一、超临界分馏原理及概况

超临界萃取技术是现代化工分离中出现的最新学科，是目前国际上兴起的一种先进的分离工艺。所谓超临界流体是指热力学状态处于临界点（Pc、Tc）之上的流体，临界点是气、液界面刚刚消失的状态点，超临界流体具有十分独特的物理化学性质，它的密度接近于液体，粘度接近于气体，而扩散系数大、粘度小、介电常数大等特点，使其分离效果较好，是很好的溶剂。超临界萃取即高压下、合适温度下在萃取缸中溶剂与被萃取物接触，溶质扩散到溶剂中，再在分离器中改变操作条件，使溶解物质析出以达到分离目的。

超临界装置由于选择了 CO_2 介质作为超临界萃取剂，使其具有以下特点：

1、操作范围广，便于调节。

2、选择性好，可通过控制压力和温度，有针对性地萃取所需成份。

3、操作温度低，在接近室温条件下进行萃取，这对于热敏性成份尤其适宜，萃取过程中排除了遇氧氧化和见光反应的可能性，萃取物能够保持其自然风味。

4、从萃取到分离一步完成，萃取后的CO_2 不残留在萃取物上。

5、CO_2 无毒、无味、不燃、价廉易得，且可循环使用。

6、萃取速度快。

近几年来，超临界萃取技术的国内外得到迅猛发展，先后在啤酒花、香料、中草药、油脂、石油化工、食品保健等领域实现工业化。

二、主要技术参数

1、分馏塔最高工作压力：30MPa（设计压力 35MPa）。

2、分馏塔容积： 800ml×4 节。

3、工作温度：室温～80℃。

4、CO_2 最大流量：0—50L/h 可调。

5、电源：三相 220V/60HZ，功率约 15KW。

The main technical parameters

1. The maximum working pressure of the fractionation tower: 30MPa (design pressure 35MPa). The

2. Volume of fractionating tower: 800ml×4 sections.

3. Working temperature: room temperature ~ 80 ℃.

4. Maximum CO2 flow: 0-50L/h adjustable.

5. Power supply: Three-phase 220V/60HZ, power about 15KW.

三、该装置的主要构成

1、CO_2 气瓶：用户自备，纯度 99.5%，且食品级。

2、CO_2 高压调频泵：3TB 50L/50MPa，泵头带冷却，三柱塞，陶瓷柱塞，柱塞冷却，自带水泵及水箱。配变频器，调节输出流量，调频范围 12~26HZ。

3、原料泵：2JX 5L/50MPa 配变频器，调节输出流量。

4、返流泵：2JX 2L/50MPa，配变频器，调节输出流量。

5、分馏塔：包括沉降池， 800ml×4 节/30MPa，材质 316L，结构形式见附图，水夹套加热，分馏塔内根据工艺条件，选装不锈钢填料。

6、分离釜：1L/26MPa，材质 316L，规格ø83*11，数量 2 套，插管至 2/3 处，保证分离效果，水夹套加热。

7、分离收集器：200ml/26MPa，材质 316L，规格ø83*11，数量 1 套，水夹套加热。

8、恒温水浴及换热器：数量 6 套，其中换热盘管 3 套，材质 316L 管，ø6*1，由AB12 工程塑料泵分别供热水给分馏塔Ⅰ Ⅱ Ⅲ、沉降池、分离釜Ⅰ、Ⅱ和原料罐。分离收集器加热温度：室温～80℃，可调。

9、原料罐：2L，材质 316L，水夹套加热，1 套。

10、制冷系统：风冷式制冷机组，制冷量 10KW，CO_2 液化，工况 4—7℃，自动开、停。配CO_2 冷凝盘管及CO_2 贮罐。

11、CO_2 流量计：金属转子流量计，量程 6.3—63L/h，数显远传，瞬时流量+累积流量。

12、压力控制及测量系统：由压力传感器和数显表组成，CO_2 泵出口和原料泵出口为带超压控制功能，设定最高压力超压停泵保护，其余储罐、分馏塔、分离釜Ⅰ、分离釜Ⅱ为压力测量功能。

13、温度控制及测量：设计恒温水浴加热器，分别为：沉降池、分馏塔Ⅰ Ⅱ Ⅲ、分离釜Ⅰ Ⅱ自动控制热水温度。测量分馏塔出口，分离釜分离釜Ⅰ、Ⅱ出口CO_2 的流体温度。

