

# **PROCESS INTEGRAL DEVELOPMENT ENG&TECH**

## **MICROACTIVITY-REFERENCE CATALYTIC REACTOR**

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## COMPANY PROFILE

PID Eng&Tech was born in 2003 as a **spin-off company of Spanish Council for Scientific Research (CSIC)** with 20 years of previous accumulated experience. The Process Control Group of the Institute of Catalysis and Petrochemistry (ICP) of the CSIC have devoted its activity since 1989 to the development of technologies dedicated to improving design, construction and operation mode of laboratory-scale reactors and micro-scale pilot plants, and their data acquisition, supervisory and control systems. **PID Eng&Tech** was awarded with the prize of the "Contest of Ideas for Spin-off companies of Researchers", granted by the Universities and Research General Management.



His Royal Highness, the Prince of Spain,  
Don Felipe de Borbón, at the inauguration

PID Eng&Tech staff is formed by experts with multidisciplinary backgrounds in chemical engineering, electronics, automated and software engineering. Launched in 2003, with a head office in the Scientific Park of Madrid, Process Integral Development Eng&Tech was able to put into practice all the experience, technological development and innovations achieved over many years of working in engineering field. From 2005, PID Eng&Tech's office and laboratories are situated in Colmenar Viejo (Madrid), near the Regional Park of Hoyo de Manzanares.

PID Eng&Tech primary **target market** are universities and research centres, both private and public sectors, which fulfil their research activities within the fields of basic chemistry, petrochemistry, environment, catalysis, agrochemistry and food technology, supercritical fluid extraction and new energies.

In addition, the company has a **worldwide net of distributors** that cover the following countries and areas: The United States of America, Canada and Mexico, Germany, Switzerland and Austria, Benelux countries, Belgium, Italy, the Netherlands, Egypt, India, Saudi Arabia and the Middle East.



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## MICROACTIVITY-REFERENCE CATALYTIC REACTOR

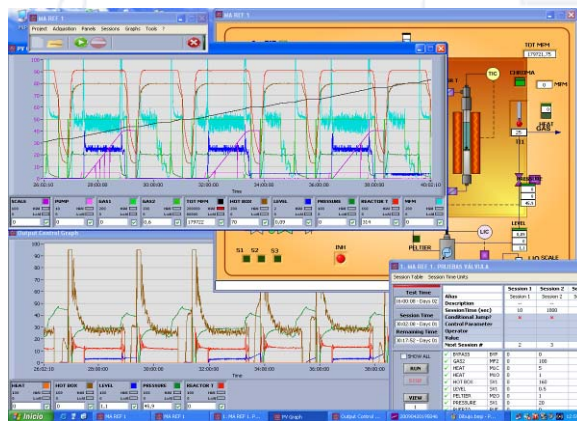
### The reference in modular catalytic reactor...

The **Microactivity-Reference** reactor (WO-2006008328 / EP-1757930 / US-2008063565) is probably the most advanced worldwide modular laboratory system for measurement of catalytic activity and selectivity. PID Eng&Tech, is a worldwide leading company at sector of Microreactors for Catalytic studies. This instrument has been developed as a standard unit that can be adapted to whatever performance is needed for catalytic testing through different configurations and options.

The **Microactivity-Reference** is a compact reactor that is completely automated. It is equipped with cutting-edge process control technology in the market. This enables the user to program a series of experiments from the computer, even on the network, and obtain real-time results with the highest degree of reproducibility and accuracy.

This equipment has been designed to save time and resources at both, catalyst development stage and factory report process during catalyst screening.

Originally designed by and for researchers at Instituto de Catálisis y Petroleoquímica of CSIC, Spain, incorporating 20 years of continuous feedback from the most prestigious Laboratory Researchers, the **Microactivity-Reference** has become an international reference with more than 170 units worldwide studying all types of catalytic reactions. It is backed by its reliability, versatility, operating simplicity and minimal maintenance.



The patented control systems have been specifically developed for this equipment. They account for operating at the microscale. There are no systems with similar characteristics in the market for working with microflows.

**Microactivity-Reference** is a universal piece of equipment that provides great versatility. It operates with flows that range from tens of ml/min to even liters/min, and pressures ranging vacuum to 100 bar (with the same pressure control valve). The reaction temperature ranges from room temperature to 100°C (using special material reactors).

**The equipments of PID Eng & Tech are certified according to European standards PED97/23/EC (pressure equipment), EN61326 EMC (Electromagnetic Compatibility) and 73/23/EEC (LVD-Electrical Safety).**



## MICROACTIVITY-REFERENCE

### the Basic unit...

**The basic unit**, the simplest one, designed for working at atmospheric pressure, includes a mixing gases unit based on MFCs with three units for the standard equipment, a tubular reactor SS316 with a 9 mm inner diameter, on the inside of a radiating oven capable of reaching 1000°C with very low thermal inertia, a reactor bypass enables isolating it while the feed analysis is being performed, and a liquid-gas separator (cooled via thermoelectric effect) at the outflow separates liquid products. The gases are fed to the analysis system via a temperature transfer line of up to 300°C.



All components that comprise the equipment are housed inside of a **hot box**. This makes it possible to keep the incoming and outgoing product feed lines at a temperature of up to 190°C. This prevents volatile products condensation, thus maintaining the entire process's path insulated, This gives stability to the flows, and prevents the presence of cold points.

The basic equipment includes the hot box, the control systems and a sophisticated safety system; which makes it a piece of equipment with an extraordinary level of reliability. Furthermore, this system may be configured by the user via a local touch-screen, independent of the computer according to a distributed control philosophy. It thus gives maximum priority to the safety of the equipment itself and its users. Finally, communication via Ethernet between the equipment and the controlling computer makes it possible to program experimental formulas, acquire system data and remote control from any workstation.

### configuring the unit...

**(P) High pressure:** This option adds a pressure control loop to the basic unit; it enables working up to 100 bar with an accuracy of 0.1bar. Pressure control is based on a servo motorized micrometric regulation valve (WO-2006021603 / EP-1775504 / US-2007241296) that gives it maximum stability in pressure control thus minimizing the piston flow pulse effect of the stream that flows across the catalyst bed. This system, developed especially for the Microactivity-Reference, deliver excellent reproducibility in experimental data.

