SUPERPRESSURE

SUPERCRITICAL FLUID EXTRACTION SYSTEM

MODEL: 46-19360-50Hz
46-19360-60Hz

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SYSTEM OPERATION

CO_2 gas or fluid from a commercial cylinder with dip tube is passed through a 5μ filter to the 10,000psi compressor. Flow and pressure control is achieved by directing the excess fluid back into compressor's suction end. This feed back function is controlled by a back pressure regulator.

The actual flow rate is controlled by a solid stem metering valve and the compressor back pressure regulator. A back pressure regulator is also used to control the separation pressure.

A second atmospheric separation maybe performed in glassware or cold trap before the gas stream passes through the flow meter indicator and is vented to the atmosphere.

The extraction, separation, and back pressure regulator temperatures are controlled and indicated independently by three (3) temperature controllers.

ELECTRICAL SPECIFICATIONS

The input power plug provided with the system is a 3 pole, 4 wire, 20 amp, 250 vac "Twist Lock". Example Hubbell Twist Lock receptacle cat. no. (7410-B) or (Leviton Cat. No. 2441)

The unit requires three-phase 220 VAC input power. A power input disconnect mounted a reasonably short distance from the system is also required.

DANGER

THE NATIONAL ELECTRICAL CODE (NEC) REQUIRES THAT AN INPUT DISCONNECT, SUCH AS A BREAKER, BE PROVIDED IN THE INCOMING POWER LINE AND BE LOCATED WITHIN SIGHT OF THE SYSTEM. DO NOT OPERATE THE SYSTEM UNTIL ALL CODE REQUIREMENTS HAVE BEEN MET. FAILURE TO OBSERVE THIS PRECAUTION COULD RESULT IN FATAL INJURY.

A blown fuse will be indicated by a total lack of power on all instrumentation.

GAS CYLINDER CONNECTION

The system is equipped with a 1/4 high pressure bulkhead fitting for commercial gas cylinder hookup. The fitting, labeled "GAS IN", is located in the lower back corner of the frame. Before connecting the system to a gas cylinder, remove the plug supplied with the inlet connection. Anytime the system is not connected to a gas cylinder, replace the inlet connection plug.

DIAPHRAGM COMPRESSOR AND FLOW CONTROL

The diaphragm type compressor supplied with this system can handle fluids of all types with absolute purity. The compressor can continuously compress supercritical fluids, gas or liquid, to 10,000 psi.
To start the compressor simply press the start button located on the motor starter and the compressor will accelerate to full speed, (approximately 58 RPM). The compressor's stroke rate, thus its flow rate, is constant. Flow rate control is achieved by sending the excess compressor capacity back into the compressor's suction end. Setting the system's flow rate involves balancing the extraction vessel metering valve setting with the feed back loop back pressure regulator setting. First allow the extraction vessel to obtain the desired operating pressure. This is achieved by closing the metering valve and adjusting the feed back loop back pressure regulator to the proper setting. Once the pressure is reached then close the

separation back pressure regulator and drain valve and slowly open the metering valve then set the separation vessel pressure. The metering valve is very sensitive and may require periodic adjustment to maintain the proper flow rate. Often a repetitive adjustment procedure between the metering valve and feed back loop regulator setting is necessary to obtain the correct extraction flow rate and pressure. Additional information pertaining to the operation, maintenance or troubleshooting of either the compressor or back pressure regulator can be found in their individual instruction manuals.

EXTRACTION VESSEL REVERSE FLOW 4 VALVE SYSTEM

The extraction vessel is equipped with four (4) hand-operated valves. These valves provide the means to reverse the flow direction through the vessel. By reversing the solvent flow through the vessel, extraction yields may increase. The valves are number 1 through 4. To provide upward flow through the vessel, open valve 2 and 3 and close valve 1 and 4. Closing valves 2 and 3 while opening valves 1 and 4 will direct the solvent flow downward through the vessel. The above valve information is shown in Table 1.

HEATER AND TEMPERATURE CONTROLLERS

The system is equipped with three (3) heater controllers. Each controller is responsible for regulating the heat supplied to the controlled system. The two controlled systems are the Extraction Vessel, Separation Vessel, and Back Pressure Regulator. Connect system to power source and turn on panel power switch. All controls that are activated are flow totalizer, flow meter, two (2) pressure transducers, and three (3) temperature controllers. Refer to Table 2 Electrical Resistance Heater Specifications.

NOTE: To set, see instruction manual. The three (3) temperature controllers can only process one thermocouple input signal.