14、触摸屏式PLC 控制采集系统：

（1） 自动控制CO_2泵、原料泵、返流泵输出流量，采集记录。

（2） 自动控制分馏塔、沉降池、分离釜Ⅰ Ⅱ加热温度，采集记录。

（3） 显示采集记录贮罐、CO_2泵出口、原料泵出口，分馏塔出口，分离釜Ⅰ Ⅱ出口压力。

（4） 显示采集记录，CO_2流量。

（5） 人机对话菜单式，表格数据，U 盘导出打印。

15、阀门、管件管线

⑴ 配手动背压阀，控制分馏塔出口压力，美国艾默生，材质 316L。

⑵ 高压阀门、管件管线：DW6，压力 50MPa，管线ø6*1，材质：接触物料为 316L，其它为 304.

The main structure of the device

1. CO2 gas cylinder: user-supplied, purity 99.5%, and food grade.
2. CO2 high pressure frequency modulation pump: 3TB 50L/50MPa, pump head with cooling, three plunger, ceramic plunger, plunger cooling, self-contained water pump and water tank. Equipped with frequency converter to adjust the output flow, the frequency range is 12~26HZ.
3. Raw material pump: 2JX 5L/50MPa equipped with frequency converter to adjust the output flow.
4. Backflow pump: 2JX 2L/50MPa, equipped with frequency converter to adjust the output flow.
5. Fractionation tower: including sedimentation tank, 800ml × 4 knots/30MPa, material 316L, structure form see attached picture, water jacket heating,

stainless steel packing is optional in the fractionation tower according to process conditions.

6. Separation kettle: 1L/26MPa, material 316L, specification ø83*11, quantity 2 sets, cannula to 2/3, to ensure separation effect, water jacket heating.

7. Separate collector: 200ml/26MPa, material 316L, specification ø83*11, quantity 1 set, water jacket heating.

8. Constant temperature water bath and heat exchanger: 6 sets, including 3 sets of heat exchange coils, material 316L tubes, ø6*1, AB12 engineering plastic pump supplies hot water to the fractionation towers Ⅰ Ⅱ Ⅲ, sedimentation tank, separation kettle Ⅰ, Ⅱ and raw material tank. Separate collector heating temperature: room temperature ~ 80 ℃, adjustable.

9. Raw material tank: 2L, material 316L, water jacket heating, 1 set.

10. Refrigeration system: air-cooled refrigeration unit, refrigeration capacity 10KW, CO2 liquefaction, working condition 4-7℃, automatic start and stop. Equipped with CO2 condensation coil and CO2 storage tank.

11. CO2 flowmeter: metal rotor flowmeter, measuring range 6.3-63L/h, digital display remote transmission, instantaneous flow+cumulative flow.

12. Pressure control and measurement system: composed of a pressure sensor and a digital display meter, the outlet of the CO2 pump and the outlet of the raw material pump are equipped with an overpressure control function, the highest pressure overpressure is set to stop the pump protection, and the

remaining storage tanks, fractionation towers, separation kettles Ⅰ. Separation kettle Ⅱ is a pressure measurement function.

13. Temperature control and measurement: design constant temperature water bath heaters: settling tank, fractionation tower Ⅰ Ⅱ Ⅲ, separation kettle Ⅰ Ⅱ automatic control of hot water temperature. Measure the CO2 fluid temperature at the outlet of the fractionation tower and the separation kettle I and II.

14. Touch screen PLC control acquisition system:

(1) Automatically control the output flow of CO2 pump, raw material pump and return pump, and collect and record.

(2) Automatically control the heating temperature of fractionation tower, sedimentation tank and separation kettle Ⅰ Ⅱ, collect and record.

(3) Display the collection pressure of storage tank, CO2 pump outlet, raw material pump outlet, fractionation tower outlet, and separation kettle Ⅰ Ⅱ outlet.

(4) Display collection records, CO2 flow.

(5) Man-machine dialogue menu type, table data, U disk export printing.

15. Valves, pipe fittings and pipelines

(1) Equipped with a manual back pressure valve to control the outlet pressure of the fractionation tower, Emerson, USA, material 316L.

(2) High-pressure valve, pipe fittings pipeline: DW6, pressure 50MPa, pipeline ø6*1, material: contact material is 316L, others are 304.

