

The micro-regulation valve

Patent WO-2006021603 / EP-1775504 / US-2007241296

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PRESSURE CONTROL OR LIQUID LEVEL CONTROL MICROREGULATION VALVE

The **micro-regulation servo positioned valve** used by PID Eng&Tech in the Microactivity-Reference reactor and pilot plants, for pressure control or for Liquid-Gas separator Level control, are based in our patent (**WO-2006021603 / EP-1775504 / US-2007241296**). This patent describes a servo-controlled valve with 8 turns of rotational movement and with a high speed and resolution that leads to the highest sensibility in flow modulation and fast response, comparing with the currently state of art worldwide.

The principal advantages using this system, for control micro-flows (laboratory plant scale) at high pressure, are the **stability**, the high **rangeability** and the **universality** of the design.

Standard commercial control valves are based on pneumatic valves, mechanical orifice-diaphragm valves or electronic devices. Each one of these systems has a big limitation when they are used at microflows & high pressure. Pneumatic valves have very low rangeability and the flow modulation is based on the change in restriction length, and not on the change in modulated orifice size. The mechanical valves are not designed for microflows because Kv and spring action lead to non-stable flows, also big dead volume is problematic with condensed liquids. Finally, electronic valves are not appropriated for use with dirty reaction products or products with vapors. In order to solve all these problems PID Eng&Tech has designed and patented its own control valve for microflows control, opening a new operational window in research scale equipment.

Stability:

Generally, the pressure in a system is controlled modulating the flow at the gas outlet. In this way, perturbations caused by pressure control valve action will be transmitted to catalytic bed generating a pulsing flow through the bed that modifies the obtained results because of the instantaneous modifications on the time on stream. The use of the PID Eng&Tech patented control system will reduce this effect over the experimental results, increasing the experimental repeatability. If compare the resolution of a pneumatic valve (with around 100 different control positions) with the servopositioned PID Eng&Tech valve (1° resolution above 360° per 8 turns), the increasing of sensibility is very important.



Rangeability:

The orifice of the control valve in the PID Eng&Tech's design is made of non-metallic material. That system allows the total sealing between the SS316 valve needle and the seat of the orifice and to control near to the close position, without damaging of the valve needle. Therefore this valve can be used for controlling micro-flows, very high pressure flows, or both simultaneously reaching very low Kv values. The material used for the orifice-trim (confidential) was developed specifically for use in this valve and the life of the valve will extend during several years of continuous work (24h/7days) at very low flows and high pressures. Since flow is modulating the orifice size, also high flows or very low pressures can be controlled with the same valve.

Universality:

The valve has been tested during years for many different operation conditions. As result its operating has been tested for high pressure or low pressure, for high flows or very low flows, for gases, liquids or mixtures, and in a temperature range from ambient temperature to 220°C. This universality in its use is one of the principal advantages of this PID Eng&Tech patented system and gives to our equipments a big added value due to the system do not need to be designed for some determinate operation conditions, and it can be used in many different type of working conditions. For these reasons PID Eng&Tech patented system is the **Universal multipurpose control valve**.

Operability:

Because of its simple design, user can easily carry out the maintenance of the valve, configure it for different uses (sensibility can be adjusted modifying the turns number), with very easy re-zero position adjust, very easy cleaning operations, cheaper spare parts sets and simple disassembling operations,..., finally it is a dream for the pilot plant users.



Technical datas

Work temperature from 0°C to 210°C
Work Pressure from atmospheric to 340 bar.
Chemical compatibility MOC: SS316, Teflon, Peek
Cv from close sealing position up to 10exp-3
CE marked



Capacitive micro-level sensor

Patent WO-2006021604 / EP-1757911 / US-2007238753

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Micro-level measurement State of the art

Users of laboratory reactors or micro pilot plants working at high pressure usually face up to a big problem when the reactions products, liquid and gas mixture, need to be separated before the outlet of the system. The liquid level inside the L/G separator needs to be measured in real time for controlling the liquid outlet of the separator.

