Introduction To The Scale Up Of Continuous Microwave Flow Chemistry

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C-Tech History

1966
Electricity Council Research Center (ECRC) established as a publicly-funded research institute

1990

ECRC privatised along with the UK energy generation industry,

becomes EA Technology

2000
Management buyout of EA Technology's Energy Division, which becomes C-Tech Innovation

Approach

→ Concept	chemistry, physics, metallurgy
→ Pilot	mechanical & electrical
→ Trials	design & build
→ Production	commissioning

Advanced Thermal Technologies

- Continuous Flow Chemistry
- Microwave Calcining
- Continuous Cooking
- Advanced Induction Heating
- RF Curing and Moulding
- Plasma Surface Modification

C-Tech Innovation

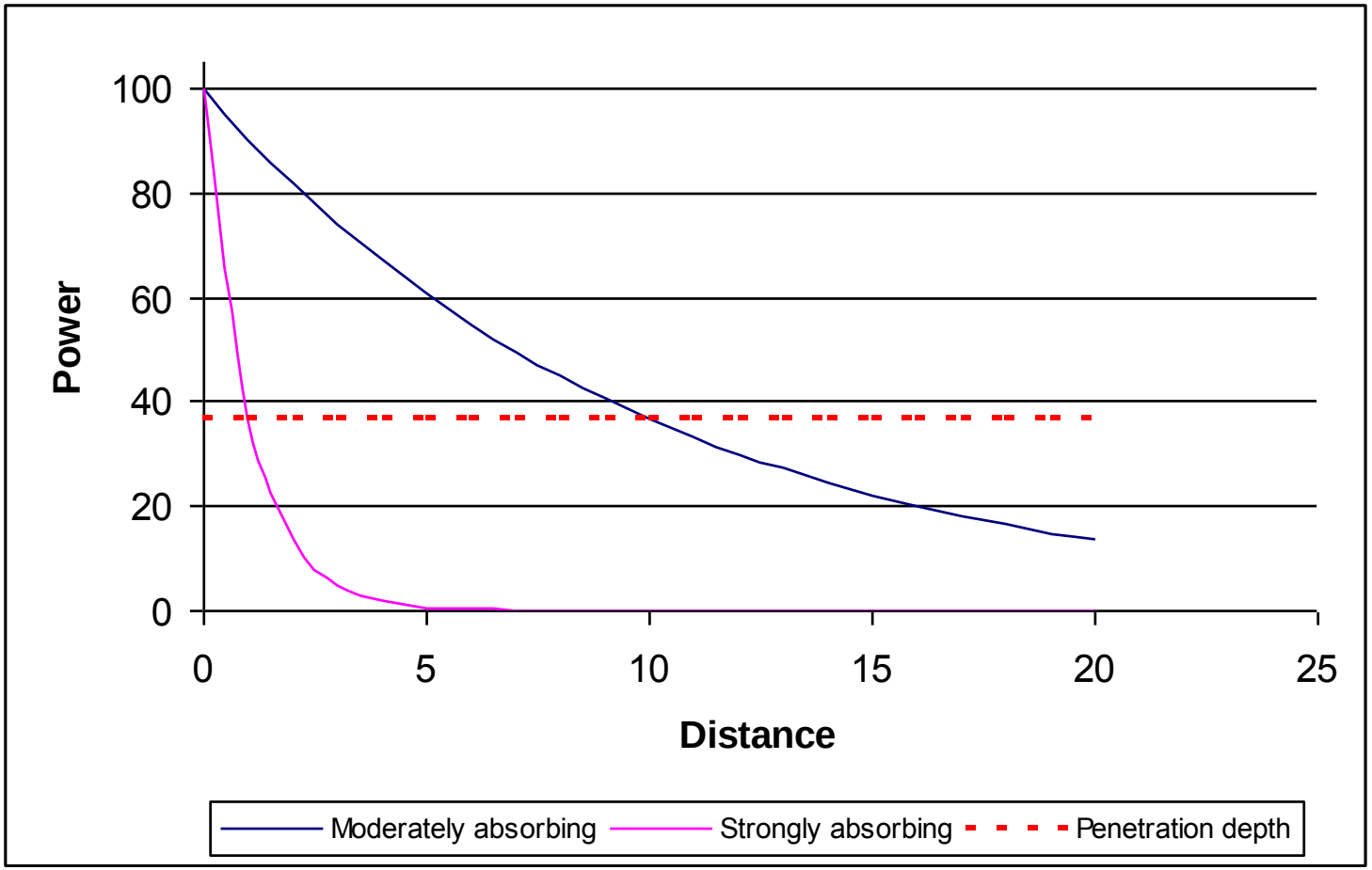
Challenges of Scaling Up Microwave Chemistry

