SOLVENT EXTRACTION

Sales Training Course

SOLVENT EXTRACTION

Solvent extraction is a method for the separation of mixtures by using the differences in the solubility of the components.

The sample is immersed in a solvent. Later on, the sample needs to be washed off with fresh solvent and the extract collected.

It is a «**solid-liquid extraction**», allowing soluble components to be removed from solids using a liquid solvent.

SOLVENT EXTRACTION

Once performed the extraction there can be 2 types of analysis:

- **Quantitative**: the total amount of the extracted matter
- **Qualitative**: which component or the quality of extracted matter

OFFICIAL METHODS

Solvent Extraction Method is **approved** by the most important **Official Worldwide Organizations and Bodies** such as:

AOAC, TAPPI, UNI, EPA, APHA, ASTM, AWWA, WEF.

Solvent Extraction analysis according to standard and official methods needs standardized conditions:

- Size and amount of the grinded sample

- Type and amount of solvent

- Extraction times

SOLID-LIQUID EXTRACTION

SOXHLET

Uses cold solvent, and takes a long time; the solvent passes through the thimble. (Goldfish, similar method with lower solvent consumption than Soxhlet);

TWISSELMAN

Hot extraction with the solvent flowing through the sample in the thimble;

RANDALL

Hot extraction by immersing the thimble into the boiling solvent to optimize speed and solvent consumption. Also known as «Hot Solvent Extraction».
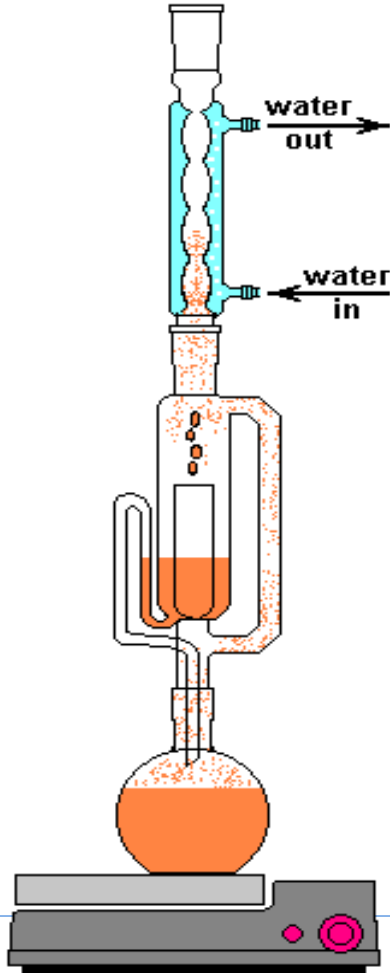
VELP MADE A CHOICE: RANDALL TECHNIQUE!

SOXHLET

The traditional technique all over the world, takes name from the German chemist **Franz Soxhlet.**
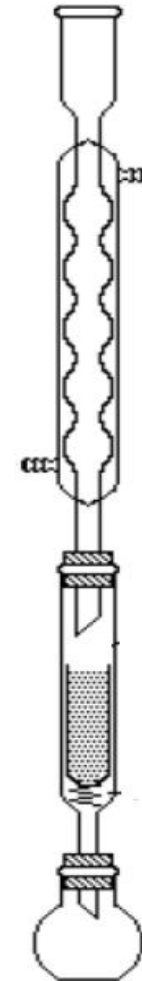
Sample **immersed** in **cold solvent.**

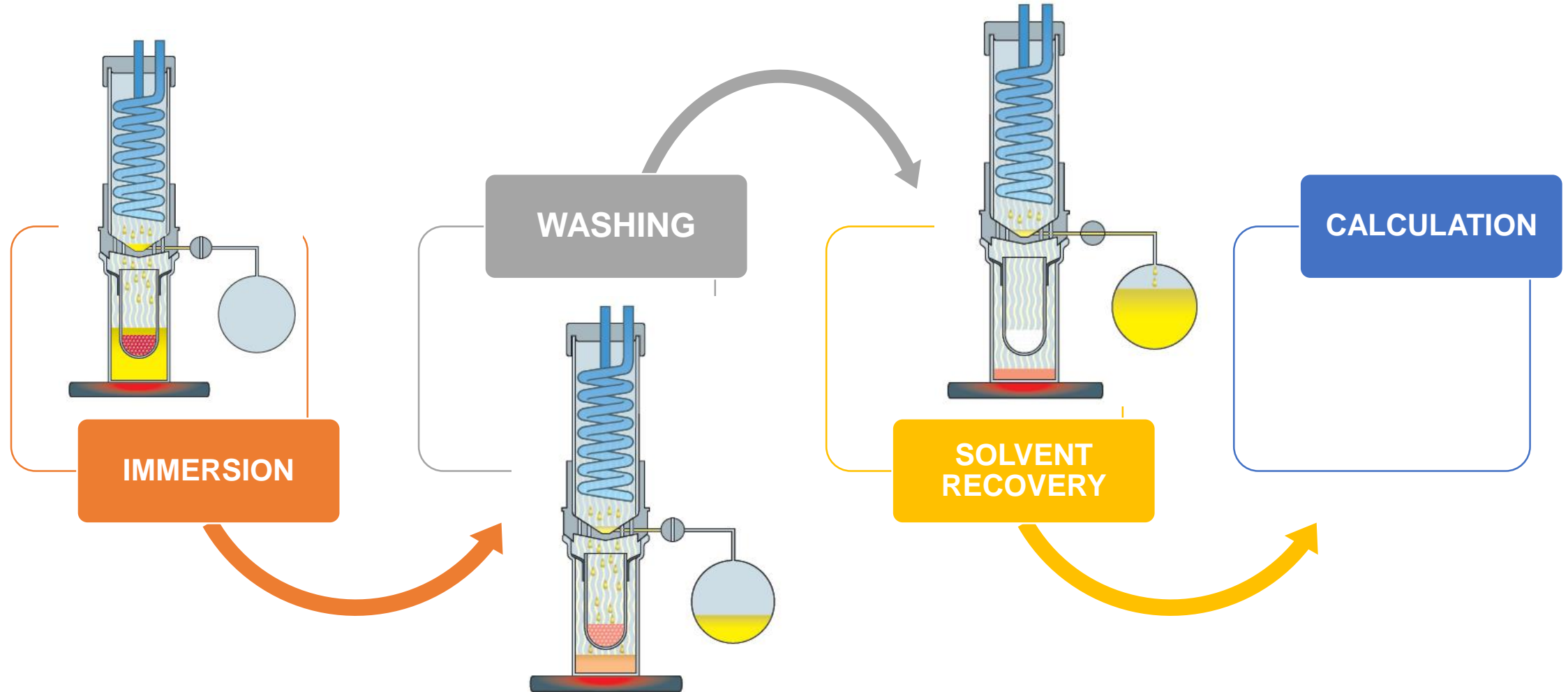A complete extraction requires **lot of hours**.

TWISSELMAN

The sample is in contact with **cold condensed solvent.**

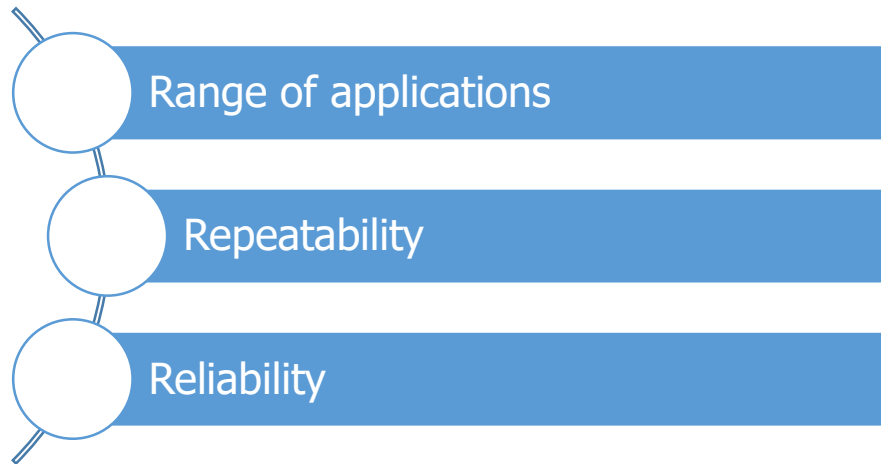A complete extraction requires **lot of hours**.
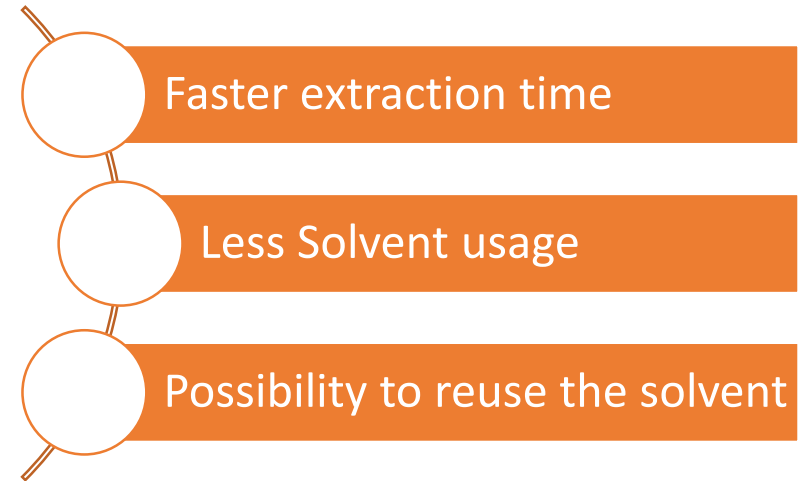
RANDALL TECHINIQUE

RANDALL vs SOXHLET

What in common:

- Range of applications
- Repeatability
- Reliability

What better:

- Faster extraction time
- Less Solvent usage
- Possibility to reuse the solvent

VELP
SCIENTIFICA®

HOT SOLVENT EXTRACTION

Solvent extraction can be performed for a wide range of applications:

FAT DETERMINATION

For both «**crude**» and «**total**» fat. Most of the food products contain fat.