The current technologies applied at these systems are based in differential pressure transmitters that measure the hydrostatic pressure of the liquid column in the vessel. According to this value, a control valve is positioned for maintaining a stable liquid level. Because of the low sensibility of these differential pressure transmitters together with their big size, it is needed a minimum amount of liquid inside the separator, (usually more than 100cc). These use of this volumes involve two important problems; first, user can never obtain liquid samples on the first stages of the reaction (some hours are needed to achieve the operational minimum level); and second, the liquid samples obtained at outlet of L/G separator is the mixture of the products corresponding to many reaction hours. Under these conditions, phenomena such us kinetic of the reaction or catalyst deactivation cannot be studied.

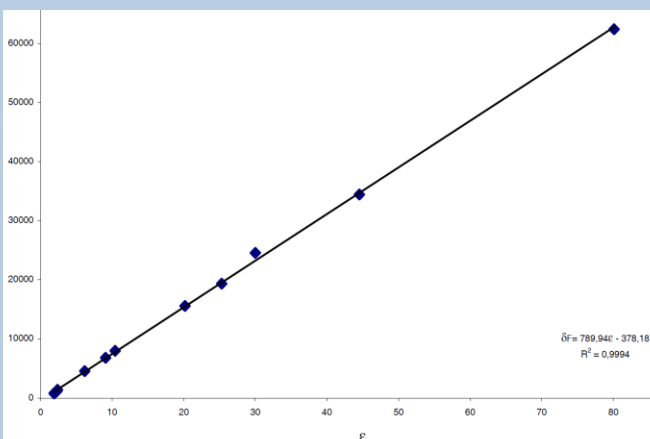
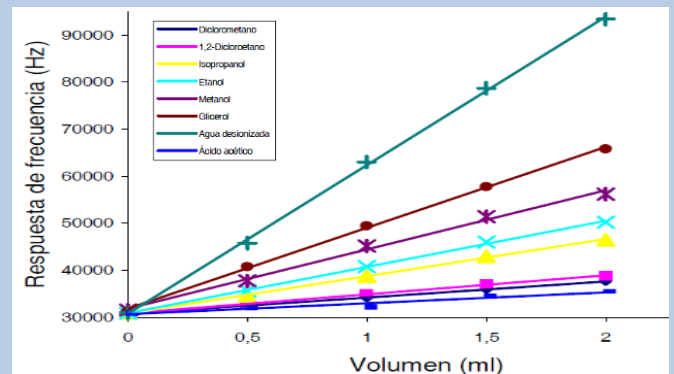
PID Eng&Tech new capacitive liquid sensor

PID Eng &Tech has devoted many years of effort to improve a micro-volume high pressure level sensor based on dielectric property of liquids. Electrical capacity of a condenser is modified when a liquid or dielectric is placed between its plates. Therefore, an oscillating RC circuit increases its oscillating frequency when there is more amount of dielectric between the condenser plates. This principle has been used for measuring the amount of liquid present inside a SS316 8mm I.D. vessel (housing) with an internal rod of 3 mm O.D., acting these both elements like a electrical condenser. These two components need to be electrically isolated, but the closure between them is designed for high pressure (400 bar). Finally, the dead volume of this system is around 0.5 to 1 cc, hundred of times less comparing with standard L/G separators. PID Eng&Tech's standard micro-L/G separator consists of the cooling system (electrical), the separator vessel and the level sensor.

Sensor response sensibility

The response of the sensor depends on the dielectric capacity of liquid. Therefore, water ($\epsilon=80$) will be detected with very more sensibility than hydrocarbons ($\epsilon=1.8$). The response of the sensor for different types of liquids and different amounts of liquids (from 0.5 to 2cc) inside the L/G separator is shown in table and graphic:

COMPOUND	FÓRMULA	ϵ (20°C)	BASE	0.5 cc	1 cc	1.5 cc	2 cc	DIF
Hexane	C ₆ H ₁₄	1,89	30923	31118	31314	31516	31710	787
Heptane	C ₇ H ₁₆	1,92	30930	31115	31330	31540	31730	800
Hexadecane	C ₁₆ H ₃₄	2,05	30960	31198	31438	31679	31914	954
Hydraulic oil			30970	31225	31522	31815	32062	1092
Mecanic oil			30988	31280	31595	31897	32170	1182
Carbon tetrachloride	CCl ₄	2,24	30955	31225	31512	31790	32060	1105
Toluene	C ₇ H ₈	2,379	30935	31245	31590	31923	32244	1390
Vegetal oil			30985	31435	31922	32403	32808	1823
Acetic acid	C ₂ H ₄ O ₂	6,17	30940	32005	32260	34511	35512	4572
Dichloromethane	CH ₂ Cl ₂	9,08	30920	32503	34191	36000	37715	6795
1,2-Dichloroethane	C ₂ H ₄ Cl ₂	10,42	30918	32790	34945	37128	38900	7982
Isopropanol	C ₃ H ₈ O	20,18	30945	34645	38808	43000	46525	15580
Ethanol	C ₂ H ₆ O	25,3	30920	35710	40800	46060	50275	19355
Methanol	CH ₄ O	30	31632	37800	45200	51452	56210	24578
Glycerol	C ₃ H ₈ O ₃	44,52	31377	40840	49470	57786	65835	34458
Deionized water	H ₂ O	80,1	31000	45810	63020	78745	93420	62420
Drinking water	H ₂ O		31000	51015	77665	109800	145700	114700



Calibrating the sensor

A clear advantage of this capacitive sensor is that response of the sensor is directly proportional to the dielectric constant of the liquid inside. This effect is showed at the graphic placed on the left side. It can be observed the response of the sensor obtained for the same amount (2cc) of different liquids represented as function of his dielectrical capacity. This linear response allows to determinate the response of a calibrated sensor when the liquid is modified. Therefore, the new liquid response can be adjusted directly from the quotient of the dielectric constants of both liquids.

Technical data

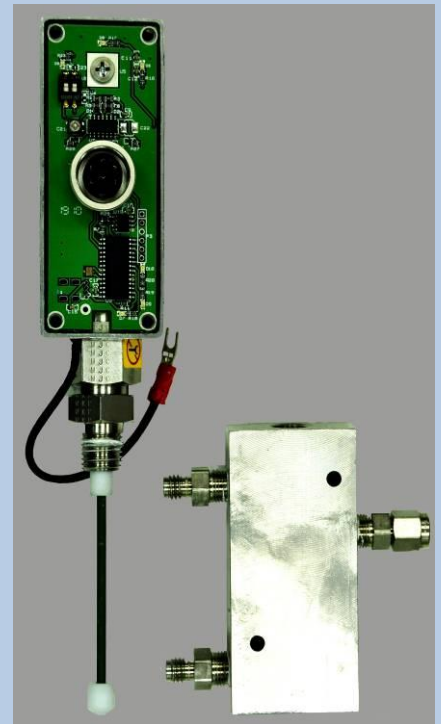
Work temperature: from 0°C to 60°C, stable

Work Pressure: from atmospheric to 400 bar.

Chemical compatibility MOC: SS316, Kalrez, Peek

Sensibility with water: recognize changes in 0.1 mm high.

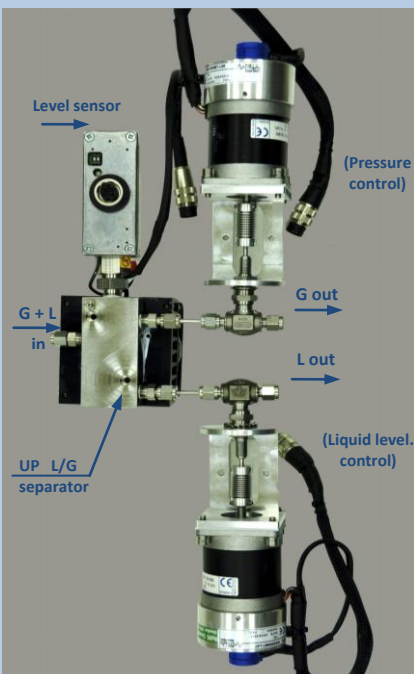
CE marked



The high pressure Liquid-Gas separator no dead-volume

Patent WO-2006021604 / EP-1757911 / US-2007238753

The **Liquid-Gas separator at high pressure without dead-volume** is one of the principal advantages of the Microactivity-Reference reactor. Traditionally the standard volumes at the L/G separators in micro-reactors have been the same used at big pilot plants, because the measurement of the level inside of the separator involves the use of differential pressure elements with low precision (zero stability) when working at high pressure. Usually the volume of these vessels is so big that appear two main problems: First it is needed too much time for starting to obtain liquid products from the reactor (and so, the analysis of reaction condensed products cannot be did during the first hours); and second, liquid products from the liquid outlet are a product mixture of some hours of operation.



