

MICROACTIVITY-Reference



What is MA-Reference?

- ✓ The **MICROACTIVITY-Reference** is an automatic and computerized laboratory reactor for catalytic microactivity reactions with reactor bypass, preheater evaporator, pressure control valve and other process layouts in hot box, which avoids the possible condensation of volatile products, at the time that preheats the reactants efficiently.
- ✓ It consists of a **BASIC UNIT** and some series of **EXTRA PACKAGES** that improve or modify its efficiency. It is a single structure that contains the electronic unit, control and MFC system and includes the hot box where the reactor and process valves are located. The system has local control and on-line remote control, based on TCP/IP Ethernet communications with distributed control structure. A complete and elaborated security system is integrated in microprocessor, independent of the computer.

What is MA-Reference?

- ✓ Strategic alliances with our customers have induced some of the most important technological solutions at the present time for a variety of catalytic processes.
- ✓ For reactions at high pressure that involve separation of gases and liquids, a new system of level control of the condensed liquid is introduced with almost no dead volume (0.3 ml), so that the sample of the liquid outlet is the condensed product mixture formed at the very last minutes of reaction.
- ✓ A wide variety of reactions has been carried out in our reactors: Hydrocracking, Hydrotreating, Isomerization, Hydrogenation, Hydrodesulphurization (HDS), Oxidation, Hydrodenitrogenation (HDN), Polymerization, Reforming (aromatization), steam reforming, etc.

