HOCHDRUCKTECHNIK UND ANLAGENBAU

SITEC SIEBER ENGINEERING AG

CH-8124 MAUR/ZÜRICH, ASCHBACH 621, TEL. +41 (0)44 982 20 70, FAX +41 (0)44 982 20 79, E-MAIL: SIEBER@SITEC-HP.CH

Supercritical Fluid Extraction (SFE)

Phase Equilibria Measurement

SITEC-Sieber Engineering AG
Aschbach 621
CH-8124 Maur/Zürich

Tel +41 44 982 20 70
Fax +41 44 982 20 79
Homepage www.sitec-hp.ch

Multipurpose High Pressure Extraction Pilot Unit (Technical Description)

This turn-key **Multipurpose Pilot Unit** is assembled in a mobile frame and is built for the

- continuous extraction of **liquid raw materials** in a counter current column B3 combined with the possibility of

- batch-wise extraction of **solid raw materials** in an extraction vessel B1

The Pilot Unit may therefore be operated continuously, extracting solutes from liquid raw material or in the batch mode extracting solutes from solid raw material.

The liquid raw material is pumped into the column B3 through an upper connector from where it drops to the bottom in counter flow with the up streaming supercritical solvent fluid. The residue is collected at the bottom of the column B3 and is automatically discharged at a rate controlled by the level controller. The extract on the other hand is carried over to the separator B2 by the supercritical gas. The liquid raw material capacity is adjustable.

The high capacity diaphragm metering pump P1 delivers contaminant free supercritical fluid. Its capacity is also adjustable from 15 – 100 %. The fluid has already reached extraction conditions when entering the column, wherein the temperature is kept constant up to the outlet by the vessel heating.

Before entering the separation vessel B2 the pressure is reduced by a control valve C1 lowering the solvent power of the carrier gas to practically zero. There are three distinct phases entering the separation vessel B2: Liquid CO_2 , gaseous CO_2, and the extract. The extract drops to the bottom of the separation vessel B2 from where it is collected and removed through a hand valve. In the separation vessel B2 a liquid level is maintained to improve the separation of the extract. The entering liquid CO_2 is evaporated continuously at moderate temperature.

Picture page 8:
Multipurpose high pressure pilot unit for the extraction of liquid and solid products (300 bar, 80 °C, 18 kg/h CO2 capacity, 2 litre extractor with 1.2 litre basket insert, 2 m column (ID 38 mm) with structured packing, 2 l/h liquid capacity, 1.2 litre final separator)

SITEC-Sieber Engineering AG
Aschbach 621
CH-8124 Maur/Zürich

Tel +41 44 982 20 70
Fax +41 44 982 20 79
Homepage www.sitec-hp.ch

In the condenser W3 the gas is condensed and in the following metering pump P1 the supercritical solvent is again brought up to the chosen extraction pressure. In the downstream heat exchanger W1 the solvent fluid is heated up to the required extraction temperature which means that the solvent fluid has already reached the extraction conditions when entering the column B3 through the bottom connector.

Flowing up the column in counter flow with the injected liquid from the top, additional extract is dissolved continuously and the closed process cycle continues.

To extract solutes from **solid raw material** the pilot unit is operated in the batch mode. The solid material is introduced into the extractor B1 in a cylindrical basket with filter elements on both ends. These filter elements retain the solids but are permeable for the solvent fluid and the dissolved extract. As in the process with liquids, the solvent fluid dissolves the solute as it flows upstream through the solid raw material.

The further steps of the process are the same as with liquid raw materials e.g. pressure reduction, separation, evaporation, condensing, pumping and heating, thus closing the process cycle again.

The spray nozzle, solution and dissolution may as an option be transmitted on TV-screens or Videos through the installed standard optical windows. In the separator B2 the phase boundaries can be observed and the liquid level is controlled automatically.

