

Ceramic Nanofiltration Setup: Design and Upscale

CIE5050-09 Additional Thesis

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1 Introduction

Surface water is one of the most significant sources for drinking water supply. Natural organic matter (NOM) is always present in surface water, and it can cause several problems in drinking water treatment. First, NOM is responsible for the odor and color in drinking water. Second, it increases the demand for chemicals, e.g., coagulants. Third, NOM reacts with disinfectants and can form harmful disinfection by-products (DBPs) (Matilainen and Sillanpää, 2010). Moreover, NOM can also reduce the efficiency of drinking water treatment for the removal of other compounds, and causes membrane fouling, biological instability and bacterial growth.

As most of NOM in natural sources is negatively charged, Anionic Ion Exchange (IEX) is one of the methods applied to remove the NOM in surface water. IEX uses ion exchange resin that has to be periodically regenerated. This regeneration process produces a waste stream with high salt and NOM concentration, called brine. The brine is difficult and expensive to be treated, and it is a drawback for applying IEX in larger scale (Verdickt, 2012).

The problem can be mitigated by reducing the volume of the waste brine. This can be achieved by recovering resources present in the waste brine, e.g., salts for IEX regeneration and NOM for agricultural use. TU Delft is studying Ceramic Nanofiltration membranes to concentrate NOM and separate it from salts, and chemical precipitation to recover a clean solution with regeneration salt (i.e., NaCl) from the membrane permeate.

The aim of this additional thesis is to upscale a previous ceramic nanofiltration setup (Fig 1), which should run overnight without supervision. This thesis is divided into two parts: (1) design and building, and (2) experiment. For the design and building part, the main modifications of the upscaled setup compared to the previous setup are (1) the use of a membrane with larger filtration area, and (2) a modified recirculation system that allows to work at constant membrane flux instead of constant pressure. The new recirculation system should guarantee higher recovery of the membrane. For the experiment stage, the aim is to test the new upscaled set-up at different feed flow rates and different concentrate flow rates. The feed in both cases is demineralized water. The initial design of the new setup is shown in Fig 2, and it will be revised and discussed in this report.

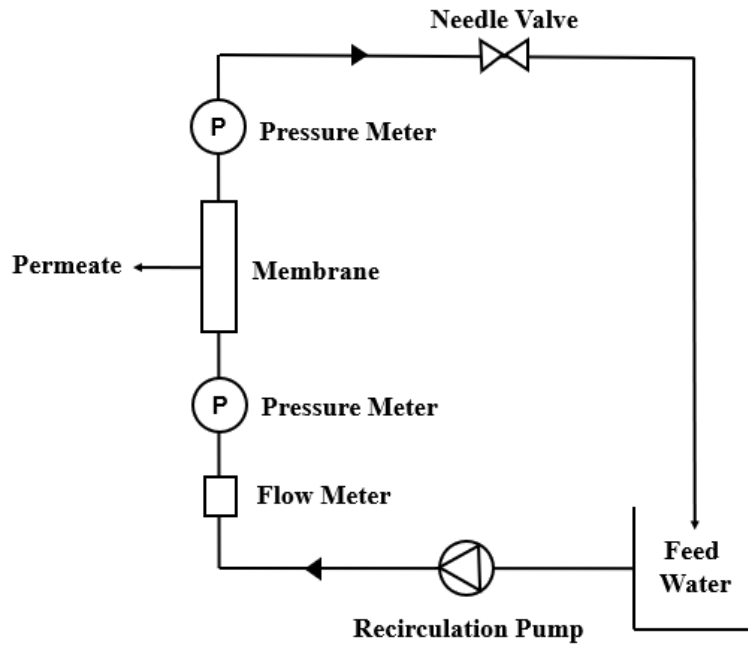


Fig 1 Previous set-up

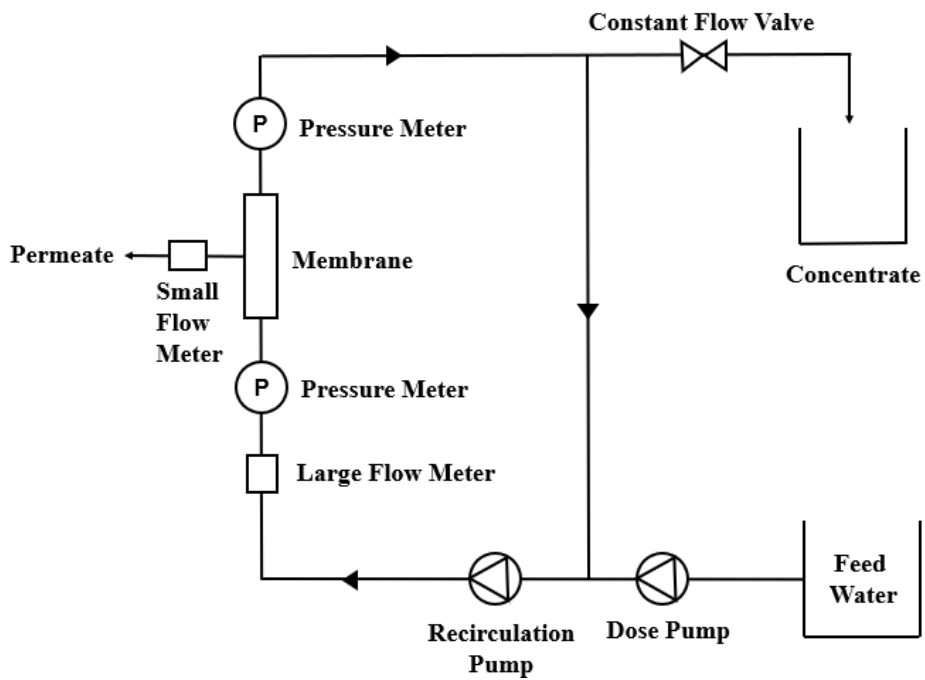


Fig 2 Initial design of the new set-up

2 Design and building part

2.1 Setup description

The experimental setup (Fig 3) includes two pumps, three ball valves, two pressure meters, two flow meters and one constant flow valve. The aim of the experimental setup

is to have a constant membrane flux system and reach 80%-90% recovery of the nanofiltration membrane. All the parts and tubes of the setup are corrosion resistant. Compared to the initial design of Figure 2, other components have been added (Fig 3): three ball valves, and a second feed water tank. The additional components are added to the initial design because air inside the system may cause damage to the two pumps. With the components of the new design, the air can be removed completely from the system before running it. The detailed procedure would be discussed later.

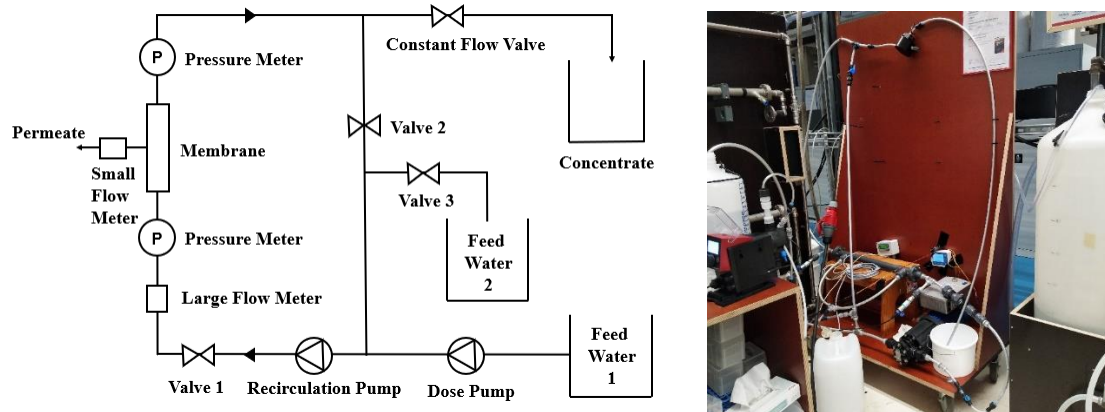


Fig 3 Experimental setup

2.2 Installation

2.2.1 Dose pump and spring load valve

The dose pump (DDA 12-10, Grundfos, the Netherlands) (Fig 4) is one of the most important equipment in the setup, because it regulates the feed flow and the pressure. This membrane pump works via pulsation to extract a constant amount of solution for each pulse from the feed tank. Therefore, it can provide a constant feed flow by adjusting the pressure of the system. Before running the dose pump, the air should be removed from a secondary outlet, which is near the inlet of the pump. The procedure of it is shown in its manual and in the Appendix. Moreover, it should be noticed that the dose pump shows a good performance under the pressure range from 2.5 to 10 bar.

However, if the pump is operated alone, the pressure difference between inlet and outlet of the pump is negative, giving an alarm. Therefore, a spring load valve (PV-G5/8-3PV/T/C X nutG5/8, Grundfos, the Netherlands) (Fig 5) is used with the pump. To overcome the resistance given by the spring inside the valve, the dose pump has to increase the pressure until reaching 2.5 bar, and after that it can run without alarm.



Fig 4 Dose pump



Fig 5 Spring load valve

2.2.2 Ceramic nanofiltration membrane

A ceramic nanofiltration membrane from Inopor (Fig 6) is used in the experimental setup. The membrane has 19 channels with a diameter of 3.5 mm. The filtration length of the membrane is 48 cm. One of the set-up requirements is that the membrane is operated at turbulent flow conditions, to avoid concentration polarisation. This means that the Reynolds number should be higher than 3500. The Reynolds number can be calculated using the formula (1). Under 25 degree Celsius, the dynamic viscosity of water is $0.9 \cdot 10^{-3}$ Pa·s and the kinematic viscosity of water is $0.9 \cdot 10^{-6}$ m²/s. Therefore, the flow rate for one channel should reach at least 0.52 L/min. The minimum flow rate for the whole membrane should be 9.87 L/min. Due to the friction along the tubes and equipments, 11 L/min or 660 L/h is set to be the requirement for the recirculation pump. In this case, the velocity of the cross flow should be around 1 m/s. Moreover, the permeability of the membrane under different TMP should be nearly the same.

$$Re = \frac{\rho v D}{\mu} = \frac{Q D}{\gamma A} \quad (1)$$

Where:

ρ is the density of the fluid (SI units: kg/m³);

v is the velocity of the fluid with respect to the object (m/s);

D is a diameter of the membrane channel (m);

μ is the dynamic viscosity of the fluid (Pa·s or N·s/m² or kg/m·s);

γ is the kinematic viscosity of the fluid (m²/s);

A is the area of the cross section.



Fig 6 Membrane with coating

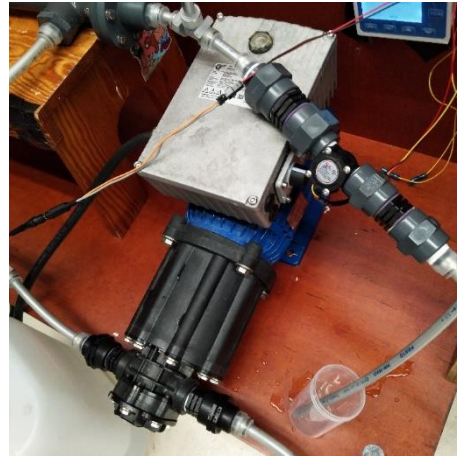


Fig 7 Recirculation pump

2.2.3 Recirculation pump

The recirculation pump (E12MBVK-X, Axflow, the Netherlands) (Fig 7) should provide high recirculation cross flow. The recirculation cross flow is regulated by adjusting the RPM. According to the discussion in section 2.2.2, the pump should provide 660 L/h cross flow in order to generate turbulent flow condition in the membrane.

