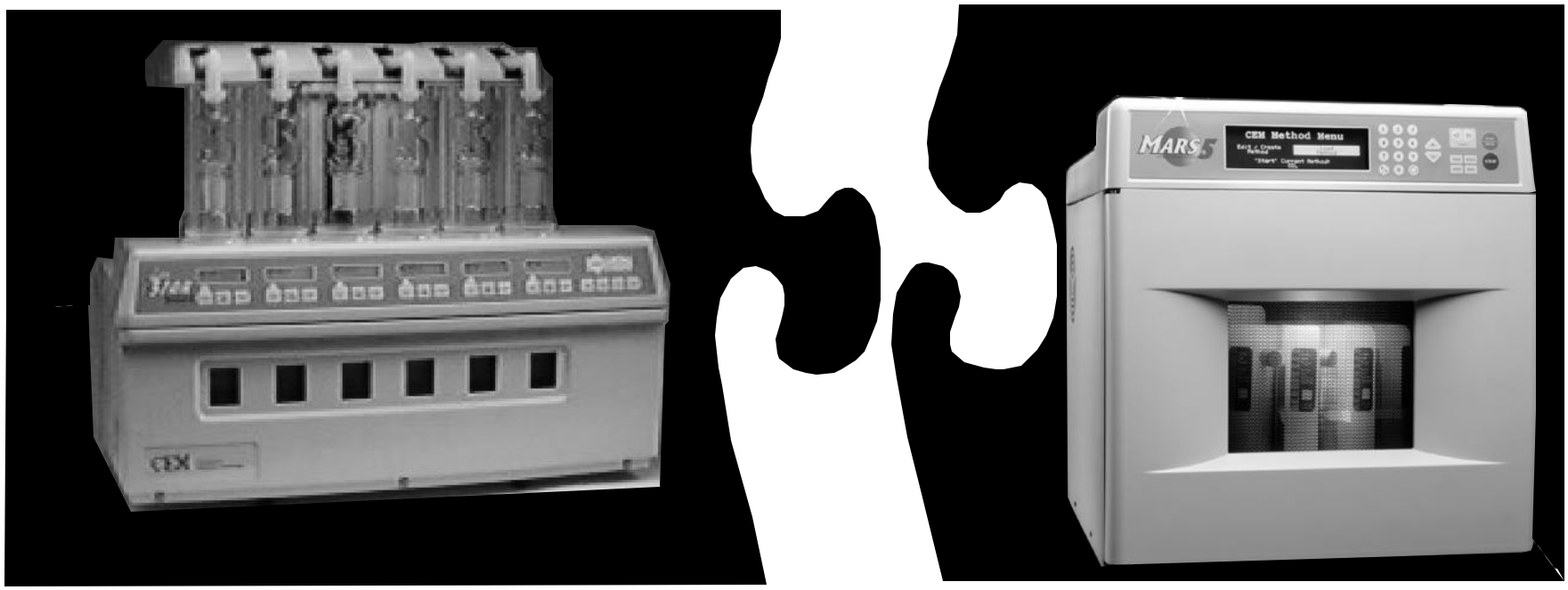
Puzzled By Sample



Preparation Problems?

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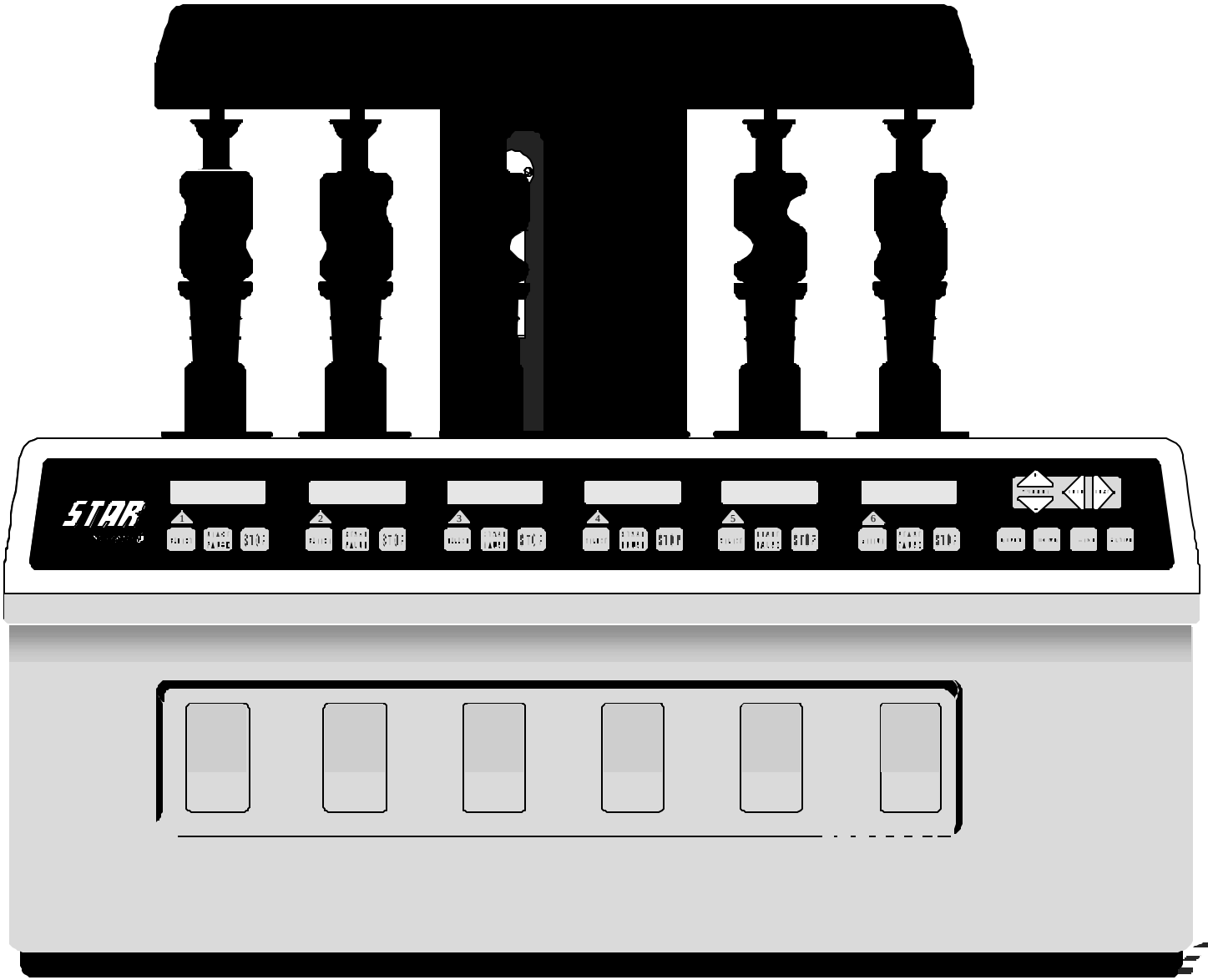
Classical Digestion Approaches

Microwave Digestion

- **Well-established technique**
  - Better Reaction Control
  - Fast, Clean, Safe, Low Blank Values
- **Conventional "Closed Cavity" & newer "Open Cavity" Systems**
  - Wide Range of Open & Closed Vessels Available
  - It's not simply an "open & shut" case!