Microwave Chemistry Scale Up at C-Tech

Case Studies

Large Production Scale Microwave Chemistry

Challenges of Scale Up

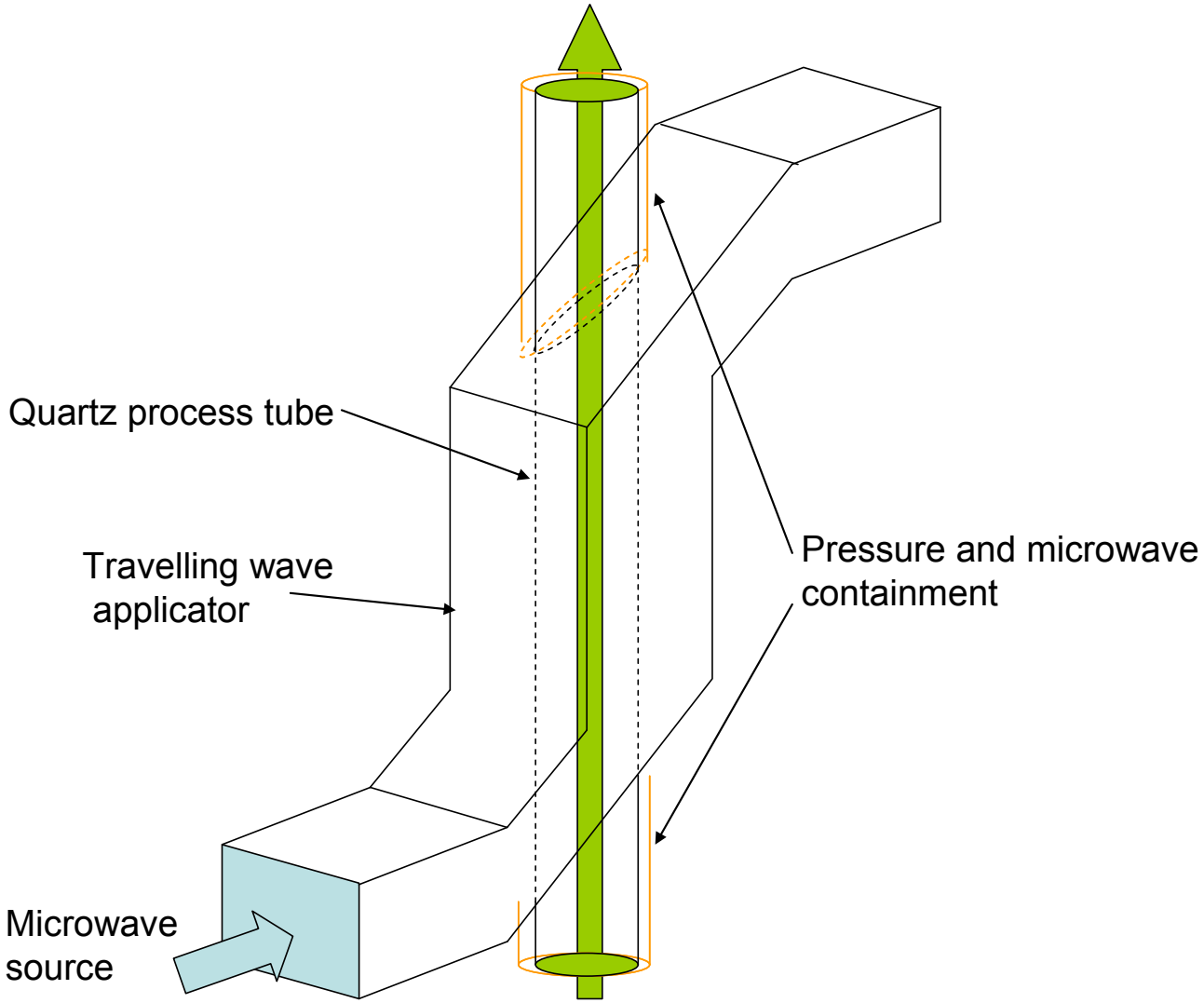


Penetration Depth = Distance through the object being heated where the incident power drops to 1/e of that at the surface

Challenges of Scale Up

- Penetration depth and uniformity
- Materials
- Measurement and control

Microwave Cavity



WHY: Microwave Flow Chemistry

Faster — Faster reaction times

Cleaner — Rapid heating and absence of wall effects results in less side reactions

Greener — Cleaner reactions means less purification/solvents/SMs. Energy saving MW flow vs batch

Safer — Less inventory of hazardous materials

Pilot Scale Reactor

- Flow rates up to 400 mL/min
- Dual feed vessels
- Pressurised receiver vessel