2.2.4 Pressure meter and flow meter

Two flow meters, a smaller one (FCH-micro-PP, Biotech, 0.03-2.5 LPM) and a larger one (ZJ-LCD-M, Sea Zhong Jiang, 1-9999 LPM) measure the permeate flow and the cross flow, respectively. Two pressure meters (GS4200-USB, ESI Technology Ltd) measure the pressure at the feed side and at the concentrate side of the membrane. The transmembrane pressure (TMP) is the average of these two pressures. The two pressure meters are connected to a computer, and the measured pressure are continuously recorded by the software (ESI). The manual for this software is shown in the Appendix 6.4. The data recorded is used to calculate the average feed pressure and concentrate pressure in each time period.



a



b



c

Fig 8 Pressure meter and flow meters.

a. Pressure meter. b. Small flow meter. c. Large flow meter

2.2.5 Constant flow valve

The constant flow valve (FC-001, Bronkhorst, the Netherlands) (Fig 9) allows to achieve the constant concentrate flow. It can operate at maximum 10 bar and the flow range is between 0.18 and 1.2 L/h. Together, the constant concentrate flow and feed flow determine the permeate flow. This means that the system is running with a constant permeate flow by automatically adjusting the pressure. In order to check the accuracy of the constant flow valve, a small setup, shown in Figure 11, is run under different pressure and feed flow rate (results in Appendix 6.1.1 Table S1). The results show that the average relative error is 4%, which is acceptable.

A balance (KERN) (Fig 10) is used to measure the weight of the water that passes through the constant flow valve. The balance is connected to the computer via an USB, and the values are recorded by a software (KERN Balance). The concentrate flow can be calculated after converting the weight of the water into volumes of water. The manual for this software is shown in the Appendix 6.5.



Fig 9 Constant flow valve



Fig 10 Balance

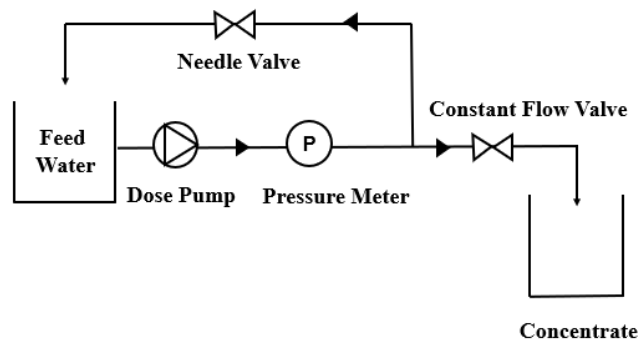


Fig 11 Small setup for testing constant flow valve

2.2.6 Hydraulic connectors

The hydraulic tubing connectors (NPQP-D-Q10-Q8-FD-P10, NPQP-D-Q8-Q6-FD-P10 and NPQP-T-Q12-E-FD-P10, Festo) (Fig 12) are resistant to pressures up to 10 bar and are resistant to corrosion. There are two types of connectors used in the setup, one-way connectors and two-way connectors, or T connectors (Fig 12). The one-way connectors are installed in both the inlet and outlet of the dose pump, the small flow meter, the constant flow valve and each ball valve. The T connectors are mainly used in the recirculation part and connecting the two pressure meters in both sides of the membrane.



Fig 12 Hydraulic connectors.

a. Two-way connector. **b.** T connector

2.2.7 Ball valves

The ball valves (VZBE-3/8-T-63-D-2-M-V15V15, Festo) are used to control the flow direction in the system. Like the hydraulic connectors, the ball valves can also operate at 10 bar and are resistant to corrosion. They are used to remove the air from the system, to protect the pumps from cavitation. The constant flow valve cannot be used for this purpose because its allowed flow rate is too low. The setup for it is shown in Fig 13. The procedure for removing air from the system is as follows.

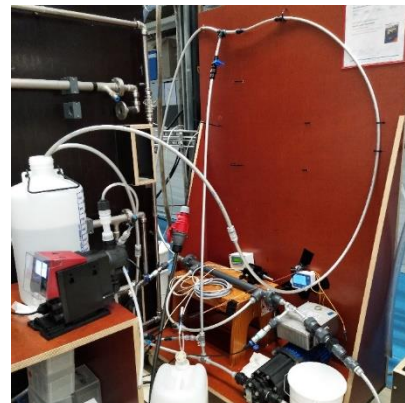
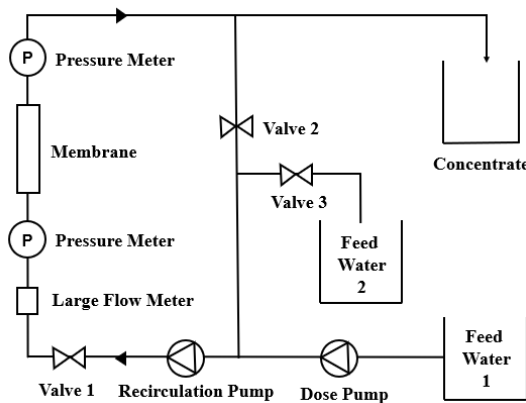


Fig 13 Setup for releasing the air in the system

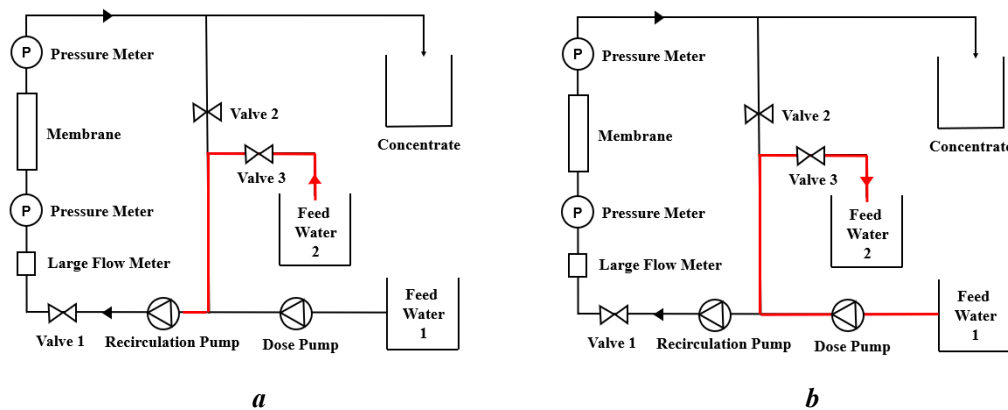
First, make sure the tube is connected to the tap of feed tank 2, and close the outlet of the permeate from the membrane. Then detach the tube connected to the inlet of the recirculation pump and open all valves and two feed water taps. Connect that tube directly to the recirculation pump when the flow goes out from it. The step is to check there is no air would enter the recirculation pump along the short route (Fig 14a).

Second, close valves 1 and 2. Then detach the tube connected to the tap of feed tank 2 and put it into the bottom of the feed tank 2 from the upper part. Then release the air from the dose pump before running it. The manual for it is shown in Appendix 6.2. The step 2 aims to release the air from the small part which is shown in Fig 14b.

Third, close valves 1 and 3. Then open the dose pump for removing the air in the recirculation part (Fig 14c).

Fourth, stop the dose pump and close valve 2. Then open the recirculation pump. In this step, the feed is from the feed water tank 2 other than tank 1. The air inside the main part of the system would be released (Fig 14d).

Lastly, close valve 3 and open the outlet of the permeate from the membrane. Then install both the constant flow valve and the small flow meter as shown in the Fig 3, and open the two pumps. The dose pump should be open first. After running the system, close the recirculation pump first and then close the dose pump.



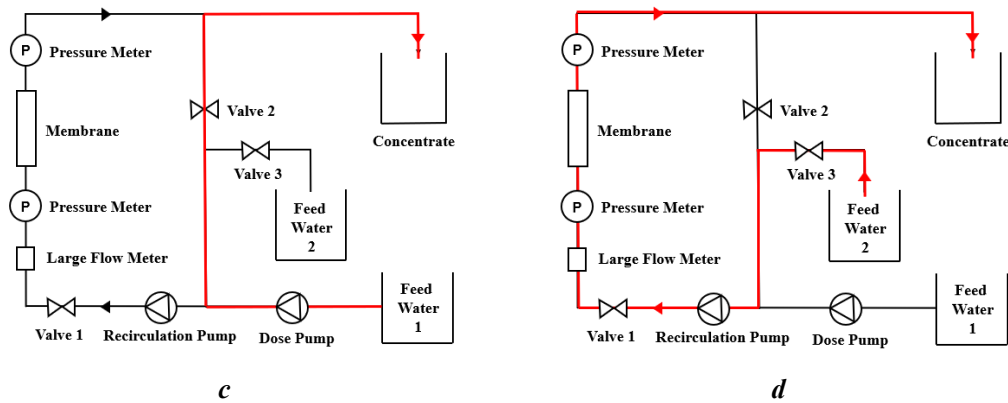


Fig 14 Procedure for releasing the air

3 Experiment part

3.1 Requirement and method

The requirement for this setup are summarized in Table 1.

Table 1 Requirement for main components

Component	Requirement
Membrane	1.Reynolds number higher than 3500 2.Same permeability 3.Recovery higher than 80%
Dose pump	Pressure between 2.5 and 10 bar
Recirculation pump	1.Cross flow higher than 11 L/min or 660 L/h 2.Cross flow velocity around 1 m/s

In the experiments, the setup is run at different feed flows and different concentrate flows. The concentrate flow is set to be around 0.25 L/h, 0.47 L/h, 1.5 L/h. by adjusting the constant control valve. Under each concentrate flow, the setup was run with different feed flows, i.e., 8 L/h, 10 L/h and 12 L/h. Each experiment runs for 10 minutes.

3.2 Results and discussion

The results of the experiments at different feed flows and different concentrate flows are shown in Table 2. The maximum cross flow is around 400 L/h (or 0.6 m/s), which is much lower than the required cross flow of 660 L/h (or 1 m/s), as shown in Table 1. This is not expected considering the pump specification, and might be due to resistance caused by the valves and by the narrowing of the tubes in the proximity of the valves of the recirculation loop. The Reynolds number, 2333, is lower than 3500, which means

that there is no turbulent flow in the membrane channels. This might cause the rejection performance of the membrane due to concentration polarisation. The recovery of the system can be higher than 80 % and can even reach 97%. The permeability under the different conditions is almost the same. Therefore, the recovery and the permeability meet the requirements.

In addition, after installing the spring load valve, the pressure inside the dose pump is in the range of 2.5 and 10 bar, as expected according to the pump specifications.

Table 2 shows that, after fixing the concentrate flow at low, medium and high flow which are around 0.25 L/h, 0.47 L/h, 1.5 L/h, and running the setup with different TMP, the work of the constant flow valve is relatively stable and accurate.