EXTRACTABLE MATTER DETERMINATION

For any other sample analysis (Oil, paraffin, resin etc.…)

SAMPLE PREPARATION

When solvent extraction comes before further tests to extract pollutants and contamination elements, mainly from environmental testing.

FAT APPLICATION

- Dairy Products and Baby Food (AOAC 963.15, AOAC 933.05)

- Cereals & Bakery Products (AOAC 2003.05;)

- Meat and Derived Products (ISO1444:1996; AOAC 991.36; AOAC 960.39)

- Fish and Derived Products (AOAC 948.15)

- Chocolate and Cacao Products (AOAC 963.15)

- Oil, Oil Seeds an Nuts (AOAC 2003.06)

- Fried Potatoes (AOAC 963.15)

- Creams, Sauces and Dressings (AOAC 920.172)

- Eggs and Derived Products (AOAC 925.32)

- Feeds (AOAC 920.39, AOAC 2003.06;AOAC 920.39B; ISO 5986-1983)

… and many more

EXTRACTABLE MATTER DETERMINATION

It is also suitable for other relevant tests
in **non-food samples** like:

- Oil and Fat in Water and Waste Water (APHA-AWWA-WPCF (1985) 16th Ed. n°503C; Standard method 5520D; EPA 1664; APAT CNR IRSA 5160)

- Hydrocarbons from Soil

- Oil and Grease in Sludge (Standard method 5520E)

- Paraffin in Wood Chips

- Fat Coating of Artificial Fertilizers

- Oil from Textiles & Synthetic Fibers

- Insulating Wool (BS 2972:1989)

- Residue of Resin in Paper

- Unsaponified & Unsaponifiable Fat in Soap (ISO 1067:1974)

- Oil in Leather (ISO 4048:2008)

… and many more

SAMPLE PREPARATION

Its real applications are extendable also to **Sample Preparation** for additional tests:

- Rocks (US EPA 3541,DIN ISO 16703 EN 14039)
- Oil and Lubricants
- Pesticides (TAPPI T408)
- Phenols
- PCB - Poly Chlorinated Biphenyls
- Dioxins
- Softeners/Additives in Plastic & Rubber
- Colorant on Textile Fibers (ASTM D2257)
- Coatings of Fertilizers
- PAH - Poly Aromatic Hydrocarbons
- EOX - Extractable organic halides

… and many more

VELP SOLUTIONS

ermes enabled

HU 6

6-place unit to simplify the preliminary hydrolysis

SER SERIES

Automatic and Semi-Automatic 3 or 6-position Solvent Extractors

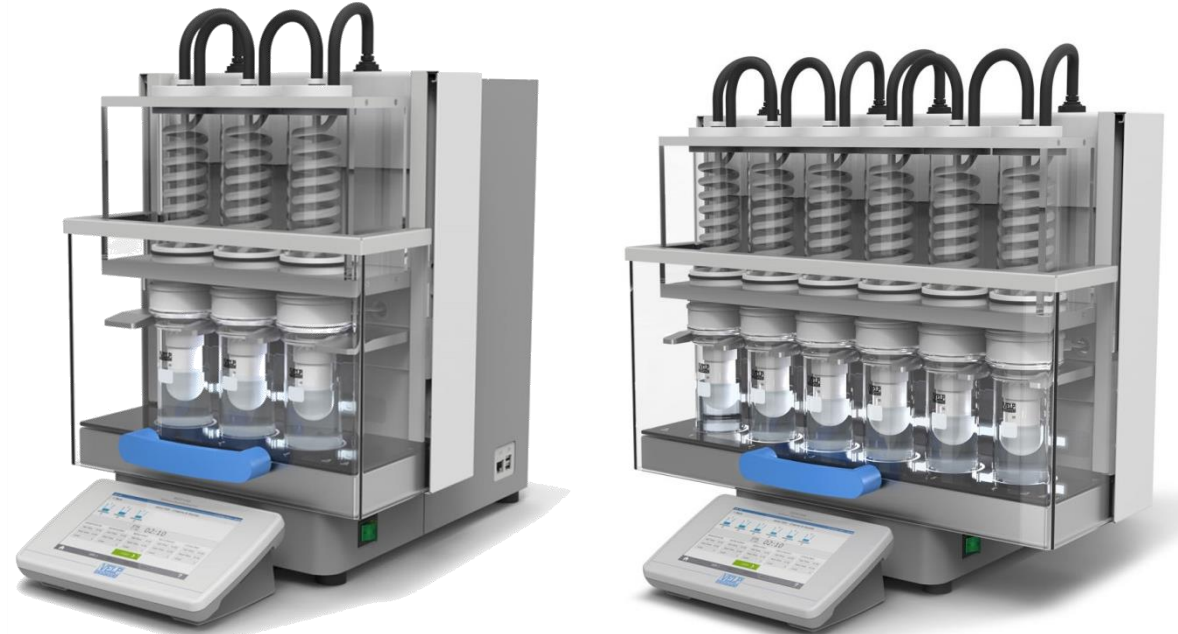VELP SCIENTIFICA

THE SER SERIES

SEMI-AUTOMATIC UNITS

FULLY-AUTOMATIC UNITS

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SER 148 3/6 Positions

Performs extractions in 3 steps

SER 158 3/6 Positions

Performs extraction in 5 steps

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SER 158 - AUTOMATIC EXTRACTION
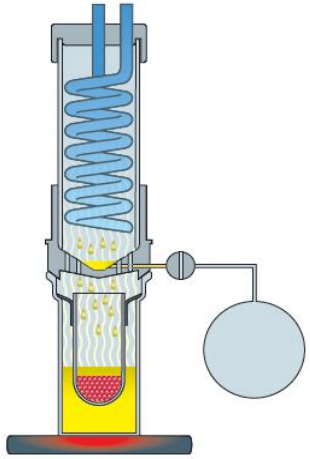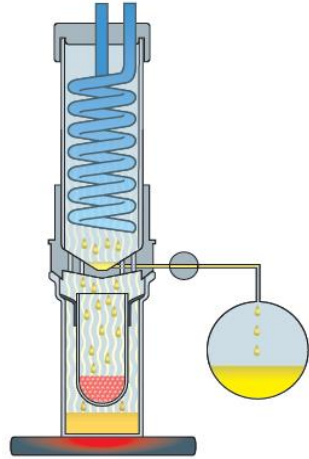
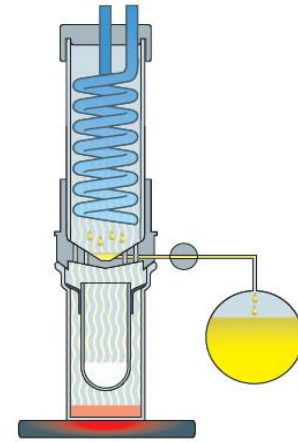| IMMERSION | → | REMOVING | → | WASHING | → | RECOVERY | → | COOLING |
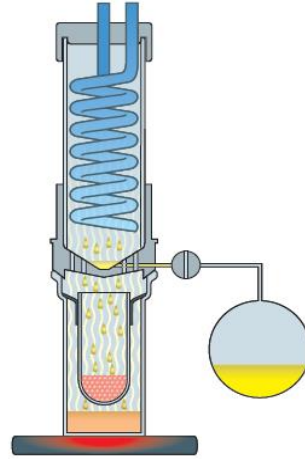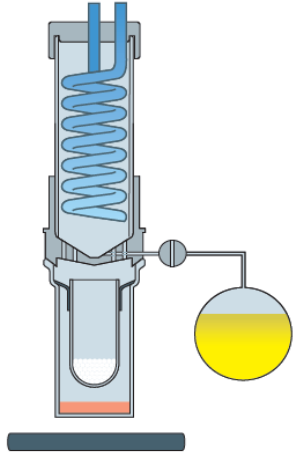
SER 158 - IMMERSION

The thimble containing **the sample** is immersed directly into the boiling solvent.

This is the most effective step of the analysis where more than 70% of the process is completed
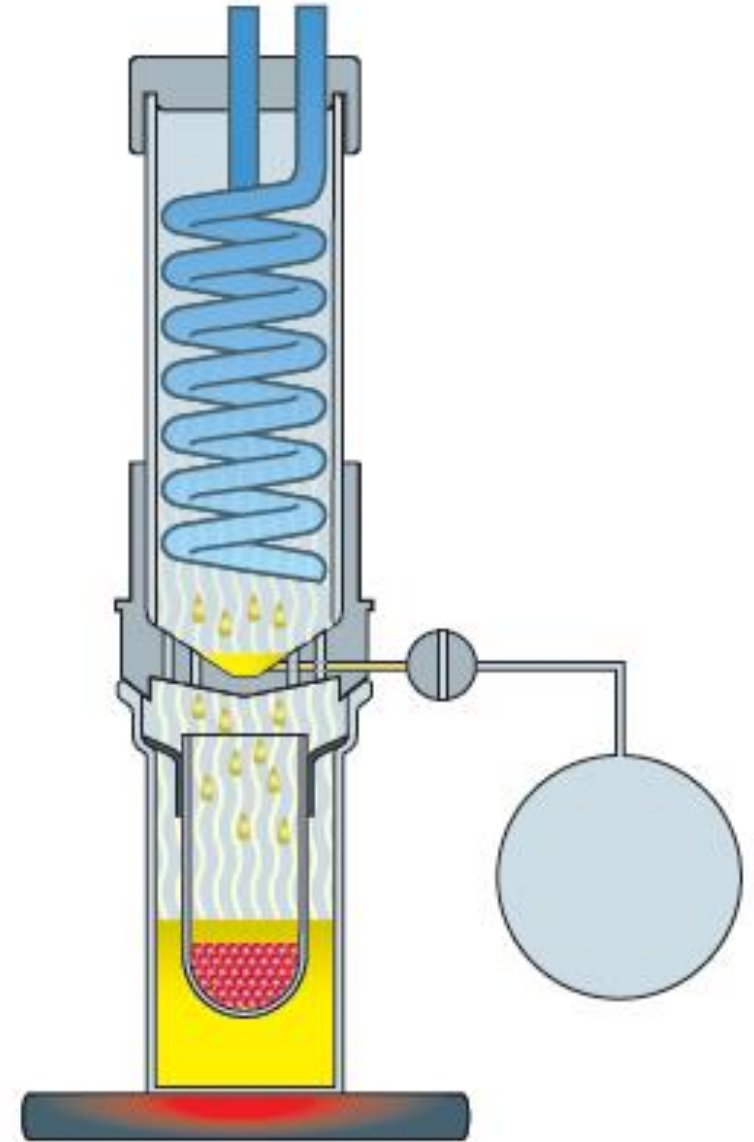
SER 158 - REMOVING

During the removing step part of the solvent is automatically removed from the extraction cup.