- Designed to process up to 20 L in a single run (can be operated for longer periods)
- Homogeneous reactions
- Light heterogeneous reactions

Equipment Specification

Features

Microwave power - 1-6 kW as standard (higher if required) Temperature

range - ambient to 250°C

Pressure - ambient to 30 bar

Flow rate - 5 mL to 1 L/min

Materials of construction - glass, fluoropolymer, stainless steel

Automatic temperature control

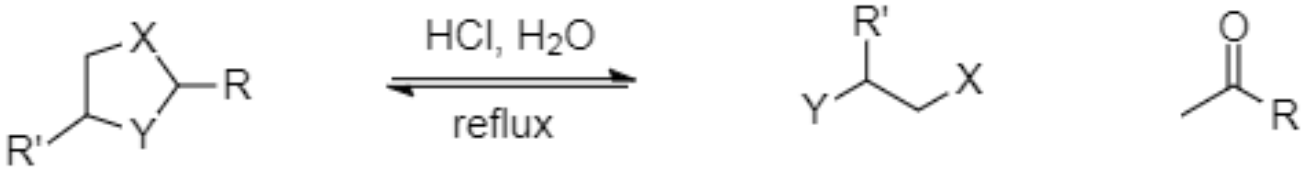
Options

Fibre optic temperature measurement

Halide resistant coating of steel parts

Complete plant or bare reactor

Case Studies



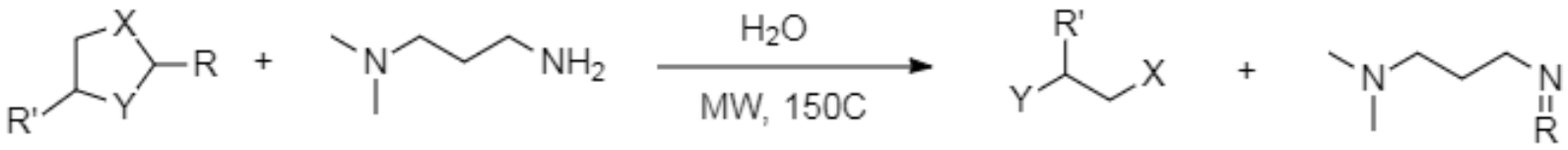
Reaction from Robinson Brothers

Equilibrium reaction - requires removal of aldehyde by-product to drive the reaction forward