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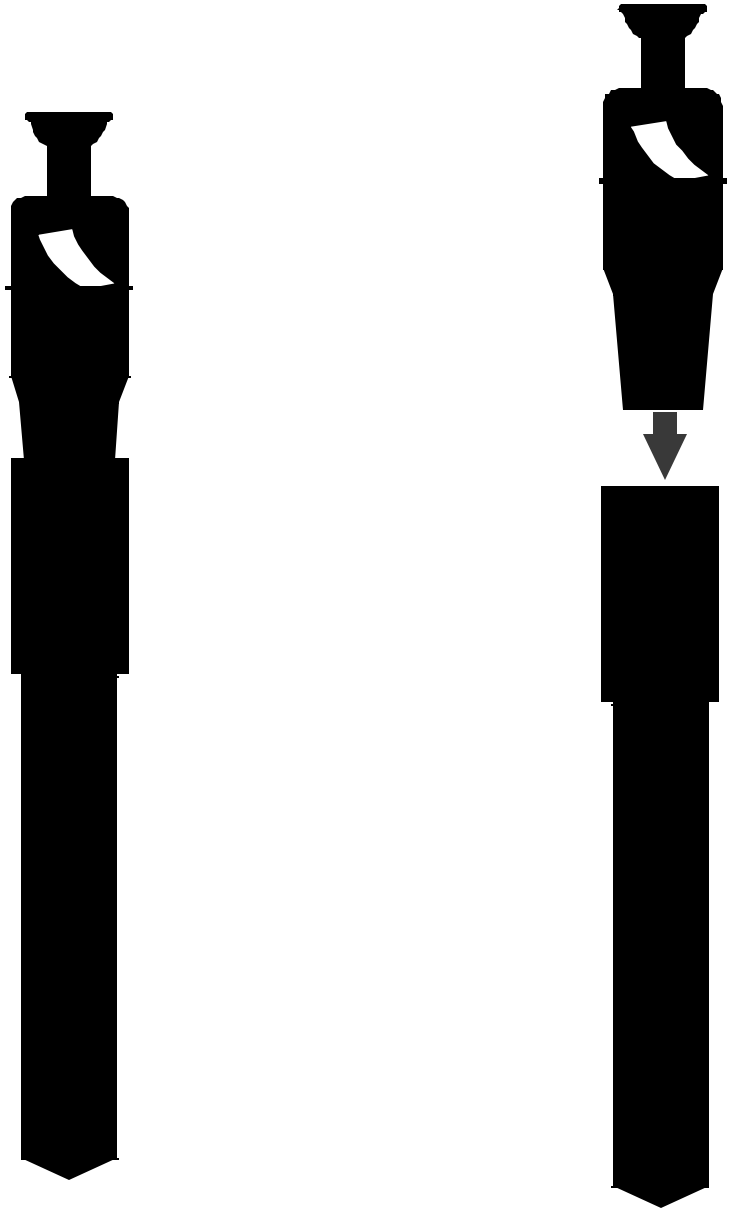
STAR System Specifications

- 2 Vessel and 6 Vessel Systems
- 430 ºC Operating Temperature
- Individual Cavity Microwave Control
- Automatic Temperature Feedback
- Acid Addition
- Vapor Containment
- Simple User Operation

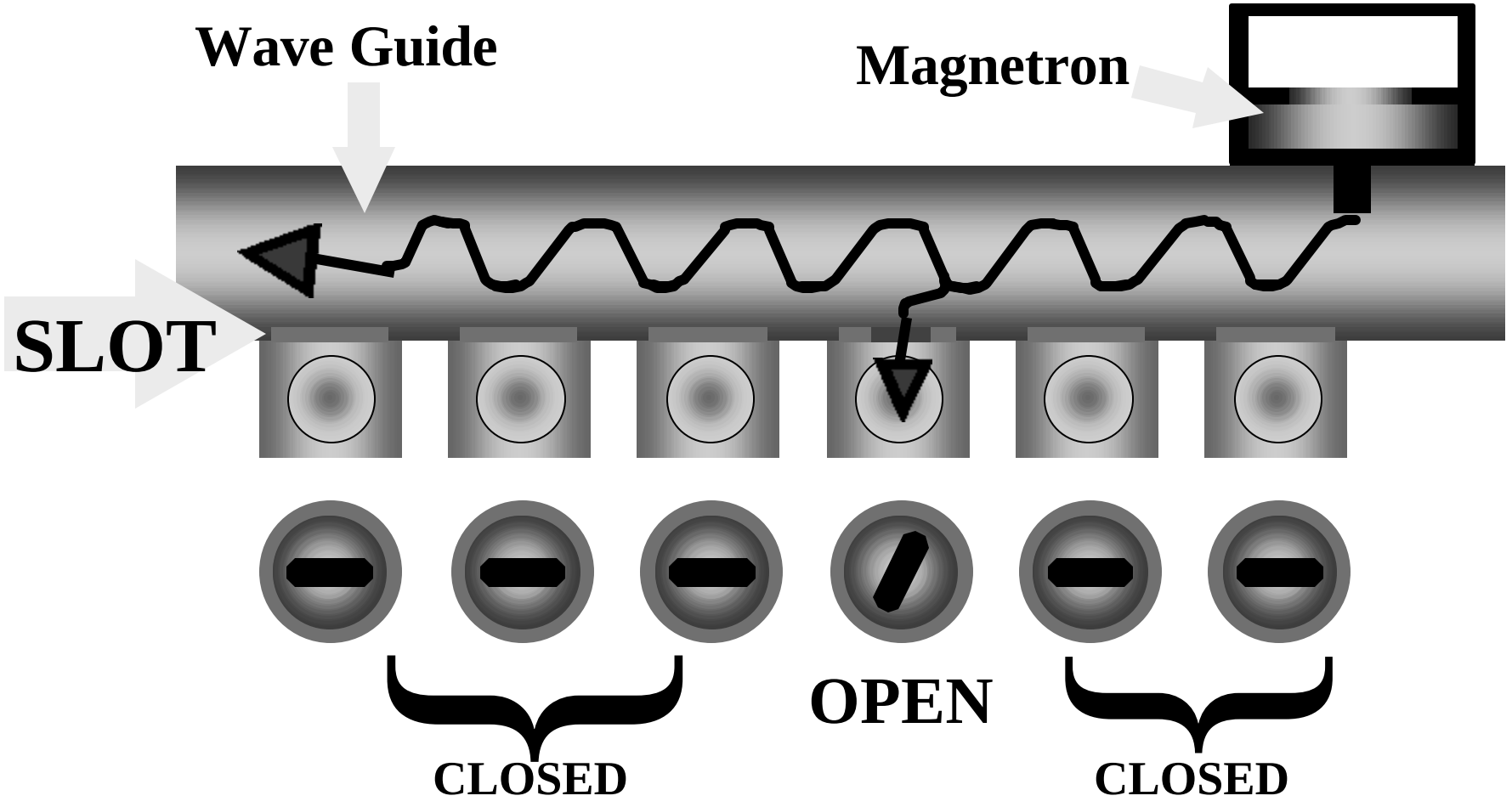
STAR Digestion Vessels

Quartz Digestion Vessel

Teflon Digestion Vessel



DISTRIBUTION OF MICROWAVE ENERGY



Slot Position Controls Passage of Microwave Energy

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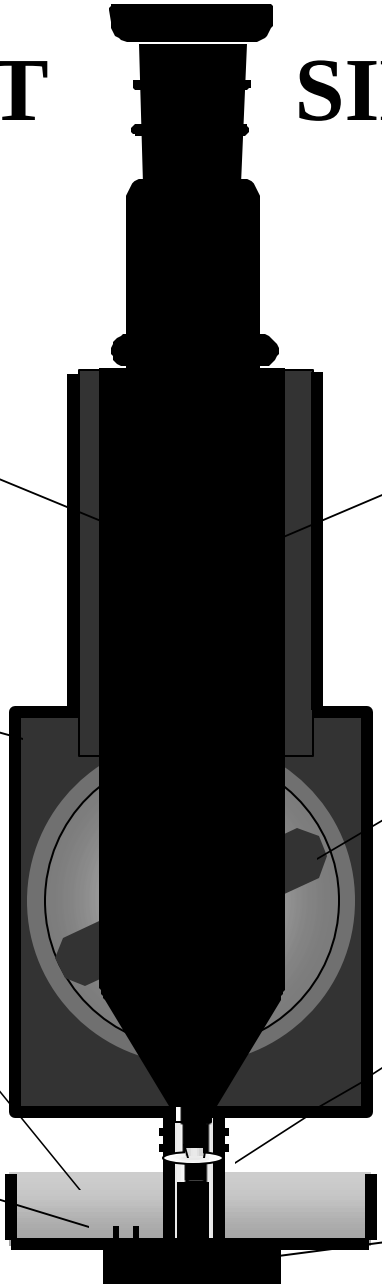
TEMPERATURE CONTROL