The level of the solvent is **automatically lowered below the extraction thimble**.

Part of the solvent is collected in the recovery tank, the rest continues to flow through the sample
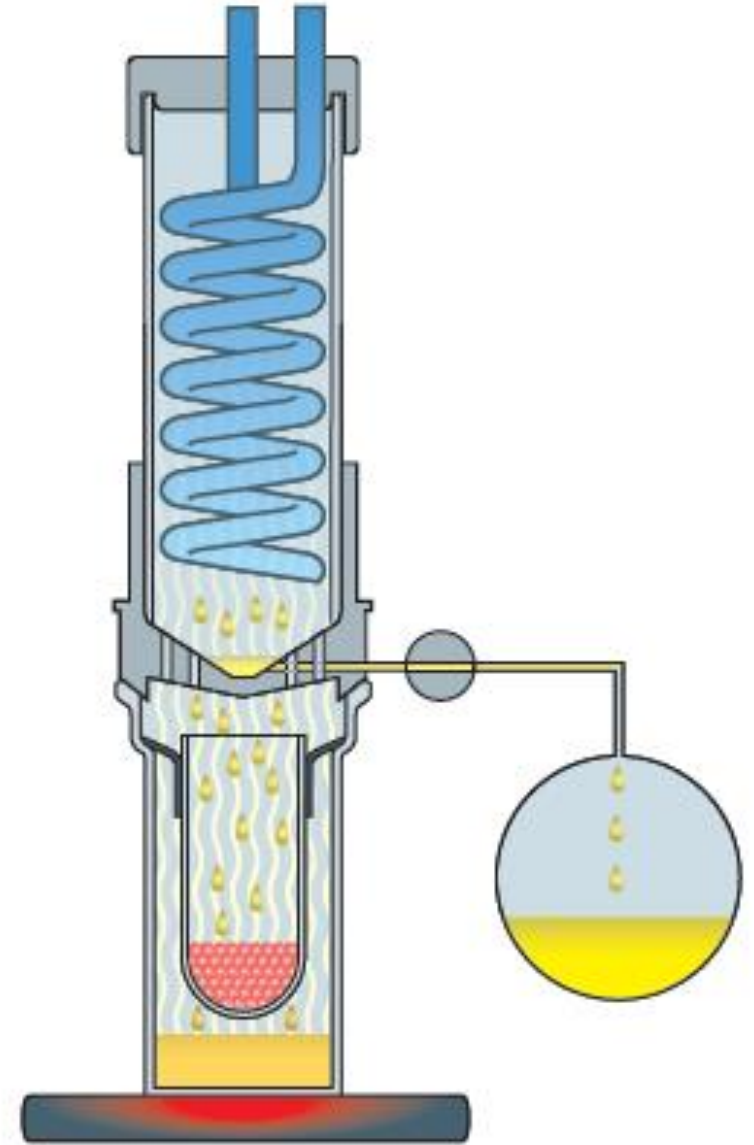
SER 158 - WASHING

The condensed solvent flows over the sample and through the thimble to complete the extraction process.

The Washing step is crucial to obtain the complete extraction.
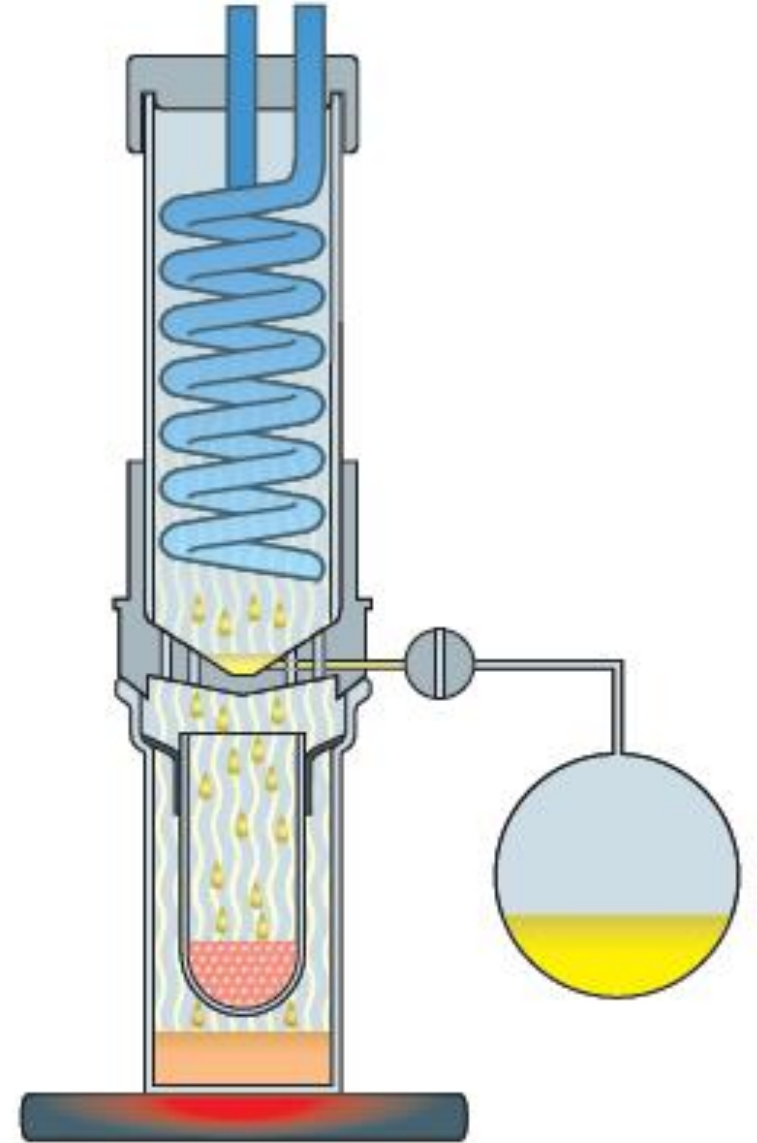
SER 158 - RECOVERY

The SER 158 continues to heat stops the condensed solvent to flow trough the thimble and drives it to the internal recovery tank.

More than 90% of the solvent is recovered in the internal recovery tank.
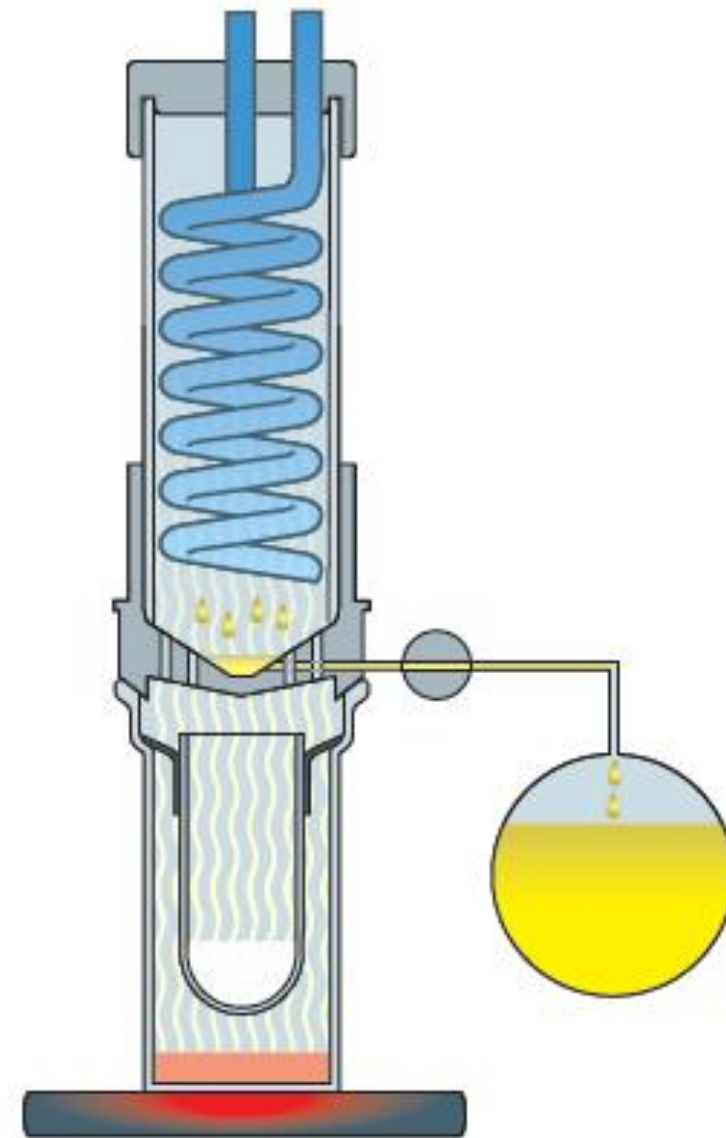
SER 158 - COOLING

The SER 158 automatically detects the ending point of the analysis and switches off the heater.