Models

**Basic
Unit**



**Extra
Packages**

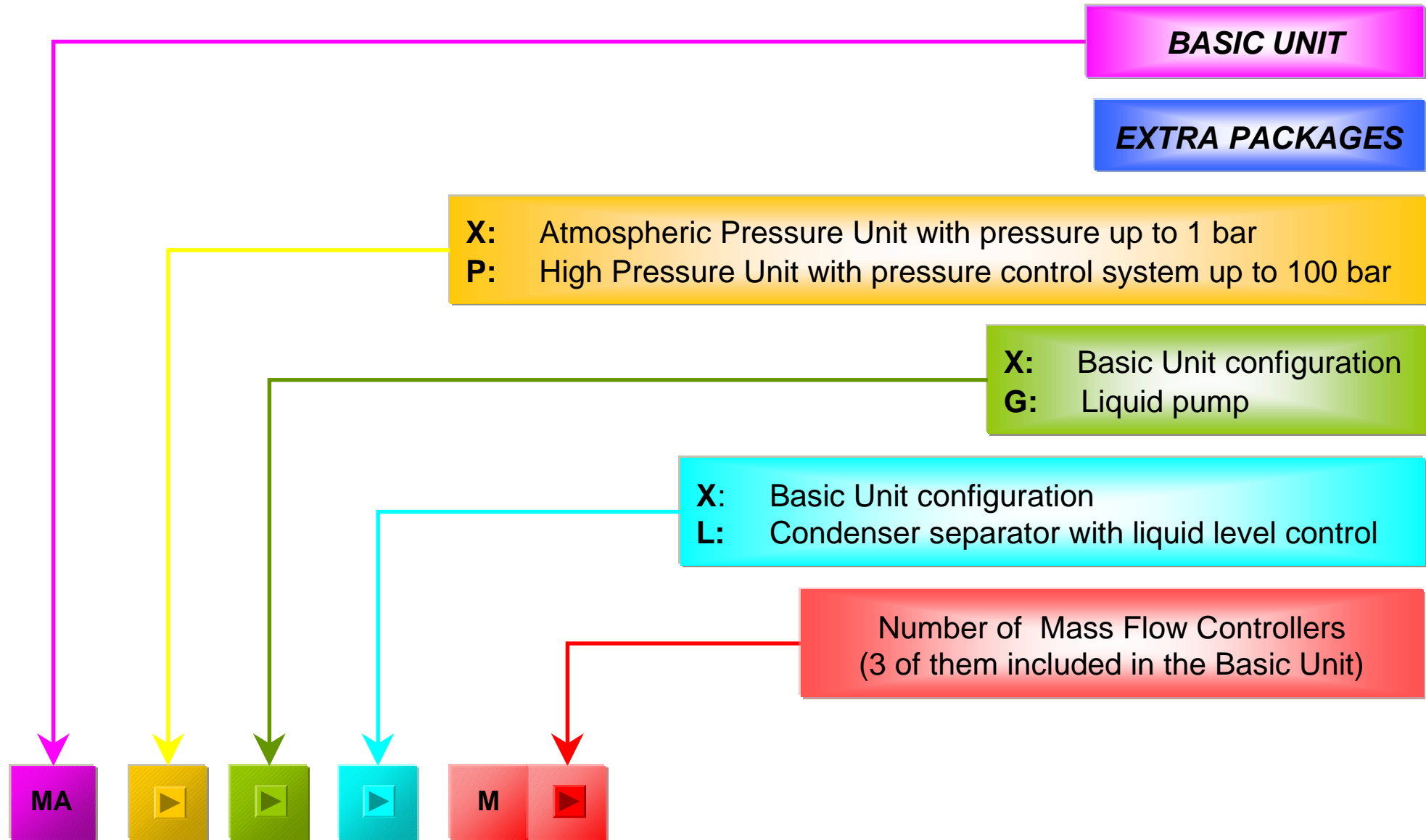


Custom-made equipment

General Information

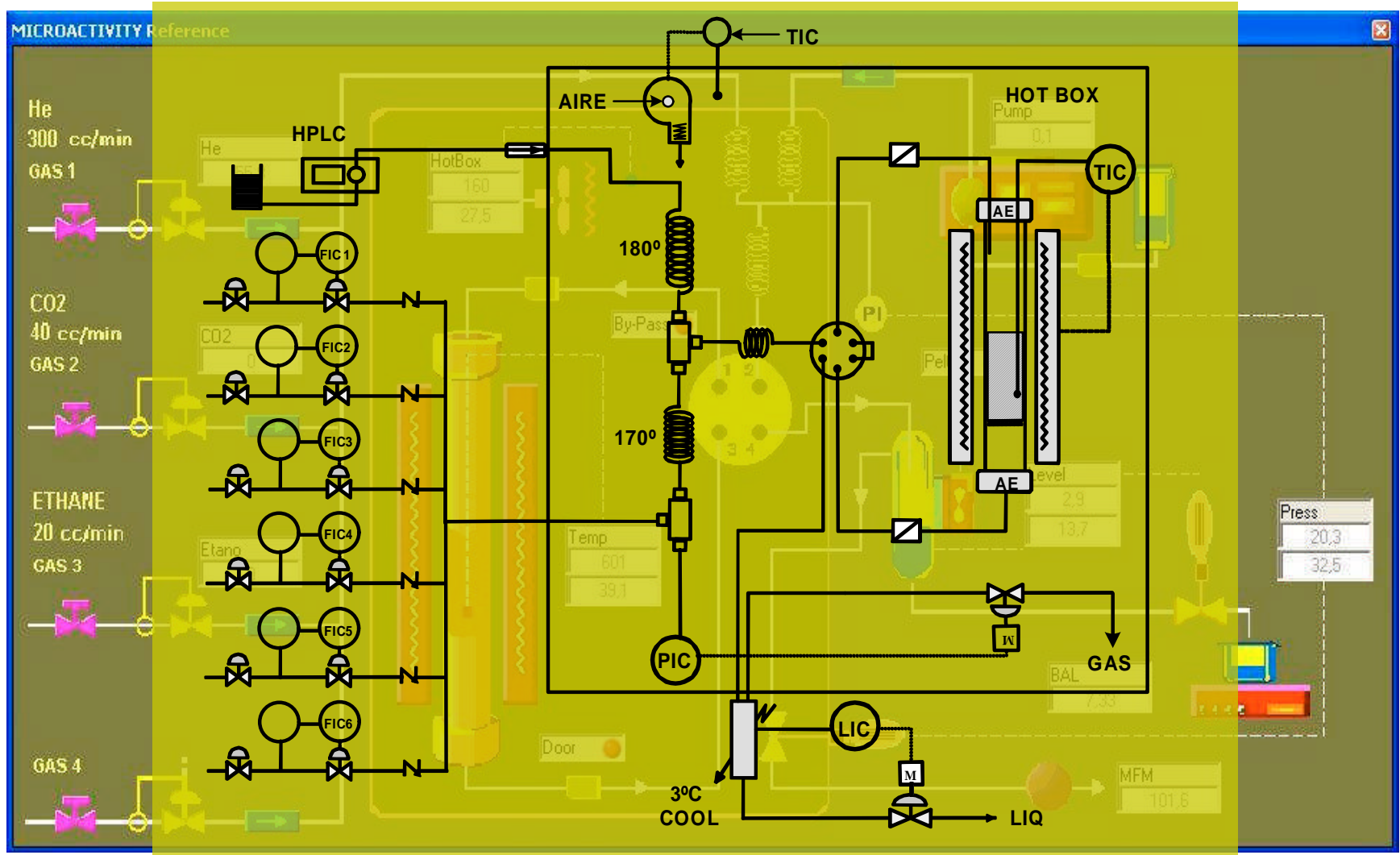
Available Configurations

General Information



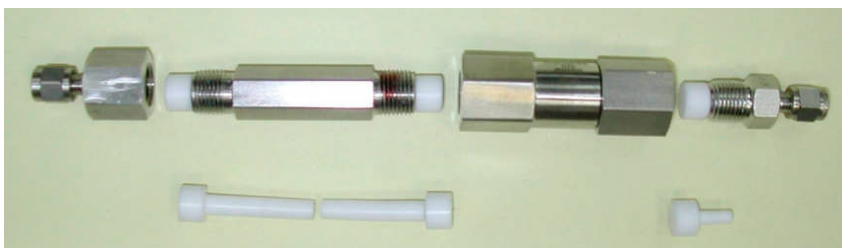
P&I Diagram

Basic Unit Features



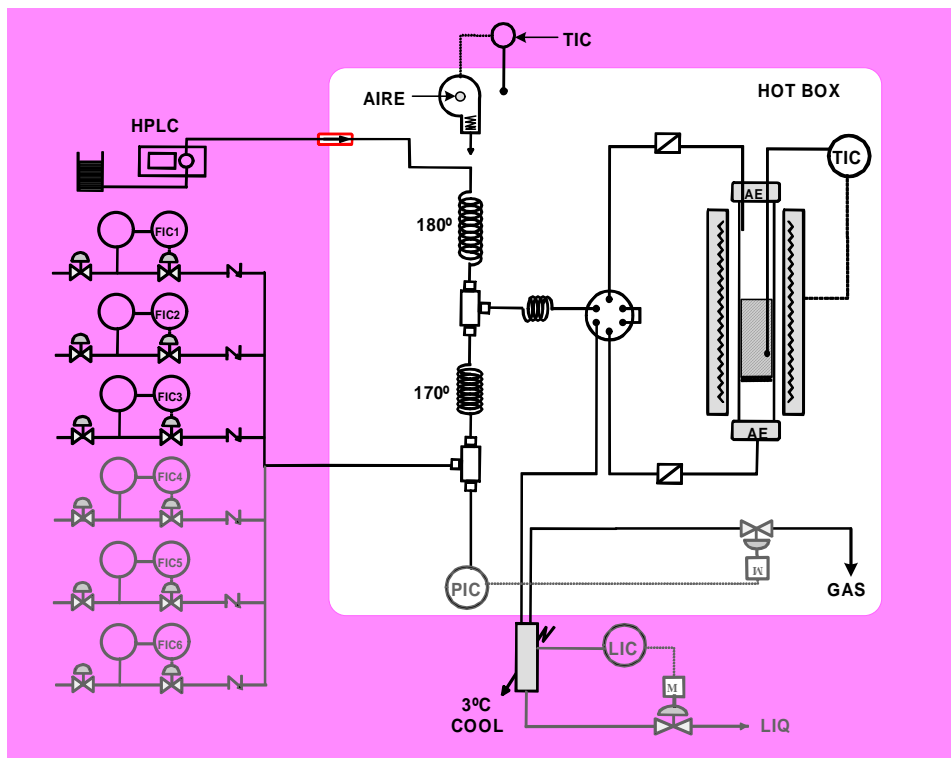
Hot Box

Avoids the possible condensation of products & reagents



Hot Box features:

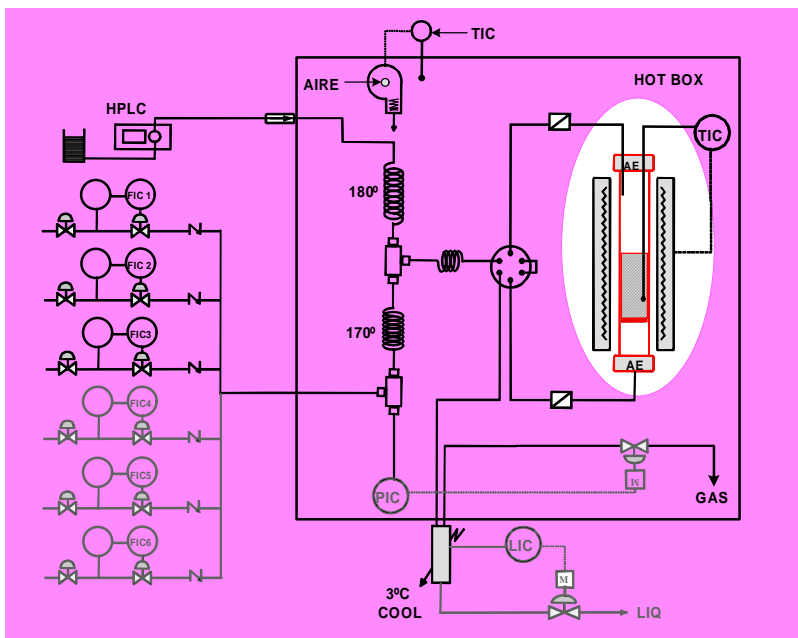
- ✓ Reactor & furnace
- ✓ Check-valve without dead volume
- ✓ VICI Valve 6 ports for reactor bypass
- ✓ Liquid evaporator
- ✓ Gas preheater
- ✓ Filters
- ✓ Electric heater
- ✓ Max. temperature 180°C



Reactor & Furnace

Fixed bed reactor with low inertia furnace

- ✓ Tubular reactor Autoclave Engineers
- ✓ Max. temperature 700°C
- ✓ Porous plate (10 μm)
- ✓ Internal thermocouple, directly in catalyst bed
- ✓ Descendent “up-down” reagents flow



Dimensions:

Length: 305 mm

Ext. diam.: 14,5 mm

Int. diam.: 9,12 mm

Material: Stainless steel 316-L

Int. volume: 20 cm³

Connections: SF562CX

Locks: AE-6F2986

Max pressure: 1350 bar at 25 °C

400 bar at 482 °C

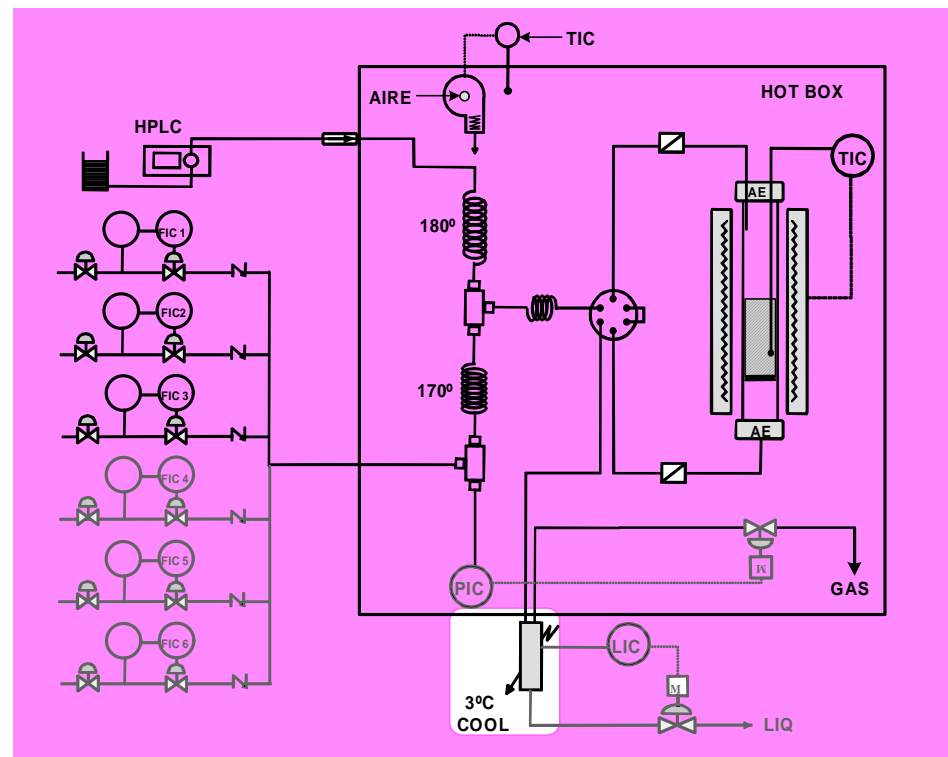
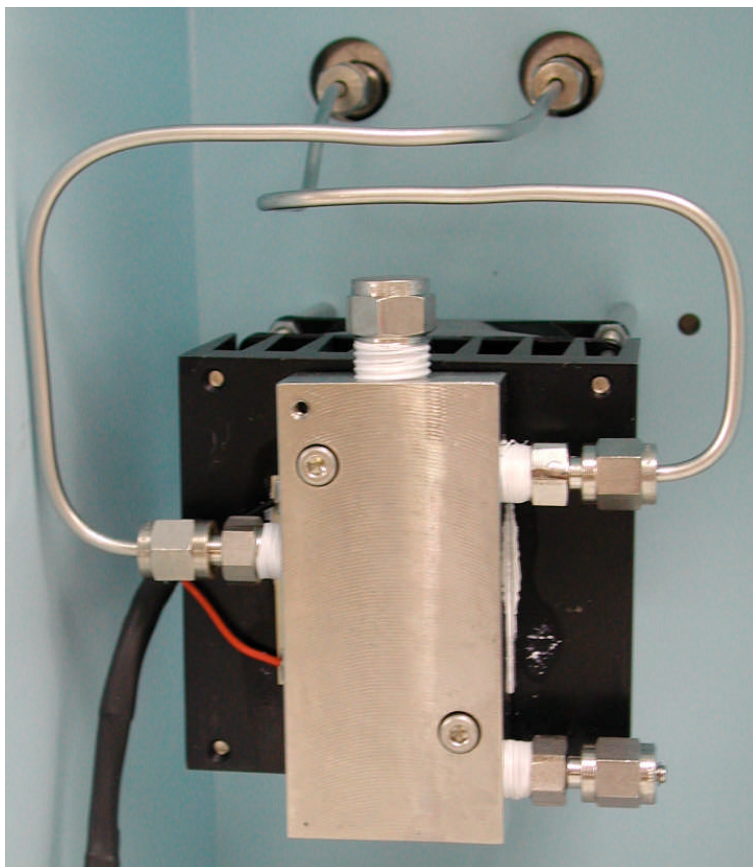