INDEPENDENT SIMULTANEOUS



Vessel Liner

Reaction Vessel

Microwave Cell

Microwave Slot

Protective Membrane

Spill Tray

Spill Detector

Temperature Sensor

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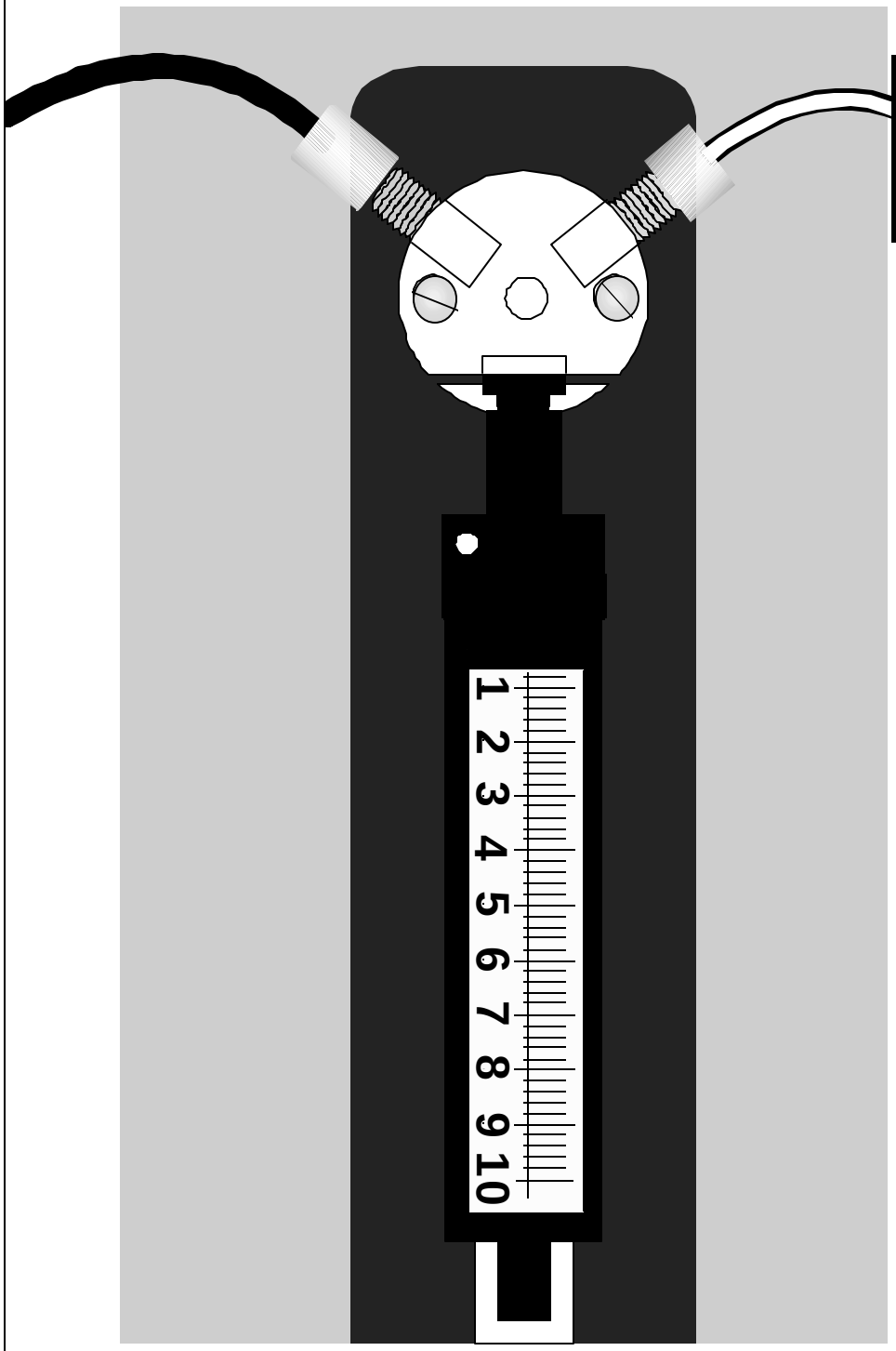


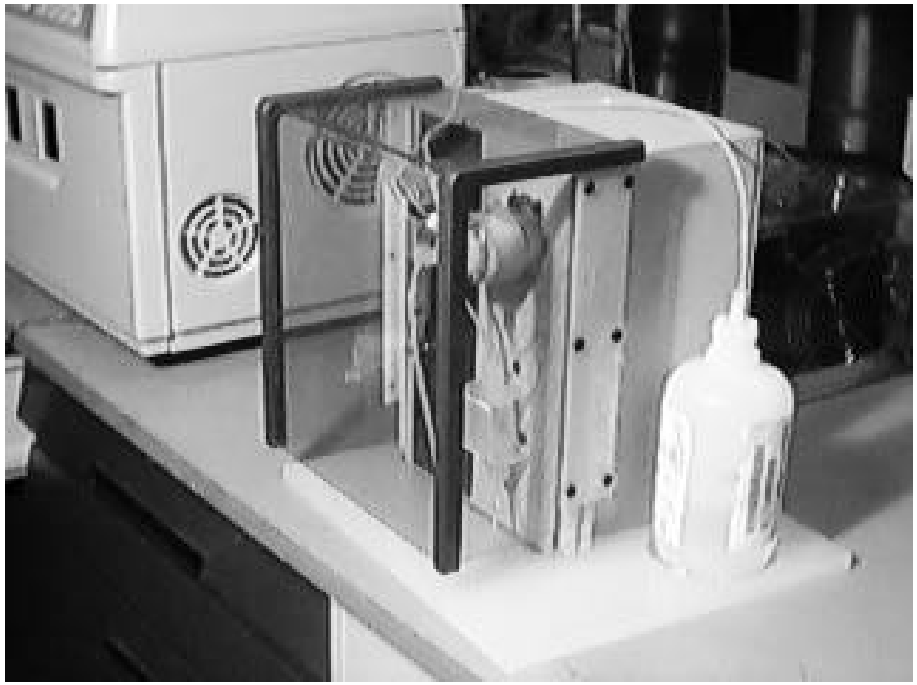
[redacted]eagent Addition Syringe

Volume
10 mL

Aliquots
0.5 mL to 50 mL

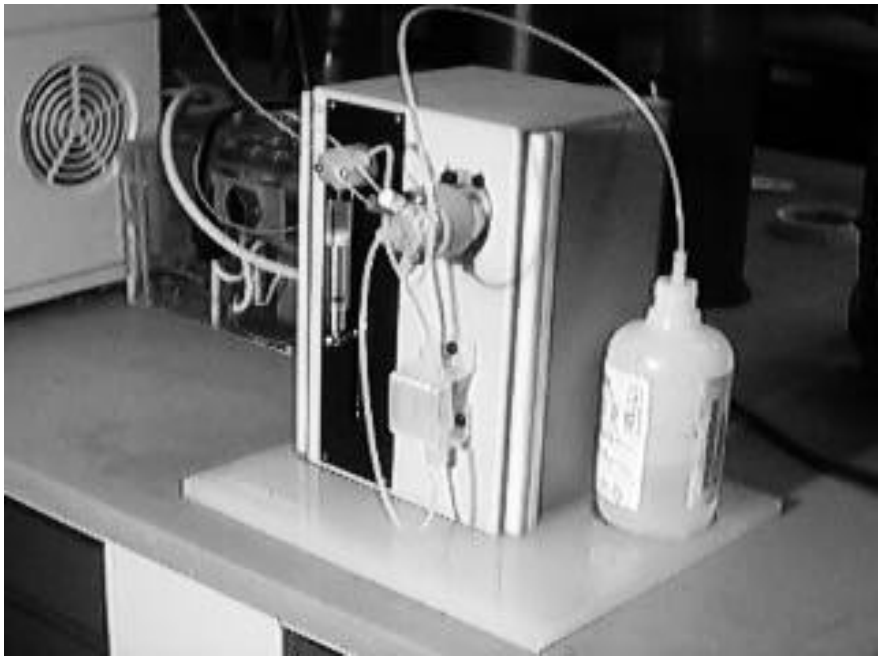
Reagent Addition

- **Up to four user defined reagents**
- **Addition of Initial Reagents**
- **Addition at the Start of a Stage**
- **Addition over Time at Parameter (TAP)**