**(G) Liquid Feed:** This option enables a liquid feed via an HPLC pump, for working in either the liquid or steam phase, and operating at pressures of up to 100 bar. The pump is installed with the necessary accessories to ensure a continuous flow that is precise and without pulse, even when the feed is to be evaporated.

**(L1) High pressure liquid-gas separator:** A **microvolume** system for liquid-gas separation at a reactor's outflow that operates at high pressure (WO-2006021604 / EP-1757911 / US-2007238753) is perhaps the Microactivity-Reference main contribution to quick evolution and development in experimentation in the field of catalysis. With a dead volume less than 0.5 ml, this system enables real time separation of condensables, thus making it possible to learn the composition of the liquid products obtained during the first reaction minutes. This makes it possible to study reaction kinetics and catalyst deactivation since the condensate during reaction is representative of the last few minutes.

**(L2) High pressure liquid-liquid-gas separator:** This is the latest evolution with which PID Eng&Tech contributes the advance in catalytic reaction control. This configuration option is an evolution of the aforementioned liquid-gas separating system. It enables separation of two non-miscible liquid fractions, e.g. for use in Fisher-Tropsch reactions (**GTL**). The high-pressure separator has no appreciable dead volume and produces three differentiated phases in real time: the condensable hydrocarbon, water and the gaseous fraction with lighter hydrocarbons.





# MICROACTIVITY-REFERENCE

## and the options...

Starting with a basic unit, and through the configuration options, the Microactivity-Reference unit can be customized for working at high pressure with the possibility of feeding liquids, or with two high-pressure liquid-gas separating models. The different **options** adapt the Microactivity-Reference reactor to match any project requirement. The know-how acquired during the execution of more than 170 installations enables us to configure a unit tailored to each user and process's specific needs. Some representative options are listed below:

- Optional reactors of 5, 13 and 17 mm in inner diameter, in Hastelloy C276, Hastelloy X, Inconel 600, Inconel 625, SS310 materials and others. Allowing adapting the unit to the specific characteristics of reaction type, temperature, pressure and reacton feed.
- The system can incorporate up to 6 MFCs without changing the original configuration. The outstream can be measured by a mass flow meter (MFM) installed at the gas exhaust or weight scales (both for the feed vessel and for the liquid outflow); thus, it is possible to monitor the system evolution and mass balance.
- In systems with high-viscosity liquids, e.g. an HVGO, the HPLC pump, feed vessel and the hot box's external path is heated at 80°C.
- A second and even a third six-port valve switches between up/down flow through the catalyst bed or, for instance, be used for bypassing the L/G separator, in the event the unit is used for reactions where there is no liquid present.
- Other options enable additional evaporators, separator temperature control, ovens with special dimensions, pressures above 100 bar, more than one liquid feed pump, fluidized beds, two or more serial units to be installed with different operating pressures, among others. It is also possible to prepare complete laboratories with 8 or 16 Microactivity-Reference units with – adaptable configuration.

PID Eng&Tech Microactivity Reactors last 6 years worldwide growth is observed in table below:

COUNTRY / YEAR	PID Eng&Tech MICROACTIVITY-Reference Reactors 1991-2009																	TOTAL	
	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007		2008
TOTALS	6	5	5	8	7	6	5	5	6	6	7	7	6	10	14	19	21	25	168
Europe																			135
Spain	6	5	4	7	6	6	4	4	6	5	6	5	5	8	6	8	11	9	111
Italy							1									1			2
Greece				1	1											1			3
France																	1		1
Germany														1			1		2
Romania																		1	1
Sweden																1		1	2
Belgium												2						1	3
Cyprus										1				1				1	3
Norway																	1		1
Finland																	1	2	3
Austria																	1		1
Slovenia														1					1
Poland																1			1
North America																			8
USA														1	2	1	2	2	8
South America																			5
Mexico			1																1
Argentina															1				1
Brazil															1	1		1	3
Asia																			7
Russia															1		2	1	4
Viet nam																		1	1
India															1	1			2
Persian Gulf																			13
Saudi Arabia								1		1			1			1	1	4	9
Jordan																		1	1
Oman																1			1
Egypt																2			2