The glass cups containing the extracts **are automatically lifted up to prevent burnings**.

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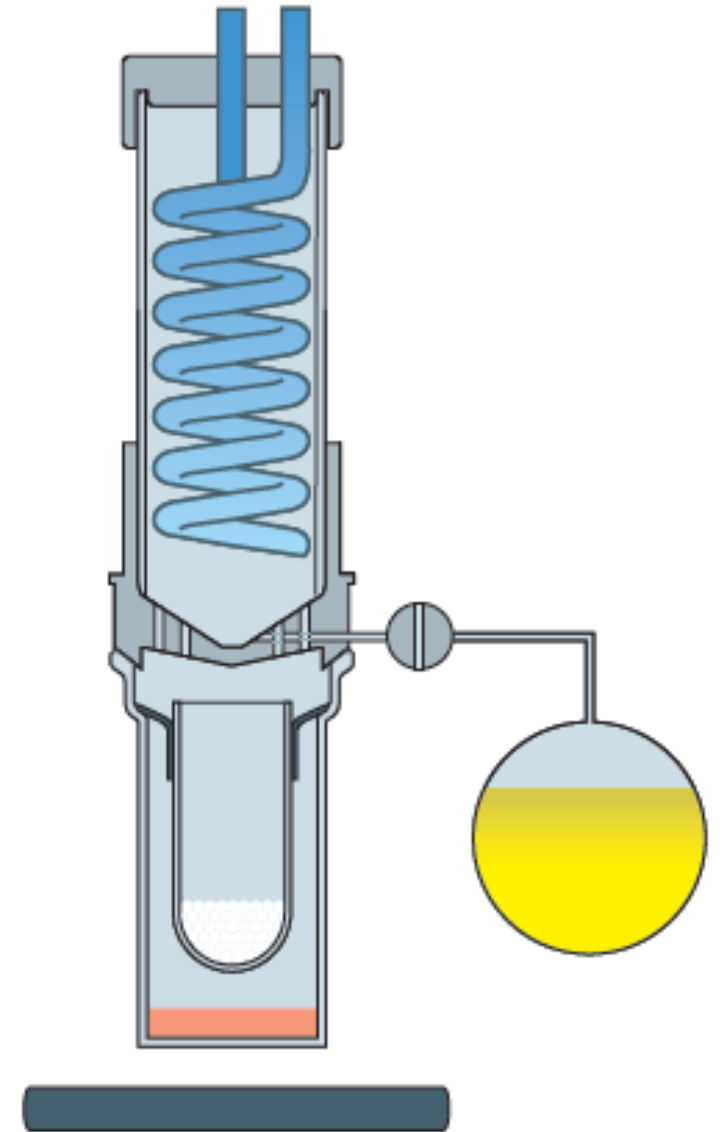
TITANIUM CONDENSER

DRASTIC REDUCTION IN THE COST PER ANALYSIS (OPEX) WITH UNMATCHED PERFORMANCE

- ① **Titanium condensers**: no need for external cooler titanium can be up to 20 times more efficient than glass

- **High recovery rate** (> 90%) thanks to titanium condensers, efficient seals and internal tank

- This brings to reduced consumption of solvent and the **lowest cooling water** need thanks to cooling water flow control
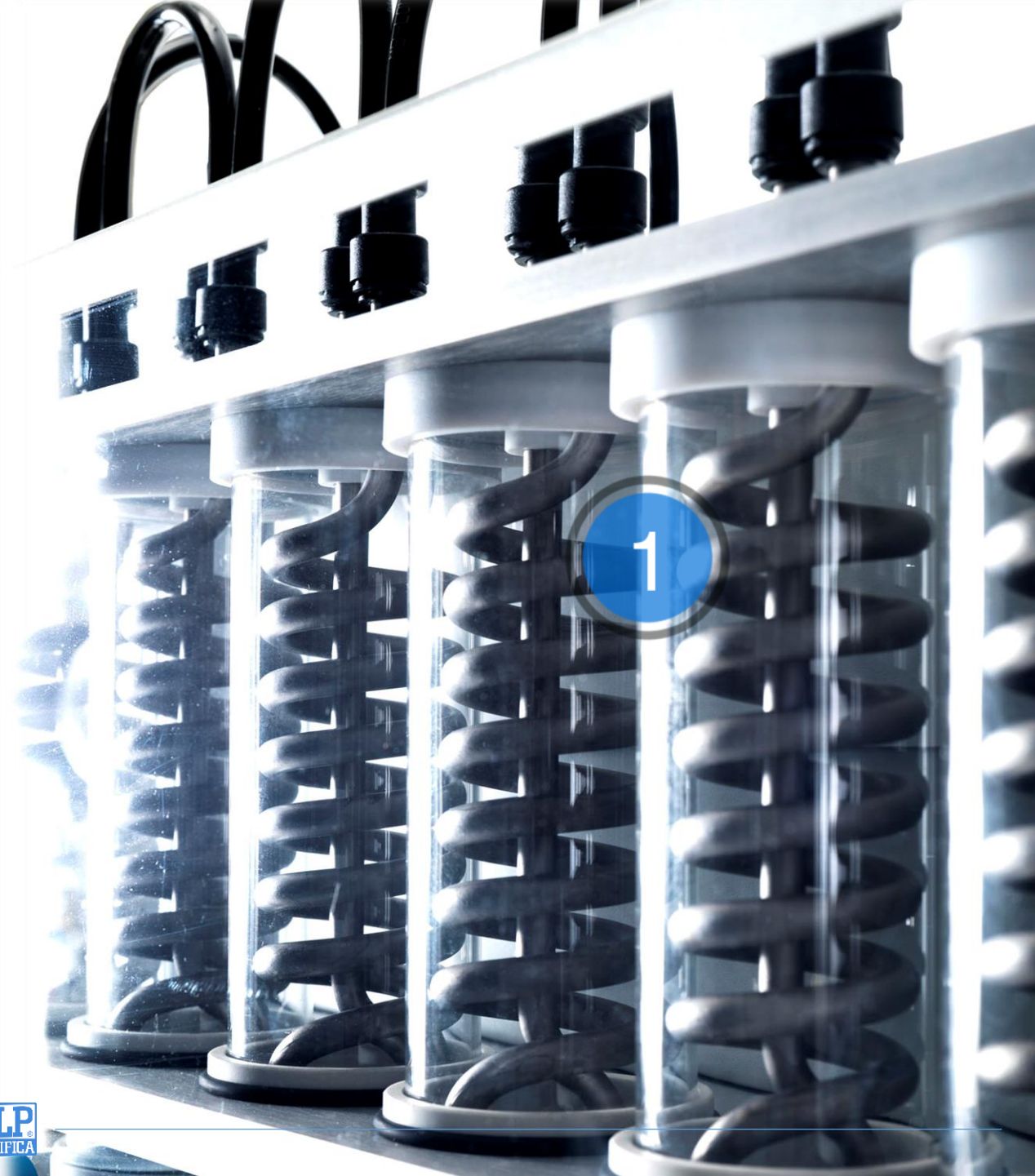
VERSATILITY

The SER 158 Series can work with all samples types thanks to its unparalleled versatility

EXTRACTION CUPS:

- Small
- STD (Glass/Metal)
- Large

THIMBLES:

- Cellulose (Small, STD and Large)
- Glass Fiber (STD)
- HU6 glass crucibles

SEALS:

- Viton
- Butyl
- Vaflon (optional): capable to work with the majority of solvents

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CUPS AND BARCODE READERS

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

The VELP glass cups (Small, STD and Large) are now provided with an **embedded barcode** that can be scanned in order to **transfer the data automatically to the ControlPad of the SER 158** by either USB or wireless connection.

The benefits of using bar code technology:

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

A00000290	A00000302	A00000303	A00000361
Extraction cup STD Ø 56x120mm 3pcs/box	Extraction cup L Ø 65x120mm 3pcs/box	Extraction cup S Ø 48x120mm 3pcs/box	Extraction cup STD aluminum Ø 56x120mm 3pcs/box

A00000364

Barcode scanner with USB socke

A00000365

Wireless barcode scanner

SOLVENTS AND GASKETS

Type of solvent	Type of Gasket	Heating Level of Glass Beaker	Boiling Stone	Boiling Point (°C)	Ignition Temperature (°C)
Acetone	Butyl	4	Required	56,5	538
Ethyl Acetate	Butyl	8	Required	77	427
Ethanol	Butyl	9	Required	78,5	423
Methanol	Butyl	9	Required	64,7	470
Chloroform	Viton	4	Required	61-62	Not Combustible
Petroleum ether	Viton	3	-	30-80	Not Specified
n-Hexane	Viton	4	-	69	234
Diethyl ether	Vaflon	1	-	34,6	180-190
Toluene	Vaflon	12	Required	110,6	536
Acetone n-Hexane 1:1	Vaflon	4	Required	-	-
Acetone n-Hexane 1:3	Vaflon	4	Required	-	-

A00000298

Grey butyl seal 3pcs/box
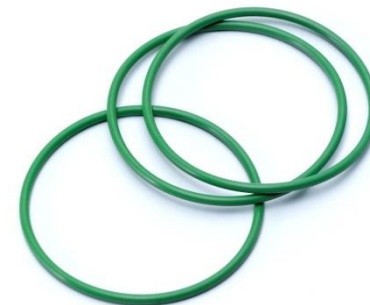
A00000297

Green viton seal 3pcs/box

A00000288

White vaflon seal 3pcs/box

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SOLVENTXPRESS

SolventXpress™ is the unique and hermetically sealed solvent dispensing system for no exposure to solvents and compatible with the majority of bottle-top dispensers.