STAR System
HF Pumping Accessory
with protective cover

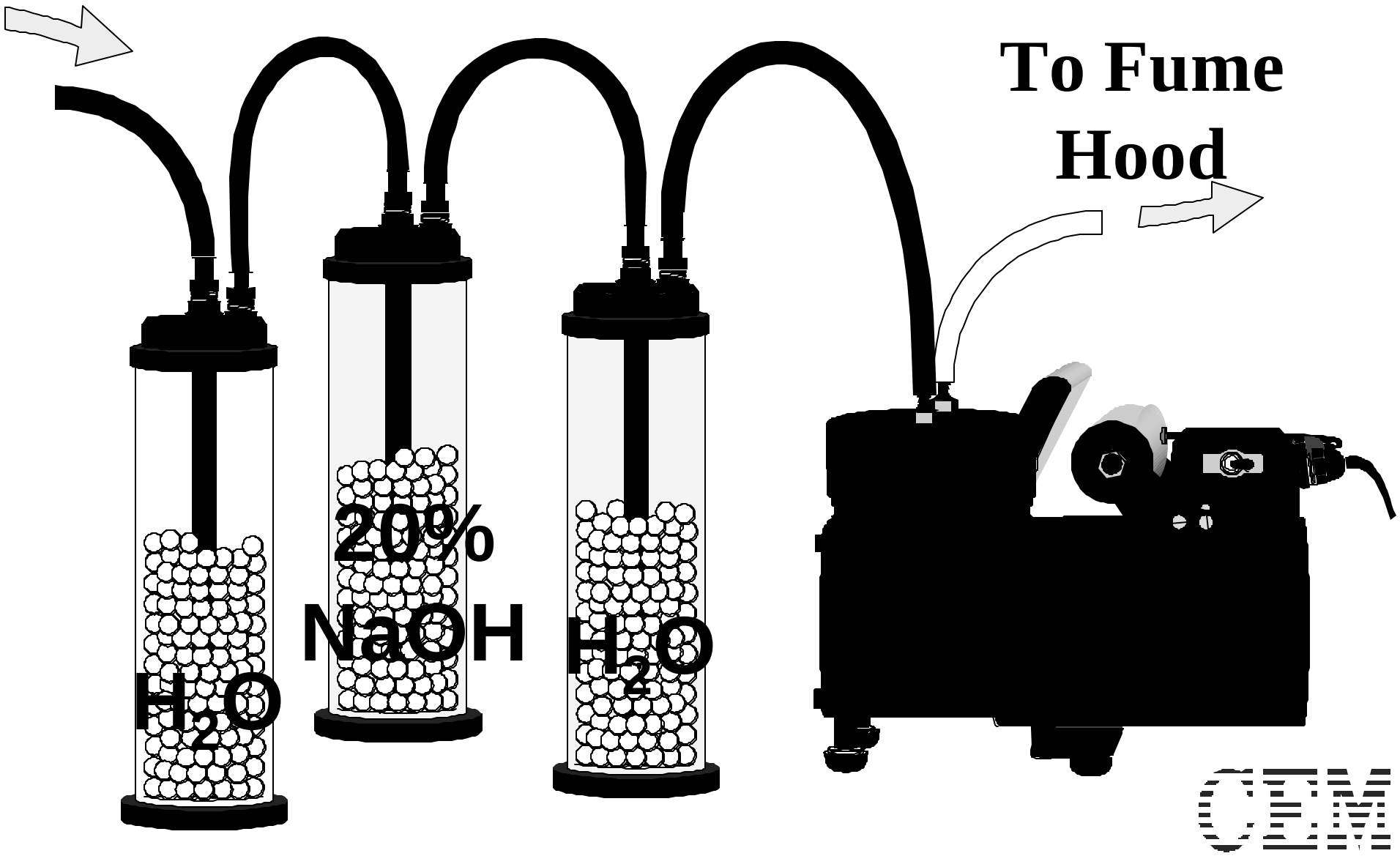
HF Pumping Accessory
with protective cover removed
to view the syringe pump and
valve



VAPOR CONTAINMENT

From Instrument Exhaust

To Fume Hood



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STAR System Programs

- **Mild Digest - Agricultural, Biological, Environmental, Paper**
- **Moderate Char - Agricultural & Biologicals with H_2SO_4, Light Oils, Foods, Plastics, Environmental, Paints, Solvents**
- **Rigorous Char - Oils, Polymers / Plastics, Resins, Solvents, Adhesives, Organic Chemicals, Asphalts, Fuels**
- **Super Char - Samples larger than 2 grams**

Technician Tasks

- **Weigh Sample into Vessel**
- **Place Vessel Into Cell**
- **Lower Vapor Containment Arm**
- **Select the Appropriate Method**
- **Press Start**

STAR Operations

Automatic, Requires No Operator Interaction

- **Add Initial Reagent(s)**
- **Microwave On (slot open) / Measure Temperature**
- **Temperature Reached / Microwave Off (slot closed)**
- **Open / Close Slot to Maintain Temperature for time prescribed by the method**
- **Add Reagents As Defined by Method**
- **Remove and Scrub Vapors**
- **Stops When Complete, Signal Operator**

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Total Digest

- **Inorganic - HF Use**
  - **Teflon Vessel / Condenser**
  - **Teflon Calibration / Method**
  - **HF Pump**
  - **Bring to Temperature/Hold**
- **Organic - Chars**
  - **Char with H_2SO_4**
  - **Oxidize with HNO_3 and H_2O_2**

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Spent Catalyst- 0.5 g (Al2O3 and SiO2)

Initial Reagent: 20 mL $HClO_4$

Stage	Ramp Time (min)	Target Temp °C	TAP (min)	Reagent (mL)	Aliquot (mL)	Add At Start
1	5:00	195	15:00	None	0	No
2	20:00	180	10:00	14 mL HF	7.0	Yes
3	0:00	140	10:00	30 mL H_2O	10.0	Yes