CAUTION

The silicone-fiberglass electrical resistance type heaters supplied with the extraction vessel have a burnout temperature of 232°C (425°F). Operating these heaters near or exceeding this temperature will result in heater failure and possible system component damage.

CAUTION

Before start up, unplug heaters on vessels and check pressure regulator, then proceed to check and set temperature controllers. Plug in heaters. To compensate for thermo lag time, i.e. that is vessel and test material density, start at low temperature and slowly increase to working temperature.

CAUTION

The silicone-fiberglass electrical resistance type heaters supplied with the extraction vessel have a low puncture resistance. Avoid situations where the heater may become pinched or otherwise damaged. When removing vessels from their mounting fixtures, remove heaters first. Failure to observe this precaution could damage heater and create a possible shock hazard.

Additional information regarding temperature controller operation, maintenance or troubleshooting can be found in the temperature controller instruction manual.

HAND OPERATED METERING VALVES

The standard system is equipped with one (1) hand-operated high pressure metering valve. This valve controls the flow rate through the extraction vessel. To provide a more accurate flow rate adjustment the valve incorporates a solid stem needle type valve stem.

Valve Maintenance

When tightening the six (6) valves packing, proceed as follows -

1. Remove the screw attaching the packing nut keeper to the valve.

2. Remove the keeper.

3. Tighten the valve packing nut.

4. Replace the valve keeper.

5. Tighten the packing nut keeper retaining screw.

NOTE: Open and close valves slowly to avoid sharp changes in pressure. Pressure shocks caused by opening and closing valves vigorously, generate additional pressure gauge stress. This additional stress might lead to early gauge and pressure transducer failure.

EXTRACTION VESSEL & SEPARATION VESSEL

The extraction & separation vessels provided with the system are rated for 9,200 psi at 100 °F. Corrosion resistant 300 series stainless steel is used to construct the wetted vessel parts. The separation vessel has a 1-1/2 inch inside diameter and an interior depth of 10-1/2 inches. These dimensions provide an interior volume equal to 0.300 liters. The vessels are fitted with J-type thermocouple which attaches to the vessel heads via a standard high pressure fitting, (see table 1 for extraction vessel specifications). The extraction vessel has a 2-9/16 inch inside diameter and an interior depth of 10 inches. These dimensions provide an interior volume equal to 0.845 liters.

WARNING

The extraction and separation vessels provided with this system were tested hydraulically at room temperature at the pressure stamped on the vessel cap, which is 13,800psi. The body caps and head were not stressed beyond their proportional limit at this pressure, and a reasonable safety factor is allowed.

It is possible, however, that certain chemical compounds or mixtures may, under the influence of moderately high pressures and temperatures, cause violent exothermic reactions which may far exceed the test pressure.

Refer to the vessel maintenance section of this manual for extraction and separation vessel assembly and disassembly instructions.

BACK PRESSURE REGULATORS

The system uses two (2) back pressure regulator to set and control the extraction and separation vessel pressure. To adjust the pressure setting, rotate the control knob either clockwise to increase the inlet pressure or counter-clockwise to lower it. Final adjustments should be made in the direction of increasing pressure. Following this rule assures the most accurate set points.

NOTE: The back pressure regulators contain Buna-N o-rings which have a service temperature range of -26°C to +74°C, (-15°F to +165°F). Operating this device outside this temperature range is not recommended.

NOTE: The back pressure regulators contain Buna-N o-rings which are not compatible with all fluids. Buna-N (nitrile) is not recommended for: halogenated hydrocarbons, nitrohydrocarbons, phosphate ester hydraulic fluids, ketons, strong acids, ozone and automotive brake fluid. Therefore exposing this device to these compounds should be avoided.

Additional information regarding back pressure regulator operation, maintenance and troubleshooting can be found in the back pressure regulator manual.

PRESSURE TRANSDUCERS AND GAUGE

The system is provided with two (2) pressure transducers. The first transducer displays the extraction vessel pressure or compressor discharge pressure. The second pressure transducer displays the separation vessel pressure the pressure gauge shows the compressor suction or inlet pressure.

Additional information regarding flow meter operation, maintenance and troubleshooting can be found in the Flow Meter instruction bulletin.

FLOW METER AND TOTALIZER

The compact gas flow meter provide linear & D.C. voltage output for recording gas flow on the totalizer. The flow meter has digital and bar graph displays.

NOTE: The flow meter is set-up and calibrated for use with CO_2 gas.