Liquid-Gas Separator

Quasi-zero dead volume

- ✓ Liquid condensation at 3°C approx.
- ✓ Peltier effect

Basic Unit Features

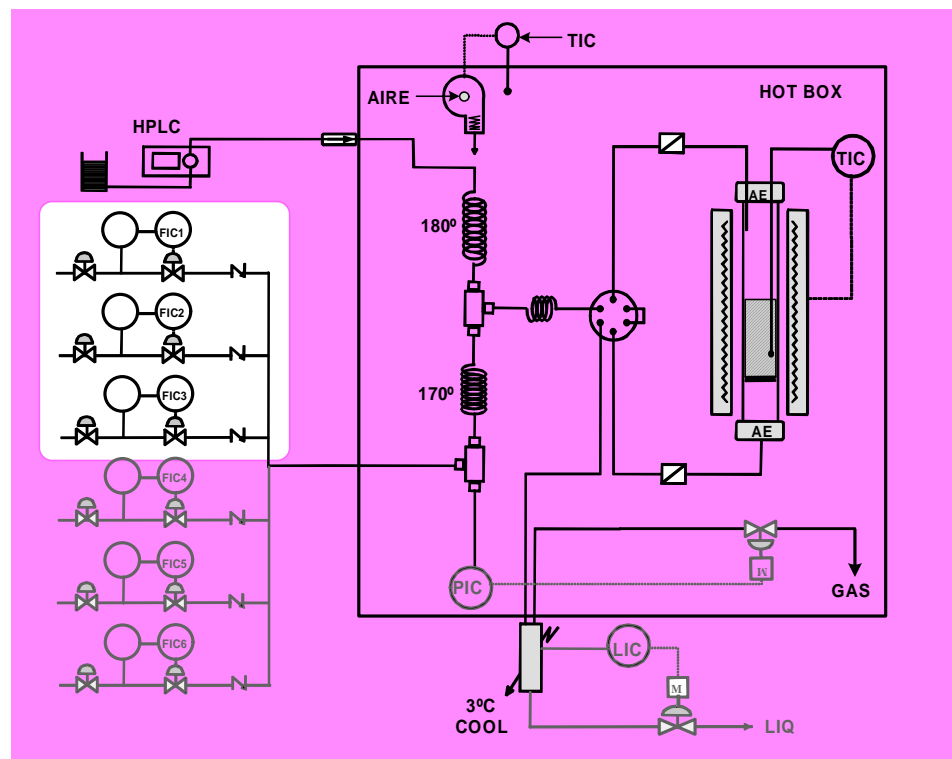
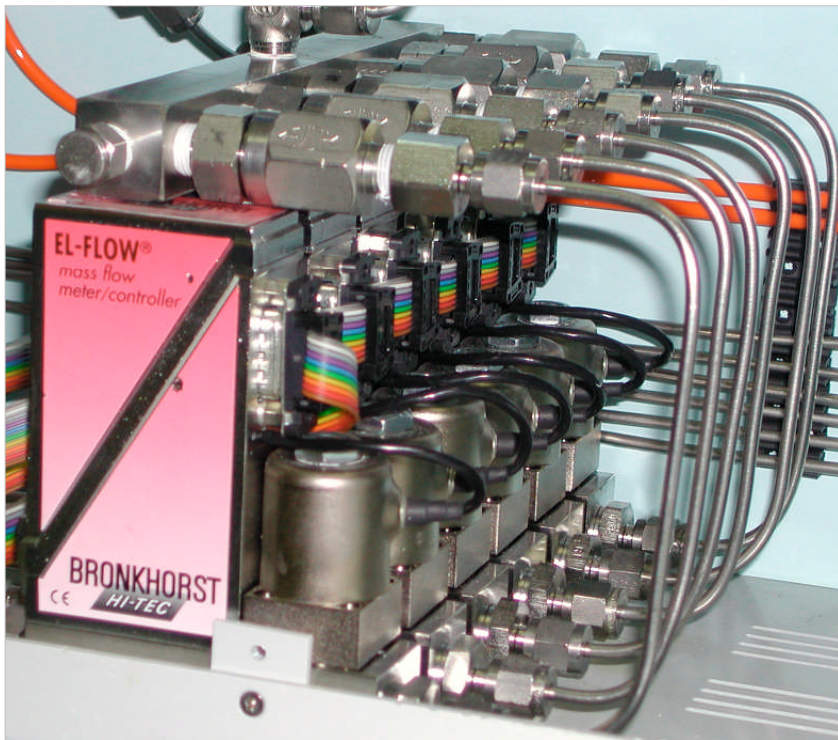


