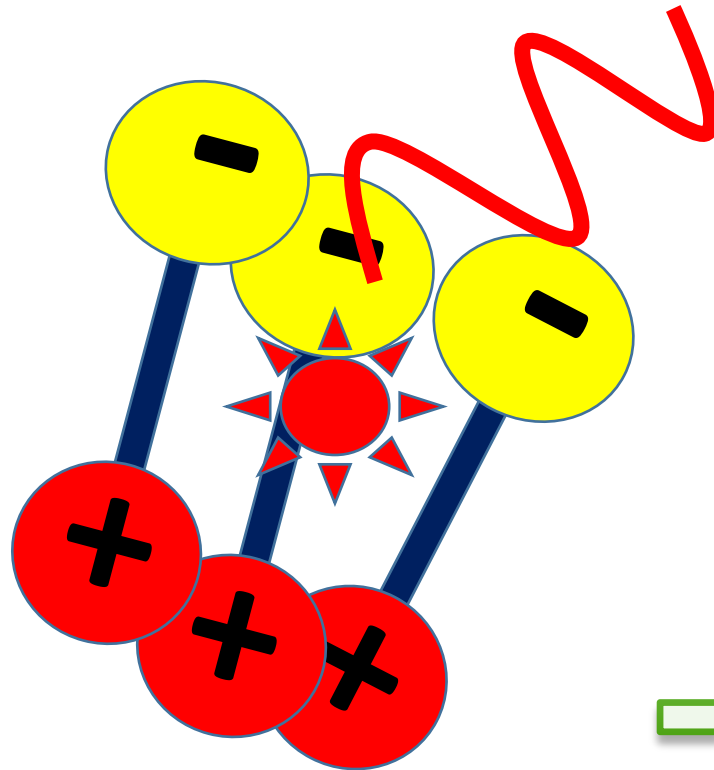
The Problem

- **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

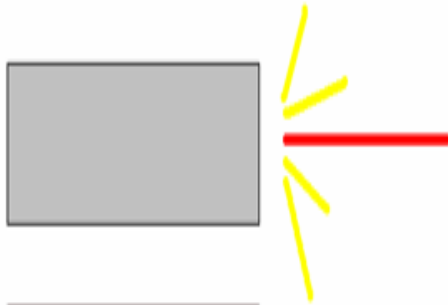
Can we use MW M. A.E for TOTAL FAT Extraction ?

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

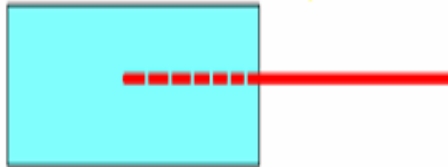
$$k = Ae^{-\left(\frac{Ea}{RT}\right)}$$


Microwaves and Heating

- The material can be :



- Reflective (metals)



- Absorbent (Water)



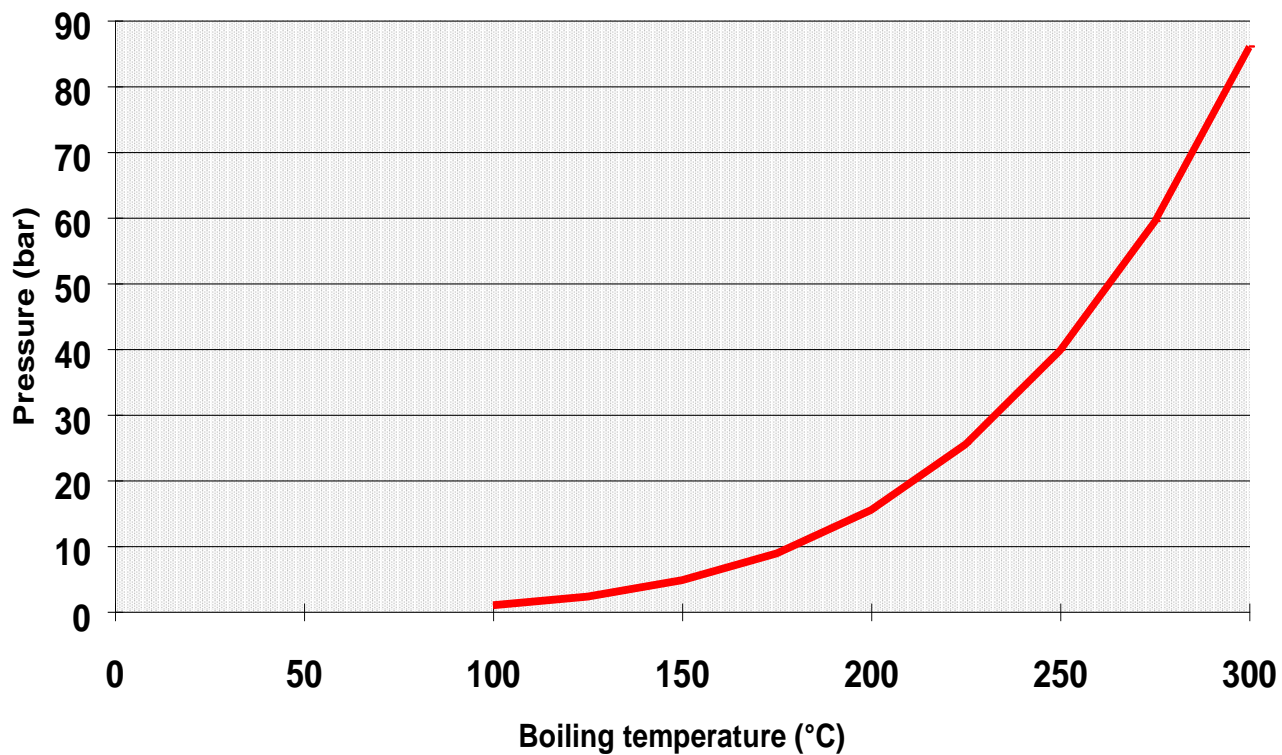
- Transparent (Hexane)

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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AEMITEQ

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