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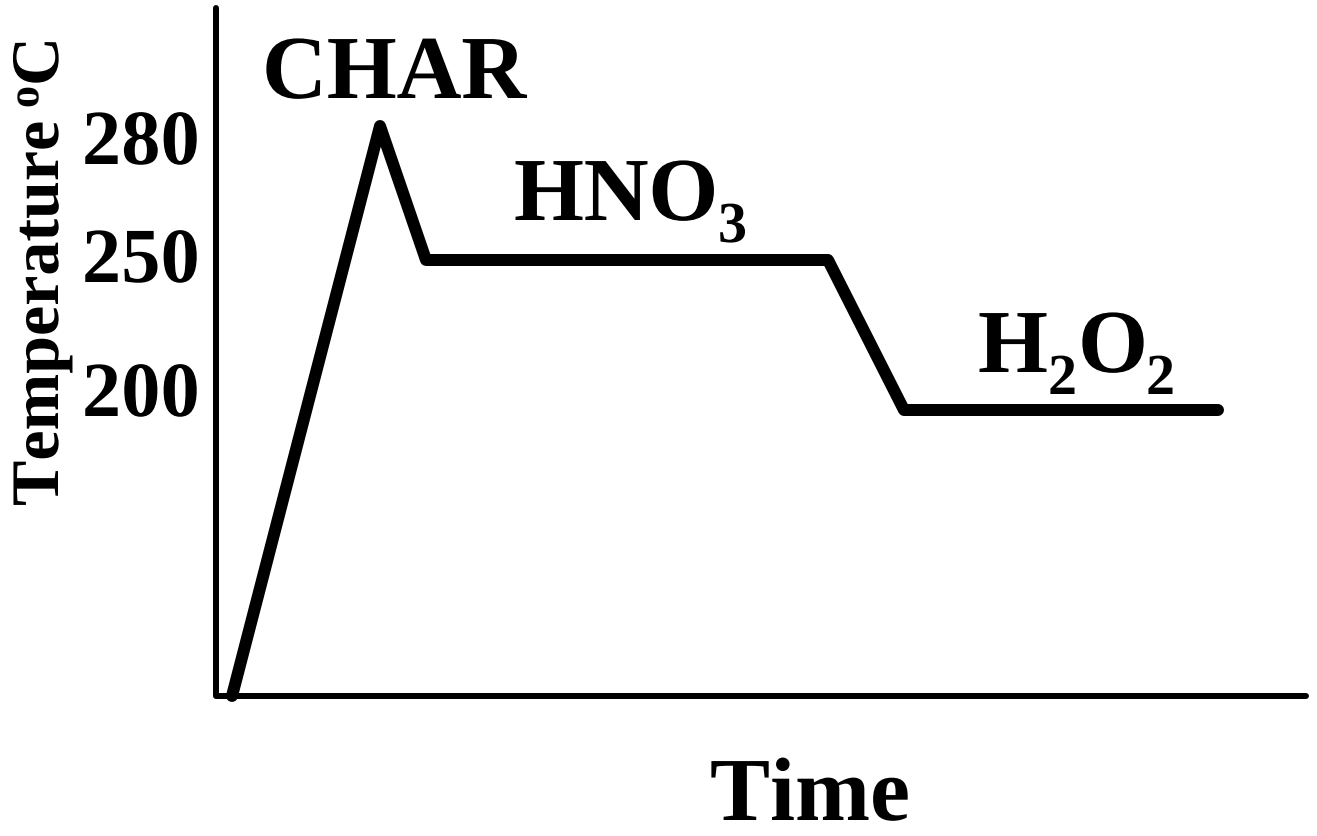
Char Followed by Oxidation



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Evaporations

- **Large Volume Reagent Evaporation**
  - **e.g. 50 mL H_2SO_4**
  - **How Clean ?**
  - **Normal Configuration**
  - **Closed Configuration**
- **Residual Reagent Evaporation**
  - **Normal Configuration**
  - **“Grooved” Air Condenser**

CEM

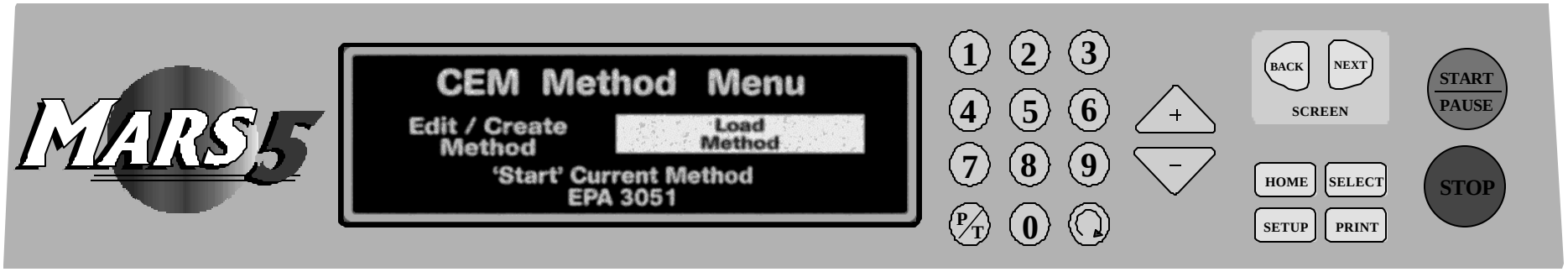

MARS 5
CEM Method Menu
STOP

Technologically Advanced Features

- **1200 watts of microwave power**
- **Continuous power at 300, 600 and 1200 watts**
- **“Auto Load” power sensing**
- **Variable Speed Stirring of all samples (opt.)**
- **Large Cavity (52% > MDS)**
- **Large, Impact-Absorbing Door**
- **Window to Observe Samples**
- **Inlet/Outlet Ports**

CEM

MARS 5 - Software



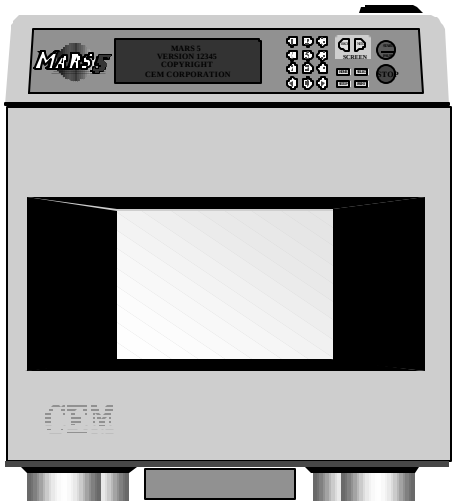
- **"Jumbo-Vision" Screen**
  - **Super clarity**
  - **Status easily read from across the room**
  - **Multi-language**
    - **Japanese, French, German, Spanish, Italian, English**
  - **Major emphasis on ease of use**
- **Built In Applications Library**
- **MARSLink Software**
  - **also multi-lingual**

CEM

New Technology for Temperature Control

EST

(electronic sensor - temperature)



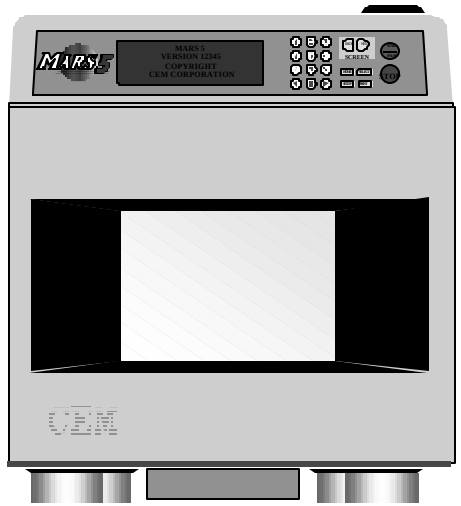
- **Durable**

  Platinum Construction
  Sapphire Thermowell
- **Reliable**
- **Accuracy +/- 1 ºC**
- **Control to 300 ºC**
- **Easy “Slide-In” Installation**

New Method of Pressure Control

ESP

(Electronic Sensor - Pressure)



- **Unique Shielded Sensor**
- **Non-Invasive**
- **Measurement to 1500 psi (100 bar)**
- **“Plug In” Connection**