Table 2 Setup under different conditions

Feed Flow, L/h	Concentrate, L/h	TMP, bar	Cross Flow, L/h	Calculated Permeate, L/h	Flux, LMH	Recovery, %	Permeability, LMH.bar
8 L/h	1.54	3.51	413.40	6.46	64.50	80.81	16.72
	0.48	3.93	412.20	7.52	75.01	93.98	17.36
	0.26	4.26	414.30	7.74	77.26	96.79	16.50
10 L/h	1.43	4.38	418.20	8.57	85.46	85.66	17.72
	0.47	4.84	410.40	9.53	95.04	95.26	17.85
	0.25	5.34	401.70	9.75	97.27	97.49	16.55
12 L/h	1.48	5.62	405.90	10.52	104.94	87.65	16.97
	0.46	6.09	400.20	11.54	115.15	96.18	17.17
	0.25	6.49	394.50	11.75	117.27	97.95	16.43

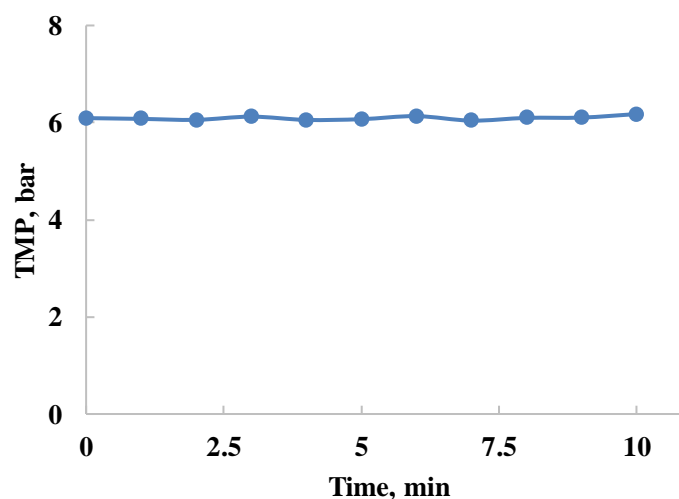


Fig 15 TMP changing within a period of 10 min

The data displayed in Fig 15 shows the TMP during one of the the experiments. In this

example, the TMP is around 6 bar and is quite stable in the time. This means that the setup is not influenced by the pulsation of the dose pump.

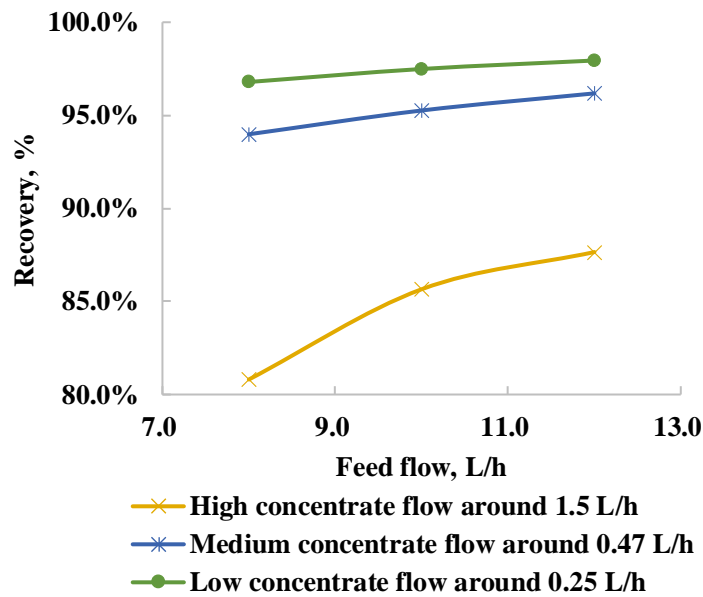


Fig 16 Recovery under different feed flows. Different colors show different concentrate flows.

Fig 16 shows that the recovery is always higher than 80%, and that the recovery increases when the feed flow increases or when the concentrate flow decreases. The recovery is the ratio between permeate flow and the feed flow, and the feed flow is the sum of the permeate flow and the concentrate flow. If the concentrate flow is fixed and the feed flow increases, the permeate flow increases as well, which leads to higher recovery. Similarly, if the feed flow is fixed and the concentrate flow decreases, the recovery increases.

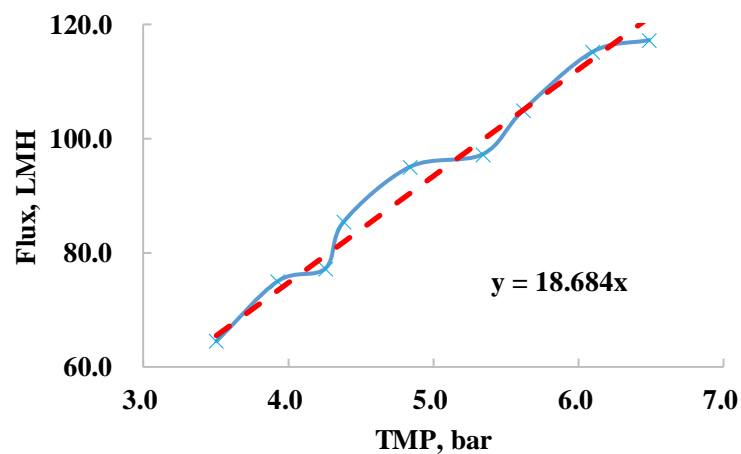


Fig 17 Flux under different TMP. Different colors show different feed flow.

Fig 17 shows that TMP increases simultaneously with the flux. The flux is the ratio

between the permeate flow and the surface area inside the membrane, which means that the flux increases when the permeate flow increases. As previously discussed for Fig 16, the decrease of the concentrate flow or the increase of the feed flow will increase the permeate flow, and will consequently increase the flux. This should cause higher TMP in the system as well. As shown in Fig 17 and Table 2, a higher the feed flow means a higher flux and a higher TMP. Table 2 shows that, at constant feed flow, a lower concentrate flow means a higher flux and a higher TMP. Hence, there is a positive correlation between the flux and TMP, as expected for membrane filtration. The slope of the fitted linear regression line, 18.684 LMH,bar, is the permeability.

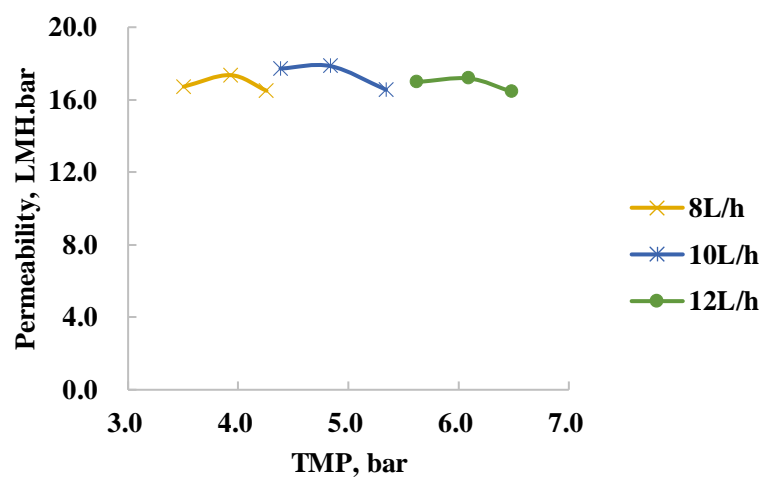


Fig 18 Permeability under different TMP. Different colors show different feed flow.

As shown in Fig 18, the permeability is always in the range of 16 to 18 LMH.bar which is similar to the slope shown in Fig 17, even when the system runs under different conditions. This means that the system is quite stable. Due to the low cross flow, the membrane cannot work in a turbulent environment. This might cause concentration polarization.

4 Conclusion

The installed setup provides suitable pressure, high recovery and stable permeability. However, some of the requirements are not met yet. Based on the discussion, the conclusions are as follows.

1. The recirculation cross flow, and thus the Reynolds number, is much lower than the target to achieve turbulent flow inside the membrane channels. This is caused by limitations of the recirculation pump.

2. One of the requirements of the setup is to work at constant-flow. The results show that this is achieved. The setup can provide high recovery and stable permeability.
3. The constant flow valve is accurate, and guarantees that the setup operates at constant fluxes with high recoveries.
4. In a constant-flow system, there is a positive relationship between flux and TMP.

With the following suggestions, the setup can be further improved.

1. During the procedure of releasing the air in the system, it is not so convenient to close the permeate and get rid of the constant flow valve each time. Some methods could be raised to make it more flexible. The method to remove the air from the system needs to be further improved.
2. The recirculation pump cannot be able to provide 660 L/h even at maximum RPM. In order to reach turbulent condition in the system, the pump should be modified.
3. The constant flow valve needs to be checked whether it can stand particles in the concentrate flow of brine.

5 References

- Matilainen, A., & Sillanpää, M. (2010). Removal of natural organic matter from drinking water by advanced oxidation processes. *Chemosphere*, 80(4), 351-365.
- Verdickt, L. (2012). Verwijdering van NOM door middel van ionenwisseling. Report Vlaamse Maatschappij voor Water Voorziening.

6 Appendix

6.1 Manual for constant flow valve

The FLOW-CONTROL is an enhanced needle valve, which contains a mechanism to eliminate pressure fluctuations, thereby creating a constant flow. It is operated just like a regular needle valve: to open, turn the control knob counterclockwise, to close, turn it clockwise.

6.1.1 Test the constant flow valve

The way to test the constant flow valve has been discussed in section 2.2.5. The relevant data is shown in Table below. Under different pressure and feed flow, the average

relative error is still about 4%, which is very accurate.

Table S1 Test data about constant flow valve

Feed flow, L/h	Pressure (Dose pump), bar	Pressure meter, bar	Weight, g	Flow rate, L/h	Relative Error	Average Error
3	2.9-3.2	1.7-2	57.32	0.34392	0%	-4%
	4-4.6	2.7-3.5	55.89	0.33534	-2%	
	4.8-5.1	3.4-4	55.1	0.3306	-4%	
	5.8-6.7	4.4-5.5	53.33	0.31998	-7%	
6	2.7-3.3	1.5-2	51.18	0.30708	0%	-3%
	3.7-4.3	2.3-2.8	50.68	0.30408	-1%	
	4.7-5.8	3.1-4.9	50.23	0.30138	-2%	
	5.8-6	4.1-4.7	48.35	0.2901	-6%	
10	2.9-3.5	1.3-2.1	50.13	0.30078	0%	-4%
	3.8-4.3	2.2-3.2	48.13	0.28878	-4%	
	4.8-5.0	3.0-4.0	48.1	0.2886	-4%	
	5.7-6.3	4.0-5.4	47.65	0.2859	-5%	

6.1.2 Points of attention

1. Check the system for leaks before applying pressure, especially if toxic, explosive or other dangerous fluids are used.
2. Be sure to apply the specified operating pressure(s). Avoid pressure shocks and bring the fluidic system gradually up to the level of operating conditions; open and close the fluid supply gently.
3. Do not pressurize the instrument beyond the maximum working pressure.
4. When closing the valve, do not tighten the control knob with excessive force. This might damage the sealing surface of the needle valve, which seriously reduces performance. The needle might even get jammed in the valve seat, blocking the flow permanently.

6.2 Manual for dose pump

Before running the dose pump, the air in the inlet route need to be released by inserting a tube in the hole near the inlet of the pump. As shown in the figure below, then press the button '100%' and turn the button which is in the middle to adjust the releasing time. Make sure there is no air flowing out from the transparent tube.

