
Characterization of ultra high vacuum system

(-by gas accumulation and SRG methods)

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Abstract

This thesis evaluates the concept of characterization process of ultra-high vacuum system, known as *SEA5 Serial Expansion Apparatus* at *RISE Research Institutes of Sweden*.

To produce precisely known pressures in the medium, high, and ultra-high vacuum range for gauge calibrations, the volume expansion system is often used for that purpose. There are different methods to determine the volume or the expansion ratios in such system. The two methods used during the characterization process are the spinning rotor gauge and the gas accumulation, by measuring the pressure before and after an expansion.

Furthermore, implementing the methods in the control software developed in LabView for fully automated characterization process. The achieved results by the methods were not completely in agreement with each other, that caused by different reasons.

Keywords: Spinning rotor gauge, Gas accumulation, Expansion ratio, Vacuum

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Nomenclature

DCR - Deceleration Rate

LTH - Lunds Tekniska Högskola

NPL - National Physical Laboratory

PLC - Programmable Logic Controller

PPM - Parts Per Million

PTB - Physikalisch Technische Bundesanstalt

RISE - Research Institutes of Sweden

SEA - Serial Expansions Apparatus

SME - Small And medium Enterprises

SP - Technical Research Institute of Sweden

Chapter 1

Introduction

This Master's thesis was carried out in Borås at RISE *Research Institutes of Sweden*. RISE is a governmentally owned research institute with a national directive to perform research and offer services, which are not financially sustainable but essential to the Swedish industry. One such service is provided by the department of Measurement Technology/National Metrology Institute, where the Pressure and Vacuum group is currently working on improving their capabilities to measure high and ultra-high vacuum.

1.1 Introduction

Accurate and precise vacuum measurement is of importance to several stakeholders, such as the life-science industry, semi-conductor manufacturing, and the process industry. An incorrectly measured vacuum can lead to a quality reduction of the product or, in the worst case the process may stop caused by the measurement uncertainty. Calibration of vacuum gauges is necessary to maintain high quality vacuum measurements. The calibration of gauges is necessary to e.g. achieve stable products, detect error in the production, fulfill quality requirements or to display correct values on meter [10].

A key concept in measuring technology is traceability. The traceability concept means that all measurements made with a calibrated measuring devices must be directly related to the defined magnitude, see the figure 1.1 below for illustration.

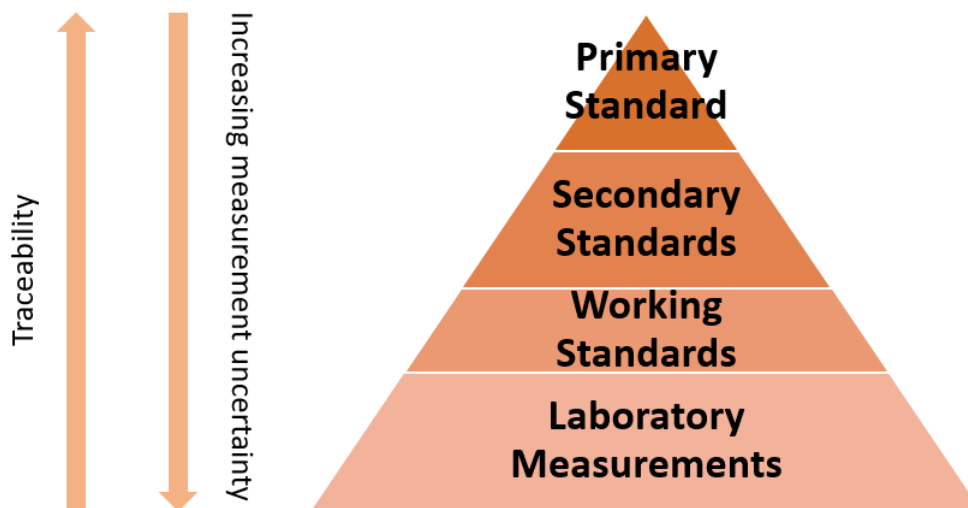


Figure 1.1: Traceability pyramid.

The primary standards are the international standards. The secondary standards link is calibrated against the primary standards. The chain's second link must be a national standard, where RISE stands for the national standards. The direct connection is obtained through a traceability chain. Each link in the chain represents a calibrated standard. All users of traceable calibrated meter can therefore be sure that they are really using the same device or measuring with same certainty. The chain can be built to any number of links. However, in each link, measurement uncertainty increases.[9]

In order to achieve a traceable reference for calibration, the static expansion is often used as a characterization method.

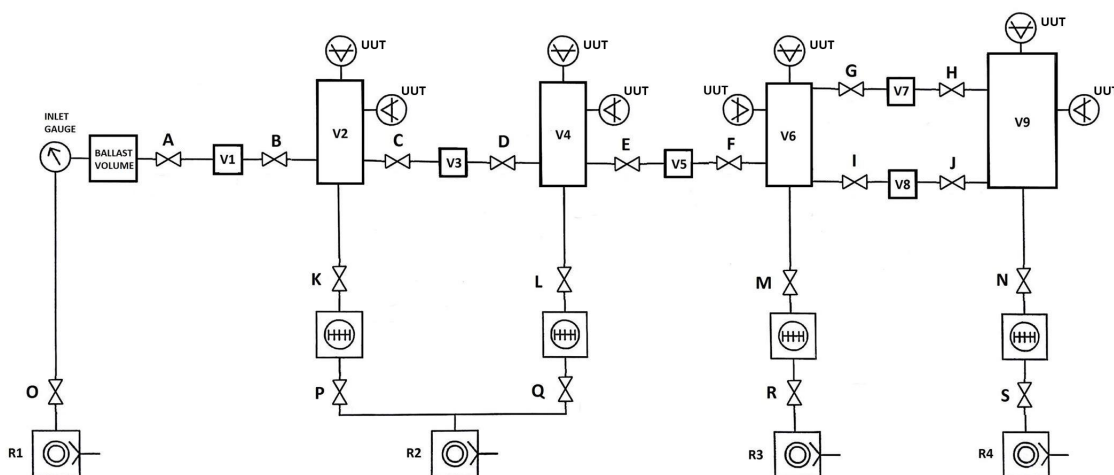


Figure 1.2: Schematic overview of SEA5 at RISE, the vacuum measurement occurs at the big volumes. UUT: unit under test