In order to use this feature is important to order the connection depending on the diameter of the dispenser tube:

☐ A00000299
Inlet
Connection1/4NPT-
tubeØ
4,3÷4,5mm

☐ A00000300
Inlet
Connection
1/4NPT-tube
Ø4,8÷5mm

VELP
SCIENTIFICA

SAFETY FEATURES

- **SafeEnd™:** the fully automated cooling operation that switches off the heating elements and lifts up the cup preventing the extracted matter overheating

- A **safe guard** protects the operator preventing spills

- A dedicated internal tank collects the used solvent

- The LED bar communicates the process status and the active positions

- Low cooling water flow-rate **warning signal**

CONTROLPAD

- Able to control up to 4 SER 158 units independently and simultaneously

- Easy to use

- Integrated yet extractable

- Immediate display of results calculation with balance connection

- Enables One-click analysis set up, Load&GO

- Customization of analysis method

- On board storage archive secures the analysis data

- No need for other external tools

CONTROLPAD SMART SOFTWARE

- Total control of the extraction process

- Intuitive data entry and programming

- Unlimited library with all the performed analyses

- Pre-installed methods

- Excellent usability trough icons and multi-lingual support

- Data export in .xls, .txt, .csv in accordance to LIMS

- USB port

- Direct access to the archive, in real time, also from remote (Ethernet connection)

- In accordance to GLP (Good Laboratory Practice)

SER 158 - LOAD & GO

The **SER 158** is designed to make analysis **easy** and to reduce to the **minimum the duration** of each step.

Simply prepare the sample and start the process with one-click.

The units are equipped with **high tech sensors** which permit **24/7 analysis**: the extractor automatically shuts down once the last analysis is finished without operator intervention.

The user only need to place the sample in the SER 158, set the program on the ControlPad™ and walk away: **"Load & Go"**

SER SERIES ACCESSORIES

Visit www.velp.com and find out the vast set of accessories for the SER 148 and 158 Series.

Thimbles, cups, seals and many more, for matching all customer expectations.

VELP
SCIENTIFICA

SER 158 – TEMS™ TECHNOLOGY

TIME SAVING

- Easy analysis set-up
- Fast solvent addition

ENERGY SAVING

- Independent heaters
- Limited water consumption, from 1.0 L/min

MONEY SAVING

- Reduced extraction time
- More than 90% recovery means lower OPEX

SPACE SAVING

- Extremely compact footprint saves bench space

SER 158 – VELP **Ermes**

The SER 158 units are connectable to the **VELP Ermes cloud platform.**

Monitor and **manage** multiple **instruments** and **consumables** 24/7 from pc, smartphone and tablet with VELP Ermes.

Real time visualization of the analysis and of the instruments' working conditions.

Notifications via email and platform to be informed about your analysis while performing other tasks

Create and Share reports

Software Updates (OTA) and **Enhanced Service Support**
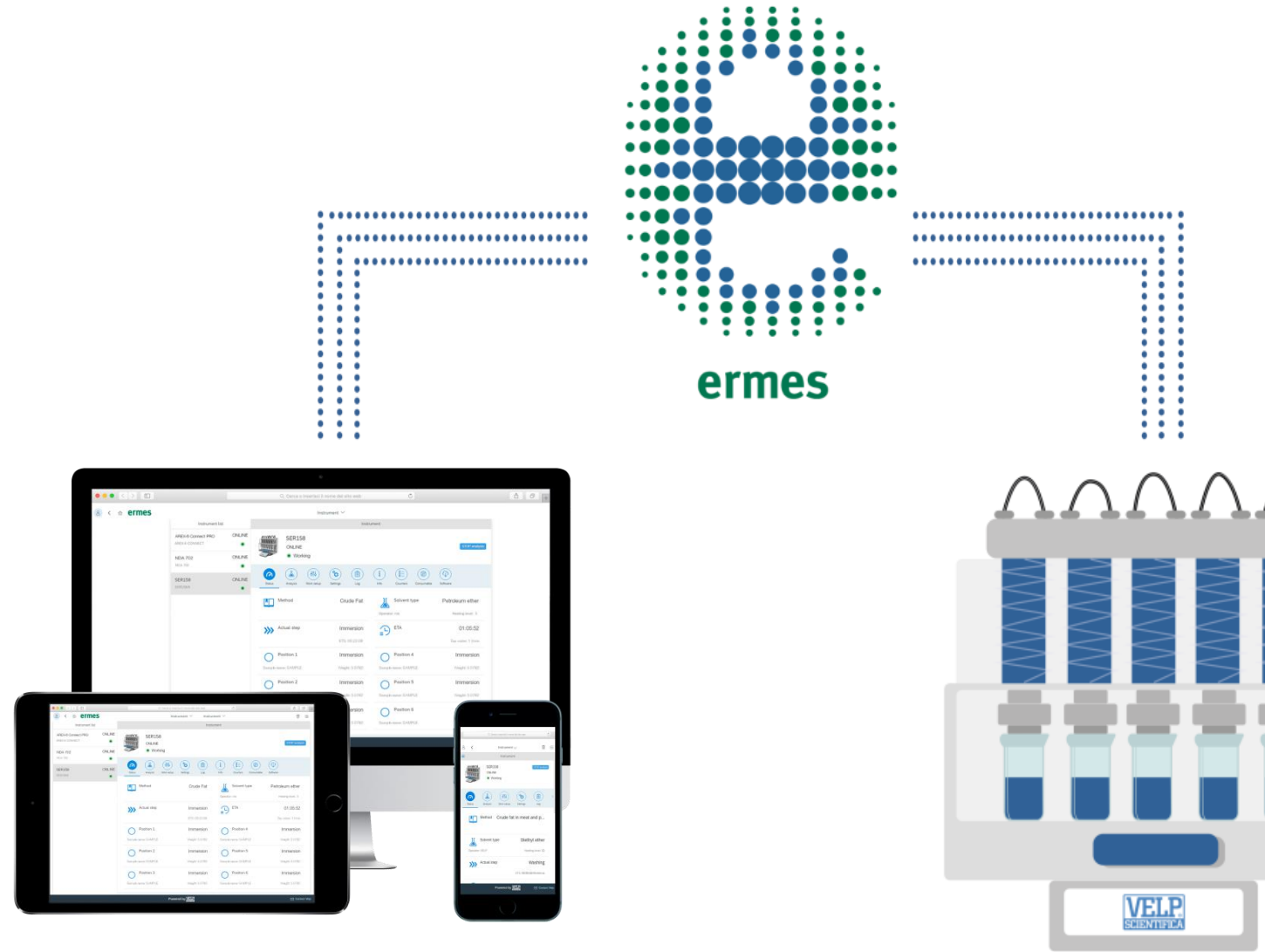
ERMES PROMOTION

1 YEAR SUBSCRIPTION ON

VELP ERMES FREE OF CHARGE

REGISTER YOUR PRODUCT ON

VELP ERMES PLATFORM

BEFORE **30/12/2019**
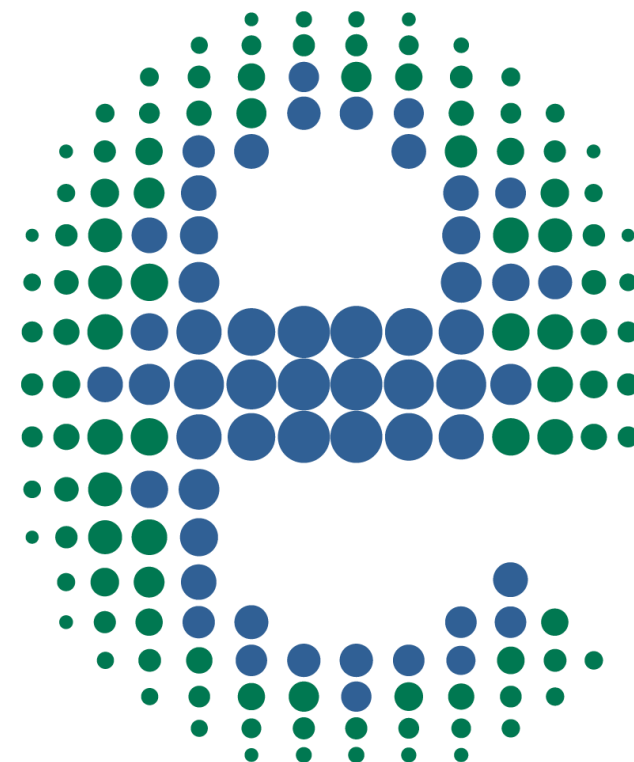
ermes
Elevating laboratories to the cloud