## MICROACTIVITY-REFERENCE WORLDWIDE USERS

<i>(more information on web site)</i>			
CUSTOMER	COMPANY / CENTER	COUNTRY	UNITS
Dr. A. Aguayo / Dr. J. M. Arandes	Universidad del País Vasco		6
Dr. A. Jiménez / Dr. Blanco	Universidad de Malaga		2
Dr. A. Pintar	National Institute of Chemistry	Slovenia	1
Dra. L. Daza	Instituto de Catálisis y Petroleoquímica / CIEMAT		7
Dr. A. Selvanathan / Dr. Debashis	SABIC Research & Technology Badadora	India	2
Dr. C. Jiménez	Universidad de Córdoba		1
Dr. Costas N. Costa	University of Cyprus	Cyprus	2
Dr. E. Gaigneaux / P. Ruiz	Catholic University of Louvain	Belgium	2
Dra. Gomez / Dr. Gilarranz / Dr. Casas	Universidad Autónoma de Madrid		4
Dr. Israel E. Wachs	Lehigh University	USA	1
Dr. J. A. Odriozola / Dr. Ollero / Dr. Munuera	Universidad de Sevilla		5
Dr. J. Cambra	Universidad del País Vasco		5
Dr. K. Sapag	Universidad Nacional de San Luis	Argentina	1
Dr. J. L. G <sup>a</sup> . Fierro	Instituto de Catálisis y Petroleoquímica		5
Dr. T. Leonnides	ICE/HT FORTH	Greece	1
Dr. J. M. Pintado / Dr. Mtnez de la Osa	Universidad de Cádiz		2
Dr. J. Pis	Instituto Nacional de Carbón		2
Dr. Khalid. S. Karim	SABIC R&D Center Riyadh, Saudi Arabia	Saudi Arabia	2
Dr. Khalid El Yahyaoui	SABIC R&D Center Riyadh, Saudi Arabia	Saudi Arabia	1
Dr. Tony Joseph	SABIC R&D Center Riyadh, Saudi Arabia	Saudi Arabia	2
Dr. L. Gandía / Dr. A. Gil	Universidad de Navarra		3
Dr. L. Gora	Institute of Polish Academy Science	Poland	1
Dr. M. Montes	Universidad del País Vasco		3
Dr. J.M. Campelo / Dr. M. Rubio	Universidad de Códoba		3
Dr. Yahia Alhamed	King Abdulaziz University.	Saudi Arabia	1
Mrs. Olga Golivkina – NEOLAB	JSC Moscow Oil Refinery	Russia	1
Dr. Miguel A. González	Universidad de Tarragona		1
Dr. Miguel Ángel Bañares	Instituto de Catálisis y Petroleoquímica		1
Dr Hoang Anh Tuan	Saigon Instrumentation Join Stock Company	Vietnam	1
Dr. Narcis Homs	Universidad de Barcelona		2
Dr Lachezar Angelov Petrov	King Abdulaziz University	Saudi Arabia	1
Dr. Van Grieken / Dra. Carrero / Dr. Serrano	Universidad Rey Juan Carlos		4
Dr. S. Al-Zahrani	King Saud University	Saudi Arabia	1
Dr. T. Cordero / Dr. L. Alemany	Universidad de Málaga		2
Dr. V. Augugliaro	Universidad de Palermo	Italy	1
Dr. V. Cortés / Dr. J. Prieto	Instituto de Catálisis y Petroleoquímica		2
Dr Sippola Vaino	NESTE OIL	Finland	3
Dr. V. V. Fadeev	YUKOS R&D Centre	Russia	1
Dr. A. Fernández	Petresa (Cepsa)		1
Mr. Brook March	Altamira Instruments	USA	1
Dr. David Coker	RTI	USA	1
Dr. E. Caló / Mrs. G. Monteleone	Enea-Cassacia	Italy	1
Dr. Gerd Rabenstein	University of Graz	Austria	1
Dra. Viviane Schwartz	Oak Ridge National Laboratory	USA	1
Dr. G. Ovejero	Universidad Complutense de Madrid		1
Dr. Victor Teixeira da Silva	Fundação COPPETEC	Brazil	1
Dr Gregg Deluga	General Electric	USA	2
Dra. Ana Gayubo	Universidad del País Vasco		1
Dr. De Chen	Norwegian University of Science & Technology	Norway	1
Dr. Kuman	Noisk	Russia	1
Dr. Jean Thivolle	Centre National de la Recherche Scientifique	France	1
Dr. J.I. Gutierrez Ortiz	Universidad País Vasco		1
Dr. Matthias Friedrich	Max Planck Institute	Germany	1
Dr. Sven Jaras	KTH University Division of Chemical Technology	Sweden	2
Dr. Ademir Donizete	Elekeiroz	Brazil	1
Dr. J. M <sup>a</sup> Arauzo	Universidad de Zaragoza		1
Dr. Valer Almasán	ITIM – CLUJ NOPOCA	Rumania	1
Dr. Mark Tsodikov	Russian Academy of Sciences	Russia	1
Dr. Maged / Prof. Fathy	El Ghonemy Group.	Egypt	2
Dr. J. A. Menendez Diaz	Instituto del Carbón		1
Dr. Philippe Barthe	Corning European Technology Center	France	1
Dra. Mónica Antunes.	Universidad Rio de Janeiro	Brazil	1
Dr. Bjorn	NYNAS	Sweden	1
Mr. Raúl Alonso Sanz	DIGEMA		1



# SCIENTIFIC PUBLICATIONS USING MA-REF

Catalysts for Chlorinated VOCs Abatement: Multiple Effects of Water on the Activity of V Based Catalysts for the Combustion of Chlorobenzene.  
F. Bertinchamps, A. Attianese, M. M. Mestdagh, Eric M. Gaigneaux  
Catalysis Today, 112 (2006) 165-168

Surface modifications of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and SnO<sub>2</sub> supports by titania grafting and their influence in the catalytic combustion of methane.  
C. Mateos, S.R.G. Carrazan, P. Ruiz  
Catalysis Today, 104, Issue 1-4 (2006) 107-111

Influence of the solid state properties of Pd/Mo<sub>x</sub>(M=Ti, Al) catalysts in catalytic combustion of methane.  
S.R. G. Carrazan, R. Mateos, V. Rives, P. Ruiz  
Catalysis Today, 104, Issue 1-4 (2006) 161-164

Origin of transient species present on the surface of a PdO/γ-Al<sub>2</sub>O<sub>3</sub> catalyst during the methane combustion reaction.  
O. Demoulin, M. Navez, P. Ruiz  
Catalysis Today, 104, Issue 1-4 (2006) 153-156

Positive effect of NO<sub>x</sub> on the performances of VO<sub>x</sub>/TiO<sub>2</sub> based catalysts in the total oxidation abatement of chlorobenzene.  
F. Bertinchamps, M. Treinen, N. Blangenois, E. Mariage, E. M. Gaigneaux  
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Modulation of selective sites by introduction of N<sub>2</sub>O, CO<sub>2</sub> and H<sub>2</sub> as gas promoters in the feed during oxidation reactions.  
O. Demoulin, I. Seunier, F. Dury, M. Navez, R. Rachwalik, B. Sulikowski, S.R. Gonzalez Carrazan, E.M. Gaigneaux, P. Ruiz  
Catalysis Today 99 (2005) 217-226

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O. Demoulin, G. Rupprechter, I. Seunier, B. Le Clef, M. Navez, P. Ruiz  
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O. Demoulin, M. Navez, F. Gracia, E. Wolf, P. Ruiz  
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Modification of Active Catalytic Sites with N<sub>2</sub>O and CO<sub>2</sub> as Gas Promoters during Oxidation Reactions.  
O. Demoulin, F. Dury, M. Navez, E. Gaigneaux, P. Ruiz  
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Understanding the Activation Mechanism Induced by NO<sub>x</sub> on the Performances of VO<sub>x</sub>/Ti Catalysts in the Total Oxidation of Chlorinated VOCs.  
F. Bertinchamps, M. Treinen, P. Eloy, A.-M. Dos Santos, M. Mestdagh, Eric M. Gaigneaux  
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Systematic Investigation of Supported Transition Metal Oxide Based Formulations for Catalytic Oxidative Elimination of (Chloro-) Aromatics. Part I: Identification of the Optimal Main Active Phases and Supports.  
Part II: Influence of the Nature and Addition Protocol of Secondary Phases to VO<sub>x</sub>/TiO<sub>2</sub>  
F. Bertinchamps, C. Grégoire, E. M. Gaigneaux.  
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The oxidizing role of CO<sub>2</sub> at mild temperature on ceria based catalysts.  
O. Demoulin, B. M. Navez, J. L. Mugabo, P. Ruiz  
Applied Catalysis B: Environmental, in press.