After that step, running the pump by pressing the arrow button which is in the left side.

The feed flow rate can be changed by using the middle button.

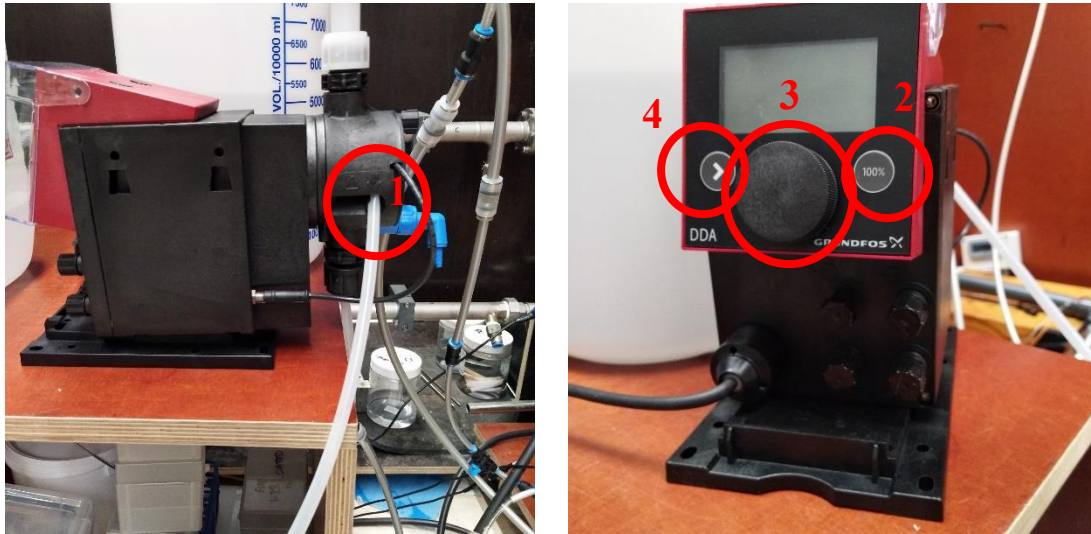


Fig S1 Dose pump

6.3 Manual for recirculation pump

There are two buttons in the side of the recirculation pump. The left button is used to control the direction of the flow. If turn the button to the left, the flow is shown as an arrow in the figure below. The other button is to control the RPM.

When running the pump, the first step is to choose the flow direction via the left button and then adjust the RPM.

Always make sure there is no air inside the pump. If there is weird noise, open the valve in the bottom of the black part to release the air.

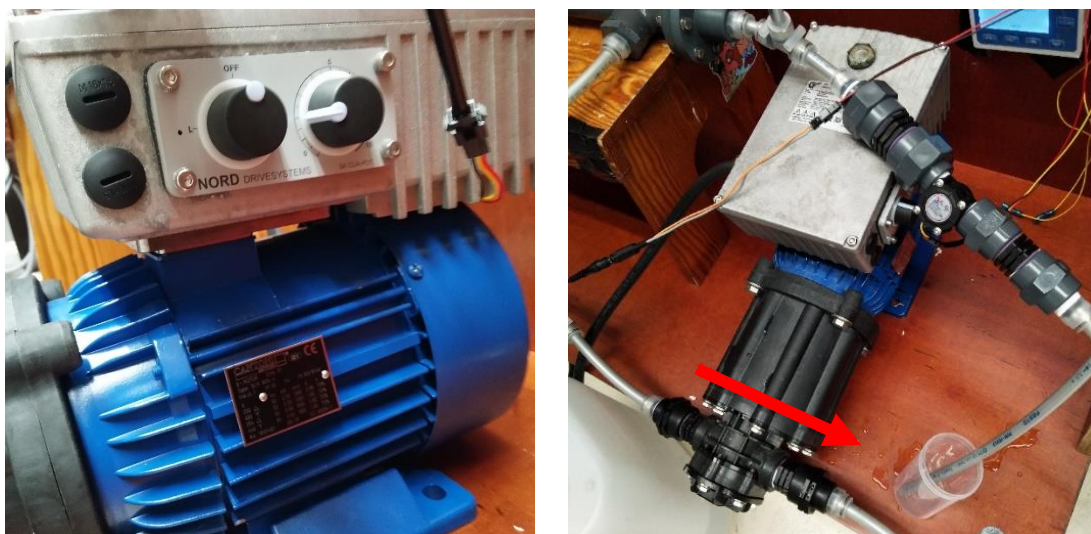
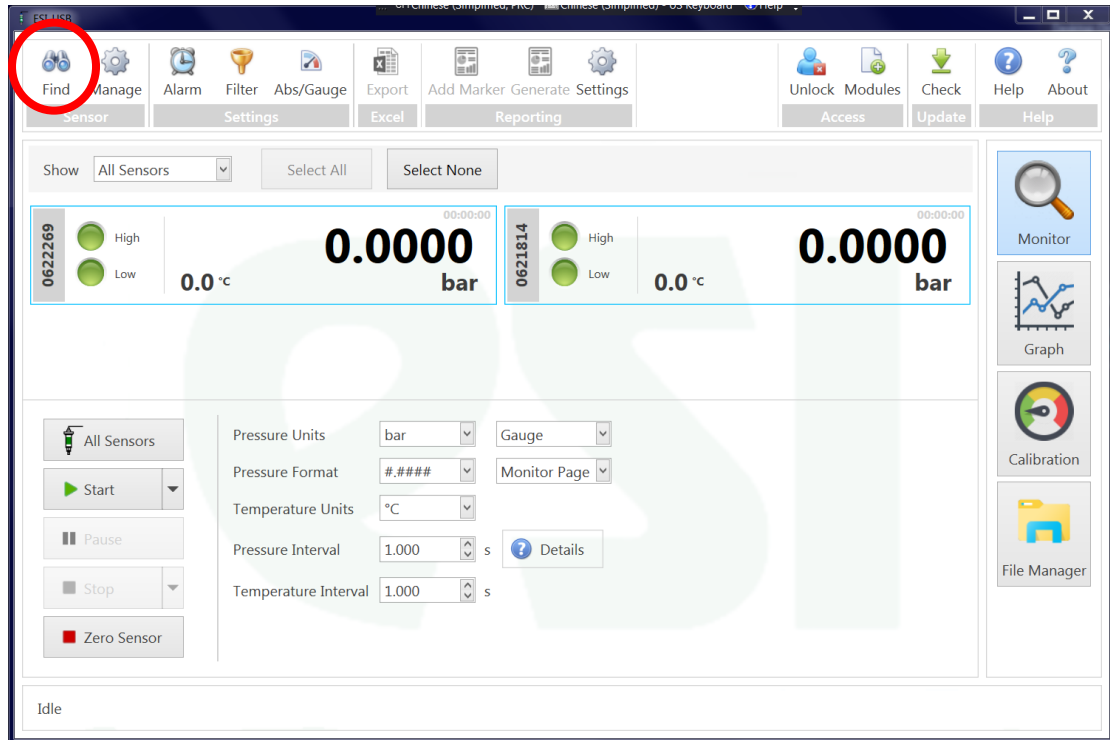


Fig S2 Recirculation pump

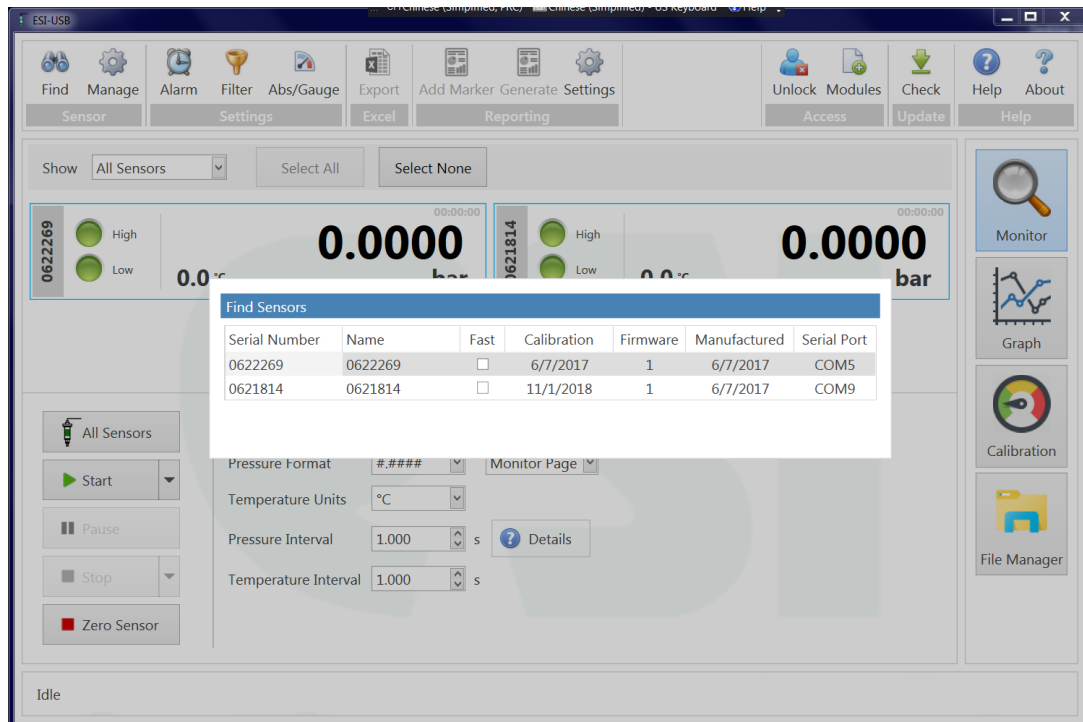
6.4 Manual for ESI

The manual for the Software 'ESI' is as follows.

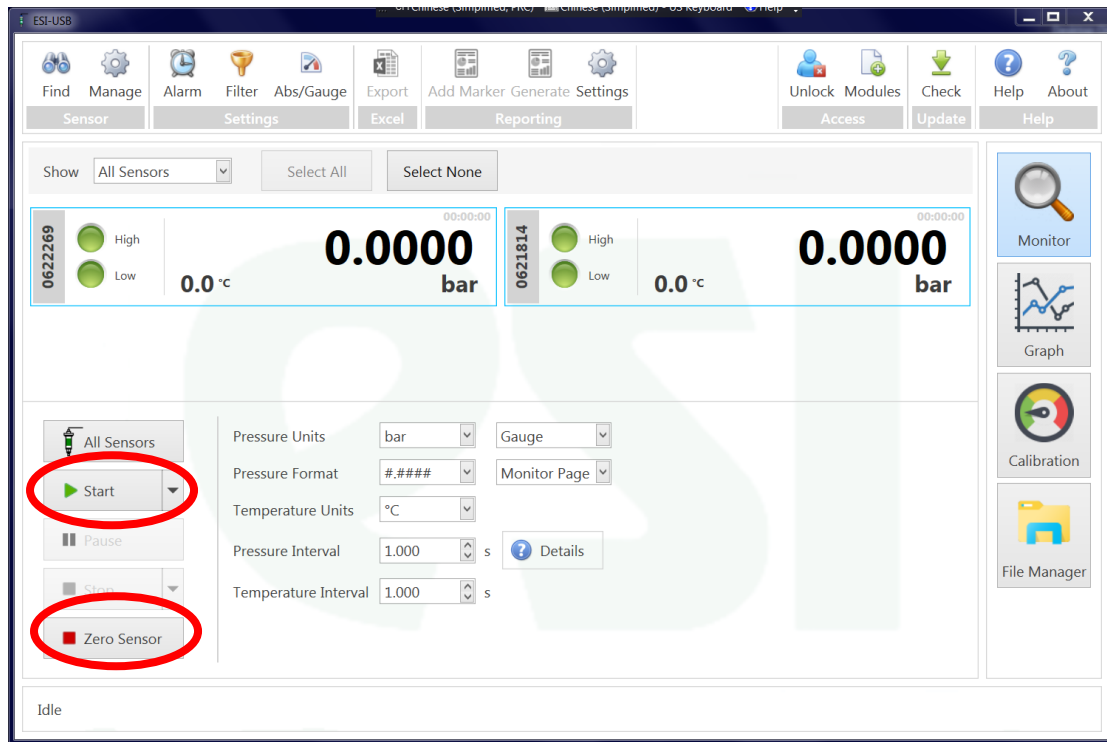
1. Open the Software 'ESI'.