RUPTURE DISC ASSEMBLIES

The system is provided with two (2) rupture disc assembly. The assembly provides overpressure protection for the extraction vessel and the extraction vessel pressure gauge. If the disc protected system should achieve a pressure exceeding the rupture disc's burst pressure, (the disc is listed to blow within $\pm$ 5.0% of rated pressure), the disc blows allowing the system to depressurize. The rupture disc assembly is tagged with a label indicating the rupture disc's bursting pressure. To order new discs specify the rupture disc assemblies catalog number and the disc's rated bursting pressure.

NOTE: The disc contained in each rupture disc assembly provides an important link to safe system operation. No attempt should be made to substitute spent rupture discs with ordinary flat metal slugs. Operating this device with "discs" of questionable origin and burst pressure could result in vessel failure should critical overpressurization result.

SPECIFICATIONS	EXTRACTION VESSEL	SEPARATION VESSEL
Catalog No.	**41-14375SP**	**41-12158**
Materials of Construction	346 Series Stainless Steel	316 Series Stainless Steel
Weight (Approx.)	59 lbs.	21 lbs.
Pressure Rating @ 100°C	9,200 psi.	9,200 psi.
Type of Seal	Flat Gasket	Flat Gasket
Outside Diameter (inches)	4-3/8	2-9/16
Inside Diameter (inches)	2-9/16	1-1/2
Inside Depth (inches)	10	10-1/2
Approximate Volume (liters)	0.845	0.300

Table 1. Pressure Vessel Specifications.

SPECIFICATION	EXTRACTION VESSEL	SEPARATION VESSEL	PRESSURE REGULATOR
Materials of Construction	Silicone & Fiber Glass	Silicone & Fiber Glass	Silicone & Fiber Glass
Voltage	220 VAC	220 VAC	220 VAC
Wattage	280	134	50
Part Number	80044017400	80044017500	80044017600
Quantity per Device	2	2	1
Total Wattage per Device	560	268	50

Table 2. Electrical Resistance Heater Specifications.

II. SEPARATION & EXTRACTION VESSEL ASSEMBLY/DISASSEMBLY INSTRUCTIONS

A. Extraction & Separation Vessel Access

Access to the extraction & separation vessel is gained as follows: (refer to drawing 41-14375SP and 41-12158)

1. Unscrew gland nuts and remove tubing from the head (4) and body (11).

2. Remove thermocouple by unscrewing the gland nut, and unplugging leads. Also remove collector ring (5).

3. Unscrew capscrews on vessel cap (9). Unscrew cap and lift off.

4. Lift off thrust ring (7) and head (4). Flat gasket (8) will come off with head (4).

B. Assembly

1. Place vessel body (11), if removed, on mounting plate. Install and tighten the two mounting plate to vessel body socket head cap screws.

2. Connect the bottom tubing and tighten gland nuts.

3. Place sample in vessel, (if the sample is a solid it should be sandwiched between two layers of fine stainless steel mesh or glass wool). This prevents the vessel's contents from entering the tubing.

4. Before replacing cap, apply thread lubricant to vessel body threads and cap screw threads. When installing cap over head and thrust ring, proceed as follows:

   a. Unscrew all cap screws until they do not protrude beyond inner face of cap.

   b. Place flat gasket in groove, no additional gasket is needed. Center the head and slip on the thrust ring.

   c. Screw cap on until it just seats on thrust ring. Screw on hand tight only. Then back up cap 1/2 to 3/4 of a turn.

   d. Screw in each cap screw finger-tight.

   e. Tighten each cap screw (preferably with a torque wrench) 1/16 turn and follow screw tightening sequence in figure 1.

   f. After once tightening all screws, repeat step (e) above.

   g. Connect the top tubing, collector ring and thermocouple. Tighten all gland nuts.

NOTE: PROPER TORQUE IS NEEDED TO SEAL THE VESSEL. GENERALLY A CAP SCREW TORQUE OF BETWEEN 25-30 FOOT-POUNDS IS SUFFICIENT TO SEAL THE VESSEL TO 10,000 PSI.

C. Maintenance

Whenever a gasket becomes nicked or work-hardened, replacement is recommended. To replace flat gasket:

1. Spring the gasket from the groove by striking it sharply several times with a cape chisel around the inner and outer circumferences (A and B in figure 2). Do not chisel down on the face of the gasket, or damage to the head will result.

2. Place a new gasket into the groove in the head. There will be a slight amount of clearance between the gasket and the groove. After several cap installations and removals, the gasket will deform and fill the groove. This deformation also forms a ridge (C) on the gasket's face as a result of the vessel body groove (D).

3. When ordering replacement gaskets refer to drawing 41-14375SP and 41-12158 for part numbers