Total Combustion of Methane on Pd/γ-Al<sub>2</sub>O<sub>3</sub> Based Catalysts Deposited On FeCrAlloy Fibers by Dip-Coating: Effect Of Pt Addition. A. Maione, F. André, P. Ruiz  
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O. Demoulin, B. M. Navez, P. Ruiz  
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Comparison of Pt and Pd alumina-supported catalysts towards the presence of H<sub>2</sub> in the feed in the catalytic combustion of methane  
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H<sub>2</sub>-assisted catalytic combustion of methane on a Pd/γ-Al<sub>2</sub>O<sub>3</sub>  
O. Demoulin, B. M. Navez, P. Ruiz

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The activation of a Pd/γ-Al<sub>2</sub>O<sub>3</sub> catalyst during methane combustion  
O. Demoulin, B. M. Navez, P. Ruiz  
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The oxidizing role of CO<sub>2</sub> at mild temperature on ceria-based catalysts  
O. Demoulin, B. M. Navez, J. L. Mugabo, P. Ruiz  
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Influence of the solid state properties of Pd/MO<sub>x</sub> (M= Ti, Al) catalysts in catalytic combustion of methane  
S. R. G. Carrazan, R. Mateos, V. Rives, P. Ruiz  
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Surface modifications of MO<sub>x</sub> oxides supports by titania grafting and their influence in the catalytic combustion of methane performances.  
C. Mateos-Pedrero, P. Ruiz.

Understanding the dynamic and transient behaviour of oxide catalysts in working conditions and the relationships with their catalytic performances.  
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The dissociative adsorption of CO<sub>2</sub> at low temperature: a new opportunity to implement the use of CO<sub>2</sub>  
E.M. Gaigneaux, F. Dury, C. Mateos, M. Navez, O. Demoulin & P. Ruiz  
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Modification of active sites by the introduction of gas promoters in the feed during oxidation reactions.  
O. Demoulin, I. Seunier, F. Dury, M. Navez, R. Rachwalik, B. Sulikowski, S.R. Gonzalez-Carrazan, E.M. Gaigneaux and P. Ruiz  
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A Selective Combinatorial Approach: Application to the combustion of methane at low temperature.  
O. Demoulin\*, M. Nave, F. Gracia, L. Bollman, E.E. Wolf and P. Ruiz  
Europacat VI, 7<sup>th</sup> European Workshop Meeting on Selective Oxidation "Innovative Selective Oxidations: Nanoscale and Dynamics Aspects" (ISO 2003), August 31-Sept 04, 2003, Innsbruck, Austria

Modulation of the active sites with CO<sub>2</sub> or N<sub>2</sub>O as gas promoter during oxidation reactions.  
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The Role of Oxygen and Hydroxyl Support Species on the Mechanism of H<sub>2</sub> Production in the Steam Reforming of Phenol and Toluene over Metal Oxide-Supported Rh and Fe Catalysts.  
K. Polychronopoulou, C. N. Costa, A. M. Efstathiou  
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P. G. Savva, G. G. Olympiou, C. N. Costa, V. A. Ryzhkov and A. M. Efstathiou  
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Novel Fe-Mn-Zn-Ti-O mixed-metal oxides for the low-temperature removal of H<sub>2</sub>S from gas streams in the presence of H<sub>2</sub>, CO<sub>2</sub>, and H<sub>2</sub>O.  
K. Polychronopoulou, F. Cabello Galisteo, M. López Granados, J. L. G. Fierro, T. Bakas, A. M. Efstathiou  
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Novel Zn-Ti-based mixed metal oxides for low-temperature adsorption of H<sub>2</sub>S from industrial gas.  
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K. Polychronopoulou, Z. Theodorou, C. N. Costa, A. M. Efstathiou  
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Effects of Support Surface Composition on the Activity and Selectivity of Pd/C Catalysts in Aqueous-Phase Hydrodechlorination Reactions.  
L. Calvo, M. A. Gilarranz, A. F. Mohedano, J. A. Casas, J. J. Rodríguez  
Industrial & Engineering Chemistry A, Vol. 44 (17) (2005) 6661 - 6667

Treatment of chlorophenols-bearing wastewaters through hydrodechlorination using Pd/activated carbon catalysts.  
L. Calvo, A. F. Mohedano, J. A. Casas, M. A. Gilarranz, J. J. Rodríguez  
Carbón A, Vol. 42 (7) (2004) 1377 - 1381

Complete oxidation of acetone over manganese oxide catalysts supported on alumina- and zirconia-pillared clays.  
L. M. Gandia, M.A. Vicente, A. Gil  
Applied Catalysis B, Vol. 38 (2002) 295-307

Platinum catalysts supported on Al-pillared clays. Application to the catalytic combustion of acetone and methyl-ethyl-ketone.  
A. Gil, M.A. Vicente, J.-F. Lambert, L. M. Gandia  
Catalysis Today, Vol. 68 (2001) 41-51

Effects of alkali-acid additives on the activity of a manganese oxide in catalytic combustion of ketones.  
L. M. Gandia, A. Gil, S.A. Korili  
Applied Catalysis B, Vol. 33 (2001) 1-8

Influence of the surface adsorption-desorption processes on the ignition curves of volatile organic compounds (VOCs) complete oxidation over supported catalysts.  
M. Paulis, L. M. Gandia, A. Gil, J. Sambeth, J.A. Odriozola, M. Montes  
Applied Catalysis B, Vol. 26 (2000) 37-46

Preparation, Characterization and Catalytic Activity in the Deep Oxidation of Acetone of Cr, Al-Pillared Saponites.  
A. Gil, M.A. Vicente, R. Toranzo, M. A. Bafares, L. M. Candia  
Journal of Chemical Technology and Biotechnology, Vol. 72 (1998) 131-136

