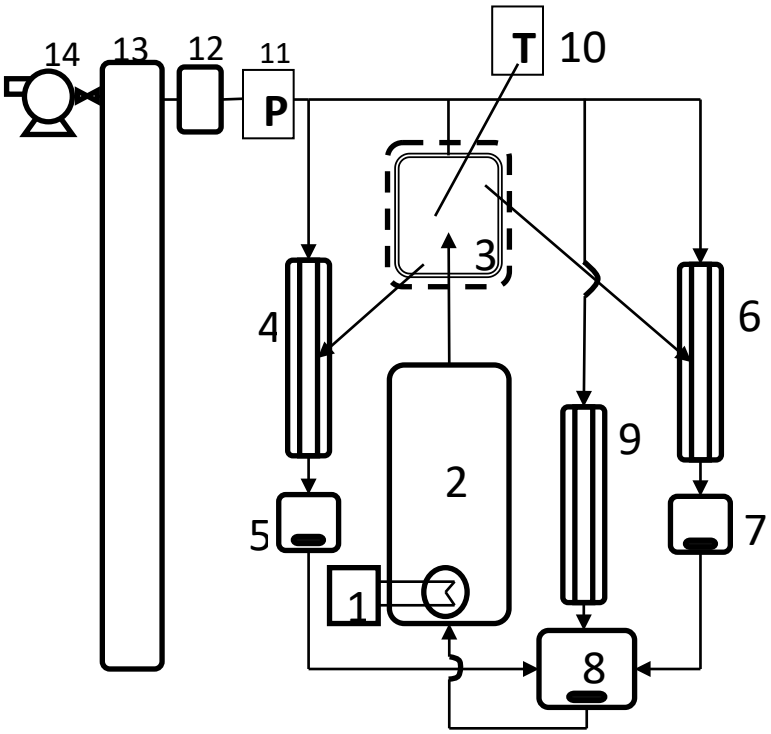
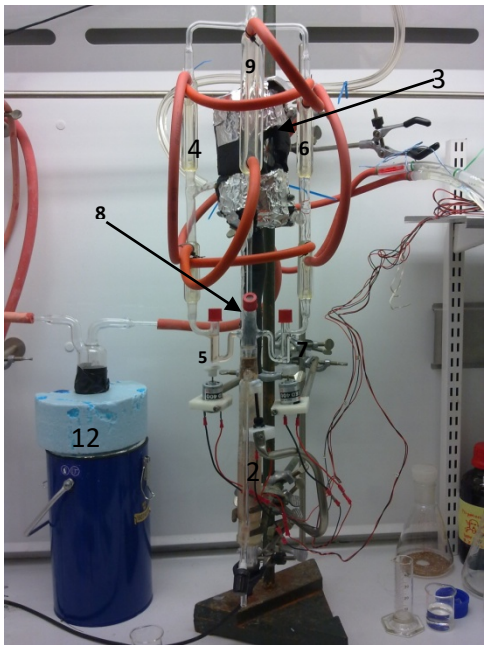
Circulation still (CS)

T: 5-200 C; P(abs): 5 – 110 kPa, liquid chemicals and their mixtures

CS is an equipment for VLE measurements at moderate temperature and pressure. The loaded chemical evaporates in the boiling chamber. The mixed liquid and vapor flow is pushed up by the formation of the gas in the boiler into the equilibrium chamber, where the mixed flow is divided into the liquid and the vapor flows. The gas flow is condensed and the liquid flow is cooled by contacting the cold walls of the condensers. Two liquid streams are passing by the sampling chambers with mixing and by pressure difference are pushed back to the boiling chamber. The pressure in the equipment is regulated by the vacuum pump.





(1) heater, (2) boiling chamber, (3) equilibrium cell, (4), (6), and (9) condensers, (5) and (7) sampling chambers with stirrers, (8) mixing chamber with stirrer, (10) temperature probe, (11) pressure transducer and indicator, (12) liquid nitrogen trap, (13) buffer tank, (14) vacuum pump.

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1. Operation procedure

1.1. Start up

A. Check the liquid level in the water bath (Lauda). Turn on the water bath and the tap water flow for cooling the condensers. Set up the cooling temperature (the temperature difference between the bath and the mixture boiling temperature should be more than 30 °C).
B. Check that the sampling and the mixing chambers are closed with new (unpunched) septa.
C. Using the water pump attached to the CS, reduce the pressure to 600 – 700 mbar and close all valves.
D. Load the solution into the CS by connecting the CS bottom tube to the solution container and opening the valve in-between. The level of the filling should be about one finger below the beginning of the tube connecting the boiler and the equilibrium cell.
E. Open the upper part of the CS (vacuum hose) into atmosphere, or adjust the pressure with the vacuum pump. A side pass of the air should be provided using the systems of valves to set up the exact pressure in the equipment. Remember to use the liquid nitrogen trap to protect the vacuum pump from the vapors from the CS.
F. Turn on heating. The boiler power regulator is to be set from 70 – 120 level. Exact value depends on the heat capacity of the solution. The heating should provide sufficient mixed flow (liquid + vapor) in the tube between the boiler and the equilibrium cell.
G. Turn on the temperature recording on the PC. Start the Ulog program (Desktop), load configuration VLE2.ulg (or other if the temperature display unit was changed). Test devise communication by clicking the display description and “test communication” button. Start logging with “►” button.
H. Turn on mixing in the liquid and the gas sampling chambers.

