

## Expansion Column Virtual Lab

### Laboratory manual for liquid-solid fluidisation experiments

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# Expansion Column Virtual Lab

Laboratory manual for liquid-solid fluidisation experiments



## Colophon

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### Virtual lab films

Onno Kramer

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1.25

## Summary

Dear students,

In the following weeks you will be introduced to the QMUL expansion column. This expansion column represents a liquid-solid fluidisation process. Fluidisation is a frequently applied unit-operation in industry. The knowledge you will gain will help you develop and improve your competence profile of a highly qualified chemical engineer.

The fluidisation experiment looks deceptively easy: you push a button, turn a valve and read the bed height. The fundamental principles behind fluidisation, however, are rather complex. This is why many research groups worldwide are concerned with unravelling the secrets behind this phenomenon.

If all goes well, you will discover that your measured data correspond with the estimated values based on hydraulics-based theory.

In industry, gas-solid fluidisation is commonly applied in processes, for example efficient mixing and heat transfer in combination with exothermic reactions. For safety reasons, we have chosen a water-particles system. Liquid-solid fluidisation is becoming increasingly popular in water treatment processes, particularly because it enables the development of more sustainable processes.

The expansion column was developed in the Netherlands at the public water cycle organisation Waternet in cooperation with dozens of chemical engineering students. More than fifty Dutch students took advantage of the expansion column to help them with their graduation activities. This is a wonderful opportunity for QMUL students, both for short Undergraduate Teaching lab experiments and for longer research projects.

If you are interested in a summer internship project in fluidised bed reactor research, please express your interest by e-mail to Onno Kramer and Edo Boek.

This simplified lab set-up is a basic version for you to gather experience in the field. So, your contribution is very valuable for QMUL. If you have any suggestions for improvement of this experiment, please inform Edo Boek, Onno Kramer and Benjamin Gridley.

Please note that this expansion column rig is unique in the UK - use your time wisely to maximise your learning opportunities.

It is crucial that you report a thorough log of your observations. For the final report, the measured data are required to do the calculations in order to answer the questions.

If you manage to solve bonus questions, extra credits can be earned.

This report can be used for both real experimental work as well as virtual lab work.

Good luck and have fun!





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# Fluidisation experiments

Chemical Engineering students increase their professional skills considerably in a short period of time, while discovering the fundamentals and applications of a fluidisation process.

Calcite ( $\text{CaCO}_3$ ) grains from a drinking water softening process are elevated in water flow to obtain a fluidised system.

They can see with their own eyes that the transition into a sustainable industrial process is possible.

## A brand new experimental set-up

In March 2019 a brand-new liquid-solid fluidisation experimental set-up has been assembled in the Chemical Engineering labs at Queen Mary University Of London (QMUL). Dutch colleagues from Waternet (Public Water Cycle Organisation of Amsterdam) in collaboration with Delft University of Technology made this possible.

This so-called expansion column was designed in the Netherlands to conduct research with the aim to produce sustainable drinking water. In addition, the apparatus is very suitable for educational purposes where students can learn to work with a frequently applied unit operation in chemical engineering: fluidisation.



Fig. 1: Installation of the fluidisation expansion column at the QMUL Engineering Building in London.

Within a day after the assembly, second year Chemical Engineering students could directly start with a Problem Based Learning (PBL) experiment using this equipment. Groups of 5 students were asked to work on an industrial full-scale challenge, which should be solved on the fly.



Fig. 2: See yourself how the pellets fluidise in the expansion column.



Fig. 3. Evaluation questionnaire to improve the expansion column.



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## Students at work!

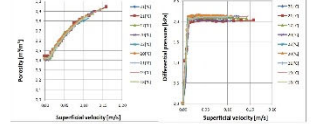


Fig. 4 and 5.: Motivated students determine the expansion characteristics of the calcite grains applied in drinking water.

Through a role-play-game, students had to supply the plant manager with an analysis of the problem. The technical issue was caused by air inclusion in a pressure sensor. The air trapped inside caused malfunctioning of the reactor and should be solved ASAP. One student had to block the water flow so that air trapping could be prevented in the highest-pressure tap. A second student had to control the water flow and a third student had to release air from the pressure sensor using a spanner. A fourth student had to monitor the pressure sensor display. A fifth student was responsible for effective communication, coordination and handling.

Students realised how hard it is to work together in a group with a healthy team spirit to obtain good results. The plant manager was watching the events closely!

After this challenge, students had to complete three assignments:

- 1) making expansion characteristics by increasing the water flow and measuring the process values;
- 2) linking fluidisation and terminal settling and
- 3) estimating an optimal process state using the specific crystallisation surface area of a full-scale drinking water softening reactor.

## Where research, education and practice come together

The expansion column is suitable for educational purposes but also very adequate for research purposes.

The module evaluation showed that students liked working with the expansion column and made it clear that it was one of the most useful PBLs this academic year.

As a result, about 10 keen students were inspired to do a voluntary summer research project, supervised by Edo Boek and Onno Kramer.



Fig. 6-10. Weighing and dosing pellets. Differential pressure. Releasing trapped air from the sensor. PBL class instructions at QMUL.

Many students in the Netherlands and now also in England have worked with expansion columns through multi-disciplinary research projects, both at UG level and in PhD projects.

Are you interested? Please do not hesitate to ask for more information. And... drinking (sustainable and reliable) water from the tap is our mission! Do you want to participate?



Fig. 11. Queen Mary University of London. [www.qmul.ac.uk](http://www.qmul.ac.uk)

### References

The expansion column was designed especially for the PhD project of Onno Kramer "Hydraulic modelling of liquid-solid fluidisation in drinking water treatment processes" carried out by Waternet, Delft University of Technology and HU University of Applied Sciences Utrecht in collaboration with Queen Mary University of London. The project was financially supported by Waternet's Drinking Water Production Department.

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# 1 Design goals and approach

## 1.1 Goals

The aim of this experiment is to determine the fluidisation characteristics of  $\text{CaCO}_3$  particles in water.