Influence of the Incorporation of Palladium on Ru/Mcm Hydrotreating Catalysts.  
D. Eliche-Quesada, J. Mérida-Robles, E. Rodríguez-Castellón and A. Jiménez-López  
Applied Catalysis B, Vol. 65 (2006) 118-126



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- Cobalt, Copper and Iron-Containing Monolithic Aluminosilicate-Supported Preparations for Selective Reduction of No with Ammonia at Low Temperatures.  
M. Brandhorst, J. Zajac, D.J. Jones, J. Rozière, A. Jiménez-López, E. Rodríguez-Castellón  
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J. Jiménez-Jiménez, M. Rubio Alonso, D. Eliche-Quesada, E. Rodríguez-Castellón, A. Jiménez-López  
Journal of Materials Chemistry, Vol.: 15 (2005) 3466-3472
- Influence of the Metallic Precursor on the Hydrogenation of Tetralin over Pd-Pt Supported Zirconium Doped Mesoporous Silica.  
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# MICROACTIVITY-REFERENCE CONFIGURATION & OPTIONS

## BASIC UNIT: MAXXXM3

Labels in photograph: Reactor TECC, 10 µm filter, AE tubular reactor, Purge separator, 10 µm filter, Electric forced convection heater, Liquid evaporator, 3 MFC, 6 port valve (6 ports), Gas preheater.

- AE tubular reactor with 10 µm porous plate: ED=14.29 mm, ID= 5.17 mm, L=305 mm
- Low thermal inertia radiant furnace
- Hot box to avoid condensations
- 6-port bypass valve
- L/G separator based on thermoelectric effect
- Hi-Tec Mass Flow Controllers for gas feeding

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## PRESSURE CONTROL: MAPXXM3

Labels in photograph: Micrometric valve for pressure control

- High Pressure Control System: Up to 100 bar
- Stability and precision in pressure control of  $\pm 0.1$  bar
- Micrometric valve servocontrolled by a stepper motor

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## HPLC PUMP FOR LIQUIDS: MAPGXM3

- HPLC liquid pump, 0.01-5 ml/min, 600 bar, Digital communications

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## LEVEL CONTROL IN L/G SEPARATOR: MAPGLM3

Labels in photograph: Condenser & level sensor, Micrometric valve, Capacitive level sensor, Condenser

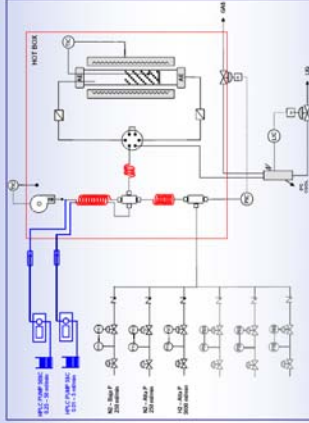
- L/G operation in continuous mode
- Recovery of liquid products in high pressure system almost in real time
- Level control in L-G separator:  $0.3 \pm 0.1$  ml
- Based on a capacitive sensor and a micrometric servocontrolled valve

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# MICROACTIVITY-REFERENCE CONFIGURATION & OPTIONS

## 2 HPLC PUMPS FOR LIQUIDS



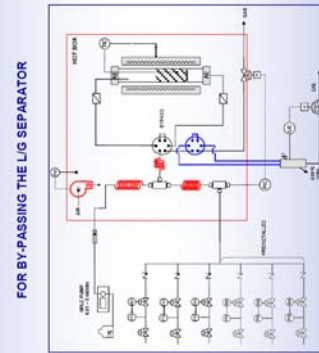
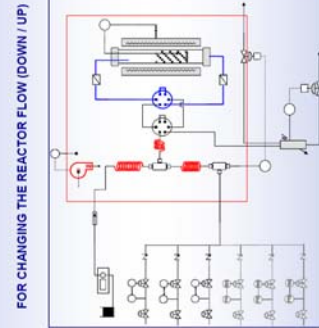
- HPLC liquid pump, 0.01-5 ml/min, 600 bar, Digital communications

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MICROACTIVITY-Reference

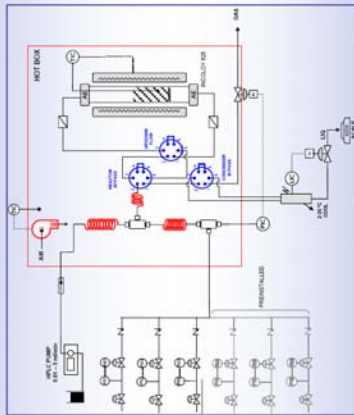
## 2nd AUTOMATIC 6 PORT VALVE



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## 3rd AUTOMATIC 6 PORT VALVE

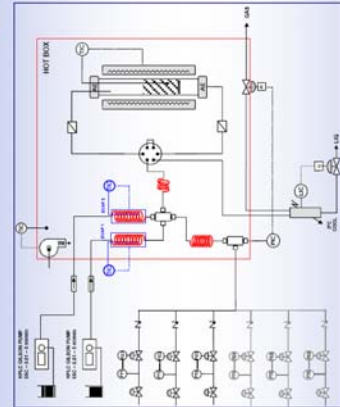


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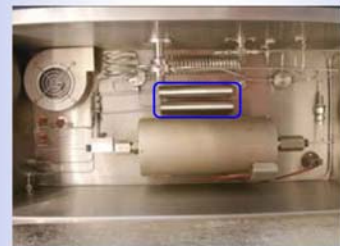
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## EVAPORATORS WITH TEMPERATURE CONTROL



- Liquid evaporators with temperature control, up to 300°C

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# MICROACTIVITY-REFERENCE CONFIGURATION & OPTIONS

## FLUIDIZED BEDS

- Fluidized bed reactor with cyclone and vessel for solid recovery
- $\Delta P$  sensor in the bed

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## SOLID FEEDING SYSTEM

- Automatic solid feeding system for high pressure reactor (up to 100 bar)

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## HPLC PUMP HEATING SYSTEM

- For feeding high viscosity liquids.
- Automatic temperature control in liquid vessel (PID control loop).
- Temperature control in pump lines and head. Max. Temperature: 75 °C.
- Gas supply and regulator in liquid vessel for pump priming.