Mass Flow Controllers

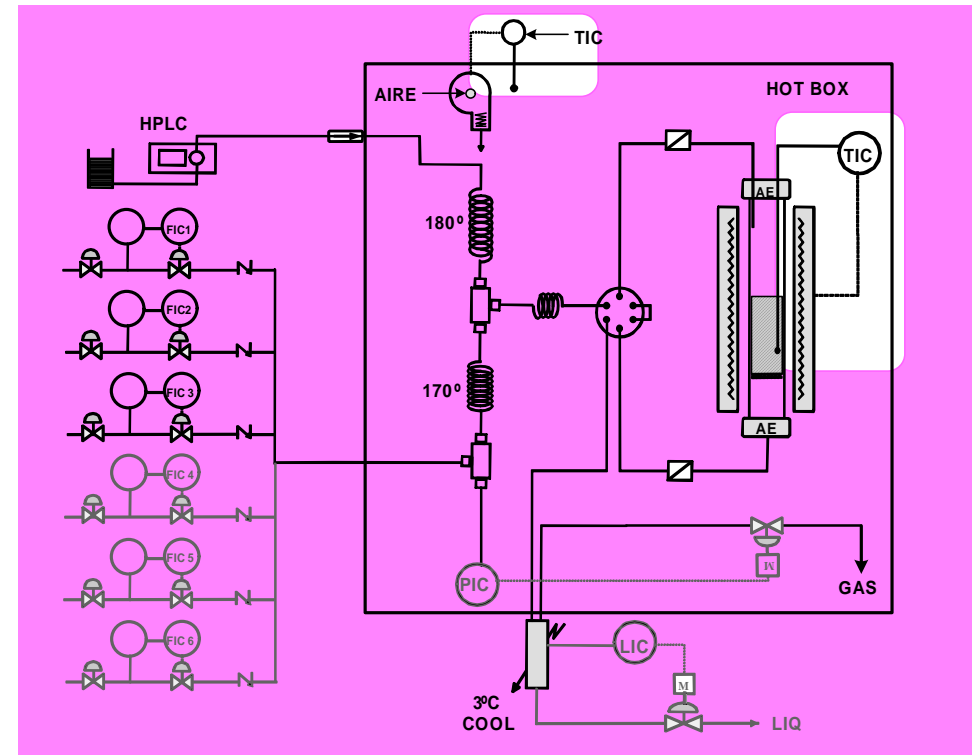
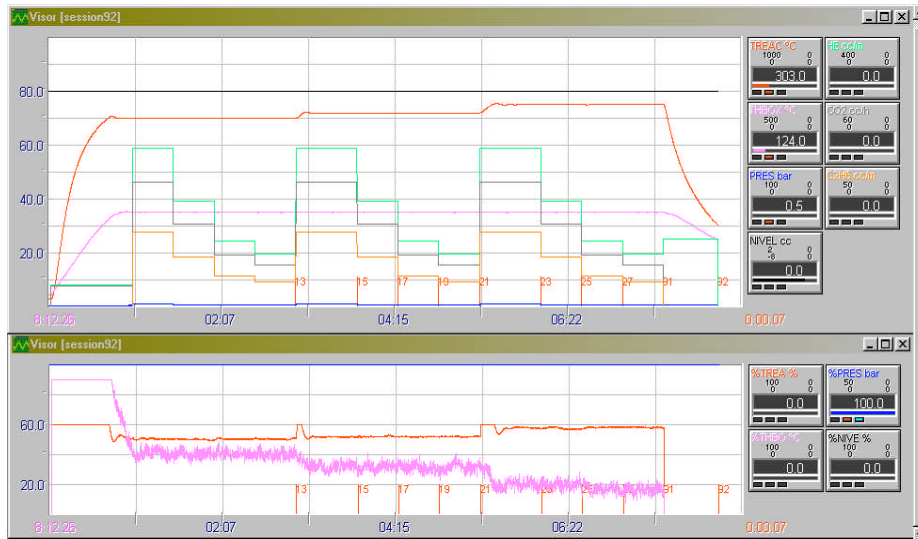
3 MFC's included in a Basic Unit

3 optional devices

Basic Unit Features



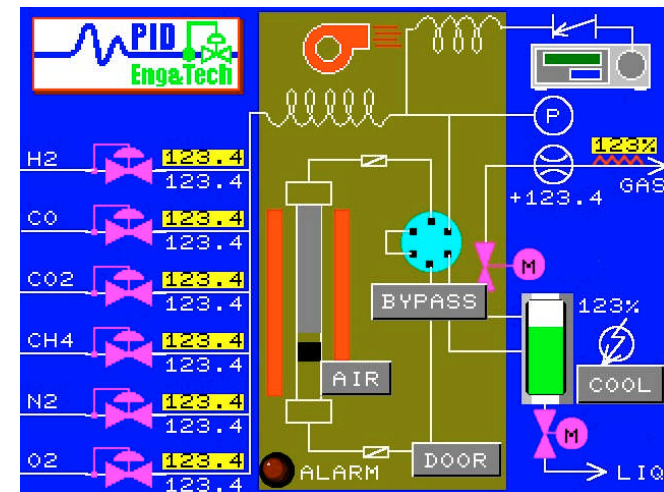
- ✓ Microprocessor for security integrated system
- ✓ 2 temperature control loops for reactor and hot box
- ✓ TOHO controllers
- ✓ Layout, fitting and valves in inox. 316L, very low dead volume
- ✓ Friendly supervision and distributed control software by PC Process@
- ✓ Remote control via Ethernet



Easy configuration: touch screen

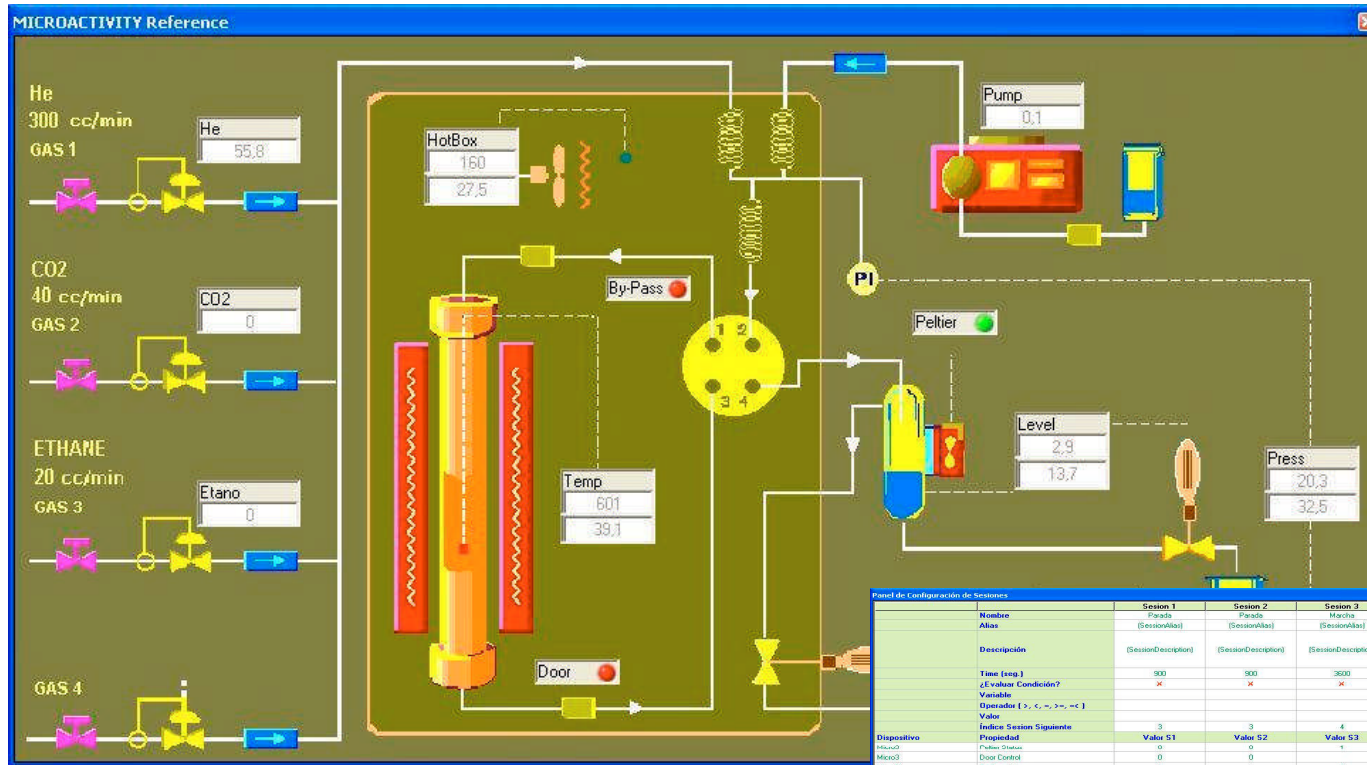
Friendly control of:

- ✓ Inlet mass flow control of the reactor
- ✓ Fast & easy configuration of temperature & pressure alarms
- ✓ Liquid level (if there is any) control in the liquid-gas separator
- ✓ Alarm system control
- ✓ Cooling control in the liquid-gas separator
- ✓ Bypass valve putting into operation
- ✓ Hot box door opening and closing
- ✓ Gas flow
- ✓ Cooler switch-on & switch-off
- ✓ Gas flow outlet data