Unique Vessel Design

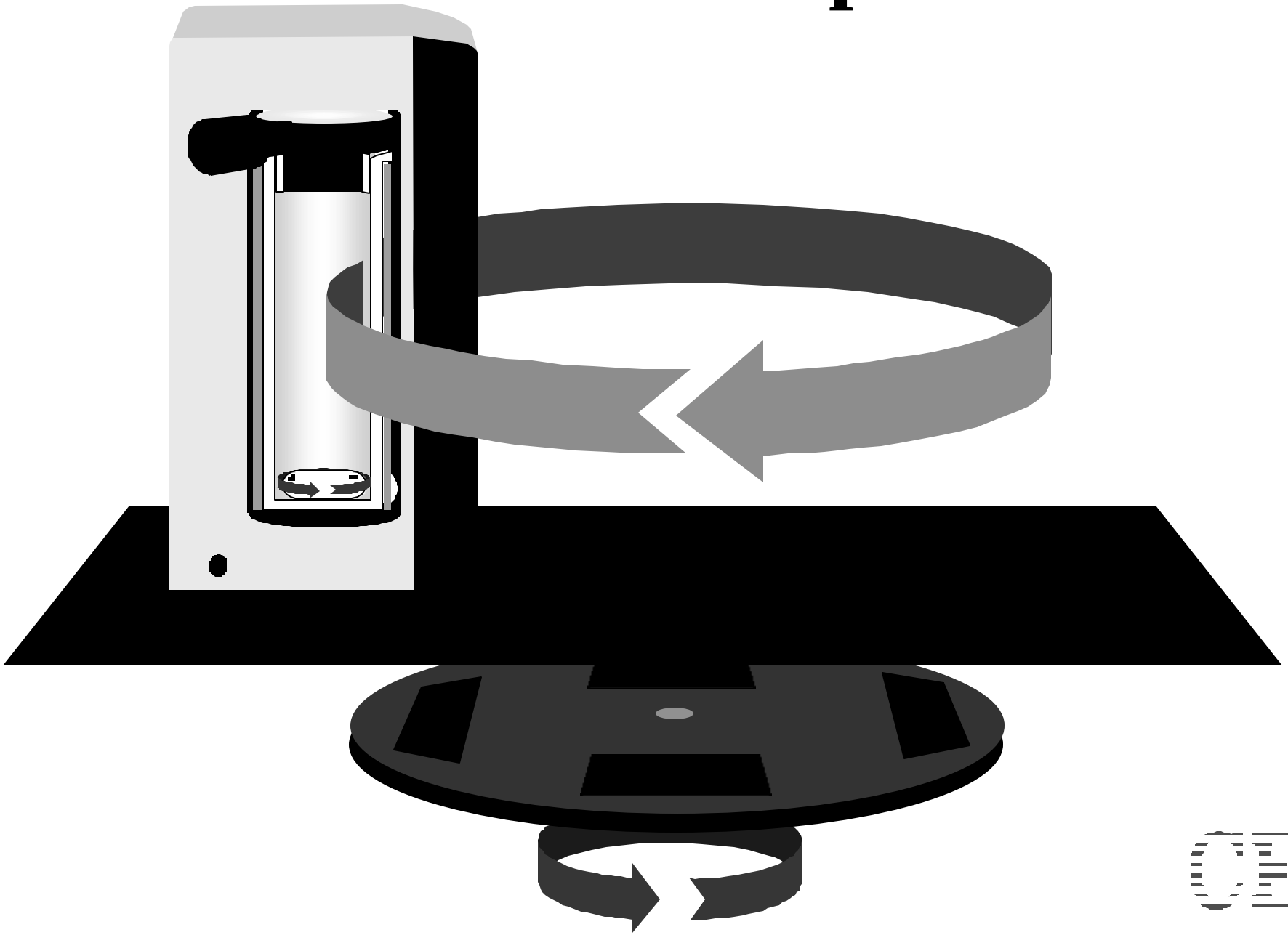
- **Easy Assembly**
- **Emphasis on Safety**
  - Frame For Axial Stresses
  - Composite for Radial Forces
- **Performance Oriented**

Advanced Vessel Technology
High and Extensive Performance

Vessel	Pressure psi	Temp. °C	Vessels on Turntable
HP 500	500	260	14
XP 1500	1500	300	12


CAUTION!
CAUTION!
CAUTION!
CAUTION!