Takes 4.5 days to produce 500kg, 30 batches per campaign

30 year old reaction

Case Studies



Reaction done in MW flow reactor

Temperatures between 120-150°C

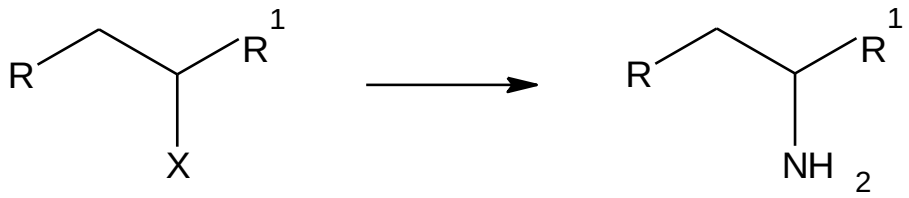
Reaction times 2-10 mins

Gave >75% conversation to product and shows much less disulfide impurity than standard reaction

Difficulties in separating product from excess DMAPA used in the reaction

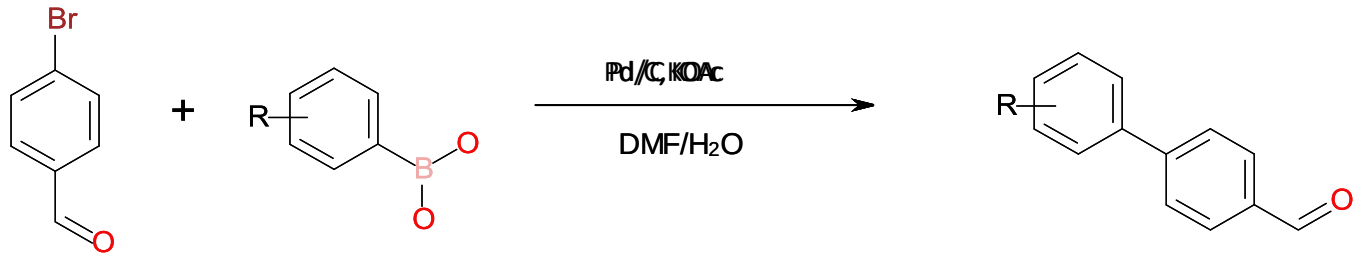
Currently investigating reduction of DMAPA eqvs and non aliphatic amines

Case Studies



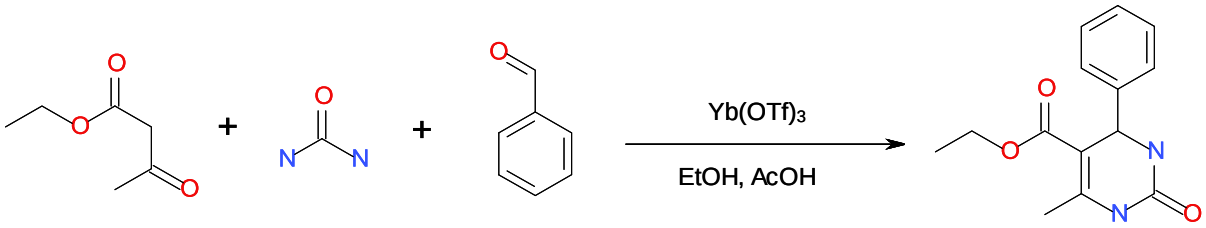
- Difficult nucleophillic amination reaction – using conventional heating
- Reaction parameters quickly defined by MW chemistry - MW lab method gives double the yield with less solvent and readily available reagents
- Following day 18 L processed in 3.5 h to give 2.6 kg
- No method development required for scale up – **saved an estimated 4 weeks in project time**