Via Ethernet distributed control

Basic Unit Features



MicroactivityReference 10

Sesión Objetos Adquisición Sesiones Ventana Ayuda

Panel de Configuración de Sesiones	Sesion 1	Sesion 2	Sesion 3	Sesion 4	Sesion 5	Sesion 6	Sesion 7	Sesion 8	Sesion 9
Nombre	Parada	Parada	Mucha	1500	Fijos	1500	Flujo2	Final	Final
Descripción	(Sesion/Descripción)	(Sesion/Descripción)	(Sesion/Descripción)	(Sesion/Descripción)	(Sesion/Descripción)	(Sesion/Descripción)	(Sesion/Descripción)	(Sesion/Descripción)	(Sesion/Descripción)
Time (sec)	300	300	300	300	300	300	300	300	1800
¿Evaluear Condición?	X	X	X	X	X	X	X	X	X
Variable									
Operador (> < = > < =)									
Value									
Indice Sesión Siguiente	3	3	4	5	6	7	4	9	10
Dispositivo	Value S1	Value S2	Value S3	Value S4	Value S5	Value S6	Value S7	Value S8	Value S9
Micro0	0	0	1						
Micro2	0	0							1
Micro3	1	1							
Micro3	0	0	400	400	400	400	400	400	400
Micro3	0	0							
Micro3	0	0							
REACTOR TEMPERATURE	0	0	500	500	600				
REACTOR TEMPERATURE	Control mode								
REACTOR TEMPERATURE	[Output] - Output 1, manipulated value								
REACTOR TEMPERATURE	[P] - Output 1, Proportional band								
REACTOR TEMPERATURE	[I] - Integral time								
REACTOR TEMPERATURE	[D] - Derivative time								
REACTOR TEMPERATURE	Setting of H limit for Output 1 MV limiter								
REACTOR TEMPERATURE	Setting of L limit for Output 1 MV limiter								
REACTOR TEMPERATURE	[SetPoint] - Setting Value SV	0	0	160	160			0	
REACTOR TEMPERATURE	Control mode								
REACTOR TEMPERATURE	[Output] - Output 1, manipulated value								
REACTOR TEMPERATURE	[P] - Output 1, Proportional band								
REACTOR TEMPERATURE	[I] - Integral time								
REACTOR TEMPERATURE	[D] - Derivative time								
REACTOR TEMPERATURE	Setting of H limit for Output 1 MV limiter								
REACTOR TEMPERATURE	Setting of L limit for Output 1 MV limiter								
REACTOR TEMPERATURE	[SetPoint] - Setting Value SV	0	0	200	200				0
PRESSURE	SV H Limit								
PRESSURE	Control mode								
PRESSURE	[Output] - Output 1, manipulated value								
PRESSURE	[P] - Output 1, Proportional band								
PRESSURE	[I] - Integral time								
PRESSURE	[D] - Derivative time								
PRESSURE	Setting of H limit for Output 1 MV limiter								
PRESSURE	Setting of L limit for Output 1 MV limiter								
PRESSURE	[SetPoint] - Setting Value SV	0	0	30	30				0
LEVEL	SV H Limit								
LEVEL	Control mode								
LEVEL	[Output] - Output 1, manipulated value								
LEVEL	[P] - Output 1, Proportional band								
LEVEL	[I] - Integral time								
LEVEL	[D] - Derivative time								
LEVEL	Setting of H limit for Output 1 MV limiter								
LEVEL	Setting of L limit for Output 1 MV limiter								
PUMP	Set Value								

Aceptar Cancelar Ayuda



Extra Packages

HIGH PRESSURE:

Up to 100 bar with a controlled pressure by means of a micrometric valve.

PUMP:

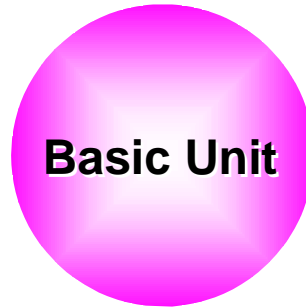
Gilson HPLC liquid pump, 0.01-5 ml/m, 400 bar. Digital communications.

LEVEL:

Liquid/gas separator with level control for operation with liquids and gases at high pressure in continuous mode, zero dead volume, based on micrometric valve servo-controlled and capacitive sensor level with approx. 0.3cm³ dead volume with precision +/- 0.1cm³ in level control. 100 bars. PID Eng&Tech design patent pending.

SCALE:

Scale for weighting liquid output in real time. Digital communications module.



Basic Unit



Extra Packages



Custom-made equipment

CEM:

MFC for liquids Hi-Tec Bronkhorst, pressurized liquid container, control and power supply. Only when HPLC pump is not possible. Includes a MFC for gas and a CEM module.

MFM:

MFM in exit gases line (for on-line and in real time supervising procedures).

CG:

Chromatographic heater line at 250°C maximum.

VICI:

2nd VICI valve, 6 ports, 2 positions. Only for special purposes.

REAC:

Construction materials of the reactor: Hastelloy C, Inconel, Monel, Titanium, and special dimensions (id= 5.2 or 13.1 mm).

High Pressure

**Up to 100 bar with a controlled pressure by means of a micrometric valve
± 0.1 bar precision**

Problems:

- = Extremely low values of Cv (pass rate)
- = A very high “rangeability” necessity
- = Presence of liquids in its vaporous state

$$C_v \approx \frac{Q}{P} \cdot \sqrt{\rho}$$

State of art:

Control valve positioned pneumatically or electrically

↪ Very low “rangeability”

Diaphragm and spring automatic valve, Tescom type

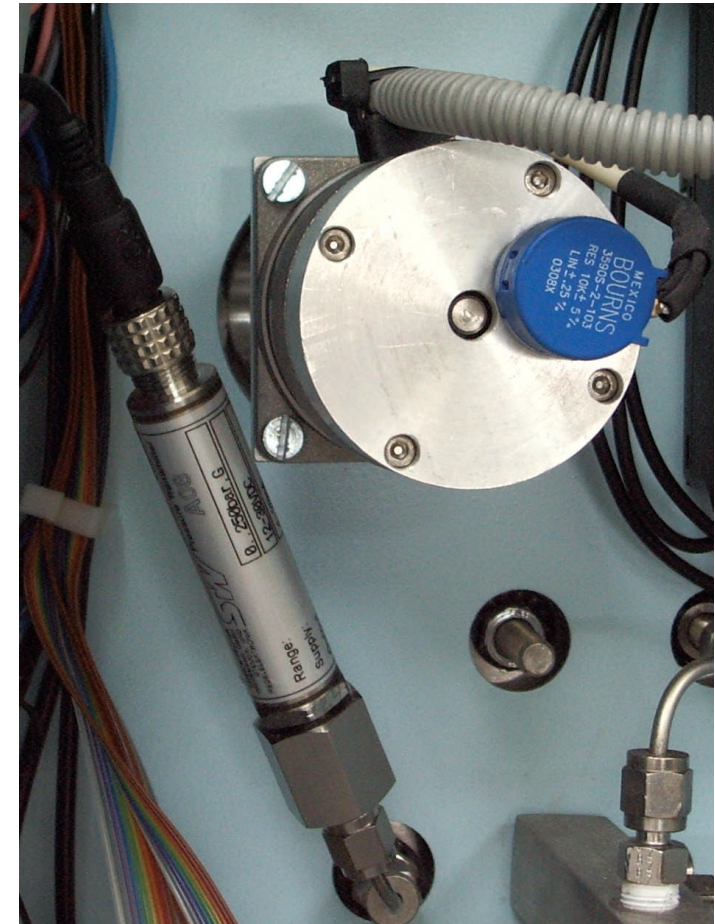
↪ High dead volume, pulsating flow, little stability

Electronic controllers, MFC type

↪ Bad results in presence of condensed and dirty products

Design based on step by step motor

- precision ± 2°
- H₂ flows of 20 cm³/min at 90 bar
 - high speed of positioning
 - friendly configuration



Pressure control performances

Obtained results:

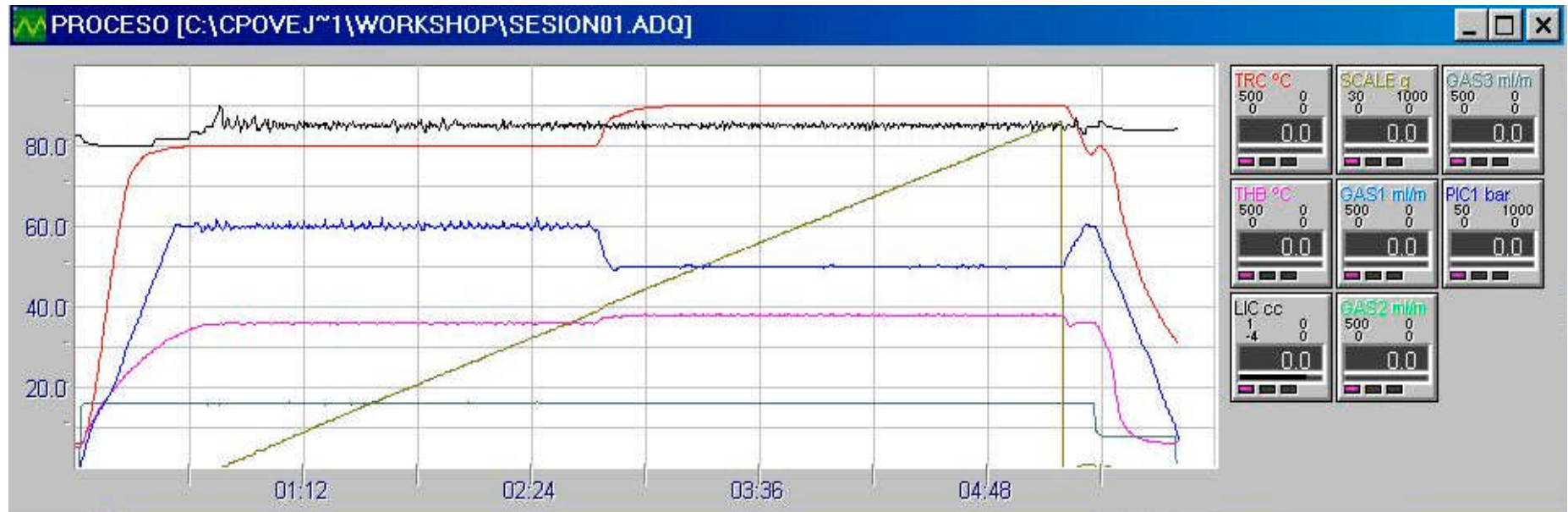
- ✓ Application to systems with gas flows including 10cm³/min of H₂@90 bar
- ✓ Admits heating and withstands liquid pass