SER 148 EXTRACTION STEPS

SAMPLE PREPARATION

IMMERSION

WASHING

VELP
SCIENTIFICA®
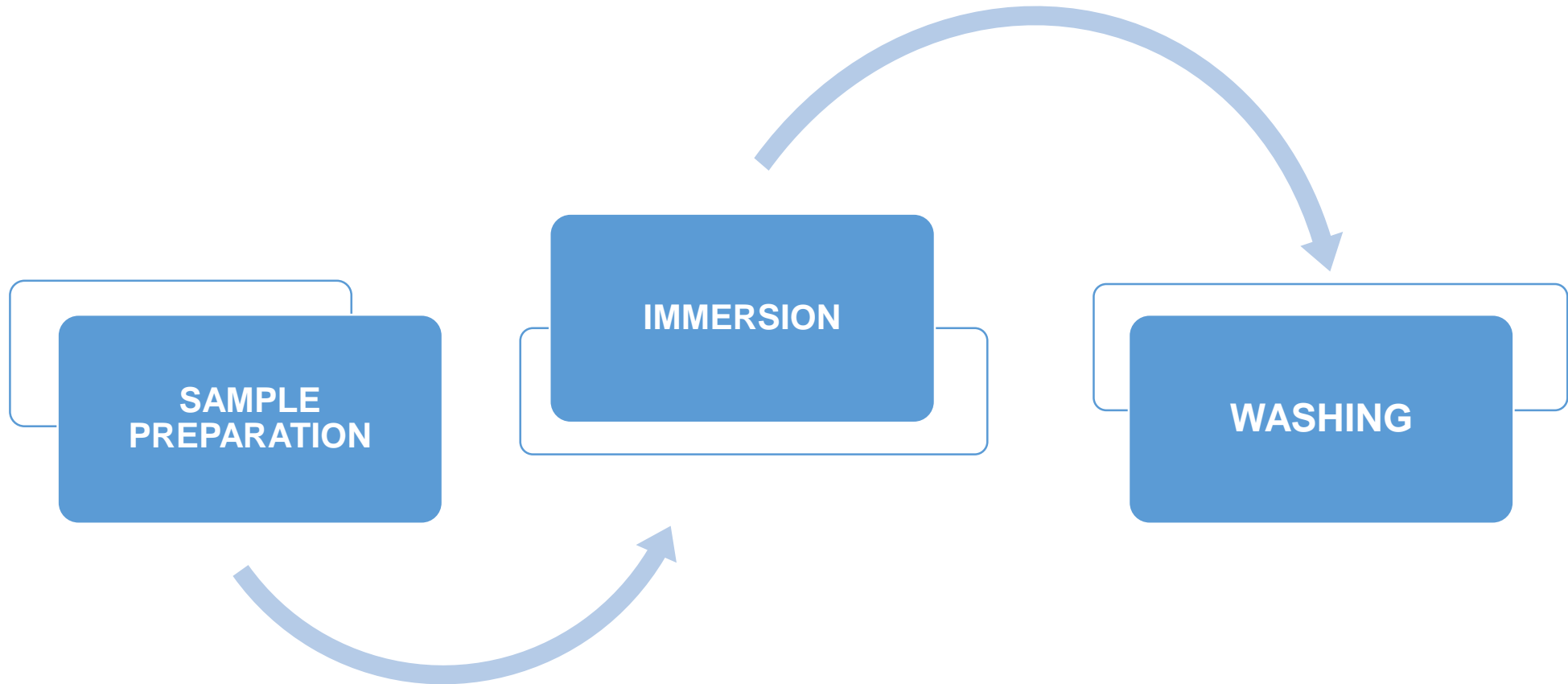
IMMERSION - SER 148

The first step of Randall Solvent Extraction consists in the **immersion** of the sample in the boiling solvent.

The Operator closes the extractor, opens cooling water in the condenser and starts the heating.

After 15-60 minutes, the operator extracts the thimble from the solvent and start the Washing step.

THIMBLE WITH SAMPLE

HOT SOLVENT

VELP SCIENTIFICA
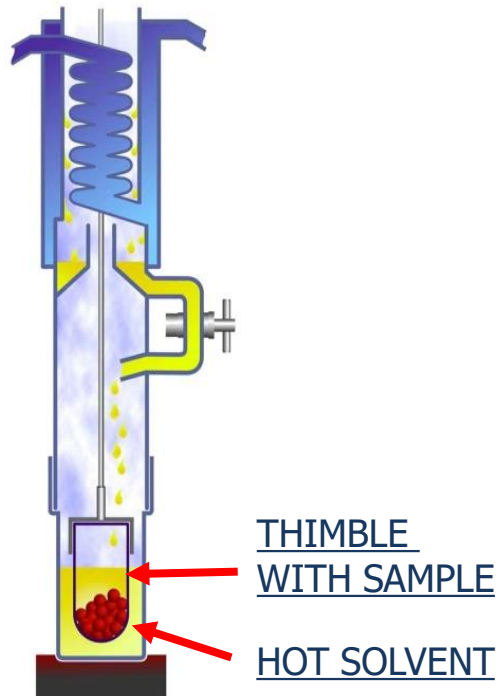
WASHING - SER 148

Note: During the Washing Step, if the used solvent has heavy vapors, the air pump can be used.

THIMBLE
WITH SAMPLE

HOT SOLVENT
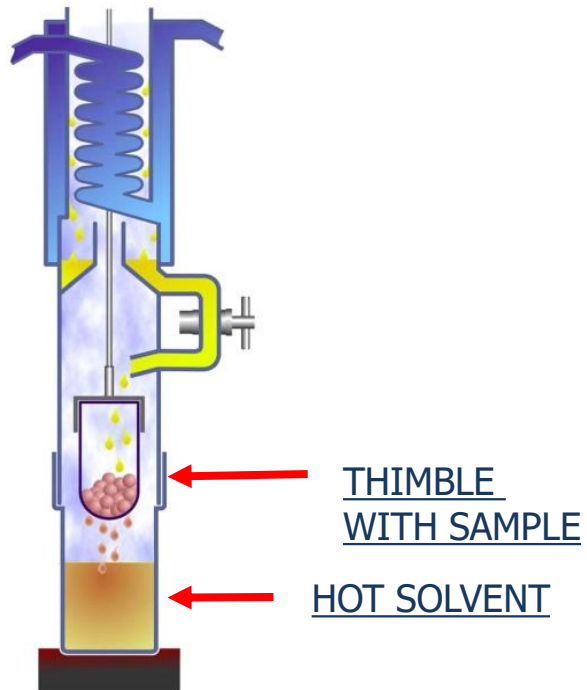
SOLVENT RECOVERY - SER 148

After **30-60 min** the washing phase ends.

The operator closes the stopcock under the condenser and evaporate the solvent from the extract collecting vessels.

To end the process the Operator desiccates in an oven the extracted matter for **20-30 min** at a temperature sufficient to evaporate all the solvent.

RECOVERED SOLVENT

EXTRACT COLLECTING VESSEL

VELP SCIENTIFICA
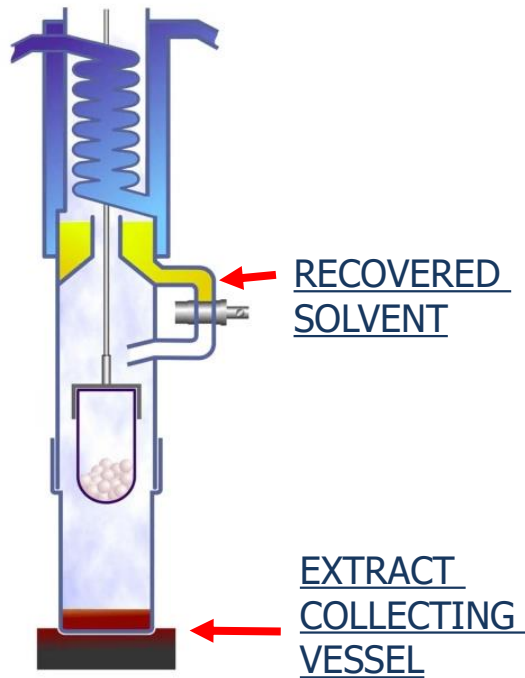
MANUAL CALCULATION

After the process has ended with the Solvent Recovery, the operator proceeds to the final calculation.

Compute the result with the following formula:

$$\% \text{ total fat} = \frac{(W-T)}{gs} \times 100$$

W = weight of the glass after extraction

T = tare of the glass

gs = grams of sample in the thimble

SER 148 SERIES - FEATURES

SAFE WORKING CONDITION

IP 55 International Protection degree.

For the operator all the risks are eliminated, in spite of the presence of an heating plate and solvents

ROBUSTNESS

Guaranteed reliability with long time in duty on the market.

PRECISION AND ACCURACY

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

SER 148 SERIES - FEATURES

FLEXIBILITY

It's possible to set and choose among **29 work programs**.

Each program is composed by 4 parameters:

- Working Temperature
- Immersion Time
- Washing Time
- Recovery Time

A **warning acoustic signal** at the end of each phase.

EASY INTERFACE

Two displays showing the working temperature and the settable parameters.

Printer connection on the front of the instrument in order to print the data.

SER 148 ACCESSORIES

TYPE OF SOLVENTS	Type of Seal	Heating Level of Glass Beaker	Boiling Point (°C)	Ignition Temperature (°C)
Acetone	Butyl A00000308	160	56.5	538
Ethanol	Butyl A00000308	210	78.5	423
Ethyl acetate	Butyl A00000308	190	77	427
Methanol	Butyl A00000308	210	64.7	470
Diethyl ether	Butyl* A00000308	110	34.6	180-190
Chloroform	Viton A00000307	160	61-62	Not combustible
n-Hexane	Viton A00000307	130	69	234
Petroleum ether	Viton A00000307	110	30-80	Not specified
Toluene	Viton A00000307	210	110.6	536
Trichloroethylene	Viton A00000307	200	86.7	Not combustible

* For longer life of seals, Vaflon seals (code A00000061) can be used.

A00000308
Butyl seal 3pcs/box

A00000061
Vaflon seal 3pcs/box

A00000307
Viton seal 3pcs/box

VELP SCIENTIFICA

SAMPLE PREPARATION – HU6 HYDROLISIS

In the large majority of food and feed products the **fat** contained are **chemically bonded** with other components.

For example the lipids are bound to proteins and carbohydrates. A direct extraction with solvents is not efficient.

The determination of the TOTAL FAT content of these samples, requires a preliminary hydrolysis using hydrochloric acid or ammonia, followed by filtration and washing, in order to free the fat molecules ready for extraction.

This procedure is required in a number of AOAC Official Methods to determine the fat content of food samples such as meat, cheese, seafood, chocolate, cereal flours, etc.