In this experiment, you determine the hydraulic fluidisation behaviour of  $\text{CaCO}_3$  lime pellets in the expansion column. By means of a stepwise increase of the water flow through the pellet bed, the pressure difference will increase to the maximum differential pressure. When the flow increases further, the differential pressure remains constant, but the bed height will increase further. At a certain point, particles will flush out of the reactor: **this should be avoided at all time.**

From this experiment, your task is to find a suitable superficial velocity of water to enable operations for drinking water softening. To this end, a large surface area of the granules must be available. However, particles should not be flushed out of the reactor (terminal settling velocity). In addition, you have to avoid a fixed bed state of particles (incipient fluidisation).

**Goal 0: plot expansion curves for the data taken from video recordings (§5.2) - 10%**

**Goal 1: plot expansion curves for the individual data set you received by email (§5.2) - 20%**

Note: you have to achieve all following goals using this data set

**Goal 2: estimate the terminal settling velocity of individual grains (§5.3) - 20%**

**Goal 3: discover the relationship between fluidisation and terminal settling (§5.4) - 20%**

**Goal 4: determine the optimal specific surface area in a full-scale reactor (§5.5) - 20%**

**Goal 5: complete the QMUL questionnaire (§5.6) - 10%**

## 1.2 Approach

You will perform a fluidisation experiment and determine a characteristic graph for this.

Preparation of part 1:

- Read the theoretical part in Chapter 2 of this manual describing the background information. This will provide you with a good picture of what, how and why.
- Perform the fluidisation experiment according to the instructions (Chapter 3).
- Complete the data in the chart tables (Chapter 7) during the experiment.
- Prepare a presentation and measurement report according to the guidelines / rubrics.
- Submit the data and report on QM+.

## 2 Theory

The fluidisation experiment with the expansion column provides insight into the operation of a liquid-solid fluidisation process. The liquid used in the set-up is water and the particles consist mostly of granules that occur in water treatment processes. Examples are  $\text{CaCO}_3$  pellets, carbon granules and sand grains. By performing hydraulic experiments, the principle of filtration, fluidisation and sedimentation is introduced.

### 2.1 Background information

#### Fluidisation

Fluidisation is a process similar to liquefaction whereby a granular material is converted from a static solid-like state to a dynamic fluid-like state. This process occurs when a fluid (liquid or gas) is passed up through the granular material. The grains will then float and move freely. Fluidisation is used extensively in the industry for the production of fertilizer granules, plastic granulate, in the petroleum industry, for transport purposes and, of course, in water purification. The advantage of fluidisation is that a large specific area per volume is created.

#### Fluidised bed reactor

In a fluidised bed process, particles are brought into a floating state in a cylindrical column. In a liquid-solid fluid bed reactor, water flows from below through the particles in an upward direction. Large and heavier grains classify more to the bottom zone of the reactor, and small and lighter grains migrate to the upper zone. Important physical quantities are measured online for controlling the reactor. In addition to the water flow and temperature, the fluidised bed height is also measured, as is the pressure difference across the reactor bed.

#### Pellet softening reactor

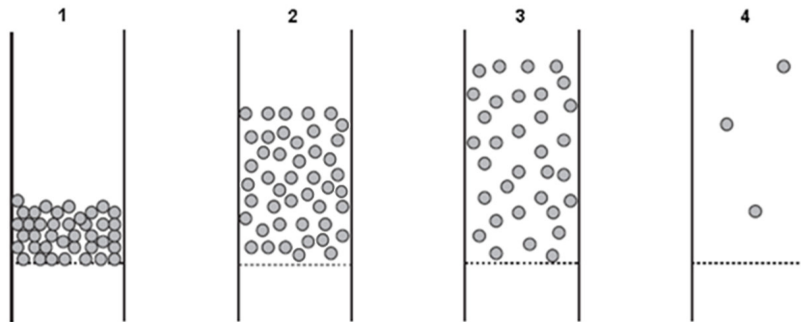
In drinking water softening, a strong base (e.g. caustic soda =  $\text{NaOH}$ ) is dosed into the reactor and mixed with water. In this reactor, grains of seeding material are present to create a large specific crystallisation surface. Due to this large specific surface area, calcium carbonate ( $\text{CaCO}_3$ ) can crystallise on the existing surface of the particles. This specific surface area is crucial and ensures that lime on the granules crystallises and does not precipitate in the water phase. The grown pellets increase in size and migrate to the lower zone of the reactor, where they are periodically drained and replaced with new graft material that is periodically dosed.

### 2.2 Hydraulics and fluidisation

To understand the experiment, the basics of hydraulics and fluidisation will be explained below. The theory is rather complex with a lot of knowledge from other disciplines such as physics, mathematics, fluid mechanics, process engineering and of course hydraulics. You need to grasp certain concepts to understand these principles. Often, the mathematics involved are complicated, but since this only distracts from the essence, simplified formulas are given which are also perfectly adequate. The following states are given when a fluid, in our case water, flows through a particle bed (see Figure 1):

1. Filtration and flow through a packed bed
2. Minimum fluidisation at maximum pressure drop
3. Fluidisation and flow through a bed of floating grains
4. Flushing and terminal settling





**Figure 1** Different states in a particle bed

The first representation in Figure 1 describes when the particles in a bed are in a fixed state. The upward water flow has insufficient force to support the particle bed. The granules are stacked at the bottom of the column. In the second picture there is homogeneous minimal fluidisation. The upward water flow has sufficient force to support the grains. The distance between the grains however is minimal. The transition point from fixed to fluidisation is called the minimal fluidisation. In case the water flow is increased, represented in third and fourth figures, the particles are moved more apart and away from each other. This phase is called expansion; the particles are fully supported by the water flow. If the water flow then increases even further, the phase of flushing occurs. Here the granules are entrained in the column by the water flow. By varying the superficial velocity, it is possible in practice to control the process of fluidisation. Because the fluidisation process is dependent on variables such as pressure difference, grain size and superficial speed, it is possible to convert the fluidisation process into mathematical equations. The superficial velocity stems from the fact that in practice the entire surface area is not available for the liquid flow due to the granules. The bed through which the water flow moves only contains a fraction of open space through which water can flow through. The superficial velocity as a function of the volume flow now becomes:

$$v_s = \frac{Q_w}{\frac{\pi D^2}{4}} \quad (1)$$

In which:

$v_s$	Superficial velocity of water	[m/s]
$Q_w$	Water flow	[m <sup>3</sup> /s]
$D$	Inner diameter of cylindrical column ( $D=5.7$ cm)	[m <sup>2</sup> ]

This open void fraction is equal to the voidage (sometimes also called porosity)  $\epsilon$  of the bed. Voidage is a very important variable in fluidisation. If you know the preliminary mass of the examined particle, you can estimate the voidage when measuring the bed height of the granules in the expansion column.

$$\epsilon = 1 - \frac{m}{\frac{\pi D^2 L \rho_p}{4}} \quad (2)$$

In which:

$\epsilon$	Bed voidage or fluid void fraction	[m <sup>3</sup> /m <sup>3</sup> ]
$m$	Particle mass	[kg]
$L$	Bed height	[m]
$\rho_p$	Specific particle density	[kg/m <sup>3</sup> ]

You can imagine that the water between the particles flows faster. This speed is called the interstitial speed  $v_i$ . You can calculate this using equation 3. In an infinitely fluidised bed or an empty column, the voidage is  $\epsilon = 1$  and the interstitial speed is again equal to the superficial velocity.



$\Delta P_{max}$	Maximum differential pressure over the particle bed	[Pa]
$L_{mf}$	Bed height at minimum fluidisation	[m]
$\epsilon_{mf}$	Bed voidage at minimum fluidisation	[m <sup>3</sup> /m <sup>3</sup> ]

Based on the particle mass, you can also calculate the maximum pressure difference:

$$\Delta P_{max} = \frac{mg(\rho_p - \rho_f)}{\frac{\pi D^2}{4} \rho_p} \quad (5)$$

When particles are monodispersed (all the same), are perfectly round and have the same specific density, the transition is clearly distinguishable. This is less the case with beds containing particles with a large particle size distribution, have an irregular shape and have different densities.

The minimum fluidisation velocity can be estimated as follows:

$$v_{mf} = \frac{(\rho_p - \rho_f)gd_p^2\epsilon_{min}^3}{180\eta(1 - \epsilon_{min})} \quad (Re_\epsilon < 2) \quad (6)$$

In which:

$d_p$	Particle diameter	[m]
$v_{mf}$	Minimum fluidisation velocity	[m/s]

After increasing the linear fluidisation velocity, the pressure drop shows a small increase before it becomes constant. This increase is due to the frictional forces between the particles which must first be overcome before the particles can fluidise separately. This pressure increase does not occur when the fluidisation speed is decreased.

In order to calculate the pressure difference, numerous formulas are given in the literature. The Kozeny equation 7 applies to laminar flow:

$$\frac{\Delta P}{L} = 180 \frac{v_s \eta (1 - \epsilon)^2}{d_p^2 \epsilon^3} \quad (Re_\epsilon < 2) \quad (7)$$

For non-laminar flow regimes, the Carman-Kozeny equation 8 can be used:

$$\frac{\Delta P}{L} = 180 \frac{v_s \eta (1 - \epsilon)^2}{d_p^2 \epsilon^3} + 2,87 \frac{v_s^{1,9} \eta^{0,1} \rho_f^{0,9} (1 - \epsilon)^{1,1}}{d_p^{1,1} \epsilon^3} \quad (Re_\epsilon < 600) \quad (8)$$

Be aware that here another Reynolds number  $Re_\epsilon$  is used: Reynolds particle number according equation 9.

$$Re_\epsilon = \frac{\rho_f d_p v_s}{\eta} \frac{1}{1 - \epsilon} \quad (9)$$

### 3/4 Fluidisation state

As the fluidisation rate continues to increase, the pressure drop across the bed will remain constant as the bed further expands upwards (region 3 in Figure 2). The height  $L$  of the fluidised bed can be many times larger than the height  $L_0$  of the fixed bed. If the particles are equally distributed over the liquid phase, we are dealing with homogeneous fluidisation. In solid-liquid fluidisation almost always homogeneous fluidisation occurs.

The degree of expansion and voidage of the fluidised bed with respect to the fixed bed can now be calculated using equation 10 and equation 11, respectively.

$$E = \frac{L}{L_0} - 1 = \frac{\epsilon - \epsilon_0}{1 - \epsilon_0} \quad (10)$$

$$\epsilon = 1 - \frac{L_0}{L}(1 - \epsilon_0) \quad (11)$$

In which:

E	Bed expansion	[%]
L <sub>0</sub>	Bed height for fixed state	[m]
ε <sub>0</sub>	Bed voidage for fixed state	[m <sup>3</sup> /m <sup>3</sup> ]

In crystallisation processes, the specific surface area is very important. Crystallisation in chemical processes is more optimal for larger surface areas. You can see in equation 12 that in case the particles are smaller, the specific surface is larger. But if the particles are too small, the risk of flushing them out of the crystallisation of chemical processes is feasible, but undesirable. This is why it is crucial to find the optimal balance. This is easier said than done. The chemical engineer is facing a great challenge to find an optimal operation point.

$$A_s = 6 \frac{(1 - \epsilon)}{d_p} \quad (12)$$

In which:

A <sub>s</sub>	Specific surface area	[m <sup>2</sup> /m <sup>3</sup> ]
----------------	-----------------------	-----------------------------------

#### 4/4 Flushing and terminal settling

As the liquid velocity is increased further, the point is approached where the particles are about to be entrained with the liquid flow (region 4 in Figure 2).