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## 2 UNITS - PARALLEL OR SERIAL WORKING MODE...

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*Dr. David Serrano, Director, Energy Institute*

**"We at NUCAT are very satisfied with the Microactivity reactor from PID Eng&Tech. Since the first contact everyone was very helpful and cordial and, above all, very fast in their responses and eager to provide us with the best solution for the problem we had.**

**When the unit arrived it performed up to expectations and according to what was asked. Some people in the lab were concerned about technical assistance because at that time there was no representative in Brazil. When the first technical problem occurred the solution was provided by a simple exchange of e-mails, showing how committed the company is in order to have a satisfied customer.**

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*Dr. José J. Pis, Carbon National Institute, CSIC*

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*Dr. Jean Thivolle-Cazat, National Center of Scientific Research, SOMC, France*

**"It is a great pleasure for me to be given this opportunity to say a few things about PID Eng&Tech. I have been closely cooperating with PID Eng&Tech and in particular with its director Dr. Jose Prieto, for the last eight years. I have to stress out from the beginning that it has been eight years of a fruitful and flawless cooperation. I have purchased from PID five different units so far and I am completely satisfied by their quality, durability and their overall performance. Based on my experience, I can assure that PID Eng&Tech is able to offer a multi-level support which starts with specialized equipment design and reaches out to maintenance and after-sale support. During these years of cooperation I have experienced only purely professional treatment by the company at the following levels:**

- Equipment Design: The experts of PID have helped me and my group with the designing of sophisticated and complex equipment, which is used for advanced scientific experiments, by providing valuable information and practical solutions to all the problems that came in our way.
- Training: The training programs given by PID consist an adequate way of understanding the operation and maintenance of their products. It is worthwhile to notice that all training programs are carried out on same unit to be purchased, something which is very helpful for the end users of the unit.
- Installation: All the units that I have purchased so far have arrived at my laboratory in a perfect state and almost ready for use. Installation requires only the necessary connections to the gasses and/or liquid supplies..... and the unit is ready for use.
- Maintenance: After attending the training program at PID, maintenance becomes very easy by all means.
- After-sale Support: **The after-sale support at PID Eng&Tech is probably the best, most professional kind of support that I have experienced so far in my career. The people at PID Eng&Tech have helped me and my group numerous times with any modifications, adjustments and changes that we need to make in our equipments. They always answer promptly and they always deliver within reasonable time. They are fast and effective.**

**Finally, I would like to thank the director and all the personnel of PID for their valuable cooperation so far and assure that our cooperation will continue to exist for many years".**

*Prof. Costas N. Costa, Deputy Coordinator, Cyprus University of Technology*

**"The best of PID Eng&Tech is that they listen to your technical requirements and offer a personalized solution quickly by adapting their standard processes. They are able to discern between your scientific and technical problems, they can offer you solutions!"**

*Dr. Mario Montes, Euskal Herriko Unibertsitatea*

**"We are getting excellent performance with Microactivity reactors and pilot plants from PID Eng&Tech".**

*Prof. Fierro, Catalysis and Petroleochemistry Institute*

**"I highly recommend PID Eng&Tech. Their Microactivity reactor is quite impressive, and I am planning to buy another unit from the same company".**

*Prof. Saeed M. Al-Zahrani, Chemical Engineer Department King Saudi University*



## OUR PRIMARY PRODUCTS AND SERVICES

### CATALYTIC REACTORS

The **MICROACTIVITY-Reference** is a modular automatic and computerized laboratory reactor for catalytic microactivity reactions with reactor bypass, preheaters, evaporator, pressure control valve and other process layouts inside a hot box, which avoids possible condensation of volatile products, at the time that preheats the reactants efficiently. The equipment consists of a BASIC UNIT and some series of EXTRA PACKAGES that improve or modify its efficiency. **This is the reference worldwide unit at catalytic studies.**



#### FOUR RUNS MICROACTIVITY-Test Unit MAT ASTM D3907

This unit is a fully automatic and computerized laboratory reactor (described in ES2011993 patent) for the analysis of Fluid Cracking Catalysts (FCC). This MAT reactor is design to perform up to four independent and consecutive FCC test, following the norms described by the standard method ASTM D3907, in automatic mode without the presence of an operator.

The operator will be able to adjust for each independent experiment stream time, catalyst/oil relationship, reaction temperature, the regeneration times and temperature, gas flow, and other parameters.

With an excellent reaction temperature control and making use of a precise HPLC pump for gasoil dosification, even for very short reaction time (10 seg), this unit carry out reaction and regeneration in-situ and consecutives stages, with coke and gases analysis. The four liquid products obtained are collected in a cooled receiver until the end of the experiments.



### MICRO PILOT PLANTS

#### POLYMERIZATION

PID Eng&Tech has worked for several years in collaboration with industrial research centers to develop and implement projects for polymerization pilot plants in both discontinuous and **continuous** mode. This has led PID Eng&Tech to a high level of knowledge on the restrictions and demands on these processes and on the technologies related to them.

PID Eng&Tech has its own solutions for operations such as continuous feed regulation of catalysts or transferring slurries, solutions that have been implemented and validated on different scales (patent pending). PID Eng&Tech, a leader in **microscale**, technology, has manufactured the first microscale factory in the world for obtaining bimodal polymers. It operates continuously with a production of 200 g/h.





# OUR PRIMARY PRODUCTS AND SERVICES

## SUPERCRITICAL EXTRACTION

PID Eng&Tech has 4 plant models for SCF with volume extractors between 350cc and 2L in capacity. They operate at a maximum pressure of 380 bar, CO<sub>2</sub> flows up to 5 l/h, and using two separators with independent control temperature and pressure contrl. More than 15 units are currently in operation.

## GASIFICATION

In the past few years, PID Eng&Tech has built several different laboratory units. They range from the 2" and 1.5 m high ones to the 8" and 4 m ones. They produce synthesis gas through the gasification of biomass, carbon or plastic waste. Accompanying manufactured solids dispenser (patent pending) makes it possible to feed the material to be gasified with high accuracy and even operating with pressurized gasifiers. PID Eng&Tech has also worked in the subsequent phases of reforming, gas-shift, COprox.