F= 60 cm³/min

F = 0,1 gr/min water

T_{hot box} = 180°C

P = 25/30 bar



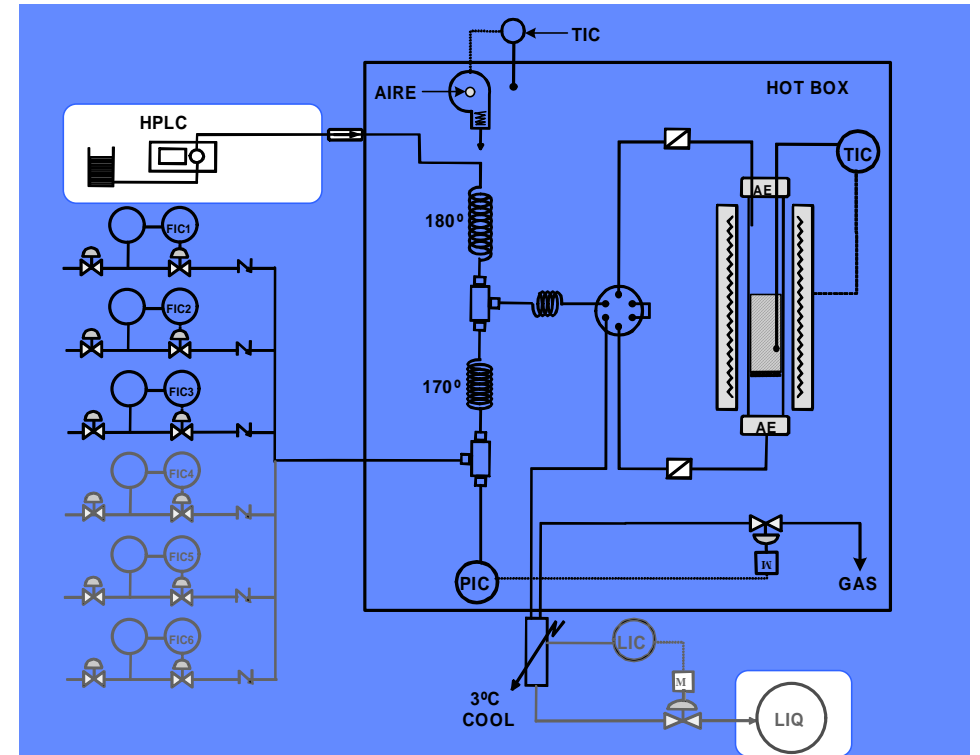
- Level in the separator (dead volume = 0,3 cm³)
- Pressure
- Liquid accumulated in outlet

Patent n° P200401348:

”Servopositioner for micro regulation valve”

Gilson Pump (scale is required)

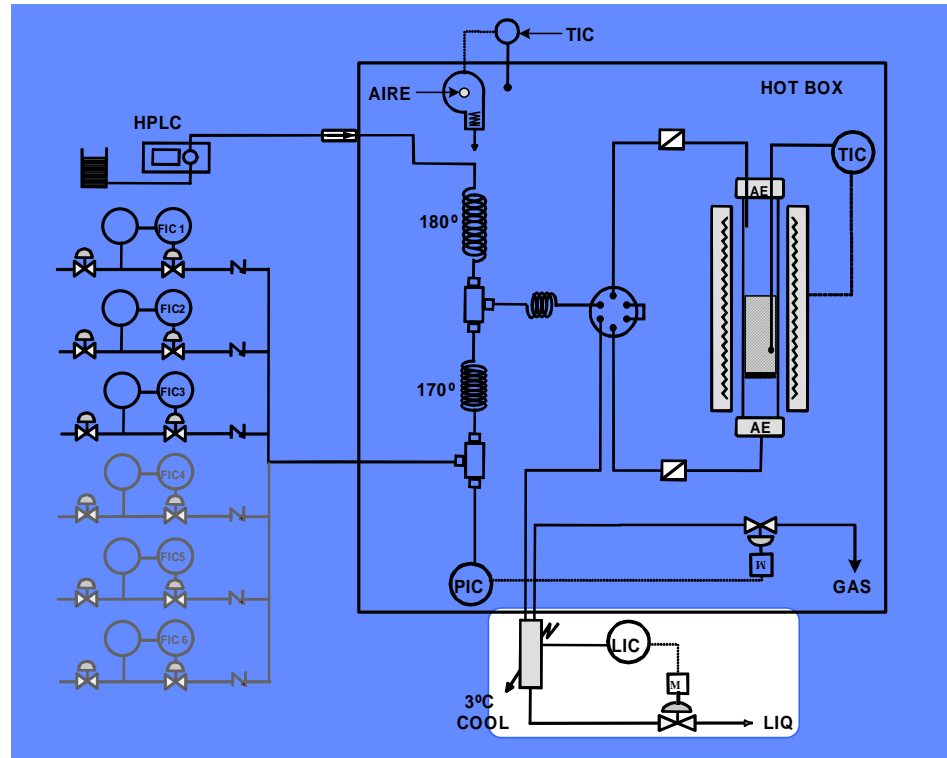
HPLC Gilson Pump, 400 bar, 0.01-5 ml/min



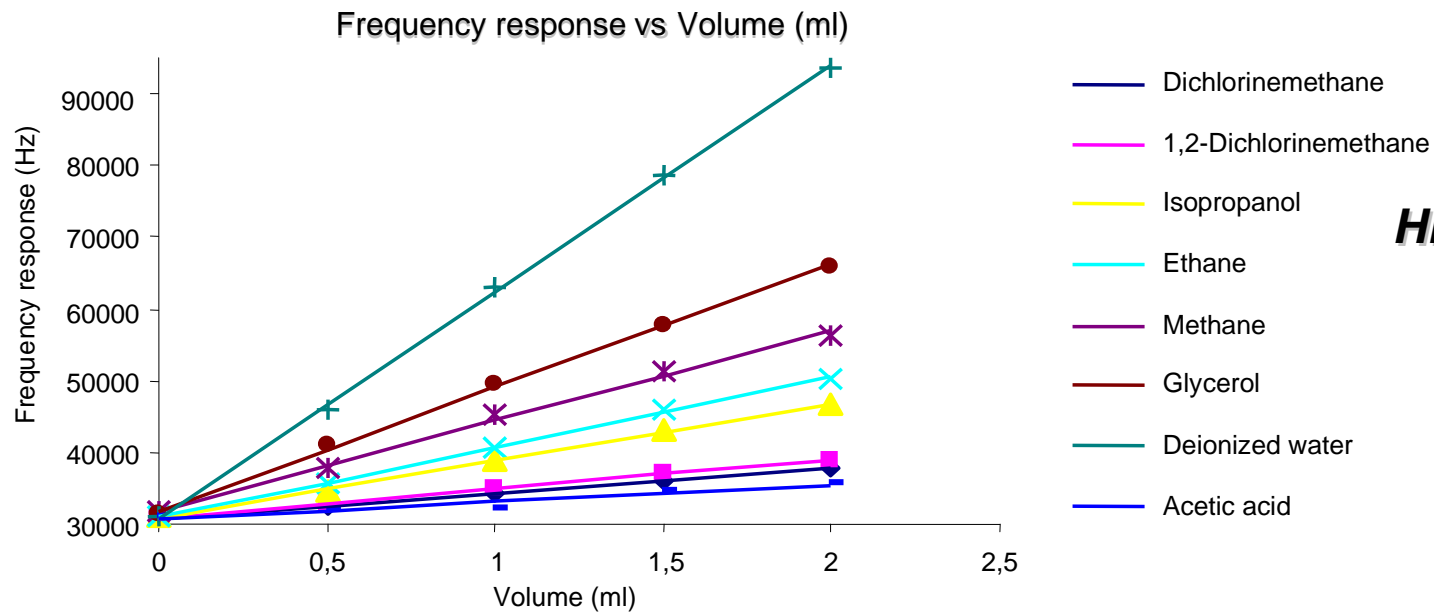
Scale for weighting liquid output in real time. Digital communications module.