The most famous equation to estimate the terminal settling velocity is given by Stokes.

$$v_t = \frac{1}{18} \frac{gd_p^2(\rho_p - \rho_f)}{\eta} \quad (\text{Re}_t < 0.2) \quad (13)$$

In which:

g	Gravitational acceleration (g=9,81 m/s <sup>2</sup> )	[m/s <sup>2</sup> ]
v <sub>t</sub>	Terminal settling velocity of one individual grain	[m/s]
η	Dynamic viscosity of the fluid	[kg/m/s]
ρ <sub>f</sub>	Specific density of the fluid	[kg/m <sup>3</sup> ]

This equation 13, however, is only valid in case the Reynolds number under terminal settling conditions is very low: Re<sub>t</sub> < 0.1.

$$\text{Re}_t = \frac{\rho_f d_p v_t}{\eta} \quad (14)$$

In case of glycerol, this will certainly be the case since the flow is considered laminar. However, this is certainly not the case for a billiard ball falling from an airplane. This involves turbulence, and Newton's Law applies:

$$v_t = 1,74 \sqrt{\frac{gd_p(\rho_p - \rho_f)}{\rho_f}} \quad (\text{Re}_t > 500) \quad (15)$$

In most cases, such as grains applied in drinking water treatment processes, both equations by Stokes and Newton are not valid, and we are dealing with a transitional area. Numerous equations have been given in the literature. The empirical equation by Lewis is rather useful in our case to estimate the terminal settling velocity of grains.

$$v_t = \left( \frac{\left( \frac{2}{15} g(\rho_p - \rho_f) \right)^2}{\eta \rho_f} \right)^{\frac{1}{3}} d_p \quad (0.4 < \text{Re}_t < 500) \quad (16)$$

A last remark:

- in laminar flow conditions  $v_t \sim d_p^2$ ,
- in turbulent flow conditions  $v_t \sim d_p^{1/2}$ ,
- and in the transitional region  $v_t \sim d_p^1$ , so in water, individual particles settle almost directly proportional to the particle size, as you can see in equation 16.



### 3 Practical work using the expansion column

In this Chapter, you will get instruction about the experimental work with the expansion column. In addition, you will be informed how to acquire data from the expansion column by watching videos.

#### 3.1 Fluidisation characteristics

In this experiment, the hydraulic fluidisation behaviour of  $\text{CaCO}_3$  pellets is determined in the expansion column. By means of a stepwise increase of the water flow through the pellet bed, the pressure difference will increase to the maximum differential pressure. When the flow increases even further, the differential pressure remains constant, but the bed height will increase further.

##### Start-up

Most probably the lab manager already has filled the reactor with grain materials. If not, do the following. Start by weighing about 1.5-2.0 kg particles. Make sure you determine the average particle diameter in case it is not given. If the lower and upper sieve mesh is given then you can calculate the 'hydraulic equivalent' or average sieve diameter with formula 17.

$$d_p = \sqrt{d_{s,1}d_{s,2}} \quad (17)$$

##### 1/4 Filtration en fluid flow through a fixed bed

Increase the water flow in small steps (e.g. 10-20 L/hr) and write down the water flow, water temperature, bed height and pressure difference in the data chart. You can convert from mbar to Pascal by dividing by 100. When measuring the pressure difference, you will notice that the pressure as shown in Figure 2 does not remain constant, but slightly increases with increasing the water flow. This is because the lower measuring point is assembled approximately 2 cm from the bottom. You must therefore use a correction factor.

$$\Delta P_{corr} = \Delta P \frac{L}{L - 0,02} \quad (18)$$

Note: the 2 cm depends on the specifications of the used experimental setup. You can find that in the provided dataset.

An old user manual has been made by Dutch students<sup>[19]</sup>.

##### 2/4 Minimum fluidisation at maximum pressure difference

Increase the water flow until you reach incipient fluidisation where the particles have just started to float. This corresponds with the minimum fluidisation velocity. Probably the smallest grains at the top of the bed fluidise just a little sooner than the slightly larger grains at the bottom. Write down both values of  $v_{mf}$ . If you observe the pressure difference, you will find that the value is constant and in principle does not rise any further. Now write down the water flow  $Q_{mf}$ , the bed height  $L_{mf}$ , the water temperature and the maximum pressure difference  $\Delta P_{max}$ .

The dynamic viscosity depends on the water temperature and can be calculated using the approximation equation 19 given by Vogel-Fulcher-Tammann<sup>[5]</sup>. We assume the specific fluid density  $\rho_f$  to be constant = 1,000 kg/m<sup>3</sup>.

$$\eta = \frac{1}{1,000}(0.83 + 0.0085 T)^{-3} \quad (19)$$

In which:

$T$  Water temperature [°C]

### 3/4 Fluidisation state

Now increase the water flow further in steps of 20-50 L/hr. Meanwhile, wait a few minutes until the bed reaches a steady state condition and ceases to expand. Write down  $Q_w$ ,  $T$ ,  $L$  and  $\Delta P$  again, and so on. You can assume  $\rho_p = 2,625 \text{ kg/m}^3$  for specific density of calcite pellets. You can also derive this specific density regarding the particles (formula 20) based on the measured pressure difference during fluidisation.

$$\rho_p = \frac{\rho_f}{1 - \frac{\pi D^2 \Delta P}{4mg}} \quad (20)$$

At high water flow where the bed  $L > 4\text{m}$ , you need to make sure that you do not flush anything out. After all, you do not want to get particles in the pump and the measurement devices. So please pay attention. If this goes well, try to increase the flow so that the granules just do not flush (flushing). You thus approach the terminal settling state.

### Device calibration

It is important that you check whether all measurement data is correct. Make sure that the pressure difference sensor contains no air and so shows a value of 0 if no water flows through the column. Create a flow versus flow chart. Does the intercept go through 0? And is the slope 1? Do this at least 3 times. The calibrated flow rate:  $Q_{\text{cal}} = (L_2 - L_1) / \Delta t \cdot 2.37$ . The relative error  $(Q_{\text{sensor}} - Q_{\text{cal}}) / Q_{\text{cal}}$  should be between  $\pm 2\%$ .

Make sure that at least one expansion (duplo is better) experiment has at least 25 measurements (bed height, pressure and temperature) at different superficial velocities (flow). Make sure you have at least 5 measuring points between 150 and 230 L/hr. After the expansion experiments have been carried out, the water flow should be shut down through switching off the pump.