PID Eng&Tech has been working during years for achieving a patented L/G separator at high pressure without **dead volume**. This system allows user to obtain liquid products since the first minutes of reaction and to obtain the products produced in the reactor just few minutes before. This system permits to obtain, first time worldwide, kinetics data's in high pressure reactions involving liquid products.

The level sensor is based on the **dielectric behavior of the liquids** (water, alcohols, hydrocarbons,...) when they are inside a micro vessel and how they modify the capacity of a condenser. A microprocessor evaluates the change in the high oscillation frequency of a RC circuit that is directly proportional to the changes in the capacity of the system, and so, proportional to the level inside the micro-vessel. Due to high sensibility of the system, changes in the level of **0.1mm** can be detected.

The system consist of a SS316 cooling condenser of 1 to 3cc of internal volume, with temperature control system if needed (from 0 to 65°C), based on a Peltier electrical cell for cooling (avoiding the use of chillers). It works simultaneously like the condenser for the condensable products, as the L/G separator and as the sensor.

The L/G separator has one inlet (gases and condensable products) and two outlets: the gases outlet at top (that usually is directed to the pressure control system), and the liquid outlet at bottom. Two patented micro-regulation valves servo-controlled are used for control the gas outlet (pressure of the system) and the liquid outlet (control the liquid level inside the L/G separator). Because of the fast response of these valves and their precision in the flow modulation, it is possible to work with less of 1cc of liquid inside the L/G separator, only the minimum volume for maintain the hydrostatic sealing inside the separator, even when the pressure can be 400 bar. This system opens a new way of catalysis studies in high pressure reactions when kinetics and catalyst deactivation are involved.

The Two Liquid phases – Gas Separator at high pressure

Patent P200930603, Patent Pending PCT/ES2010-070559 for WO/EP/US

The **Two Liquid Phases-Gas Separator at high pressure without dead-volume** is one of the most recent contributions of PID Eng&Tech and ICP-CSIC to the advance in micro-scale catalytic reactions control. In the last years, there is a significant the increase in the study of **Fisher-Tropsch** reactions (**GTL**). In these processes, water together hydrocarbons products are present in reactors outlet, as consequence of the reaction stoichiometry. In this type of reactions, where water and liquid hydrocarbons are mixed, two liquid phases appears in the liquid-gas separator because of the **non-miscibility** of both phases.

A modification of our patented system for *one liquid phase – gas* separator has been developed for solving this application, obtaining from the L/G separator 3 streams simultaneously: liquid 1 outlet (water), liquid 2 outlet (hydrocarbon) and gas outlet.. The same patented level sensor and micro-regulation valves are used for control the internal levels of water and hydrocarbon At the same time, with very low dead volume. The system is composed by two level sensors, two valves (one for each liquid outlet) and a third valve for gas outlet (usually, the pressure control valve).

The Two Liquid Phases-Gas Separator can work at 400 bar, from 0 to 65°C and the precision in control can be adjusted, for each one of the liquid phases, with volume inside the separator of **1cc +/-0.3cc**. Since condensation of liquid products occurs at high pressure, and the residence time is too short, equilibrium is not reached, so separation of products is really effective.

This type of system can also be used in reactions where a few amount of water is obtained or mixed with the reaction products and liquid hydrocarbons need to be separated at real time for online analysis by chromatography. This is also a new open door for the automation possibilities for High Throughput Reaction Systems. These patented products are property of PID Eng&Tech and are not provided as spare products, only like part of the PID Eng&Tech equipments.