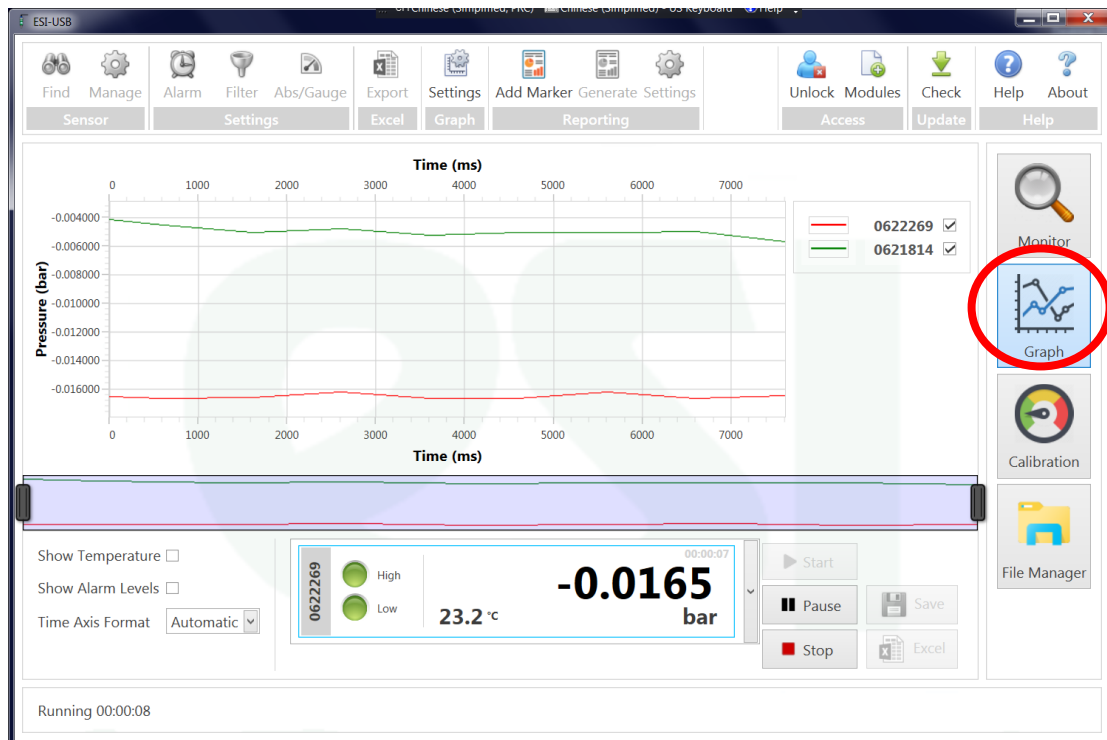
2. Press the 'Find' to search the connected two pressure meters.



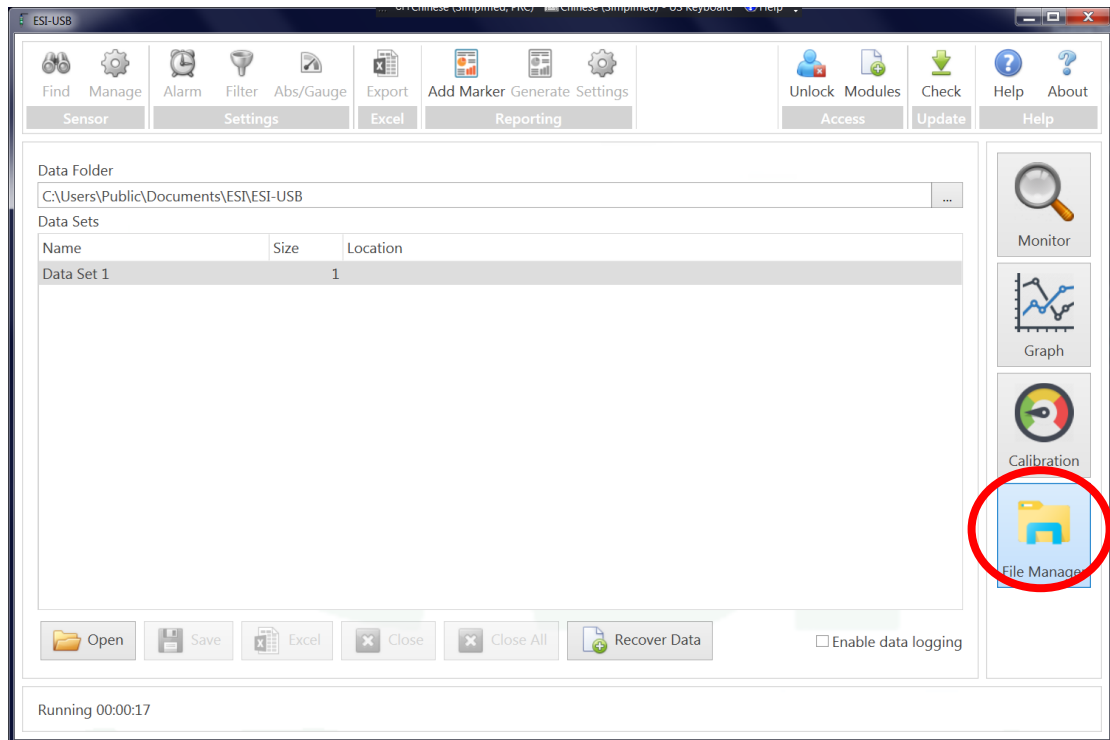
3. Before connecting the pressure meters to the setup, press 'Zero Sensor' to make it zero. Then connect the pressure meters to the setup. After that, press 'Start'.



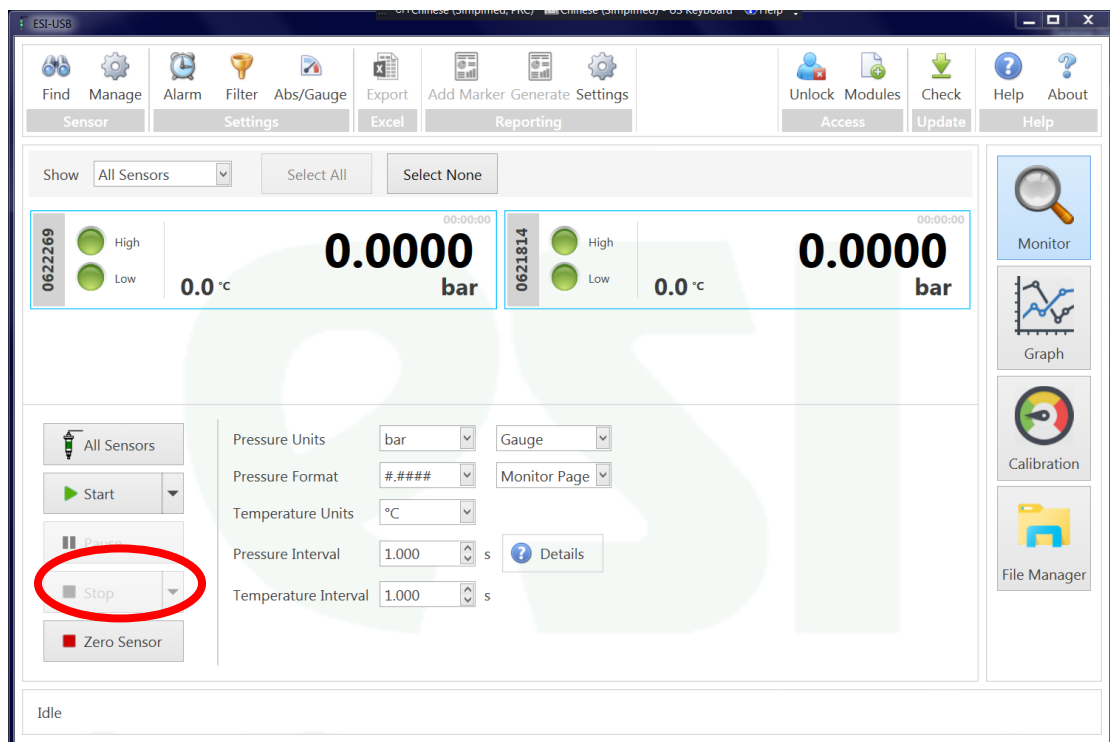
4. Press 'Graph'. The pressure data can be displayed in the figure.