Create a graph with the voidage on the y-axis and the superficial velocity on the x-axis. Also plot the pressure difference against the superficial velocity on the x-axis on the second secondary y-axis as shown in x-axis. What do you notice?

Make a graph where you plot the specific surface area (equation 12) against the superficial velocity on the y-axis. What do you notice? What do you think is a good superficial velocity to operate optimal softening?

Tip: try to make the surface as large as possible but make sure there is no risk of leaching and certainly no fixed bed condition. A lower limit of the specific surface is approximately  $2,500 \text{ m}^2/\text{m}^3$ .

### 4/4 Flushing and terminal settling

At a later stage, terminal settling experiments will also be a part of this experiment. For now, you should only make a theoretical estimation of the terminal settling velocity of particles.

Optional: If you succeed, you can also include the theoretical equation 8 in the graph.

Calculate the terminal settling velocity using the given equation by Stokes, Newton and Lewis (equation 16). Indicate which calculated value is valid according to the Reynolds terminal for the given conditions.

### 3.2 Relationship between terminal settling and fluidisation

Create a log-log diagram of the superficial velocity against the voidage. Determine the slope  $n$  and the intercept  $v_E$  of this curve. The principle is based on the most popular Richardson-Zaki model<sup>[25]</sup>.

$$\ln(v_s) = \ln(v_E) + n \cdot \ln(\epsilon) \quad (21)$$

In which:

$v_E$	Extrapolated superficial velocity	[m/s]
$n$	Fluidisation index, also called the hindering index	[-]

## 4 Video demonstration of virtual lab work using the expansion column

Here you will find a demonstration how to operate the expansion column in a series of short video recordings. You will carry out several measurements following instructions in the recordings. When you have provided correct measurements, showing your proficiency in working with the virtual expansion column, you will each get an individual data set, measured in the lab by previous generations of students. Using this data set, you must complete the design goals set in Chapter 1.1.

Before watching the videos, you are expected to have read Chapter 2: Theory. You have to complete a couple of multiple-choice quiz questions before you can access the videos.

Source<sup>[16]</sup>: Virtual Lab: Fluidisation Experiments, MOOC for students chemical, civil or mechanical engineering“, 4TU.ResearchData, The Netherlands, 2020. <https://doi.org/10.4121/12881009>

### 4.1 Your hosts Onno and Cas demonstrating the expansion column at Waternet

The virtual lab starts with an introductory talk by your hosts Cas van Schaik and Onno Kramer from Waternet, Amsterdam. Waternet is a public drinking water company in the Netherlands, ensuring that all customers have access to safe and tasty drinking water from the tap. One of the steps in drinking water treatment is removing  $\text{Ca}^{2+}$  and  $\text{CO}_3^{2-}$  ions from the water supply, a process known as water softening. This is achieved in a fluidised bed reactor, where the  $\text{Ca}^{2+}$  and  $\text{CO}_3^{2-}$  ions precipitate / crystallise on the calcite pellets in the reactor. We built the QMUL expansion column, as a lab scale fluidised bed reactor together with Dr. Edo Boek, just over two years ago, from scratch. Two generations of students have used the expansion column so far to learn about handling this pilot-plant scale lab experiment. Working with the live expansion column requires teamwork - you have to carry out several measurements at the same time, log your data and make sure that the equipment is working properly. Unfortunately, you will not be able to work in the lab this year due to COVID.

Therefore, we will demonstrate how this experiment works using video-recorded virtual lab experiments. Please pay close attention - you will be asked to carry out several measurements during the demonstration.

The video contains useful information to understand the principles of a fluidised bed reactor at a water treatment plant.



Topic: Hosts talk

Time: 5:54

File: Virtual lab - fluidisation experiments - Hosts talk.wmv

URL: <https://youtu.be/78Do7rouXQI>

### 4.2 Acquiring expansion data from video recordings of lab experiments

To be able to acquire expansion data, 7 videos were recorded at the pilot plant facility of Waternet in Amsterdam. In the videos you will see the different states of a liquid-solid fluidised bed. Please watch these videos carefully and write down the sensor values of the differential pressure in [mbar], the flow rate in [L/h], the temperature in [°C] and the visually observed bed height of the grains [m].

You will see that the pressure sensor values on the displays is not constant and shows fluctuations. Try to estimate the best average value and the data spread. Use error bars to visualise the degree of fluctuations. Regarding the bed height, with increasing water flow rate, it will become more difficult to measure the transition from particle bed to the open fluid phase. Figure out how to determine the best representation of the fluidised bed height, including error bars due to bed height fluctuations. Explain which strategy you have considered.

Finally, plot your data for the 7 different flow rates in Excel, showing both bed height and differential pressure as a function of flow rate in a single graph. Include your error bars. What do you observe?





Point 0: Starting point

Time: 0:32

File: Virtual lab - fluidisation experiments - Point 0.wmv

URL: <https://youtu.be/QhJfoj9AGDY>



Point 1: Fixed bed

Time: 0:41

File: Virtual lab - fluidisation experiments - Point 1.wmv

URL: <https://youtu.be/3Xfgkdlg1EE>



Point 2: Minimum fluidisation

Time: 1:04

File: Virtual lab - fluidisation experiments - Point 2.wmv

URL: [https://youtu.be/e\\_H74k60j\\_E](https://youtu.be/e_H74k60j_E)



Point 3: Fluidised state

Time: 0:45

File: Virtual lab - fluidisation experiments - Point 3.wmv

URL: <https://youtu.be/tFXCYlieM4Y>



Point 4: Fluidised state

Time: 1:06

File: Virtual lab - fluidisation experiments - Point 4.wmv

URL: [https://youtu.be/Jff\\_3ujBEYE](https://youtu.be/Jff_3ujBEYE)



Point 5: Intermediate fluidisation

Time: 0:56

File: Virtual lab - fluidisation experiments - Point 5.wmv

URL: [https://youtu.be/7m\\_Qzn25Dto](https://youtu.be/7m_Qzn25Dto)



Point 6: Maximum fluidisation

Time: 1:10

File: Virtual lab - fluidisation experiments - Point 6.wmv

URL: <https://youtu.be/y8alks2jo5U>

## 5 Final design report

### 5.1 Knowledge gained and lessons learned

In your report, describe what you have learned from this experiment. In your opinion, which competencies are needed to be able to successfully work with the expansion column?