四、开机前的准备工作

1、首先检查电源、三相是否完好无缺。（220V/60HZ）

2、制冷机内水箱加 30％乙二醇＋70％纯水。（氯离子含量<25mg/L），并接好与CO_2 泵头的冷却水管。

3、CO_2 气瓶压力保证在 5～6MPa。（室温较低时可用电热圈加热）

4、检查管路接头以及各连接部位是否牢靠。

5、将各加热水箱加入去氯离子水，不宜太满，离盖 2-3cm。

6、准备好液体样品装入原料罐。

4. Preparation before starting

1. First check whether the power supply and three phases are intact. (220V/60HZ)

2. Add 30% ethylene glycol + 70% pure water to the water tank in the refrigerator. (Chloride ion content <25mg/L), and connect the cooling water pipe with CO2 pump head.

3. The pressure of the CO2 gas cylinder is guaranteed to be 5-6MPa. (It can be heated by electric heating ring when the room temperature is low)

4. Check whether the pipe joints and the connection parts are firm.

5. Add deionized ionized water to each heated water tank, not too full, 2-3cm away from the cover.

6. Prepare the liquid sample into the raw material tank.

五、开机操作顺序（详见阀门面板上流程图及说明书流程图）

1、接通电源，开电源开关，三相电源指示灯都亮，说明电源正常接通。

2、开制冷机组电源，延迟几分钟后开始制冷，制冷温度上下限已设定好，不需调整，自动控制。

3、开始加温，将分馏塔、分离釜加热开关接打开，并设定好工艺要求的温度。（第一次加水时要及时补充夹套用水量）

4、开始制冷的同时，将 CO_2 气瓶阀门打出开，通过开阀门 2 使 CO_2 气瓶内的 CO_2 进入净化器、冷盘管和贮罐，CO_2 进行液化。液态 CO_2 通过 CO_2 泵、阀门 4 及换热器进入沉降池底部或沉降池上部接口。（工艺选择进口）

5、CO_2 增压:该装置出厂时已将 CO_2 出口保护压力设定在 30MPa，原料泵保护压力设定在 30MPa，一般情况下不需再设定。

6、设定 CO_2 泵工作频率（即流量范围）一般宜在 12—26HZ 之间，与工艺条件有关。

7、开阀门 4 CO_2 进入分馏塔底部（沉降池上口或下口端，工艺选择），开阀门 10、12（此时关闭阀门 9、11）CO_2 与原料进入分离釜Ⅰ进行分离。开阀门 13 溶质 CO_2 进入分离釜Ⅱ再次分离；开阀门 15、1 CO_2 回路循环。调节背压阀门 12 控制分馏塔出口压力，调节阀门 13 控制分离釜Ⅰ出口压力，调节阀门 15 控制分离釜Ⅱ压力（一般工艺阀门 15 不需调整，为常开式）。

8、待分馏塔、分离釜Ⅰ、分离釜Ⅱ CO_2 循环的压力、温度参数稳定与工艺参数要求相似时，再开始进液体原料。（油类物质）

9、原料物质进行精制分馏，原料通过原料泵进入分馏塔的上中下进口端，由阀门 5、6、7、8 根据工艺条件分别选择原料进口。

10、精制出的物质在分离釜Ⅰ收集，如果未达到质量要求可选择分离收集器收集，再用返流泵进入分馏塔顶部再次精制分馏。

11、阀门 3 为分馏塔放空阀。阀门 9 为分馏塔倒气阀，阀门 11 为分馏塔与分离釜Ⅰ倒气阀，阀门 14、16 分别为分离釜Ⅰ Ⅱ放空阀。

Fifth, start-up operation sequence (see the flow chart on the valve panel and the flow chart of the manual for details)

1. Turn on the power, turn on the power switch, the three-phase power indicator lights are on, indicating that the power is normally connected.

2. Turn on the power of the refrigeration unit and start cooling after a delay of a few minutes. The upper and lower limits of the refrigeration temperature have been set without adjustment and are automatically controlled.