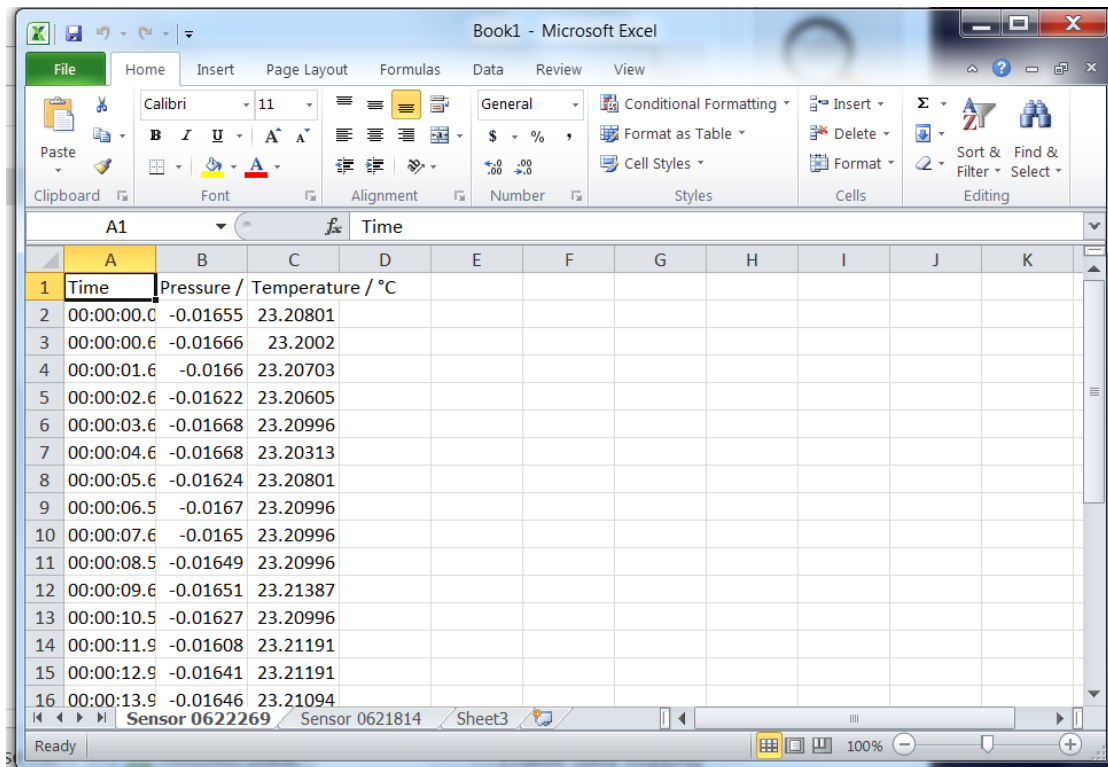
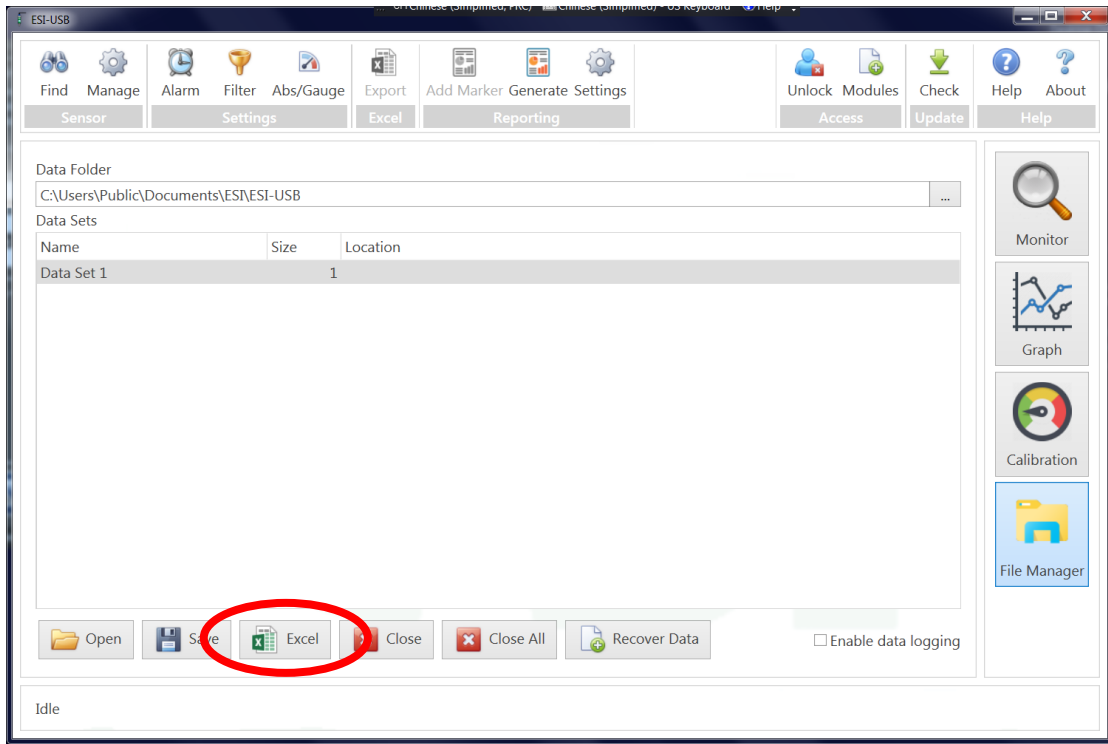


5. Press 'File Manager'. The page is shown in the figure below.

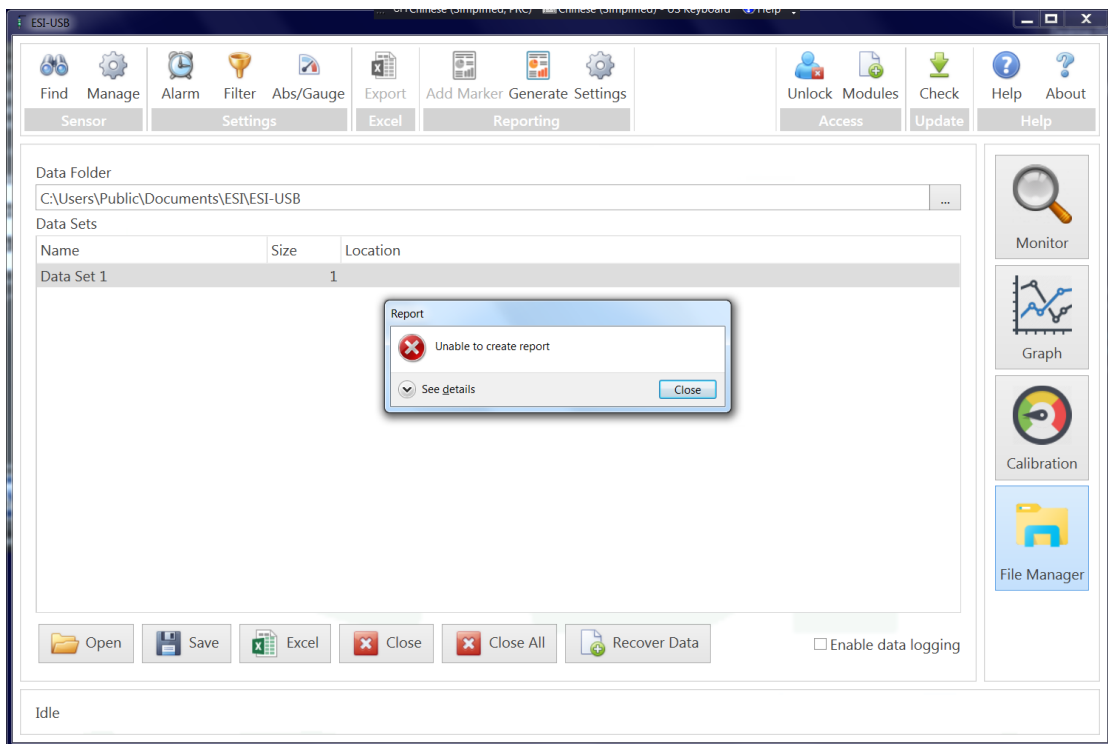
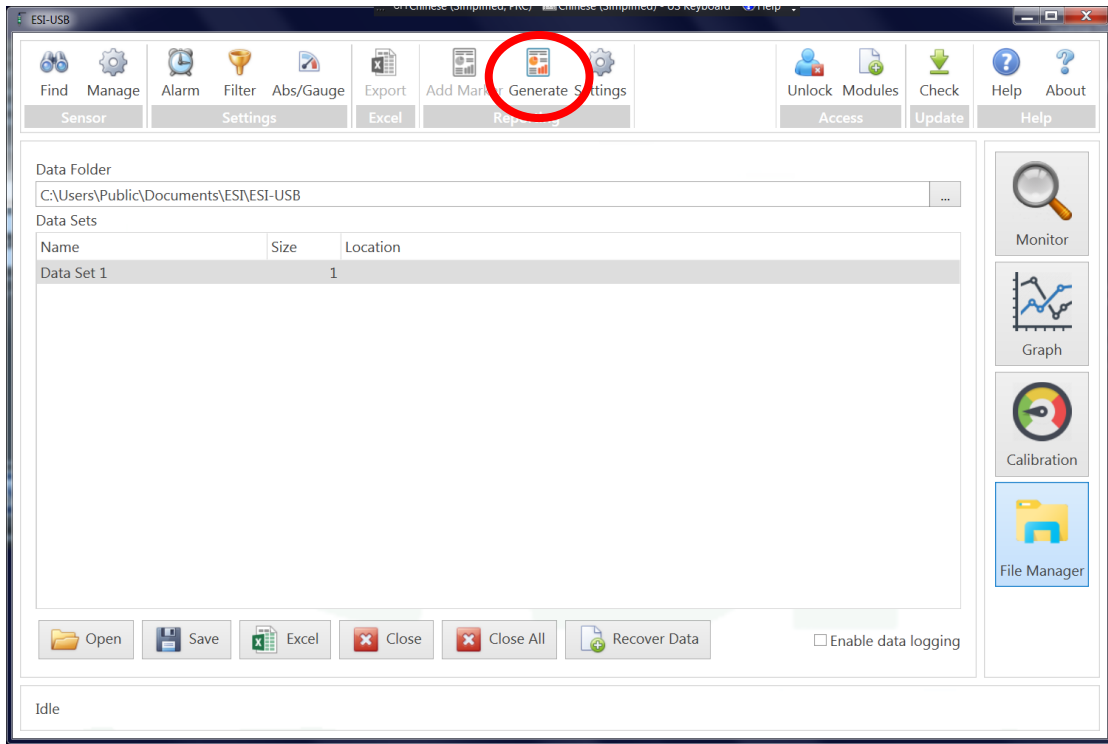


6. Press 'Stop' in the mainpage. Then press 'Excel' in the 'File Manager' page. Then the data is displayed in the Excel document. Save this document.