Compose a comprehensive dataset in Excel including SI units and error bars. Distinguish between experimental data and calculations, e.g. by including data in yellow boxed cells and calculations by default in black.

In your final report, describe your results regarding the design goals defined in Section 1:

**Goal 0: plot expansion curves for the data taken from video recordings (§5.2) - 10%**

**Goal 1: plot expansion curves for the data set you received by email (§5.2) - 20%**

Note: you have to achieve all following goals using this data set.

**Goal 2: estimate the terminal settling velocity of individual grains (§5.3) - 20%**

**Goal 3: discover the relationship between fluidisation and terminal settling (§5.4) - 20%**

**Goal 4: determine the optimal specific surface area in a full-scale reactor (§5.5) - 20%**

**Goal 5: complete the QMUL questionnaire (§5.6) - 10%**

**5.2 Goal 1: Plot an expansion curve**



Topic: Mini lecture about composing an expansion curve  
Time: 4:12  
File: Virtual lab - fluidisation experiments - Goal EC.wmv  
URL: <https://youtu.be/sesRJ8YwCJE>

**5.3 Goal 2: Estimate the terminal settling velocity of individual grains**



Topic: Mini lecture about terminal settling.wmv  
Time: 3:48  
File: Virtual lab - fluidisation experiments - Goal TS.wmv  
URL: <https://youtu.be/yrgVoAKs5Uc>

**5.4 Goal 3: Discover the relationship between fluidisation and terminal settling**



Topic: Mini lecture about terminal settling and fluidisation  
Time: 3:48  
File: Virtual lab - fluidisation experiments - Goal RZ.wmv  
URL: <https://youtu.be/d2P75Qp8-hU>

**5.5 Goal 4: Determine the optimal specific surface area in a full-scale reactor**



Topic: Mini lecture about specific surface area  
Time: 3:04  
File: Virtual lab - fluidisation experiments - Goal SSA.wmv  
URL: <https://youtu.be/AMn1LPwJJ98>

## 5.6 Goal 5: Complete the QMUL questionnaire

In 2019 with the help of our Dutch colleagues from Waternet, we installed a liquid-solid fluidisation column for you in the laboratory of the School of Engineering and Materials Science. With this experimental set-up, called the expansion column, it is possible to discover the phenomenon of fluidisation, a frequently used industrial unit operation.

The expansion column is slightly different compared to other more standard facilities in the lab. For example, with this expansion column it is possible to work in groups, apply problem-based learning strategies\*) and solve technical and technological challenges with role-playing games. Finding effective pragmatic solutions and at the same time providing sustainable long-term answers to important questions is a vital part of a chemical engineer's distinctive competences and therefore extremely important for developing a successful future career. Last but not least, the expansion column is an adequate apparatus that is suitable for conducting high-quality research and producing accurate data sets.

You will probably not be surprised that we are very enthusiastic about the opportunities offered by the expansion column, but do you share our enthusiasm? As we would very much like to validate our own opinion, we are curious to hear about yours: your views are extremely important and will be highly valued.

In order to improve our curriculum for the benefit of future students, we kindly invite you to share your opinion about the current programme. The questions below can be answered by indicating your level of agreement with the statements presented (choosing a number on a five-point scale), by supplying feedback, by describing how you would qualify your own competences, and finally by answering a number of open questions.

All information will at all times remain strictly confidential. Only anonymous numerical results will be used to evaluate the programme; these results will be published.

Thank you for your consideration and your time.

QMUL Questionnaire: <https://goo.gl/forms/TOD3ZfMNIjp72OIW2>



### **5.7 Future opportunities (BONUS)**

This experimental set-up is relatively elementary. Present an outline of the options which could be applied to improve this set-up. To give you some examples: temperature control, gas inlet, pressure difference sensors over the reactor height, filtration (reversed flow), ...

### **5.8 Assessment (BONUS)**

To improve this set-up for the benefit of future students, share your opinion and your findings concerning this experimental work with the expansion column, i.e. hints, tips and tricks etc.

### **5.9 Communication**

In line with QMUL guidelines, you need to write a final report about the theoretical and practical aspects regarding the expansion column. Please submit your pdf reports on QM+. Also please email the Word version of your final product to Edo Boek and cc to Onno Kramer.



## 6 References

### 6.1 Bibliography

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## 6.2 Wikipedia websites

<https://en.wikipedia.org/wiki/Fluidization>  
[https://en.wikipedia.org/wiki/Fluidized\\_bed](https://en.wikipedia.org/wiki/Fluidized_bed)  
[https://en.wikipedia.org/wiki/Fluidized\\_bed\\_reactor](https://en.wikipedia.org/wiki/Fluidized_bed_reactor)  
<https://en.wikipedia.org/wiki/Porosity>  
[https://en.wikipedia.org/wiki/Settling#Single\\_particle\\_drag](https://en.wikipedia.org/wiki/Settling#Single_particle_drag)  
[https://en.wikipedia.org/wiki/Drinking\\_water](https://en.wikipedia.org/wiki/Drinking_water)  
[https://en.wikipedia.org/wiki/Stokes%27\\_law](https://en.wikipedia.org/wiki/Stokes%27_law)  
[https://en.wikipedia.org/wiki/Calcium\\_carbonate](https://en.wikipedia.org/wiki/Calcium_carbonate)  
[https://en.wikipedia.org/wiki/Kozeny%E2%80%93Carman\\_equation](https://en.wikipedia.org/wiki/Kozeny%E2%80%93Carman_equation)  
<https://en.wikipedia.org/wiki/Porosity>  
[https://en.wikipedia.org/wiki/Reynolds\\_number#Laminar%E2%80%93turbulent\\_transition](https://en.wikipedia.org/wiki/Reynolds_number#Laminar%E2%80%93turbulent_transition)  
<https://en.wikipedia.org/wiki/Settling>  
[https://en.wikipedia.org/wiki/Surface-area-to-volume\\_ratio](https://en.wikipedia.org/wiki/Surface-area-to-volume_ratio)  
[https://en.wikipedia.org/wiki/List\\_of\\_dimensionless\\_quantities](https://en.wikipedia.org/wiki/List_of_dimensionless_quantities)  
[https://en.wikipedia.org/wiki/Close-packing\\_of\\_equal\\_spheres](https://en.wikipedia.org/wiki/Close-packing_of_equal_spheres)