Level Control

Liquid-gas separator with level control for operation with liquids and gases in continuous mode, zero dead volume, based on micrometric servocontrolled valve and capacitive sensor level with approximately 0.3 cm³ dead volume and ± 0.1cm³ precision in level control. 100 bars. PID Eng&Tech design patent pending.

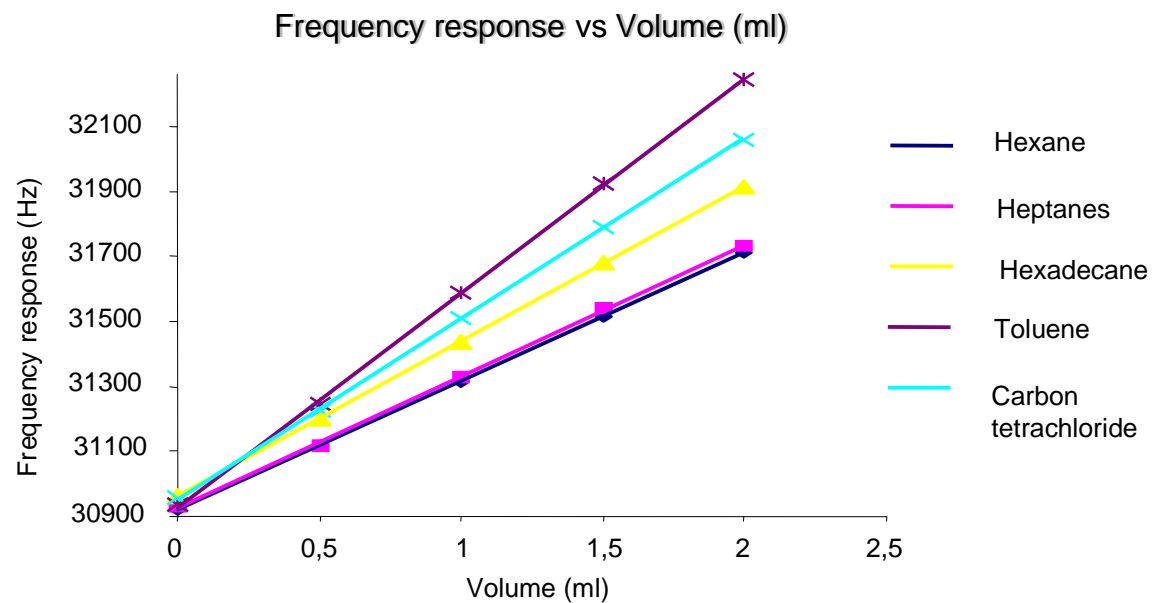


Level sensor response



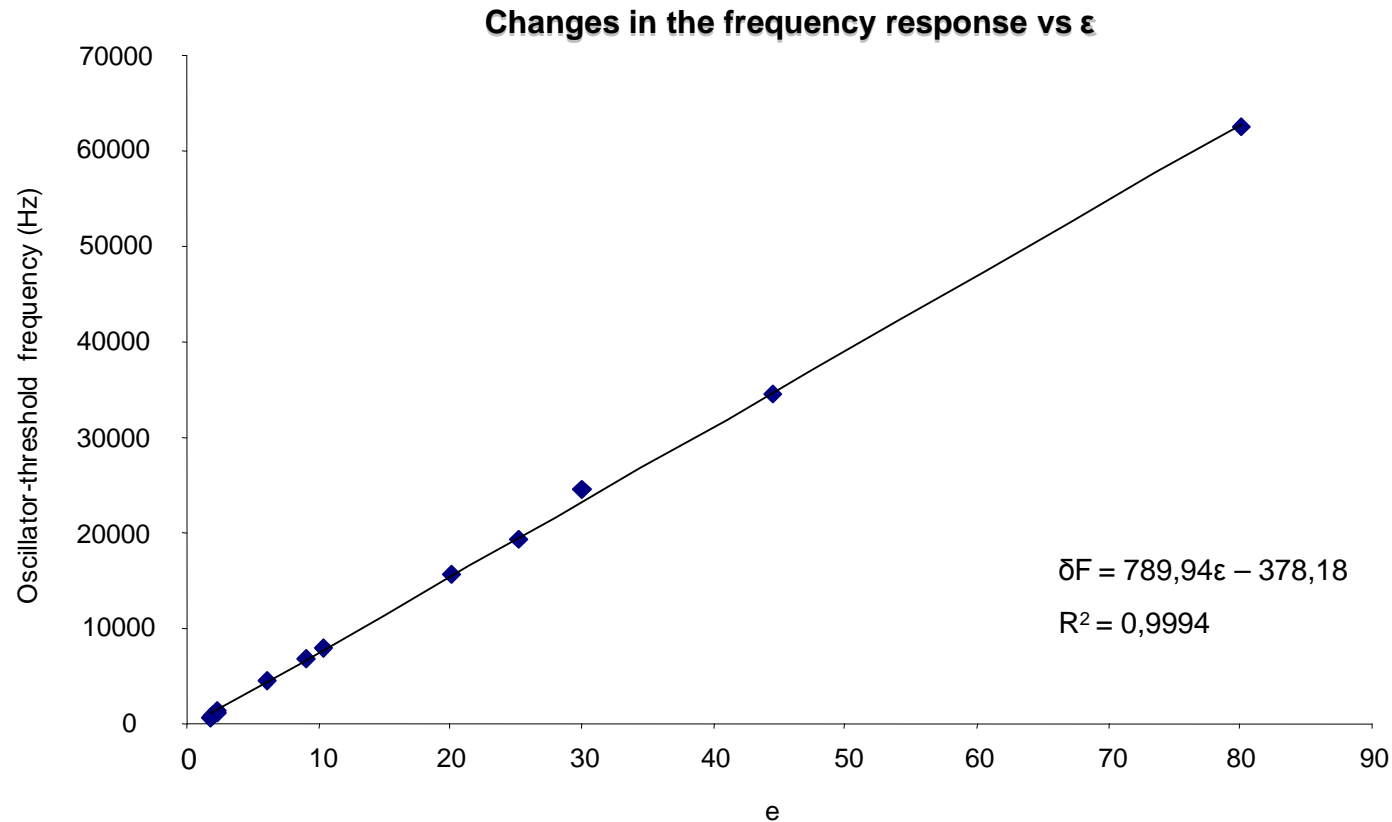
High sensibility response
 $\epsilon = 3$ a 80 :
 $\delta f = 20.000$ a 5000Hz

