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SUPERCRITICAL EXTRACTION PILOT PLANT FOR SOLID SAMPLES IN 350 CC VESSEL AND LIQUID SAMPLES IN COUNTER-CURRENT COLUMN

Automated and computerized laboratory-pilot plant for extraction of solid samples by means of supercritical CO_2.

The system is a modular-type built for easy return to factory for service. Each module (feed section, Extractor module and two Separator modules) includes process system and electronic control system. All these systems are supervised for distributed control system PC based. The security systems are integrated in each one of these modules.

Feed system

- **Dosapro Milton** Roy CO_2 pump, 4.7 l/h, 380 bar, SS-316 hydraulic membrane, refrigerated head. Inverter for computerized control. Check valves, filters and other components.
- **Refrigerator unit** (-10ºC) for cooling CO_2 line feed and CO_2.
- **Dosapro Milton** Roy Co-solvent pump 0.3 l/h, 100 bar, SS-316 piston head. Inverter for computerized. Check-valves, filters and components.
- **Back pressure** system and bypass for flow measures at high pressure in Co-solvent pump.

Extractor

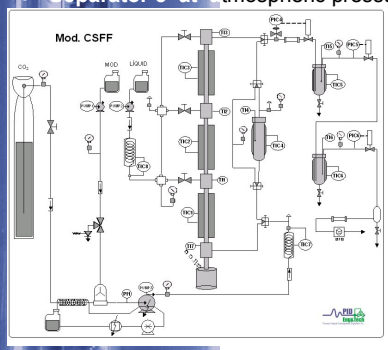
- Furnace for CO_2 preheater. Two control actions: heating by electrical power and cooling by furnace opening and closing.
- 350 cc Head Line vessel, 400 bar, easy closure system, for solid sample. Porous plate 20 microns. Quick connectors for agreeable work.
- Furnace for control temperature of extraction operation, internal thermocouple. Two control actions: heating by electrical power and cooling by furnace opening and closing.
- Bypass system (two-three way valves) for cleaning procedures.
- Pneumatic security valve put into operation by pressure control system.
- Pressure control system based on micrometric regulation servocontrolled valve. High precision in pressure control and fast response. Maximum pressure 340 bar.

Column System

- Dosapro Milton Roy liquids pump 2.5 l/hr, 380 bar, SS-316 hydraulic membrane head. Inverter for computerized control. Check valves, filters and other components.
- Preheater for liquid input.
- 3 valves for zone inlet selection.
- 1700 mm column with three zones and three inlets (for select one), inside diameter 19mm, inox 316, filled with inox 316 balls (5 mm diameter), independent temperature control for each zone.
- Receiver in bottoms with level control for liquid output (capacitive level sensor), with servo-controlled micrometric valve for liquid output control.
- Pneumatic security valve put into operation by pressure control system.
- Pressure control system based on micrometric regulation servocontrolled valve. High precision in pressure control and fast response. Maximum pressure 340 bar.

Three Separators

- **40 cc Head Line** vessel, 400 bar, easy closure system, for extracts collection. Valve for sample.
- **Furnace for control** temperature of separation operation, internal thermocouple. Two control actions: heating by electrical power and cooling by furnace opening and closing.
- **Pressure control** system based on micrometric regulation servocontrolled valve. High precision in pressure control and fast response. Max pressure 220 bar in Separator 1, 120 bar in Separator 2.
- **Separator 3 at** atmospheric pressure and MFM for CO_2 flow measurement.



Test

- The system will be tested during 24 hours at 360 bar closing (except MFM and rupture discs).
- The system will be tested during 4 hours at 340 bar in Extractor, 220 bar in Separator 1 and 120 bar in Separator 2, in operation mode, with 3 l/h CO_2 and

Distributed control system

- All module control systems are linked with PC computer by means of Process@ software for remote control with digital communications. The system can be controlled manually or automatically.
- Process@ software allows the operator to design automatic procedures for the process run.
- Plant has several independent safety levels: automatic switch-off in case of any problem, pressure and temperature security systems, all that based on electronic or mechanical devices and independent of PC.

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