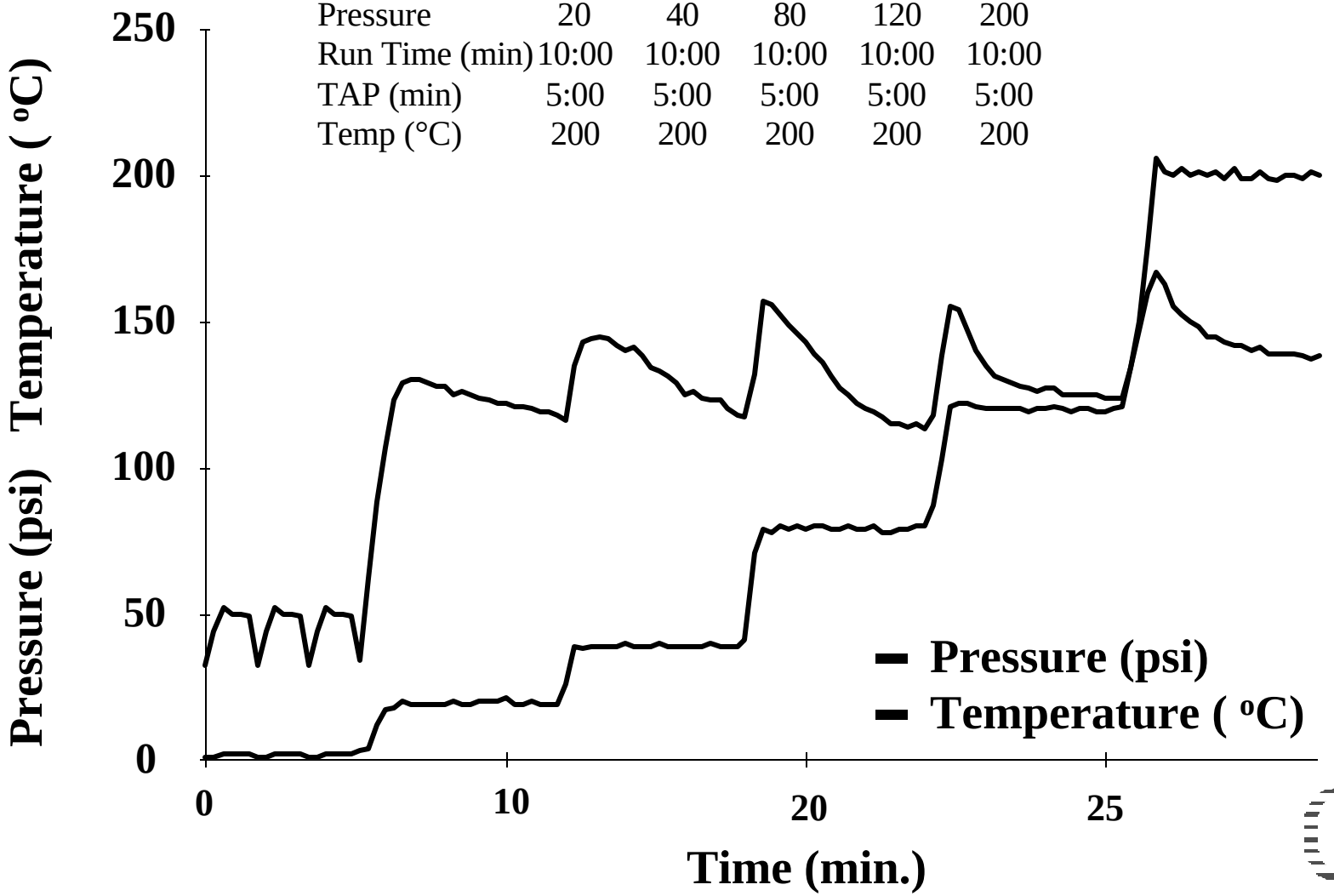
All Vessel Variable Speed Stirring



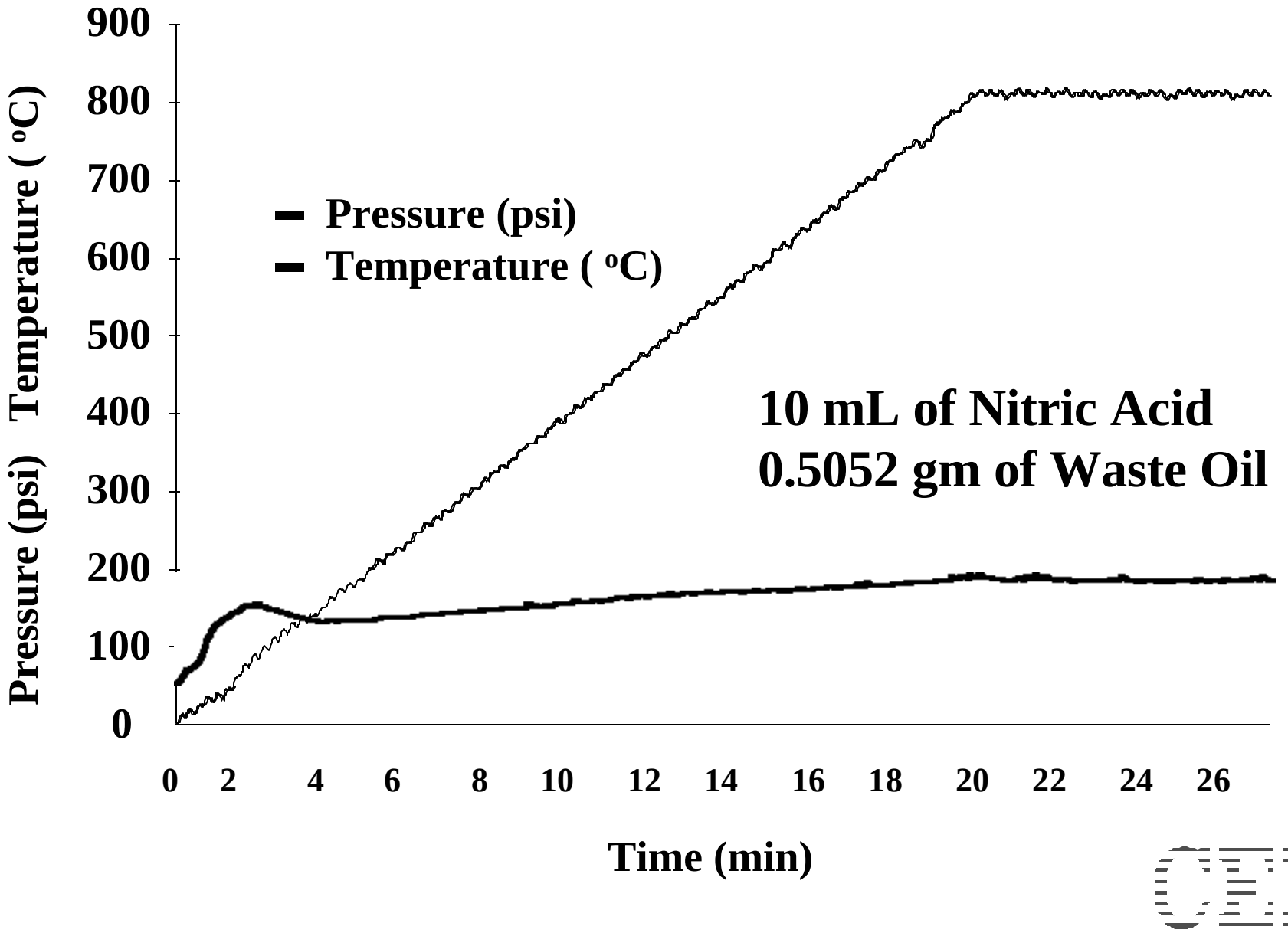
Digestion of Waste Oil

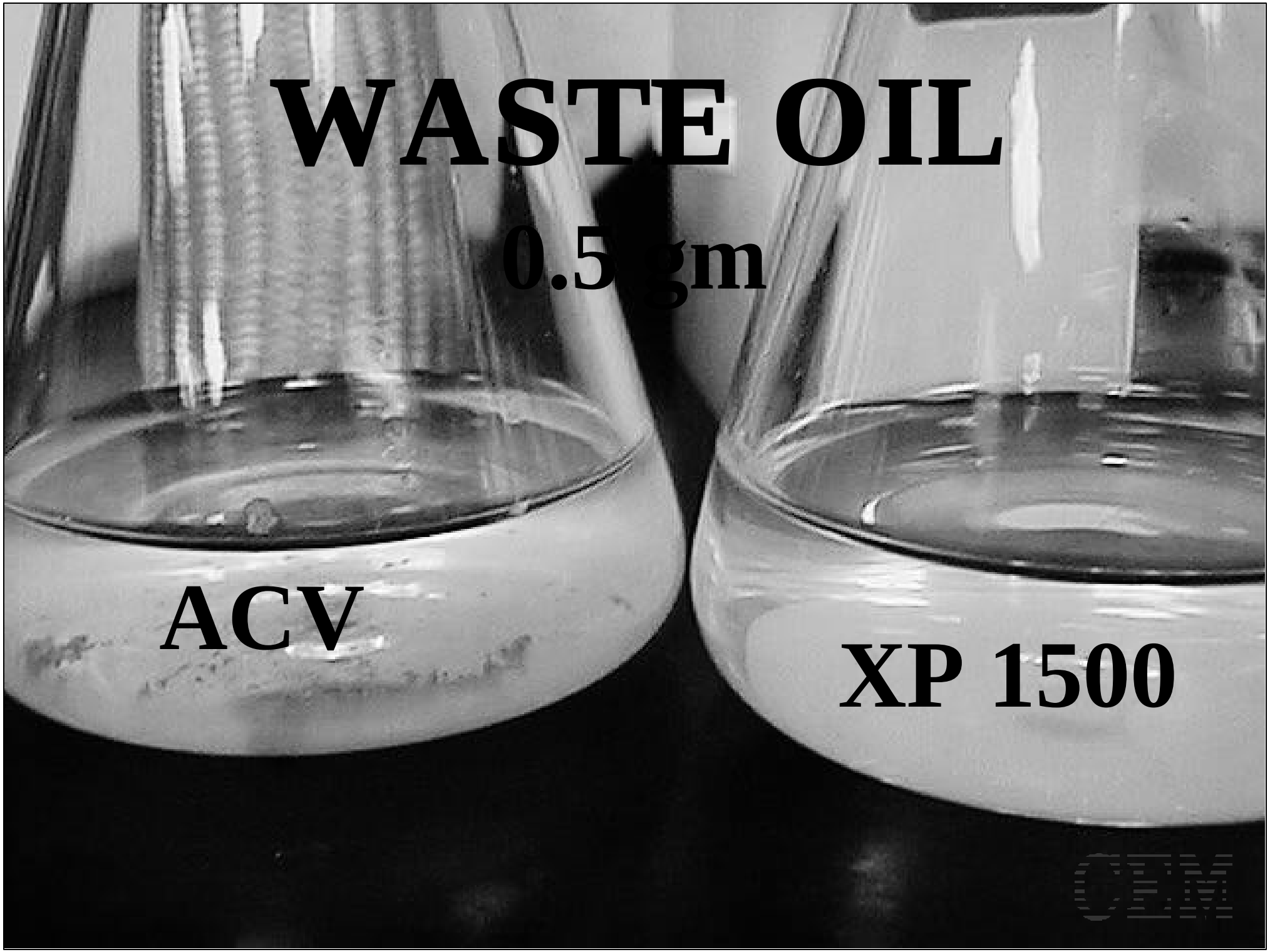
MDS-2100, 1 ACV, 0.5053 grams oil, 10 mL Nitric