## 7 Appendices

### 7.1 Additional information about the expansion column



Topic: Info system start up (in Dutch).wmv

Time: 0:46

File: Virtual lab - fluidisation experiments - Info Start up.wmv

URL: <https://youtu.be/1-ZsUwrwFak>



Topic: Info system shut down.wmv

Time: 0:13

File: Virtual lab - fluidisation experiments - Info Shut down.wmv

URL: <https://youtu.be/SRpDqH2CCMo>



Topic: Info pellets withdrawal (in Dutch).wmv

Time: 7:27

File: Virtual lab - fluidisation experiments - Info Pellet aftap.wmv

URL: <https://youtu.be/Vjs9SqTvlgo>

7.2 **Fillable**

To help you to compose an accurate data set, you may use the following tables.

**Remember to use proper SI units.**

**Table 1** Data chart fluidisation-experiments basics.

<b>Grain type</b>			
<b>Description</b>	<b>Variable</b>	<b>Value</b>	<b>Comment</b>
Lowest sieve diameter	$d_{z,\min}$ [mm]		
Highest sieve diameter	$d_{z,\max}$ [mm]		
Average sieve diameter	$d_p$ [m]		
Particles mass	$m$ [kg]		
Inner column diameter	$D$ [m]		
Fixed bed height	$L_0$ [cm]		
Particle volume	$V_k$ [L]		
Minimum fluidisation flow	$Q_{mf}$ [L/hr]		Smallest particles
			Average value
			All particles
Maximum pressure difference	$\Delta P_{\max}$ [mbar]		
Minimum fluidisation bed height	$L_{mf}$ [cm]		
Specific density particles	$\rho_p$ [kg/m <sup>3</sup> ]		

**Table 2** Calculations.

<b>Description</b>	<b>Variable</b>	<b>Value</b>	<b>Equation</b>
Average particle diameter	$d_p$ [m]		Equation 17
Voidage fixed bed	$\epsilon_0$ [m <sup>3</sup> /m <sup>3</sup> ]		Equation 2
Voidage at minimum fluidisation	$\epsilon_{mf}$ [m <sup>3</sup> /m <sup>3</sup> ]		Equation 2
Maximum pressure difference over particle bed	$\Delta P_{\max}$ [Pa]		Equation 5

**Table 3** Data chart fluidisation-experiments.

Counter	Water flow [L/hr]	Bed height [cm]	Temperature [°C]	Pressure difference [mbar]	Pressure difference [cm H <sub>2</sub> O]
1					
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**Table 4** Calculations fluidisation experiment 1/2.

<b>Experiment</b>	<b>Superficial velocity [m/s]</b>	<b>Voidage [m<sup>3</sup>/m<sup>3</sup>]</b>	<b>Pressure difference [Pa]</b>	<b>Specific density particles [kg/m<sup>3</sup>]</b>	<b>Dynamic viscosity [kg/m/s]</b>	<b>Specific surface area [m<sup>2</sup>/m<sup>3</sup>]</b>
<b>Nr.</b>	<b>Equation 1</b>	<b>Equation 2</b>	<b>Equation 18</b>	<b>Equation 20</b>	<b>Equation 19</b>	<b>Equation 12</b>
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**Table 5** Calculations fluidisation experiment 2/2.

<b>Experiment</b>	<b>Logarithm Voidage [-]</b>	<b>Logarithm Superficial velocity [-]</b>	<b>Slope fluidisation index n [-]</b>	<b>Intercept Extrapolated superficial velocity <math>v_E</math> [m/s]</b>
<b>Nr.</b>	<b>Equation 1</b>	<b>Equation 2</b>	<b>Equation 21</b>	<b>Equation 21</b>
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**Table 6** Data chart flowmeter calibration.

<b>Experiment</b>	<b>Water flow Observed value [L/hr]</b>	<b>Calibrated water volume V [s]</b>	<b>Time <math>\Delta t</math> [s]</b>	<b>Water flow calibration <math>Q_w = V / \Delta t</math> [L/hr]</b>
<b>Nr.</b>				
1				
2				
3				
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10				