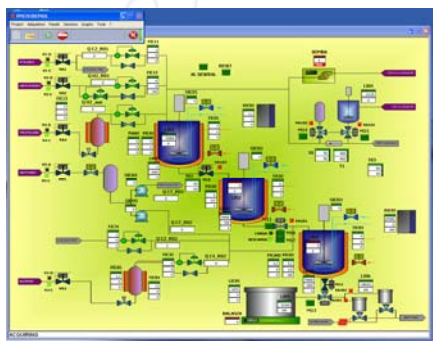
## TAYLOR MADE PILOT PLANTS



Improving competitiveness in the industry is sharply marked by developing new products with high quality features and a high added value. Continuous and automatic pilot plants that can simulate industrial processes at the laboratory level with reasonable scalability are essential for seeking new products, improving the quality of existing ones and developing new processes. These systems' high degree of complexity, their high number of operating variables and the interrelationship among them requires an exhaustive study of the instrumentation and control in order to attain results provided by these systems that are representative and reproducible.

An initial commitment toward development, the experience accumulated during 20 years carrying out projects, and strong involvement with customers during the implementation and operation stages accredit PID Eng&Tech as specialists in designing and building pilot plants and laboratory reactors.

## THE SOFTWARE



PID Eng&Tech is a leading supplier of Supervisory Control and Data Acquisition (SCADA) solutions for laboratory reactors and pilot plants. Our systems include the Process@ software making use of the Ethernet remote control. The operator is able to set all the devices configuration parameters: set points, alarm values, calibration parameters settings, etc. Process@ can also save sessions that are used in order to automate the processes. The main innovation of Process@ application is its ability to manage different manufacturer protocols.



## CONGRESS PARTICIPATION



As International marketing activities, PID Eng&Tech has participated in several congresses around the World.

From 2003 the Company opened new markets and starts its Internationalization in Europe, Middle East, Asia, EEUU and South America. The most important business activity of PID during these 6 years was based on **Customer confidence activities** and **Congress assistance**. Nowadays we are looking for covering through Distributors the most strategic markets for our products.

PID Eng&Tech has attended the following events:

- **ICC 2008. 14th International Congress on Catalysis** 13-18 July 2008. COEX, Seoul, Korea.
- **WPC 2008. 19th World Petroleum Congress** 29 June-3 July 2008. IFEMA, Madrid, Spain
- **ISFL 2008. 6th International Symposium on Fuels and Lubricants** 9-12 March 2008. New Delhi, India.
- **PITTCON 2008. Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy** 2-6 March 2008. New Orleans, USA.
- **EUROPACAT VIII.** 26-31 August 2007. Turku, Finland.
- **PITTCON 2007. Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy.** February 25 - March 2 2007. Chicago, Illinois, USA.
- **OPERANDO II.** Second International Congress on Operando Spectroscopy. 23-27 April 2006. Toledo, Spain.
- **EXPOQUIMIA 2005.** 14-18 November 2005. Fira de Barcelona, Spain.
- **EUROPACAT VII. Catalysis: A Key To A Richer & Cleaner Society.** 28 August - 1 September 2005. Sofia - Bulgaria
- **VII SEMINARIO ITALIANO DE CATALISI.** 19-24 June 2005. Verbania-Pallanza, Italy.
- **NORTH AMERICAN MEETING ON CATALYSIS 2005.** 20-25 May 2005. Philadelphia, Pennsylvania, USA.
- **XXXVIII JAHRESTREFFEN DEUTSCHER KATALYTIKER.** 16-18 March 2005. Weimar, Germany.
- **13th INTERNATIONAL CONGRESS ON CATALYSIS.** 11-16 July 2004. Palais des Congrès, Paris, France.
- **BIOTECHNICA 2003.** October 2003. Hannover, Germany.



PID Eng&Tech will attend these Congresses during the next four years:

- |                    |  |                     |
|--------------------|--|---------------------|
| ■ 2009 7-12 June   | <b>National America Society of Catalyst</b>            | San Francisco (USA) |
| ■ 2009 1-4 Sept.   | <b>EuropaCat IX</b>                                    | Salamanca (Spain)   |
| ■ 2009 13-17 Sept. | <b>South America Congress on Catalysis</b>             | Sao Paulo (Brazil)  |
| ■ 2009 21-23 Oct.  | <b>COST Chemistry Workshop</b>                         | Benahavis (Spain)   |
| ■ 2010             | <b>SICAT (Iberoamericano)</b>                          |                     |
| ■ 2011             | <b>EuropaCat X</b>                                     | Czech Republic      |
| ■ 2012             | <b>15<sup>th</sup> International Catalyst Congress</b> | Munich              |



# ACCREDITATIONS/CERTIFICATIONS

**ISO 9001:2000**

**ISO 14001:2004**



## European Directives

### PED - Directive 97/23/EC

Our equipments meet the Directive 97/23/EC of the European Parliament and of the Council of 29 May 1997 on the approximation of the laws of the Member States concerning pressure equipment.

Conformity assessment applied procedures, according to the equipment category:

Equipment Category	Assessment Module	CONFORMITY ASESMENT PROCEDURES
I	A	Internal production control
II	A1	Internal manufacturing checks with monitoring of the final assessment
III	B+C1	EC Type-examination + Conformity to type
IV	B+F	EC Type-examination + Product verification
Unit	G	EC Unit verification



### EMC - Directive 2004/108/EC (and former Directive 89/336/EEC modified by the Directives 91/263/CEE, 92/31/CEE, 93/68/CEE and 93/97/CEE)

Our equipments meet the European Directive 2004/108/EC on the approximation of the laws of the Member States relating to Electromagnetic Compatibility. Its conformity has been checked with the Harmonized Standard EN 61326:1997 (and its amendments EN 61326/A1:1998 and EN 61326/A2:2001) for "Electrical equipment for measurement, control and laboratory use – EMC requirements" approved by the European Standardisation Organisation CENELEC.

Tests of EMC immunity according to the standard EN 61326:

- EN 61000-4-2: Electrostatic discharge immunity test.
- EN 61000-4-3: Radiated, radio-frequency, electromagnetic field immunity test.
- EN 61000-4-4: Electrical fast transient/burst immunity test.
- EN 61000-4-5: Surge immunity test.
- EN 61000-4-6: Immunity to conducted disturbances, induced by radio-frequency fields.
- EN 61000-4-11: Voltage Dips, Short Interruptions and Voltage Variations Immunity Tests.