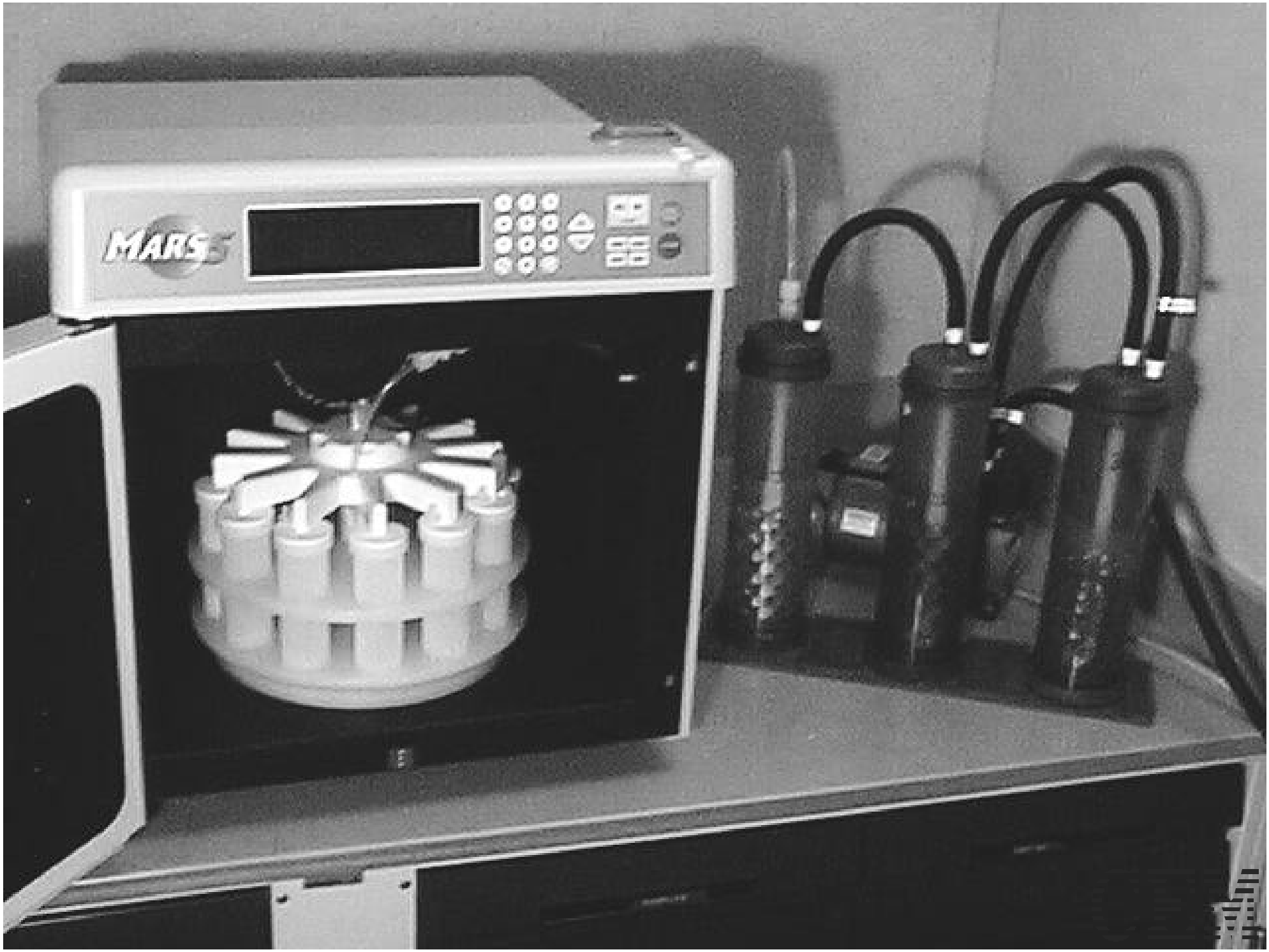
Stage	1	2	3	4	5
Power (%)	35	35	35	35	35
Pressure	20	40	80	120	200
Run Time (min)	10:00	10:00	10:00	10:00	10:00
TAP (min)	5:00	5:00	5:00	5:00	5:00
Temp (°C)	200	200	200	200	200

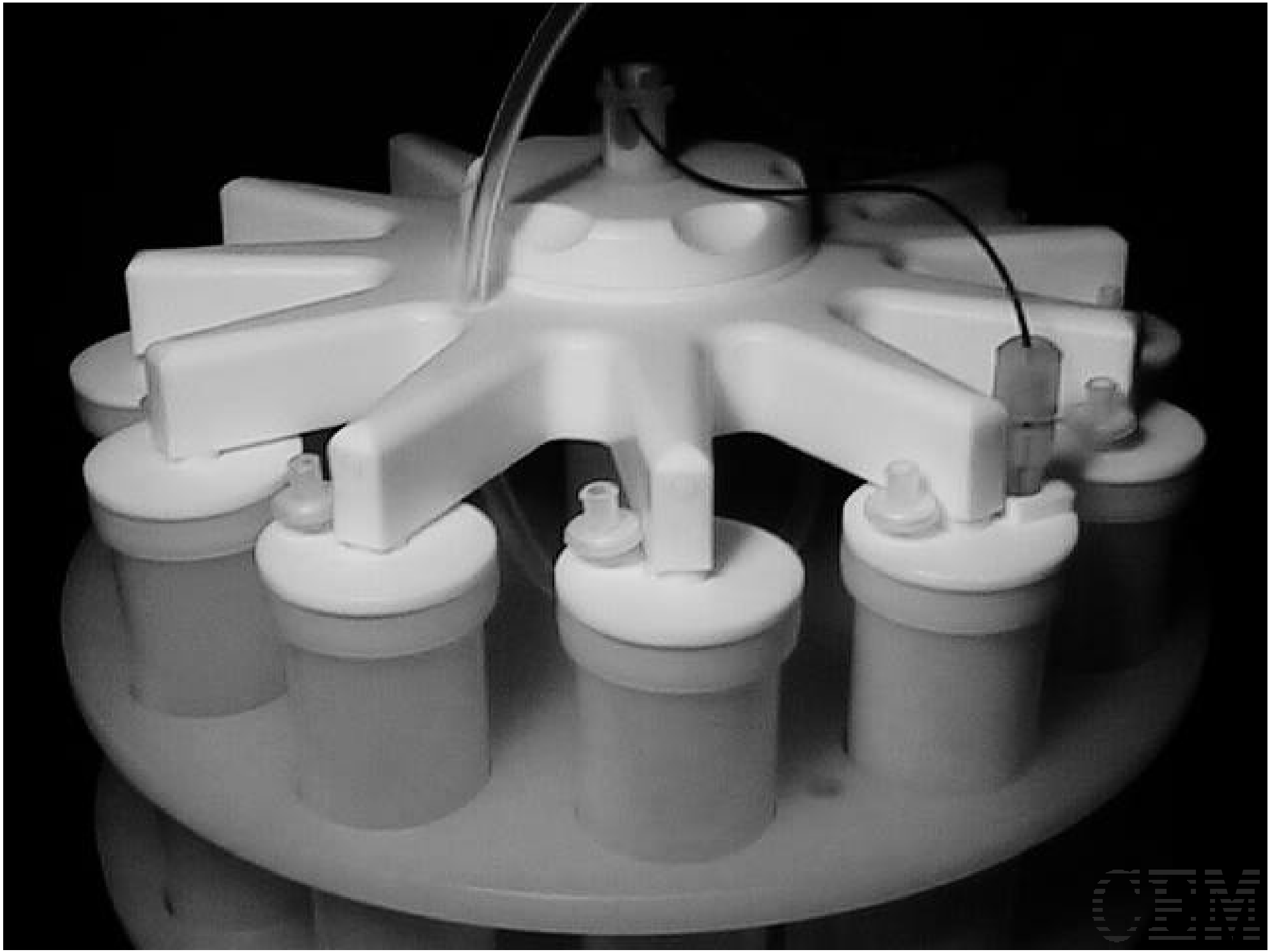


Waste Oil Digestion in XP-1500




WASTE OIL
0.5 gm
ACV
XP 1500


MARS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Heating Program*

	Stage 1	Stage 2
Power:	100%	60%
Time:	2:00	35:00
Temp:	BP - 5°C	BP + 2°C
TAP :	0:01	35:00

*** Heating Program for 12 Vessels with 15 mL of acid per vessel**

BP = Boiling Point of the Solution

CEM

Evaporation Performance

Number of Vessels:	12
Starting Acid Volume:	15 mL
Final Acid Volume:	< 0.5 mL
Heating Time:	30-35 min.
Acids Tested:	HNO_3, HF and HCl

Key Considerations

- **Sample Matrix, Size, & Reagents**
- **Elements of Interest**
- **Throughput Requirements**

STAR System Pluses

- **Ease of Use**
- **Safety (no pressure, no acid handling)**
- **High-Temperature**
- **Sample Size**
- **Labor Savings (semi-automated)**

Closed Vessel Pluses

- **Safety**
- **Regulatory Approvals**
- **Speed (per sample, not thruput)**
- **Results (volatiles, complete digestion)**
- **Environmental (no hood, little acid used)**
- **Labor and Cost Savings**

Conclusion

- **"One Size Fits All" is no longer good enough for all sample types.**
- **Match your needs (matrix, size, throughput, elements) to the:**
  - **microwave instrument best-suited**
    - **Conventional "Closed Cavity" or new "Open Cavity"**
  - **most cost-effective open or closed vessel technique**