3. Start heating, turn on the heating switch of the fractionation tower and separation kettle, and set the temperature required by the process. (The water consumption of the jacket should be replenished in time when adding water for the first time)

4. While starting refrigeration, open the valve of the CO2 gas cylinder. By opening the valve 2, the CO2 in the CO2 gas

cylinder enters the purifier, cold coil and storage tank, and the CO2 is liquefied. Liquid CO2 enters the bottom of the sedimentation tank or the upper interface of the sedimentation tank through the CO2 pump, valve 4 and heat exchanger. (Imported process selection)

5. CO2 supercharging: The CO2 outlet protection pressure has been set at 30MPa and the raw material pump protection pressure has been set at 30MPa when the device leaves the factory. Under normal circumstances, it does not need to be set again.

6. The working frequency (ie flow range) of the CO2 pump should generally be between 12 and 26 Hz, depending on the process conditions.

7. Open the valve 4 CO2 enters the bottom of the fractionation tower (upper or lower end of the sedimentation tank, process selection), open the valves 10 and 12 (close the valves 9 and 11 at this time). CO2 and raw materials enter the separation kettle Ⅰ for separation. Open the valve 13 and the solute CO2 enters the separation kettle II for separation again; open the valve 15 and circulate the CO2 circuit. Adjust the back pressure valve 12 to control the outlet pressure of the fractionation tower, adjust the valve 13 to control the outlet pressure of the separation kettle I, and adjust the valve 15 to control the pressure of the

separation kettle II (general process valve 15 does not need to be adjusted and is normally open).

8. When the pressure and temperature parameters of the CO2 cycle of the fractionation tower, separation kettle I and separation kettle II are stable and similar to the requirements of the process parameters, the liquid feedstock is started. (Oily substance)

9. The raw materials are refined and fractionated. The raw materials enter the upper, middle and lower inlets of the fractionation tower through the raw material pump. The valves 5, 6, 7, 8 are used to select the raw material imports according to the process conditions.

10. The refined material is collected in the separation kettle I. If the quality requirements are not met, the separation collector can be selected for collection, and then the reflux pump is used to enter the top of the fractionation tower to refine and fractionate again.

11. Valve 3 is a venting valve for the fractionation tower. Valve 9 is a degassing valve of the fractionating tower, valve 11 is a degassing valve of the fractionating tower and separation kettle Ⅰ, and valves 14 and 16 are the bleed valves of the separation kettle Ⅰ and II, respectively.

六、注意事项及故障处理

1、此装置为高压流动装置，非熟悉本系统流程者不得操作，高压运转时不得离开岗位。如发生异常情况，要及时停机，关闭电源检查。

2、制冷机组：详见说明书。

3、CO_2 高压泵、原料泵、返流泵的使用保养详见说明书。

4、CO_2 流体系统

(1) CO_2 泵运转时应检查泵头是否有冷却水循环以及柱塞冷却是否正常。（自带水箱和微型水泵）

（2）CO_2 泵开始加压时，应等冷箱内达到制冷温度 CO_2 液化后开始工作，同时打开泵头放空阀门进行放空。

5、加热及控温系统

（1）开机时需检查三相电源是否正确，禁止缺相运行。

（2）每次开机（每班）都要检查各加热水箱的水位，不够时及时补充（因高温蒸发），否则会烧坏电热管和水泵。同时要检查三相水泵电机是否转动正常，特别长期未开机，防止水垢卡死转轴而烧坏电机。

Six, matters needing attention and troubleshooting

1. This device is a high-pressure mobile device. Those who are not familiar with the flow of this system shall not operate it, and shall not leave their posts during high-pressure operation. If an abnormal situation occurs, it should be shut down in time and the power supply should be turned off to check.

2. Refrigeration unit: see the instruction manual for

details.

3. For the operation and maintenance of CO2 high-pressure pump, raw material pump and return pump, please refer to the instruction manual.

4. CO2 fluid system

(1) When the CO2 pump is running, check whether the pump head has cooling water circulation and whether the plunger cooling is normal. (Bring your own water tank and micro water pump)