## ACCREDITATIONS/CERTIFICATIONS

Tests of EMC emission according to the standard EN 61326:

- EN 61000-3-2: Limits for harmonic current emissions (equipment input current up to and including 16 A per phase).
- EN 61000-3-3: Limitation of voltage changes, voltage fluctuations and flicker in public low-voltage supply systems, for equipment with rated current  $\leq$  16 A per phase and not subject to conditional connection.
- EN 55011 (CISPR 11): Industrial, scientific and medical (ISM) radio-frequency equipment – Radio disturbance characteristics - Limits and methods of measurement.



### LVD - Directive 2006/95/EC (and former Directive 73/23/EEC)

Our equipments meet the European Directive 2006/95/EC on the approximation of the laws of the Member States relating to Electrical Safety (Low Voltage). Its conformity has been checked with the Harmonized Standard EN 61010-1:2001 for "Safety requirements for electrical equipment for measurement, control, and laboratory use - Part 1: General requirements", approved by the approved by the European Committee for Electrotechnical Standardization (CENELEC).

Test of Electrical Safety according to the Standard EN 61010-1:

- Marking and documentation.
- Protection against electrical shocks.
- Protection against mechanical hazards.
- Mechanical resistance to shock and impact.
- Protection against the spread of fire.
- Equipment temperature limits and resistance to heat.
- Protection against hazards from fluids.

### MACHINERY - Directive 98/37/EC

Our equipments meets the Directive 98/37/EC of the European Parliament and of the Council of 22 June 1998 on the approximation of the laws of the Member States relating to machinery.

### ATEX - Directive 94/9/EC

Our equipments should not be used in potentially explosive atmospheres.

The Directive 94/9/EC (related to the approximation of the Member States concerning equipment and protection systems for its use under potentially explosive atmospheres) in its chapter I, article 1, section 4, establish that:

- "Excluded from the application ambit of this Directive are those equipments which are destined to be used under no commercial settings where the potentially explosive atmospheres are created in rare occasions only as consequence of a fortuity escape of gas."

The application Directives 94/9/EC, from may 2000, establish in the section 4.1.2. a) that:

- "It is considered that an equipment only enter in the application ambit of the Directive if it is destined (in its totality or partial) to be used in potentially explosive atmosphere. The fact that in the interior of the equipment deliberately, could be a potentially explosive atmosphere has no relevance".

**RoHS - Directive 2002/95/EC**  
Restriction of Hazardous Substances.



**WEEE – Directive 2002/96/EC**  
Waste Electrical and Electronic Equipment.





## R&D ACTIVITIES & PATENTS

PID Eng&Tech experiences continuous growth and strives to expand its product line through various research projects and research investments. PID Eng&Tech in collaboration with **Catalysis and Petrochemistry Institute (CSIC, Spain)** has been working in new investigation lines and in the development and improvements of the products. This investment has the following objectives:

- **LEADERSHIP:** development of devices and instruments that solve the problems inherent in microscale (components of limited dimensions, small volumes or quantities of matter, etc.). PID is a World leader in microscale systems.
- **INNOVATION:** Control systems development capable of manage the variables related to the micro-process, allowing the stability and safety of the process in addition to automating the operation of the plant. Moreover, PID takes part in numerous technological programs and projects.
- **QUALITY and SECURITY:** Analysis of risks that make necessary the incorporation of devices that guarantees the safety in all the phases of the process. PID has certification ISO 9001 and ISO 14000 that is the result of fulfillment of all the instructions that are applicable to its products. In addition, with all equipment PID supplies the Declaration of Conformity that is demanded by the European Institutions.

Some of the R&D governmental projects subsidized by CDTI, IMADE, MITYC and MEC are:

### NEOTEC PROJECT

- "Instrument and equipments for specific processes of chemical research", 2004-2005. CDTI.



### PIE PROJECT

- "Investigation and development of a monitorized system and advance control for bioreactors".
- "Research and development of an automated and computerized microreactors system for combinatorial chemistry, with an "in situ" analysis", 2005.
- "Research and development of an automated lab-scale reactor for catalytic microactivity studies of chemical reactions", 2004.



### PROFIT PROJECT

- "Technical viability study for monitorization and advance control system, automatic, integrated and universal for bioreactors to laboratory scale, pilot and semi-industrial with sterilization " In situ " ", 2006.
- "Research and development of a new catalytic Microactivity reactor", 2004.
- "Research and development of an automated pilot plant for extraction at supercritical conditions", 2005

### TORRES QUEVEDO PROGRAM

- "Viability study for development an automated unit for the study and evaluation of catalysts in the process FCC (Fluid Catalytic Cracking)", 2005.
- "Critical and technical viability studies for investigation and posterior development of microreactors system for combinatorial chemistry with "in situ" analysis", 2005.
- "Critical and technical viability studies for investigation and posterior development of SCADA system for bioreactors", 2004.

### PATENTS

CSIC (Spanish Council for Scientific Research), proprietor of the patents listed below, granted PID Eng&Tech, under the Patent License Agreement, to make, have made, use, sell, offer to sell and export products.

- Automated reactor for catalytic microactivity studies: ES-2245238 / PTC-ES2005/070079 / WO-2006008328 / EP-1757930 / US-2008063565
- Servopositioner for microregulation valve: ES-2245239 / PCT-ES2005/070080 / WO-2006021603 / EP-1775504 / US-2007241296
- Capacitive level sensor for reduced volume systems: ES-2249139 / PCT-ES2005/070081 / WO-2006021604 / EP-1757911 / US-2007283753
- Modular integrated elements for processes control: ES-2032182
- Computerized Unit for FCC catalysts studies and evaluation: ES-2011993
- Method of immobilizing hydrocarbons inside submerged containers or of transporting said hydrocarbon to the surface, using the properties of supercritical fluids at a great depth: WO-2004065526 / EP-1595786 / NO-20053945 / CA-2514171 / US-2006016828
- Immobilising or removing hydrocarbons inside sunken tanker ships, by delivering fluid into tank and then degassing the resulting supercriticalfluid: ES-2213476 / ES-2214974