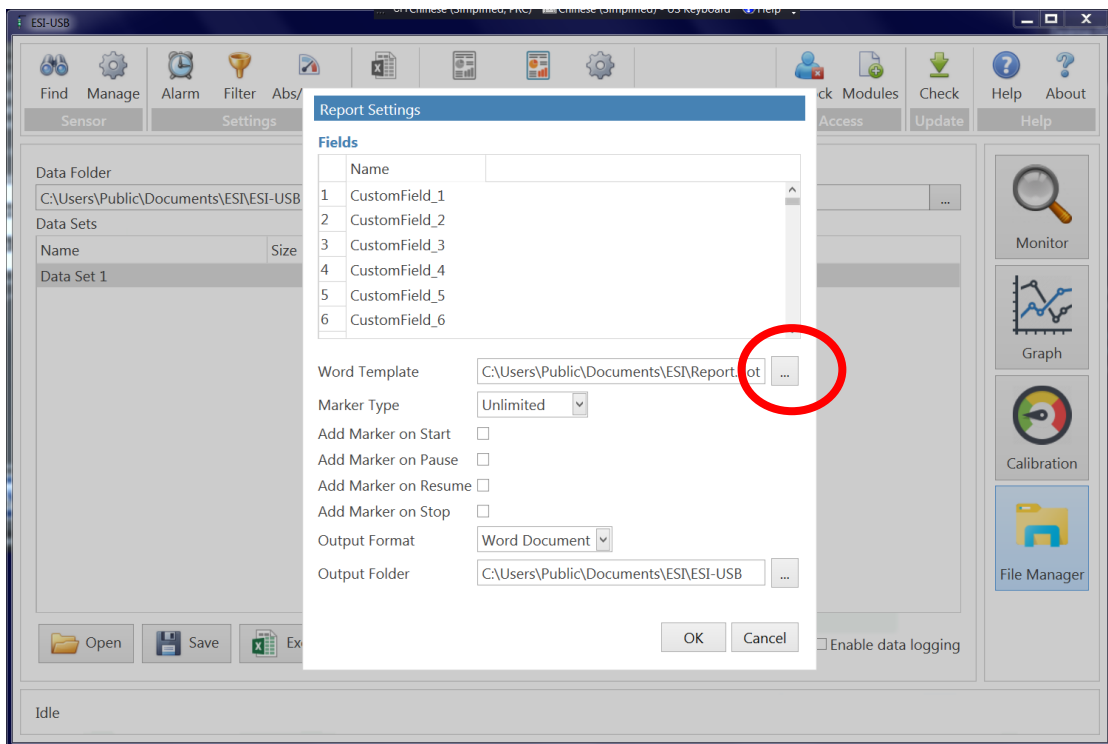
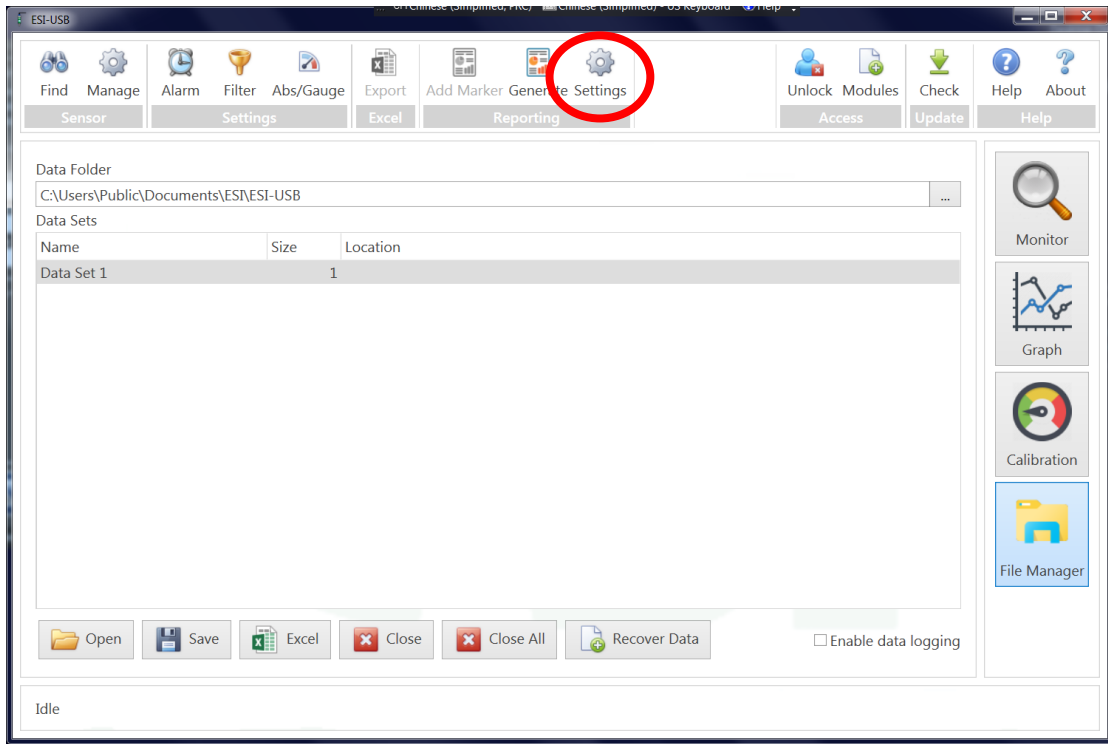


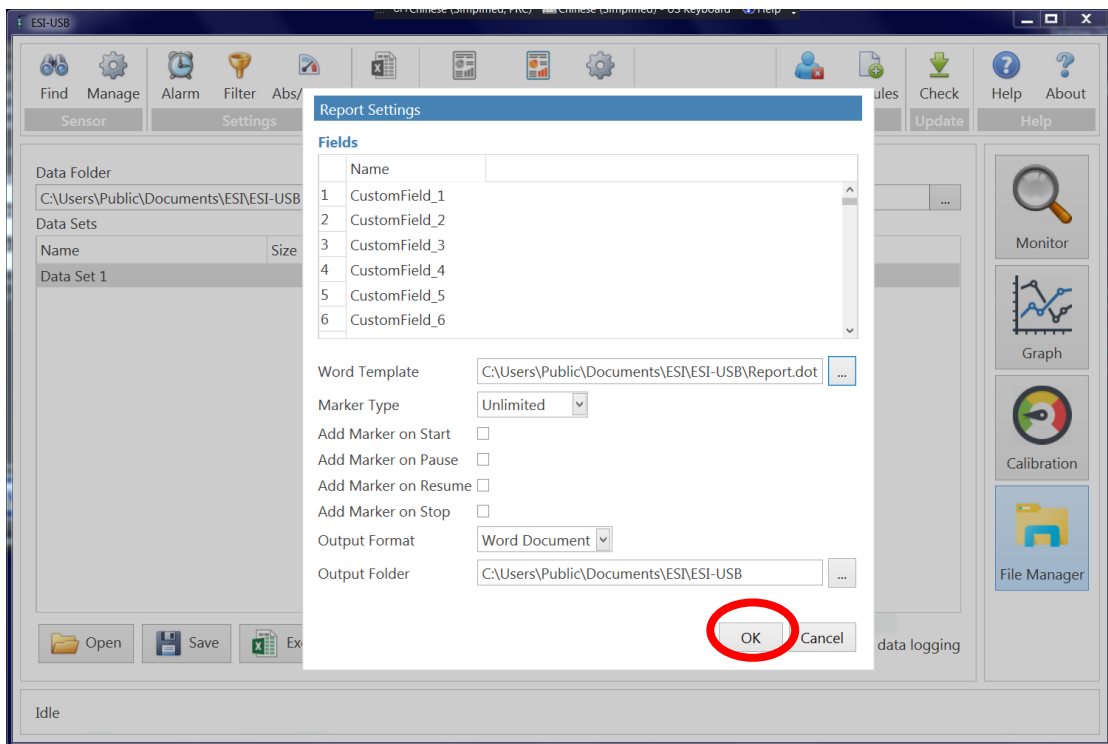
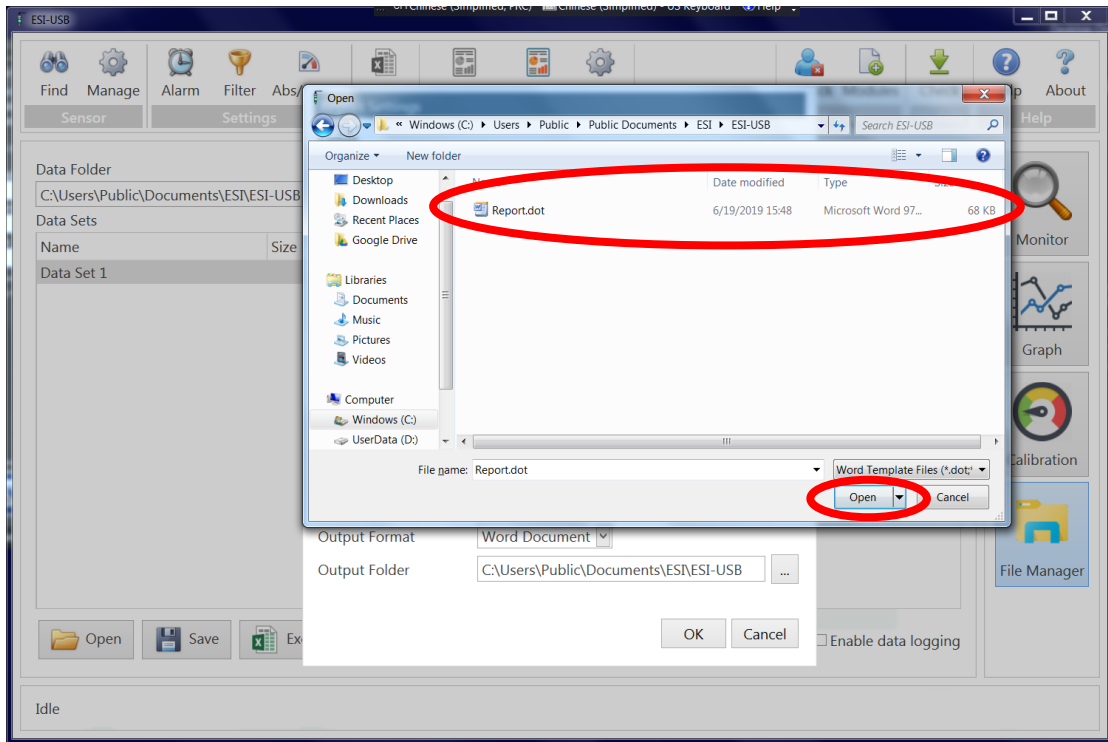