Case Studies



- Thermal reaction
  - 92% conversion to product after 2h
- MW reaction
  - 295 mL/min (~1 min residence time)
  - Reaction went to 98% completion (analysis by GC/MS)
  - 4.08 kg/h of product
- Reaction run in pilot scale reactor in Sweden
  - Continuous operation –- 32 hours
  - 140 L of reaction mixture giving 22 kg of isolated product

Case Studies



- Thermal reaction
  - 35% conversion after 8 h

- MW reaction
  - Reaction run at 75 mL/min (~4 min residence time)
  - 71% conversion to product
  - 307 g/h of product

Case Studies

•MW flow chemistry allows direct scale up from lab scale to plant scale without time consuming process development step

•MW flow chemistry allows rapid heating of solvents (sometimes above their boiling point)

- •Higher yields
- •Shorter reaction times
- •Less side products

C-Wave - Continuous Microwave Flow Reactors

- Temperature up to 250°C
- Pressure up to 20 bar
- Flow rates up to 200 mL/min
- Dual feed vessels
- Dual receiver vessels
- Designed to process up to 10 L in a single run (can be operated for longer periods)
- Homogeneous reactions Light heterogeneous reactions

- Temperature up to 180°C
- Pressure up to 20 bar
- 0.22L capacity
- 6kW

C-Wave - Continuous Microwave Flow Reactors

20 bar, 215°C

20 mL capacity

1 kW, 2450 MHz

Hastelloy and quartz glass

ATEX rated

450 W solid state generator

@2450 MHz

20 bar

2 x 10 L pressurised receiver vessels

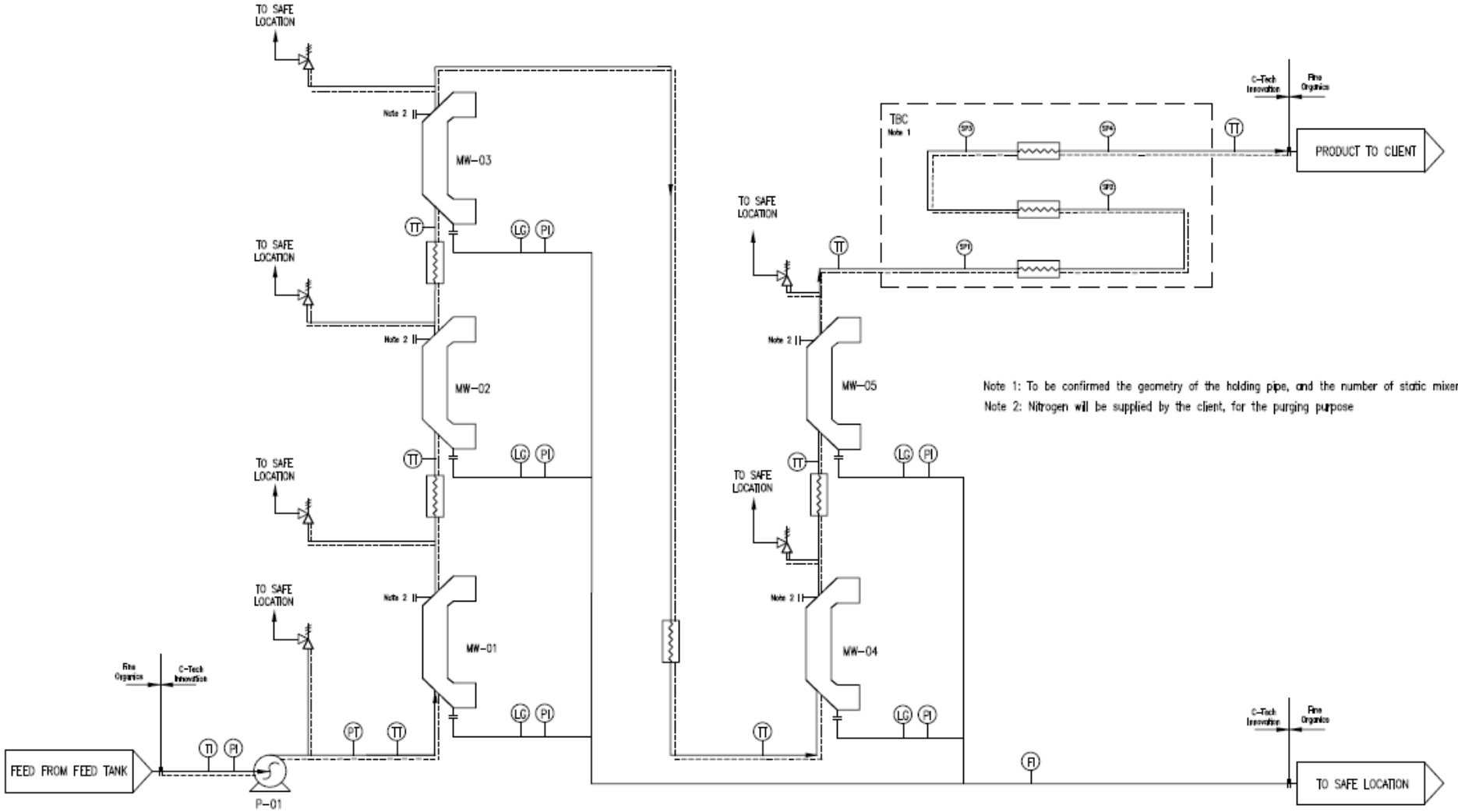
17 mL/min, 30 sec residence time

From 0 - 180°C in a single pass

Scale Up

- Scale up to 2000 kg/h throughput
- Maintain reaction temperature for 5.5 mins
- Requires 120 kW
- Designed as 4 separate modules with independent pumping, MWpower supplies and MW power control