SAMPLE PREPARATION – HU6 HYDROLISIS

ACID HYDROLYSIS
Meat, seafood, cocoa, cereal meals, eggs, nuts, bread derived products, pasta, seeds and related products. Animal feed

BASIC HYDROLYSIS
Milk and related products like cheese, fresh cheese, mozzarella, condensed milk, sugar lactose, etc.

SAMPLE PREPARATION – HU6 HYDROLISIS

Put some soya beans into the cereal mill to obtain grinded sample.

Weigh the sample and place it into the test tube along with reagent (acid or basic solution).

Soya Beans

Cereal Mill

Grinded Sample
(**1 mm Particle Size**)

Test tubes

SAMPLE PREPARATION – HU6 HYDROLISIS

The Hydrolysis Unit HU6, is the **ideal solution for the acid and basic hydrolysis** of food and feed samples prior to solvent extraction

The samples are heated in test-tubes in an aluminum heating block offering excellent thermal homogeneity.

The tubes are lifted down in the heating block And The sample inside is heated up.

CRUCIBLE

PUMP

SUCTION MANIFOLD

TEST TUBE WITH SAMPLE

WASTE BOTTLE

VELP
SCIENTIFICA
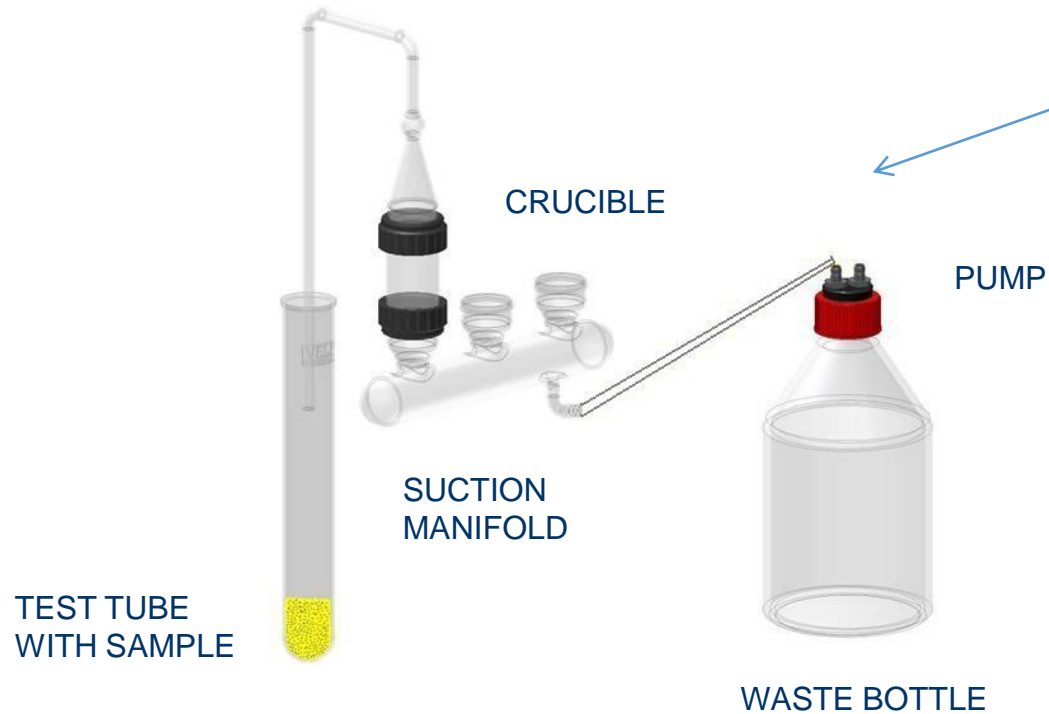
SAMPLE PREPARATION – HU6 HYDROLISIS

The hydrolyzed sample is then filtered in a glass crucible and washed with warm de-ionized water to eliminate traces of hydrochloric acid or ammonia

Thanks to the vacuum pumps the filtration time is reduced and is assured the operator safety thanks to the reduction of acid-basic fumes during boiling.

CRUCIBLE

PUMP

TEST TUBE

SUCTION MANIFOLD

WASTE BOTTLE
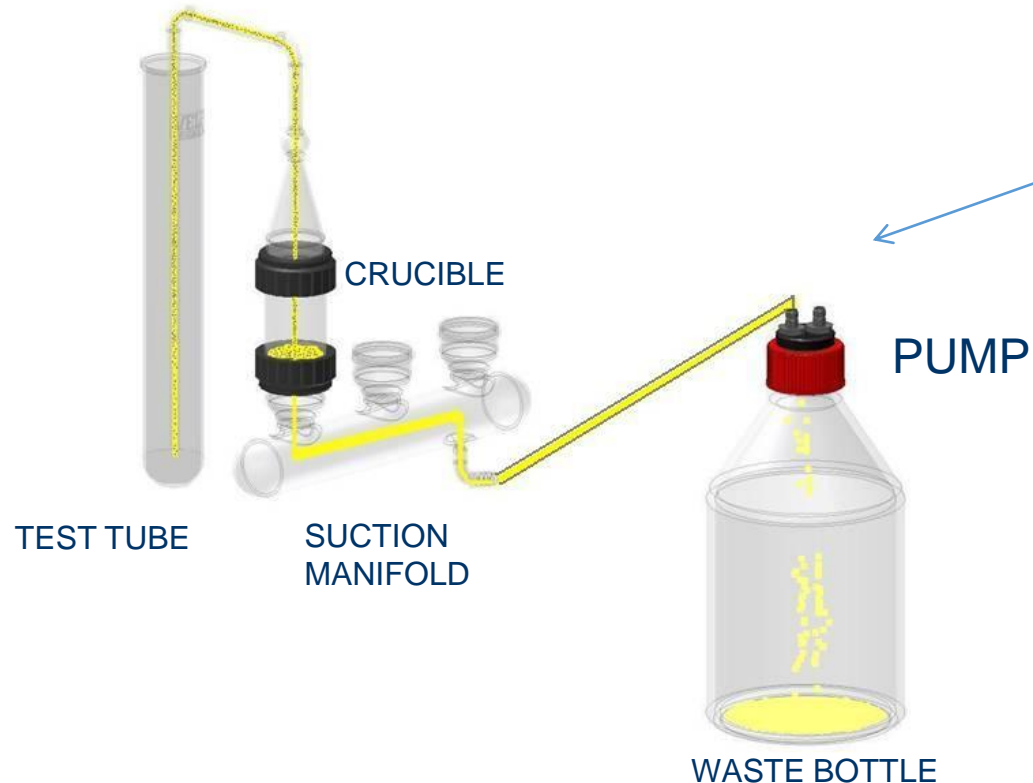
HU6 HYDROLISIS RECAP

THE COMPLETE INTEGRABLE SOLUTION FOR HYDROLYSIS AND SOLVENT EXTRACTION FOR FAT DETERMINATION

Test tubes with sample
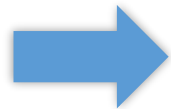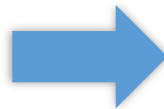
Hydrolisis unit

Hydrolyzed sample
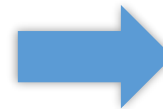
VELP Solvent Extractors

SAMPLE PREPARATION – HU6 HYDROLISIS

The glass crucible containing the hydrolyzed and washed sample is oven dried before extraction.

The crucible is then transferred directly to the SER Solvent Extraction Unit avoiding any possible sample loss and ensuring improved accuracy of results.

MAKE THE RIGHT CHOICE

Development plan: Does your customer want to grow?
Ask him:

- Number of samples

- Range of application

- Samples per single run

- …

Try to make an exercise:

- Is the money saved purchasing a SER 148 comparable to the cost of a dedicated operator?

- How much does it cost to repeat an analysis on an overheated sample?

- Which implications includes a wrong manual result calculation?

MAKE YOUR CHOICE

SER 148 SERIES

Semi Automatic: the operator can control every single step of the analysis;

Robustness: longer time in the market and guaranteed reliability;

Low Budget required

Target customer: medium throughput.

SER 158 SERIES

No need of a dedicated operator: the SER 158 is fully automatic

No result calculation error: the final results are automatically calculated and stored on the ControlPad

Time Saver: fast, precise and accurate analysis 24/7 ☐ Load&Go

Target Customer: higher throughput

SER 148 – SALES ARGUMENTS

KEY FEATURES

Semi Automatic: you can control every single step of the analysis

Robustness: longer time in the market and guaranteed reliability

Low Budget required

Target customer: medium throughput

Single Heating Element

MITIGATIONS

Requires operator supervision

Calculations made by operators: require operator accuracy.

Careful Sample handling: requires operator supervision during all extraction phases.

SER 158 – SALES ARGUMENTS

KEY FEATURES

No need of operator supervision: the SER 158 is fully automatic

No manual result calculation: the final results are automatically calculated and stored on the ControlPad

Time Saving: fast, precise and accurate analysis 24/7 ☐ Load&Go

Target Customer: high throughput

MITIGATIONS

Needs enough extra-budget

VELP
SCIENTIFICA

APPLICATION NOTES

Application notes are available on our website.

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

SALAMI

Salami is crude or cooked meat obtained after a long period of seasoning, with the addition of chopped fat and several kind of ingredients as salt, garlic, wine, herbs in a spice mix.

Reference methods: ISO 1444:1996, AOAC 991.36, AOAC 960.39

Sample preparation:

Cut the salami in slices and grind it with a blade grinder for few seconds. Dry it in an oven at 105°C in order to remove the moisture. Record the weight of the sample before and after the desiccation in order to calculate the loss of humidity.

- Fat labeled value: **34 g / 100 g**

Put 3 - 4 g of desiccated sample directly in the VELP extraction thimbles.

Dry the empty extraction cups in an drying oven (105 °C) for 1 hour. Cool them in a desiccator, weigh the Tare, then place the extraction cups containing the thimbles directly on the SER 158.