7.3 Piping and instrumentation diagram

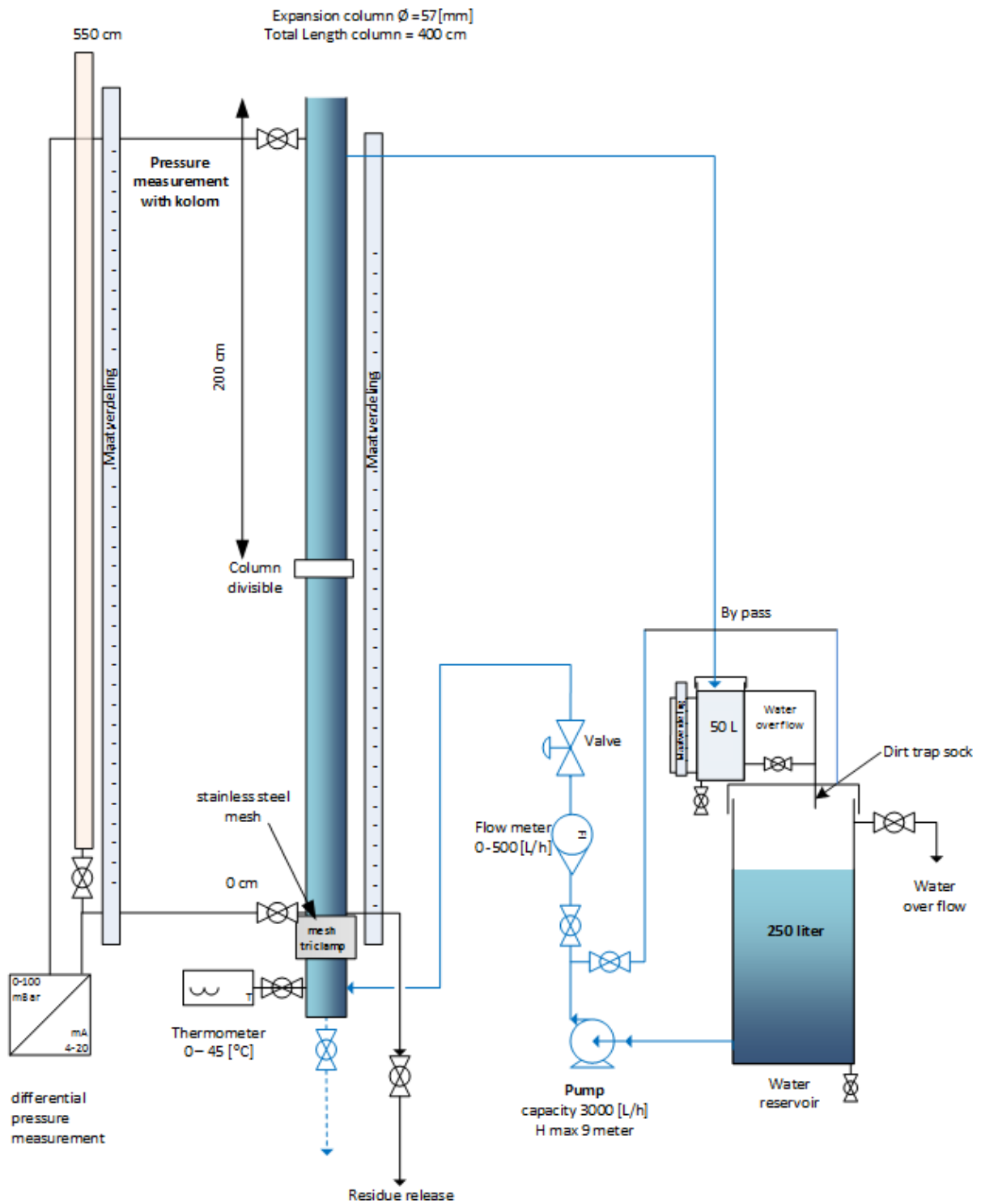


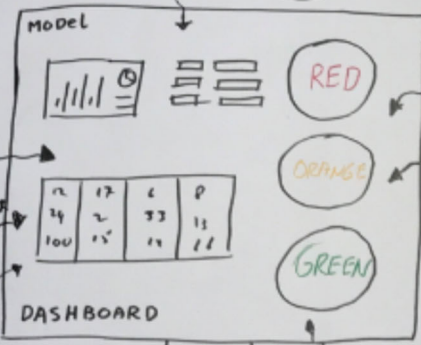
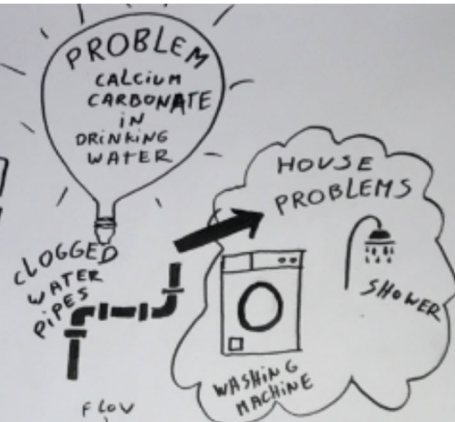
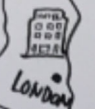
Figure 1 QMUL Expansion column.

# PILOT SOFTENING



WATERNET.NL

Queen Mary  
University of London

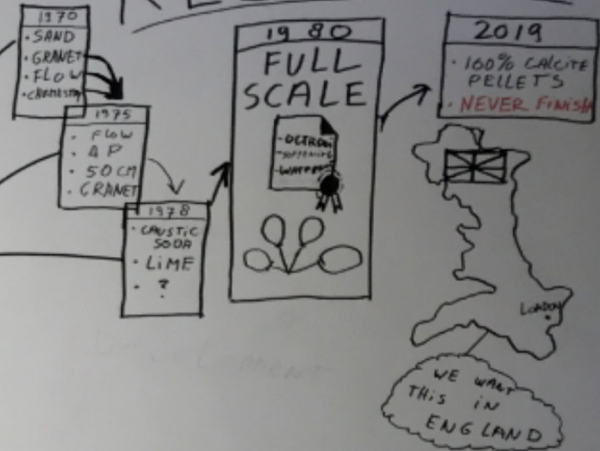


FIXED BED  
FLUID BED  
2 WEEKS

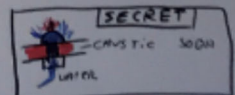
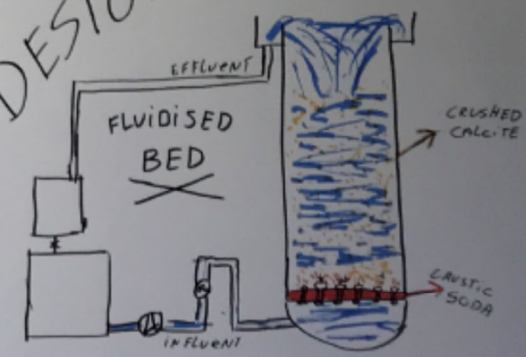


- MATERIALS**
- ALUMINUM PROFILE
  - PRESSURE DIFFERENCE
  - BARRELS
  - DRIP TRAY
  - PUMP
  - FLOW METER
  - CONTROL VALVE

# RESEARCH



# DESIGN



**Figure 2** QMUL Expansion column design drawing by Michel Colin (project leader pilot plant Amsterdam).

# **Expansion Column Notes**