Low sensibility response
 $\epsilon = 1,1$ a 3 :
 $\delta f = 800$ a 1.500 Hz



Level sensor calibration

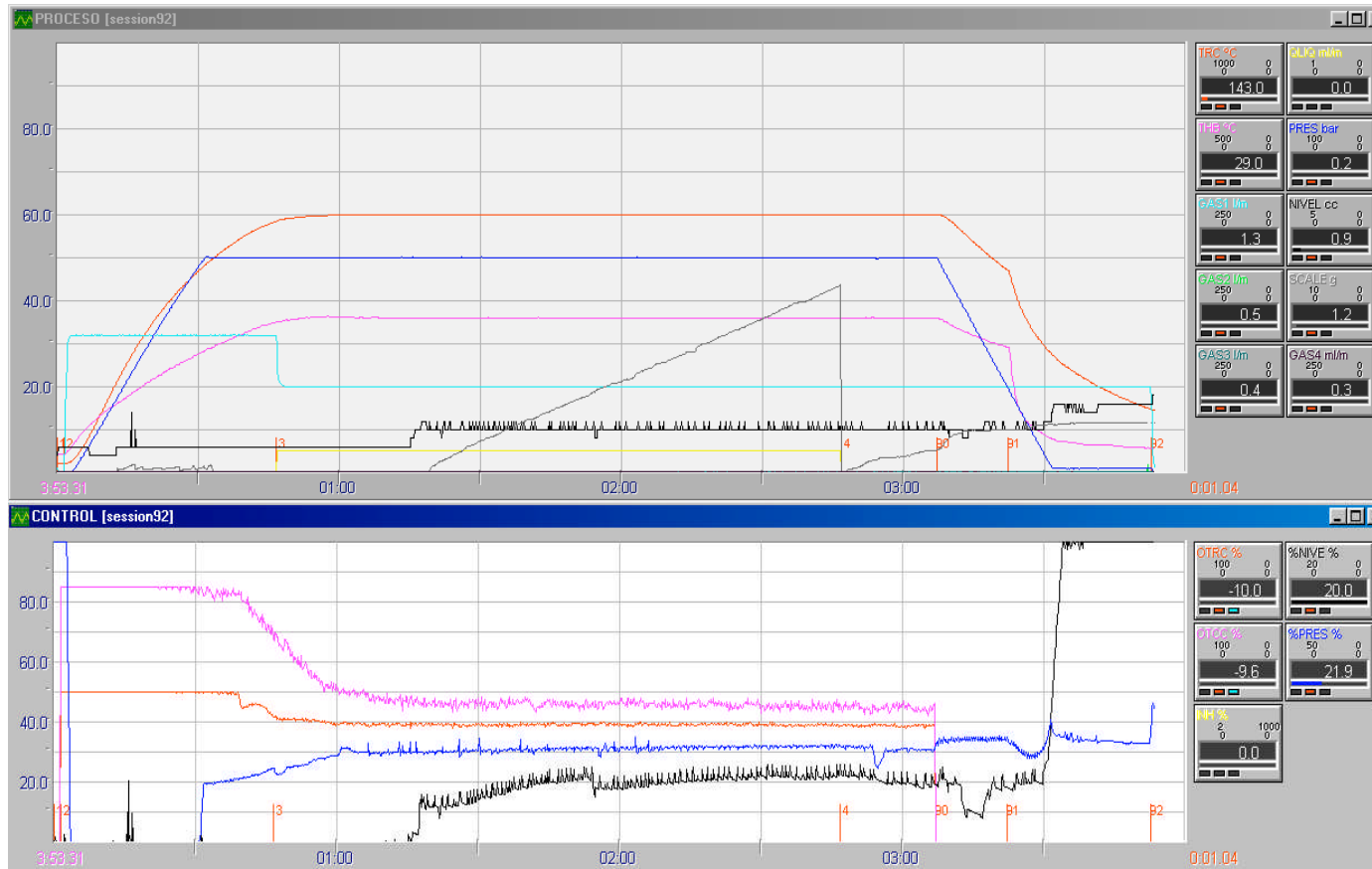
Level sensor response to a dielectric constant



These results permit foresee the sensor response in presence of any liquid, as a function of its dielectric constant.

Level sensor performances

Obtained results



P = 50 bar

F = 50 cm³/min H₂

Q = 0,02 gr/min H₂O

Patent n° P200401349:

“Capacitive level sensor for reduced volume systems”

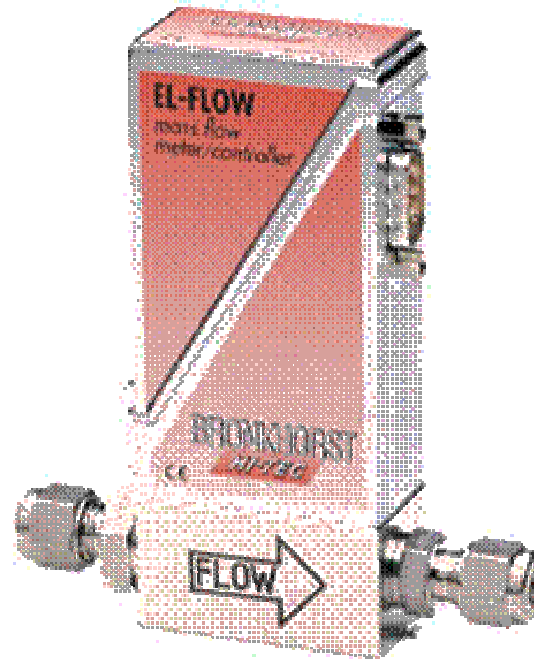
Others



MFC for liquids Hi-Tec Bronkhorst, pressurized liquid container, control and power supply. Only when HPLC pump is not possible. Includes a MFC for gas and a CEM module.



MFM in exit gases line (for on-line and in real time supervising procedures).



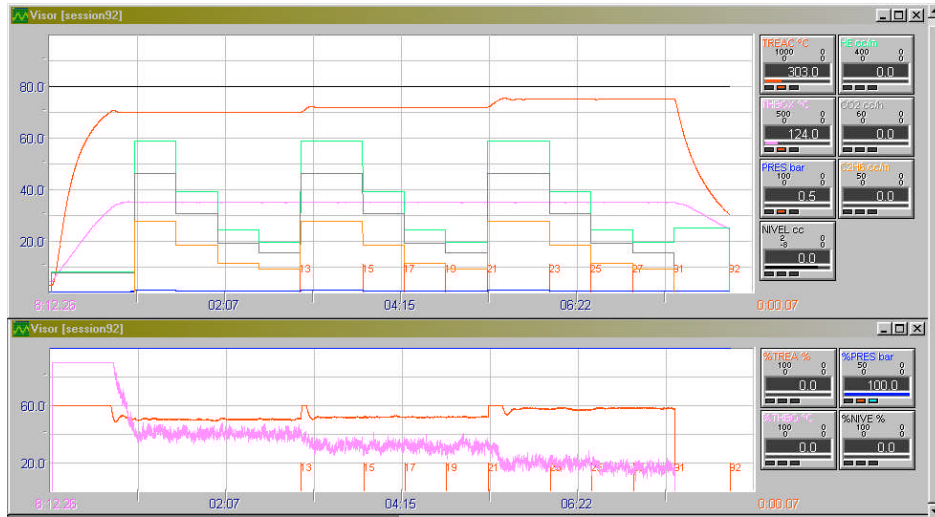
Construction materials of the reactor:

- ✓ Hastelloy C
- ✓ Inconel
- ✓ Monel
- ✓ Titanium
- ✓ Special dimensions (id= 5.2 or 13.1 mm).

2nd VICI valve, 6 ports, 2 positions. Only for special purposes.

Automation Possibilities

Application



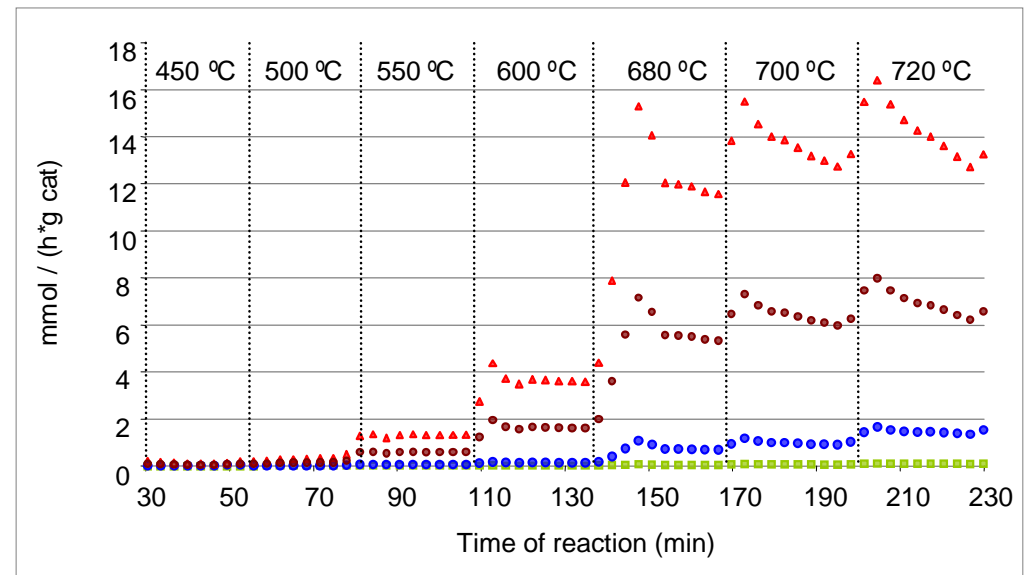
**Heterogeneous reaction -
CaCeO₂-FD catalyst**
C₂H₆:CO₂:He = 10:20:170 ml/min

The use of the rapid analysis technique (MicroCG) allows to have the results at our disposal never before obtained in this type of equipment.



High speed CG analysis

- CH₄
- Ethylene
- ▲ CO
- Water



Advantages

- ✓ The experimental reproducibility, consequence of the system stability, permits to use this equipment as a “reference unit” between laboratories.
- ✓ High degree of automation
- ✓ Quasi-zero dead volume
- ✓ ± 0.1 bar precision in pressure control
- ✓ $\pm 1^{\circ}\text{C}$ precision in temperature control
- ✓ 0.3cm^3 dead volume with $\pm 0.1\text{cm}^3$ precision in level control

About us

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