1.2. Measurements

A. Wait for the temperature equilibration (30-60min, or until the temperature is almost stable in accordance with the temperature figure on the computer).
B. Record the equilibrium temperature and pressure.
C. If the mixture is investigated, take the GC samples: with syringes, take 0.6 – 1.5 ml samples from the sampling chambers. The best way is to take the samples simultaneously, but they can be also taken in series with the minimal difference in time. For volatile compounds the samples should be taken with the valve syringes and transferred to GC bottles instantly (a cold solvent can be placed into the GC bottle in advance to keep the volatile component in the sample).
D. Add the portion of the original solution or one of the component into the mixing chamber. The added volume should be equal to the withdrawn volume.
E. For isothermal measurements, when one component is added, the composition of the mixture change and the pressure is to be adjusted to keep the measurement at the same temperature.
F. Change the pressure of the system, if a new VLE point at different temperature or pressure to be measured.
G. Run the GC samples to determine the phase compositions.
H. Return to step A

I. The template for the processing the measurement results is not available.

1.3. Shutdown

A. Turn off heating (0 value of the boiler power regulator) and UNPLUG! the regulator.
B. Turn off the mixing in the sampling chambers
C. At reduced P measurements, turn off the vacuum pump. Disconnect the nitrogen trap and pour the liquid nitrogen back to the main liq. nitrogen container.
D. After the boiling of the solution stops, turn off the cooling (water bath and tap water).
E. Wait for the cooling of the CS and discharge the solution (if no further measurement with this solution is planned).

1.4. Equipment cleaning

A. **Regular cleaning**: Following the procedure of starting up and shutdown, clean the equipment with the boiling of a suitable solvent (2-3 times). The cleaning solvent should boil for 30-40 minutes inside the CS to clean all internal surfaces. The suitable solvent after an organic solution measurement can be acetone (first portion can be the technical grade and last - the analytical grade acetone). Acetone is to be used at the end of cleaning. The leftovers of the acetone are to be removed from CS by applying vacuum.

2. Risk assessment (possible hazardous scenarios)

<table>
<tr><th>Risk action</th><th>Hazard (factor of danger)</th><th>Impact</th><th>Prevention tools</th></tr>
<tr><td>An overheating</td><td rowspan="3">Pressure build up</td><td>Break of the glass equipment</td><td rowspan="2">The operation instructions</td></tr>
<tr><td>An insufficient cooling</td><td rowspan="2">Release of the chemicals</td></tr>
<tr><td>All valves are closed</td><td>Protective clothes</td></tr>
<tr><td rowspan="2">A withdrawing of a hot solution</td><td rowspan="2">Temperature</td><td>Skin burn</td><td>Protective clothes</td></tr>
<tr><td>Eye injury</td><td>Protective glasses</td></tr>
</table>

What to do IF

1. Turn **OFF HEATING** and unplug it.
2. **OPEN** vacuum hose into atmosphere & turn OFF the vacuum pump;
3. Other instructions are in accordance with injury type and/or chemical type

3. Maintenance

3.1. Regular services (including cleaning between the measurements)

A. **Regular cleaning**: see section 1.4
B. **General cleaning**: is made with base and acid water solutions (1M NaOH and 1M HCl) following the procedure described in 2.1.A
C. **Oil addition / replacement** to the temperature probe pocket has to be done ones in half a year.

D. **Calibration of the pressure meter.** The reference pressure meter is to be attached close to the main pressure meter. The pressure is adjusted with the vacuum pump and two pressure meter values are recorded.

E. **Calibration of the temperature meter.** There are three approaches to the temperature uncertainty estimation.
   1. is to use MIKES calibration certificate (usually it is the smallest value).
   2. Is to calculate the oscillation of the temperature before the sampling/recording based on the temperature recording.
   3. Is to determine boiling temperature of the distilled water and compare it with the literature value (pressure correlation for the pressure meter should be taken into account).

3.2. Replaceable parts

Part name	Supplier/ amount	Approx. price	Live time / storage need
Septa FOR13-425, Teflon/sil, 10/50	ThermoScientific		2 month of meas. / 1 pack (100)
Syringes, 1 ml with lock	Hamilton	200	1-2 years / NO