Extraction procedure:

Immersion Time: 20 min	*Recovery Time*: 10 min
Removing Time: 10 min	*Cooling Time*: 5 min
Washing Time: 20 min	Petroleum Ether 40-60 °C, 100 ml

SALAMI- RESULTS

Tare (g)	Sample on w.m. (g)	Total (g)	Extract (g)	Extract (%)
124.8150	4.4195	126.3380	1.5230	34.461
123.3130	4.0970	124.7256	1.4126	34.479
122.1910	3.9338	123.5340	1.3430	34.140
124.4620	4.0330	125.8377	1.3757	34.111
123.2250	4.0513	124.5880	1.3630	33.644
125.3890	3.8306	126.7040	1.3150	34.329
			Average ± SD%	**34.194 ± 0.311**
			RSD% **	**0.909**

Fat Labeled Value: 34g / 100 g

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

VELP SCIENTIFICA

MAYONNAISE

Mayonnaise is a white or pale yellow cream used as dressing for salads, fish or meat dishes. Commercial product has a fat content ranging between 70-80%, however an handmade mayonnaise can reach 85%.

Reference method: AOAC 920.172

Sample preparation

Mix ≈20 g of mayonnaise and put 1g directly in the VELP extraction thimbles. Add 2 g of sodium sulphate anhydrous and mix using a glass rod. Clean the glass rod using a piece of defatted cotton and place it into each thimble, over the sample.

- Fat labeled value: **72 g / 100 g**

Position the empty extraction cups in an drying oven (105 °C) for 1 hour. Cool them in a desiccator, weigh the Tare then place the extraction cups containing the thimbles directly on the SER 158.

Extraction procedure

Immersion Time: 15 min   *Recovery Time*: 10 min

Removing Time: 8 min   *Cooling Time*: 5 min

Washing Time: 15 min   Petroleum Ether 40-60 °C, 100 ml

MAYONNAISE - RESULTS

Tare (g)	Sample (g)	Total (g)	Extract (g)	Extract (%)
128.2772	0.9956	128.9970	0.7198	72.30
129.7454	1.0891	130.5305	0.7851	72.09
127.1787	1.0125	127.9150	0.7363	72.72
128.4261	1.0863	129.2118	0.7857	72.33
126.7936	0.9903	127.5146	0.7210	72.81
127.8123	1.0856	128.6033	0.7910	72.86
			Average ± SD%	**72.52 ± 0.32**
			RSD% **	**0.44**

Fat Labeled Value: 72 g / 100 g

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

TEXTILE FIBER

Reference method: ASTM D2257

Sample preparation

Cut the cotton tissue in small pieces (5-10 mm) and put 10 g of sample directly in the VELP extraction thimbles (Large).

• Expected oil content: **< 0,5%**

Position the empty extraction cups (Large) in an drying oven (105 °C) for 1 hour. Cool them in a desiccator, weigh the Tare then place the extraction cups containing the thimbles directly on the SER 158.

Extraction procedure

Immersion Time: 20 min   *Recovery Time*: 30 min

Removing Time: 40 min   *Cooling Time*: 5 min

Washing Time: 30 min

Petroleum Ether 40-60 °C, 180 ml

TEXTILE FIBER - RESULTS

Tare (g)	Sample (g)	Total (g)	Extract (g)	Extract (%)
142.2534	10.0015	142.2711	0.0177	0.177
142.6725	9.9231	142.6899	0.0174	0.175
143.7172	9.9254	143.7348	0.0176	0.177
143.7381	10.0567	143.7558	0.0177	0.176
144.7079	9.9665	144.7255	0.0176	0.177
140.9712	10.1493	140.9890	0.0178	0.175
			Average ± SD%	**0.176 ± 0.001**
			RSD% **	**0.47**

Expected oil content: < 0.5 %

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

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PCB in Sediments

Polychlorinated biphenyls PCB are ubiquitous contaminants considered persistent and <u>bio-accumulative</u> in the environment.

Reference method: UNI EN 16167:2012; EPA 1668C-2010

3 steps analysis:

1. **Solvent extraction by SER158**
2. **Purification**
3. **GC-MS analysis**

Cooperation with IRSA – CNR Water Research Institute in Italy.

PCB - Extraction

Sample:

Certified reference materials:

1. **CNS329** – "PCBs and PBDEs on Fresh Water Sediment"
2. **CRM961** – "PCB Congeners - Clay Soil"

The certified reference material were lyophilized, ready for the extraction.

Put 0.5 g of sample in the VELP extraction thimbles with a spatula.

Extraction procedure with SER158

Immersion Time: 60 min   *Recovery Time*: **0 min**

*Removing Time*: 10 min   *Cooling Time*: 4 min

Washing Time: 60 min

n-hexane:acetone (75:25 v/v), 100 ml

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PCB - Purification

- Concentrate the extracts to 1-2 ml by an evaporation system at 40 °C and at a pressure of 300 – 400 mBar.

- Purify the extract through a multilayer glass columns (ø 1.5 cm, h 15 cm) filled from the bottom with 2 g of the acidified silica, 2 g basic silica and 2 g activated Florisil®, prepared as indicated in the UNI EN 16167: 2012.

- Condition the columns with 25 mL of n-hexane, then load the extract.

- Collect the eluate and reduce this fraction to a volume of 50-100 µL with a gentle nitrogen stream at a temperature of 50°C.

- Finally transfer the extract to an autosampler vial for GC-MS analysis.
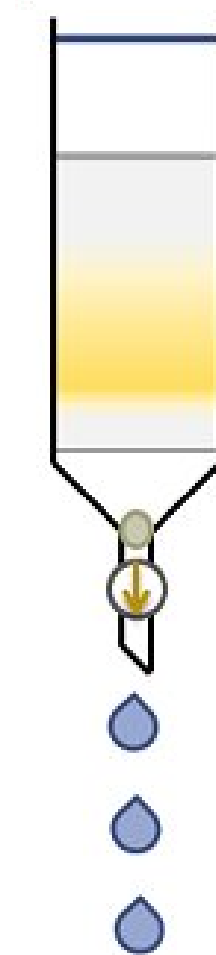
PCB – GC MS

GAS CHROMATOGRAPH

Injector	PTV
Injection type	Splitless
Time (min)	2
Volume	1 µL
Injection Temperature (°C)	60
Time (min)	0.1
Transfer Ramp (°C sec⁻¹)	8
Final Temperature (°C)	270
Cleaning Temperature (°C)	300
Column:	Supelco SLB-5ms 60m X 0.25mm X 0.25µm
Carrier gas	Elio 6.0
Flow (mL min⁻¹)	1.3 ml min⁻¹
Isotherm 1 (°C)	60
Time 1 (min)	1.5
Ramp 1 (°C min⁻¹)	30
Isotherm 2 (°C)	210
Time 2 (min)	0.1
Ramp 2 (°C min⁻¹)	10
Isotherm 3 (°C)	15

MASS SPECTROMETER

Acquisition	SRM
Temperature Source (°C)	300
Temperature Transfer Line (°C)	300
Time (min)	0.1
Transfer Ramp (°C sec⁻¹)	8
Final Temperature (°C)	270
Cleaning Temperature (°C)	300
Polarity	+
Ionization	EI, 70 eV
Collision Gas	Argon 6.0
Emission current	50 µA

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RESULTS on Sample 1

CNS329 *"PCB and PBDE on Fresh Water Sediment"*

PCB congeners	Extraction System SER 158/6		Certified values		Confidence range
	Mean (ng/g)	SD (SD, *n=4*) ±	Mean (ng/g)	SD (SD, *n=4*) ±	
PCB 52	257.2	11.3	230	29.6	195-265
PCB 180	112.9	6.3	104	34.5	68.8-138
PCB 153	148.7	4.3	133	26.1	110-155
PCB 138	233.4	14.9	226	4.99	220-231
PCB 118	192.3	13.4	175	26.1	149-201
PCB 101	414.5	28.2	390	99.6	291-489

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RESULTS on Sample 2

CRM961 "*Clay Soil*"

PCB congeners	Extraction System SER 158/6		Certified values		Confidence range
	Mean (ng/g)	SD (SD, *n=4*) ±	Mean (ng/g)	SD (SD, *n=4*) ±	
PCB 52	100.3	1.5	85.9	18.2	67.8-104
PCB 180	127.9	6.4	116.0	11.6	105-128
PCB 153	143.7	3.0	137.0	18.5	120-154
PCB 138	146.8	3.7	130.0	22.8	108-153
PCB 118	184.7	9.9	173.0	19.9	154-191
PCB 101	115.9	0.9	106.0	11.0	95.2-116
PCB 81	175.5	24.7	205	35.8	171-238
PCB 77	197.1	44.4	23	31.5	197-249
PCB 126	201.3	32.3	213	26.6	188-238
PCB 189	245.5	43.5	247	60.2	184-309
PCB 167	206.8	36.0	236	43.7	194-279
PCB 123	158.3	28.4	170	24	147-194
PCB 114	159.6	33.3	183	28.9	153-213
PCB 105	137.2	28.1	147	18.5	130-164

CONCLUSION

VELP SER Series is ideal for the:

- FAT CONTENT QUANTIFICATION
- EXTRACTION OF MATTER FROM INDUSTRIAL SAMPLES
- SAMPLE PREPARATION FOR QUALITATIVE ANALYSIS

Benefits:

- Fast: Randall technique up to **5 times faster** than Soxhlet (hot solvent vs. cold solvent)

- Accurate: in accordance with the expected values, and complying worldwide **official methods**

- Precise with a low relative standard deviation (**RSD < 1%**), that means high repeatability of the results.

- Safe: **no exposure to solvent and low solvent consumption** - limited cost per analysis

- Load and go: **full traceability** with automatic result calculation and on-board archive

- Connectable by Ermes: **Real time visualization of the analysis**

Thank You

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