Scale Up


TO SAFE LOCATION
Note 2
MW-03
TT
LC
PI
TO SAFE LOCATION
Note 2
MW-02
TT
LC
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TO SAFE LOCATION
Note 2
MW-01
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Note 2
MW-05
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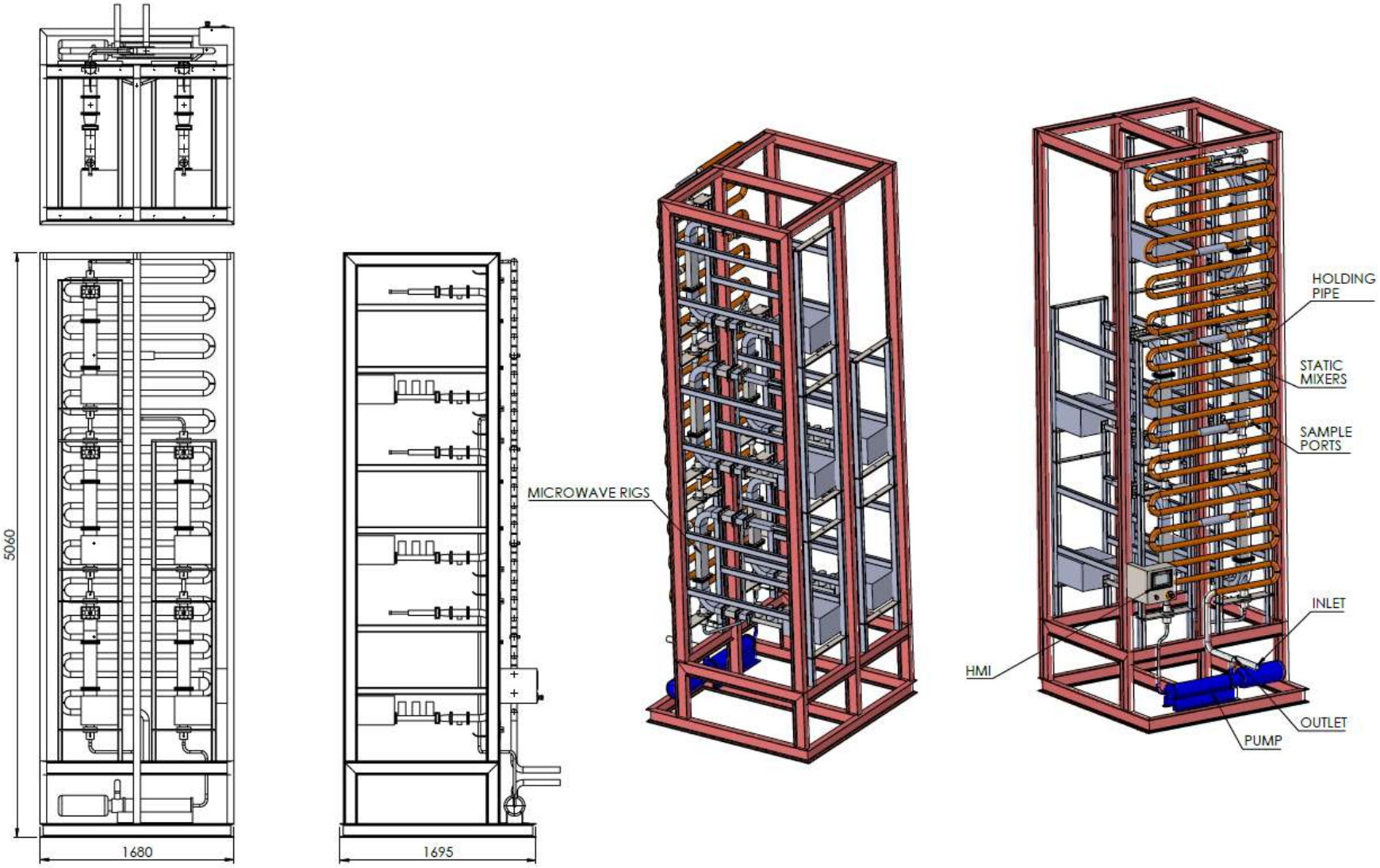
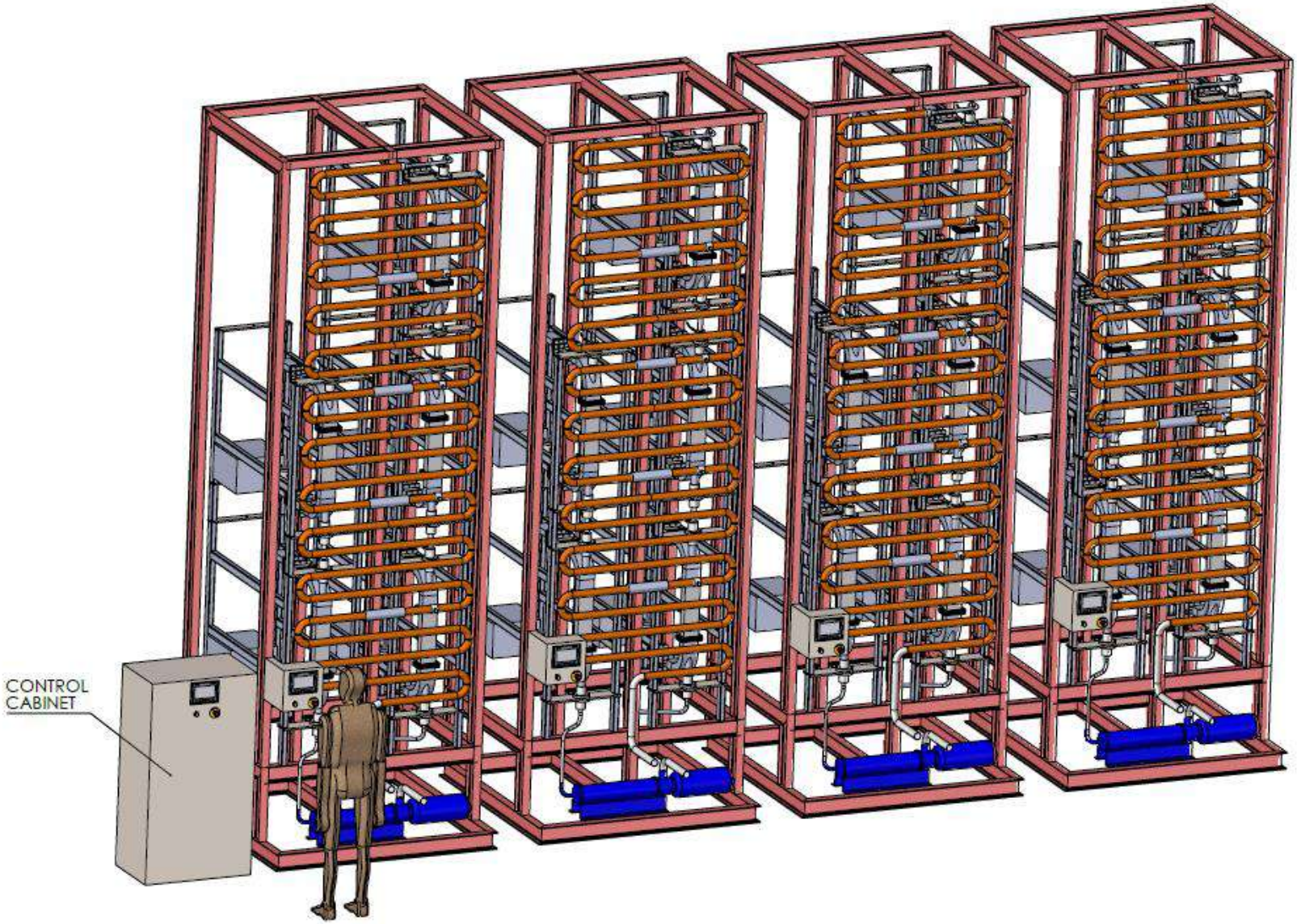
TO SAFE LOCATION
Note 2
MW-04
TT
LC
PI
TBC
Note 1
SP3
SP4
SP2
SP1
TT
C-Tech Innovation
Fine Organics
PRODUCT TO CLIENT
Fine Organics
C-Tech Innovation
FEED FROM FEED TANK
TI
PI
P-01
PT
TT
FI
C-Tech Innovation
Fine Organics
TO SAFE LOCATION
Note 1: To be confirmed the geometry of the holding pipe, and the number of static mixers