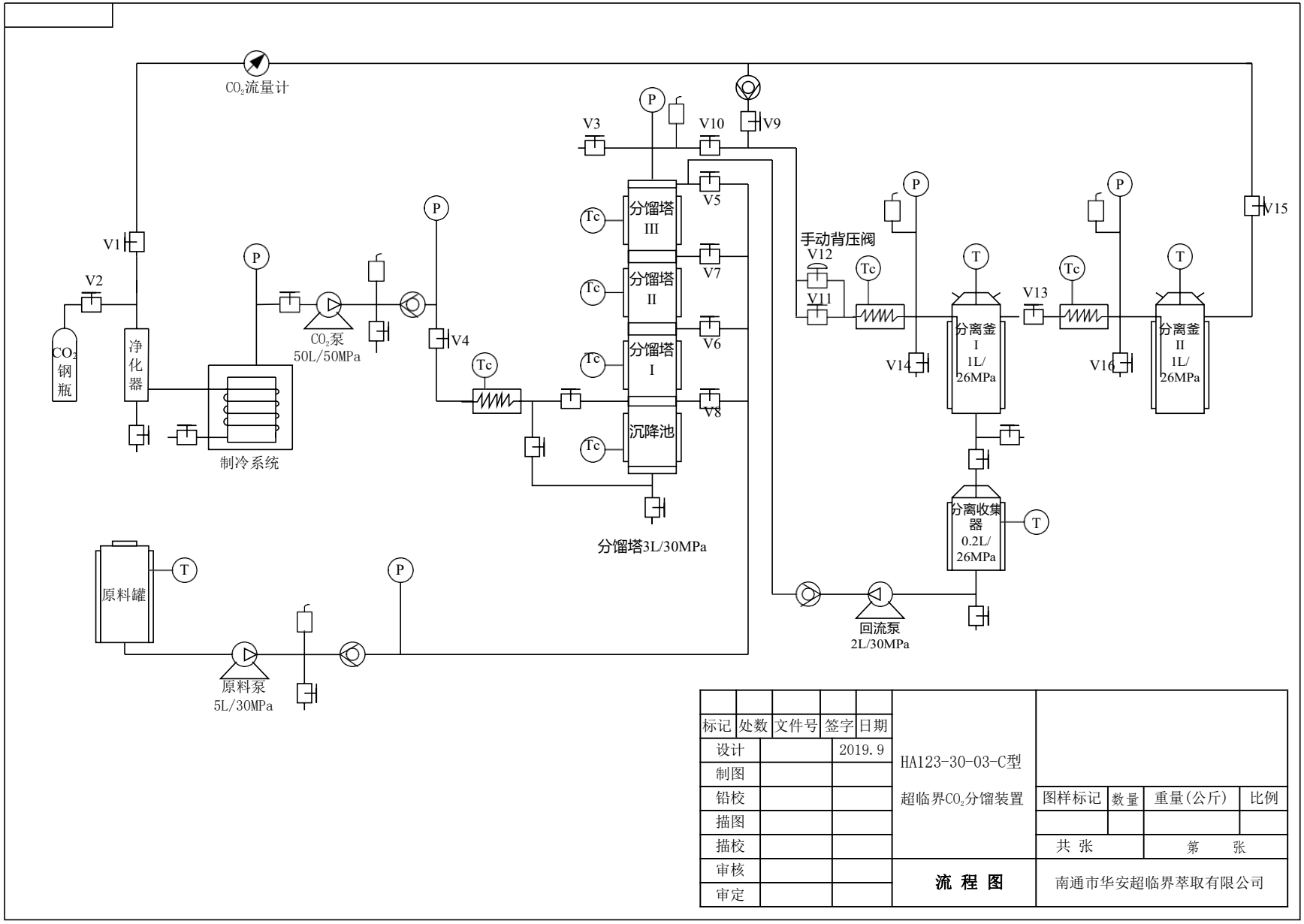
(2) When the CO2 pump starts to pressurize, wait for the CO2 to liquefy in the cold box and start working. At the same time, open the pump head vent valve to vent.

5. Heating and temperature control system

(1) It is necessary to check whether the three-phase power supply is correct when starting up, and it is forbidden to run without phase.

(2) Check the water level of each heated water tank every time it is turned on (each shift), and replenish it in time when it is not enough (due to high temperature evaporation), otherwise it will burn out the electric heating tube and the water pump. At the same time, it is necessary to check whether the three-phase pump motor rotates normally, especially if it has not been turned on for a long time, to prevent the scale from jamming the rotating

shaft and burning the motor.



标记	处数	文件号	签字	日期		
设计				2019.9	HA123-30-03-C型 超临界CO_2分馏装置	图样标记 / 数量 / 重量(公斤) / 比例
制图						
铅校						共 张 / 第 张
描图						
描校						
审核					流 程 图	南通市华安超临界萃取有限公司
审定						