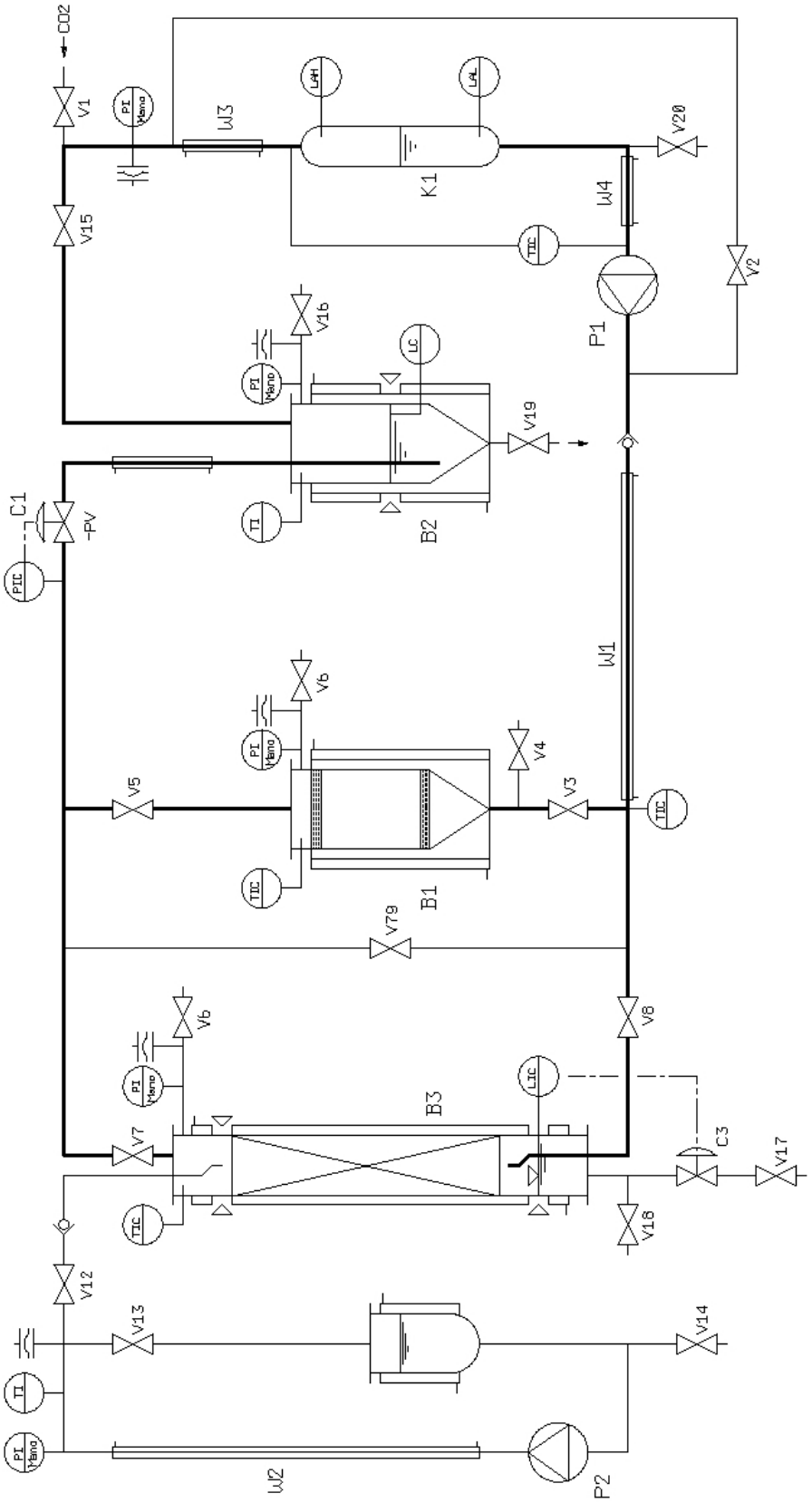
The vessels are easily accessible and the closures are hand operated. The extract from the separator B2 is subtracted manually and the residue from the bottom of the column continuously.

The temperatures are controlled by separate heaters; the extraction pressure by a triggered valve system which eliminates the clogging risk by the extract in the control valve seat.

For reliable scale-up and economical reasons the design of the system is based on pumping the supercritical fluid rather than compressing it in the non-relevant and costly gaseous state. Built for preparative work, the unit can also be used for screening and analytical research. For certain industries such as pharmaceuticals it can even be used as a full production unit. The capacity of this extraction unit is large enough for the production of batches for market testing.

All relevant data are indicated on digital displays on the front panel flow sheet and, as an option may be brought up to a Personal Computer using the SITEC data acquisition system.

Various options are available, such as larger vessel capacities of 2 litre, 4 litre, 10 litre and 20 litre; longer columns; higher pressures, temperatures and mass flows, several vessels in parallel, in series or in the carousel mode.


CO2
V1
PI Manno
W3
LAH
LAL
K1
V20
W4
V15
TIC
V2
P1
V16
LC
PI Manno
V19
B2
TI
C1
PV
PIC
W1
V6
PI Manno
V4
V5
V3
TIC
TIC
B1
V79
V6
V8
PI Manno
LIC
B3
V7
C3
V17
TIC
V18
V12
V13
V14
TI
PI Manno
W2
P2

Standard Design and Options (Please mark required data)

Max. operating pressure:		**300 bar**	500 bar	(700 bar)
Fluid capacity:	10 l/h	☐	☐	☐
	18 l/h	☐	☐	☐
	30 l/h	☐	☐	
	50 l/h	☐	☐	
	100 l/h	☐		

Max. operating temperature: ☐ **80 °C** ☐ 120 °C ☐ 150 °C ☐ 200 °C
☐ .....

Supercritical solvent: ☐ **carbon dioxide** ☐ .....

Extraction vessel capacities:
☐ **1 litre with 600 ml basket insert**
☐ 2 litre with 1.2 litre basket insert
☐ 4 litre with 2.4 litre basket insert
☐ 6 litre with 3.9 litre basket insert
☐ 10 litre with 7 litre basket insert
☐ 20 litre with 14 litre basket insert
☐ .....

Number of extraction vessels: ☐ **1** ☐ 2 ☐ 3 ☐ .....

Extraction column diameter:
☐ ∅ **38 mm**
☐ ∅ 50 mm ☐ ∅ 65 mm ☐ ∅ 90 mm
☐ .....

Extraction column length:
☐ **2 m**
☐ 3 m ☐ 4 m ☐ 5 m
☐ .....

Liquid educt capacity:
☐ **2 l/h**
☐ 4 l/h ☐ 10 l/h ☐ 18 l/h ☐ .....

Options:

☐	Mass-Flowmeter for	☐ carbon dioxide (recommended), ☐ liquid product	☐ entrainer	
☐	Intermediate separation system(s)	☐ 1	☐ 2	☐ 3
☐	Entrainer system			
☐	Data acquisition by PC			
☐	Continuous recovery of	☐ extract	☐ entrainer	
☐	Preparation of rack for RETROFIT of a(n)	☐ extractor	☐ column	☐ separator
☐	Colour camera system with endoscope			

SITEC-Sieber Engineering AG
Aschbach 621
CH-8124 Maur/Zürich

Tel +41 44 982 20 70
Fax +41 44 982 20 79
Homepage www.sitec-hp.ch