Note 2: Nitrogen will be supplied by the client, for the purging purpose

Scale Up


5060
1680
1695
MICROWAVE RIGS
HOLDING PIPE
STATIC MIXERS
SAMPLE PORTS
INLET
HMI
OUTLET
PUMP

Scale Up



Scale Up

- Continuous flow microwave generator in 4 modular sections
- 5 x 6kW microwave generators @ 2450 MHz in each module section
- 20mm inside diameter quartz glass reactor tube with >1000mm heat application length
- Holding section with 5.5 minutes residence time at required flow rate

- Automatic control of outlet temperature, easy to use touch screen facility. Manual control option for microwave power also available, power can be set from 10% to 100%
- Working temperature up to 230°C
- Working pressure up to 10 bar
- Pressure relief and leak detection system
- Temperature and power analogue outputs
- Data logging of all process data of up to 50 data points with 20,000 entries each

Conclusions

Large scale microwave chemistry is possible

Its already being done at pilot scale

Continuous flow not batch

Materials and mechanical design are as important as microwave cavity design

Benefits

Faster reactions, more throughput

Cleaner - no hot oil, no fouling of hot surfaces

Greener - higher yields, less by-products, less catalyst, less waste

Safer - lower chemical inventories, easy temperature control

High throughput and continuous operation

Handles liquids and light slurries

Excellent chemical resistance

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