7. If you want to generate a report, press 'Generate'. There would be an error which is tackled in next step.

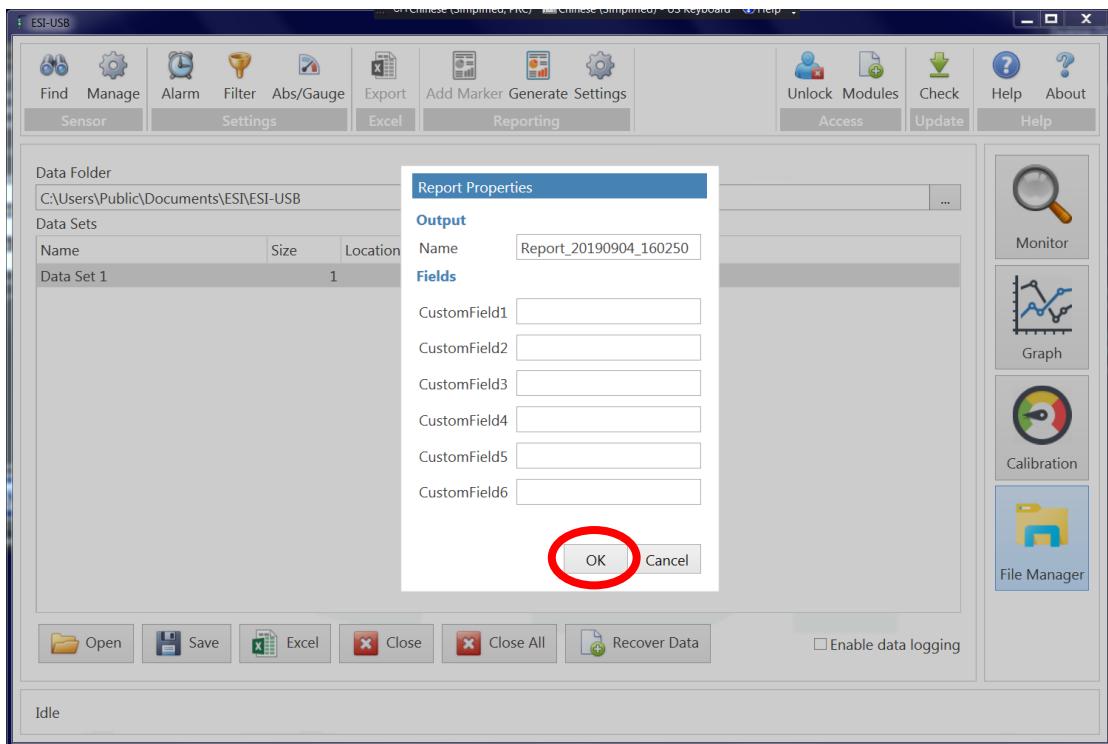
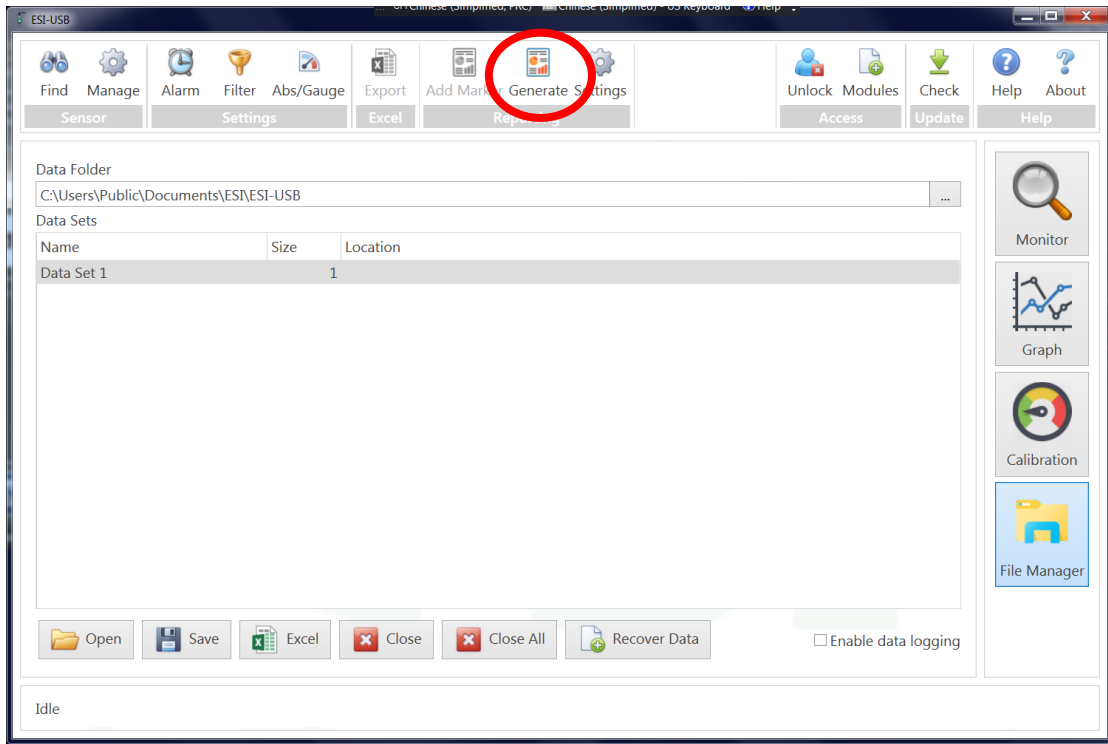


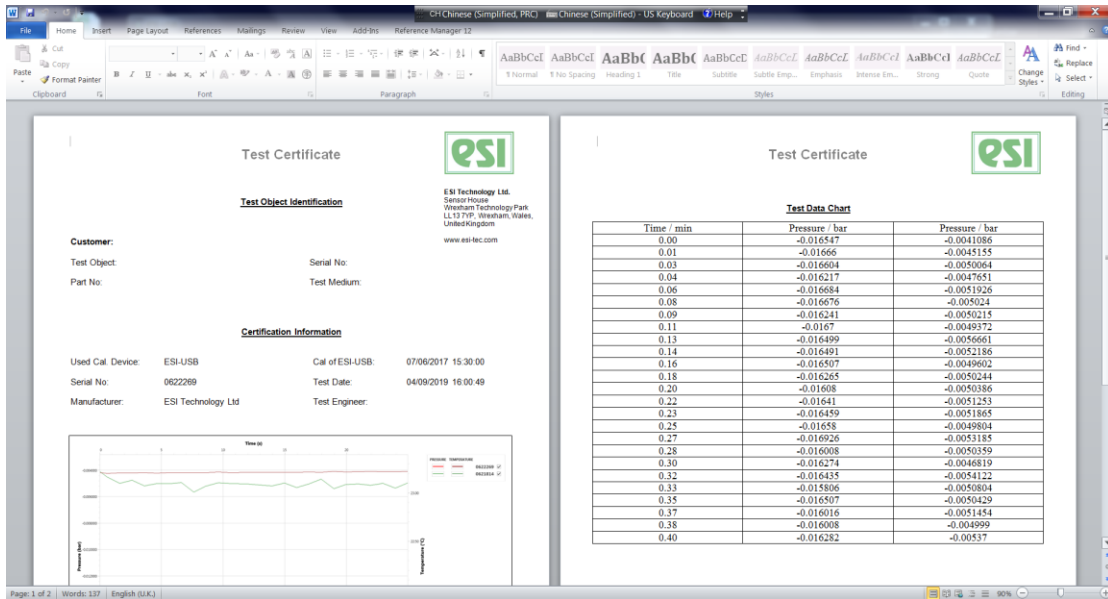
8. Press 'Setting' to find the document called 'Report.dot'.





9. Press 'Generate' again. The report could be shown successfully. Lastly, save this document.

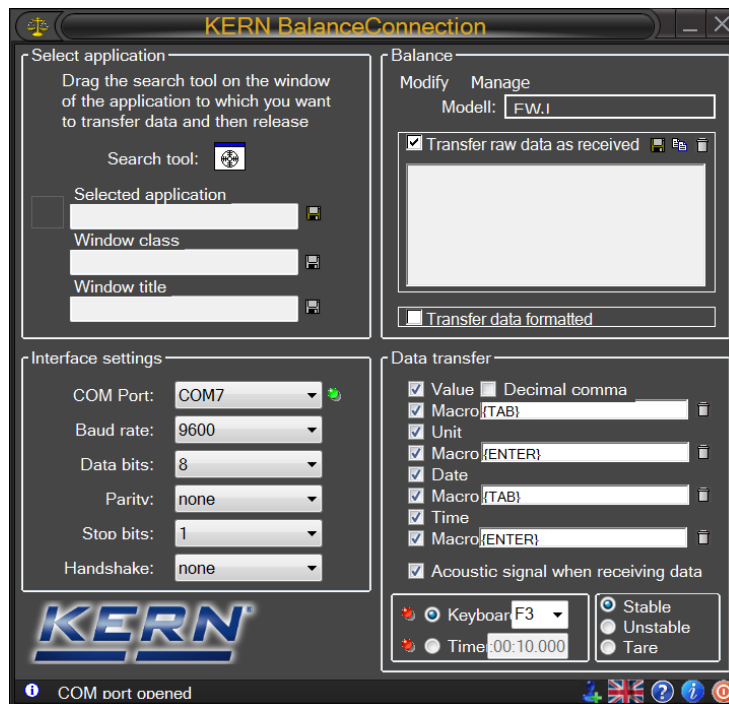




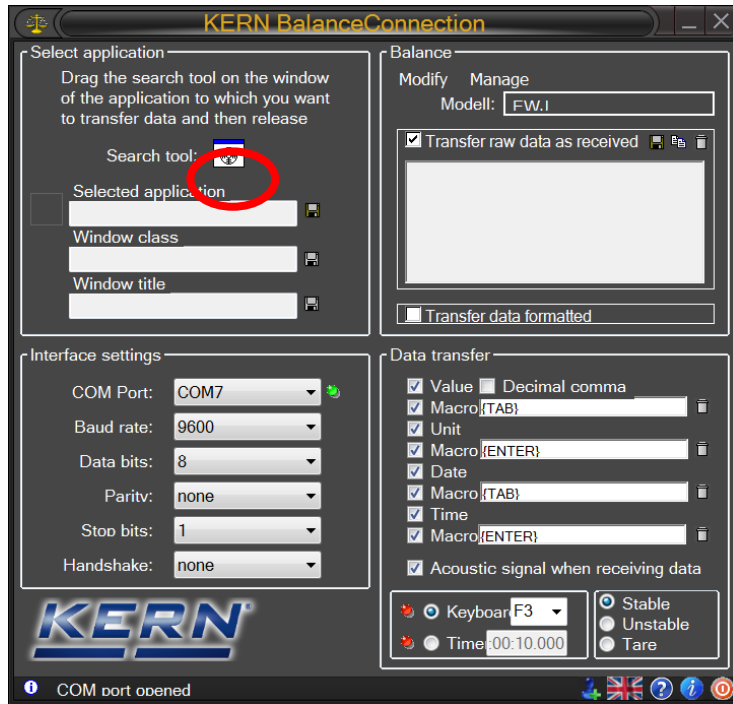
6.5 Manual for KREN

The manual of the balance Software 'KERN' is as follows.

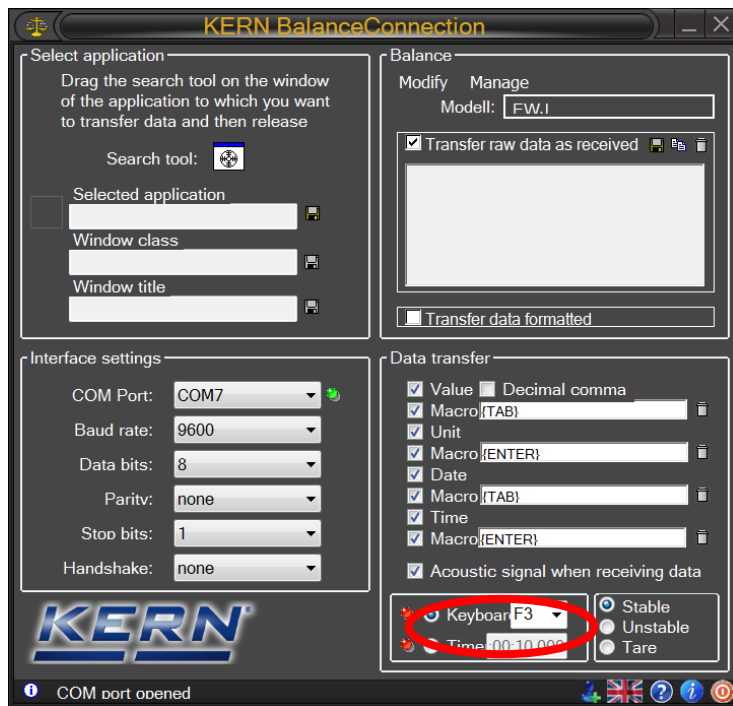
1. Open the Software 'KERN'.



2. Open an Excel document and then drag the 'Search tool' to this Excel page.



3. Press the 'F3' as shown in the Keyboard in order to record the weight data. The data will be recorded in the Excel sheet automatically.



6.6 Manual for large flow meter

For calibrating the large flow meter, set the K value:

1. Press the “menu” several times shortly until the interface of K value setting (see the figure below). It shows “_ _ _ _” at the top-right of the display. The current K value is shown in the center of the display.
2. Press and hold “set” button for 3 seconds. Then, we need to input the password “1234” in the “_ _ _ _” at the top-right corner of the display. Firstly, the left “_” among the “_ _ _ _” starts to blink. By pressing the + or -, we can set the number. By press the “set” the next “_” starts to blink and is ready to be set.
3. If the incorrect password is input, it beeps. If the password is correct, the K value is ready to be set. Follow the same steps using buttons +, -, and “set” to input the K value.