A static expansion pressure standard is typically characterized in terms of the ratio between the density of an expanded sample of gas and the density of the initial sample. Typically the system performs a series of gas expansions from smaller to larger volumes

where the ratio between each expansion is known and the final pressure can be calculated from the initial pressure and the ratio of each expansion. The figure 1.2 above illustrate the characterization principal.

The pressure and vacuum group at RISE has two static expansion standards, labeled Serial Expansions Apparatus (SEA). SEA3 is used for medium to low vacuum and SEA5 for high vacuum and ultra-high vacuum. Both systems are functional, but so far only SEA3 has undergone a full evaluation. During this thesis, SEA5 will be characterized to obtain the ratios by two methods, Spinning Rotor Gauge (SRG) and the gas accumulation. Those methods will be presented later in more detail during the report.

1.2 RISE

RISE, Innventia, SP and Swedish ICT have united together into RISE - Sweden's research and innovation partner for business and society at the beginning of 2017.

"As a strong, independent research institute, RISE offer a wide range of research and innovation services and cross-border business areas for SME (Small And medium Enterprises) , industry, academia and the public sector. In global collaboration, RISE enable renewal and value-creating innovations for increased competitiveness and sustainable social development."

RISE has 2200 employees and are located in 30 locations in Sweden from north to south within different areas. RISE is owned by the Swedish state and in 2017 RISE established its headquarter in Gothenburg [8].

1.3 Aims and Challenges

The aim of the project is to develop a method of characterization for SEA5 based on current best practice standards, and to execute a measurement of the expansions ratios of SEA5. SEA5 has five specific expansions ratios. The aim is to characterize them by two different methods, gas accumulation and SRG. These ratios are necessary to enable reliable instruments calibration later on by SEA5. When the ratios have been determined, the methods should also be included in the control software to allow for an automatic self-characterization. The system is developed in the U.K. by NPL and delivered to RISE in 2008. The system SEA5 has since 2008 undergone several upgrades and modifications. The control software has changed to LabView and the logical controller to Siemens S7. Many of the essential system functions have not yet been implemented and this project seeks to automate the characterization and ratio determination procedure. As with many systems in the high vacuum range a major challenge will be to control system leaks and maintain a low system base pressure. This in turn will require lengthy pump out times, which will make the measurement procedure time-consuming.

1.4 Thesis Outline

This thesis will start by covering some historical background on the idea of vacuum and technological development. It will also discuss the modern definition of vacuum. From this a more in depth discussion on the relevant theory will be covered in chapter 3. The chapter 3 will present the kinetic theory of gases and the ideal gas law. In chapter 4, a detailed look at SEA5 is presented together with the relevant measurement devices needed to realize the two ratio determination techniques. After the introduction of the experimental system the ratio determination methods are discussed in more detail in chapter 5. A study of previous work within the field of serial expansion systems and in particular the gas accumulation and SRG techniques will be presented. It also covers a third method called the gravimetric technique which might be of interest for the reader. The final part of this chapter will present the work done to implement these techniques in the already existing LabView code. This section will present the bulk of the efforts that went into producing the results. Lastly, the results will be presented in chapter 6 which also covers a discussion of the challenges that were addressed during the project.

Chapter 2

Background

In this chapter some historical input will be presented and the definition of vacuum and the use of it. Finally, different pressure ranges and relevant applications of each respective range will also be discussed.

2.1 The Historical Journey of Vacuum

The vacuum concept was discussed as early as 460-375 B.C. by the Greek philosopher Democritus. He assumed that the world consisted of many inseparable particles, which he called atoms. In Greek, atoms mean indivisible. Democritus assumed an empty space between atoms, a kind of micro-vacuum which the atom was oriented in according to some general laws of the mechanics. Distinctions in the shape, orientation and preparation of the atoms would be the variations of microscopic objects.

Democritus together with his teacher Leucippus could be considered the inventors of the vacuum concept. According to them, the vacuum space was a criterion for the variations in our world, as it allowed the atoms to move freely. Today's modern physical view is quite close to their idea.

Experiments on their idea came as late as the 1600's when Italian scientist were preparing to build a water system in Genoa, which forced a discussion about atmospheric pressure in the water pipes. The year was 1644 when the Florence professor Evangelista Torricelli presented an experiment which has been fundamental to latter progressions in science.

Torricelli's attempted experiment consisted of the following; he filled a 100 centimeter long tube with mercury, of which the open end was closed with a fingertip, see figure 2.1 below which shows a drawing of the Torricellian device.

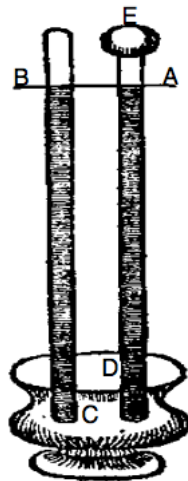


Figure 2.1: Torricelli's vacuum experiment in 1644. The level AB of mercury in both tubes C and D was equal, independent of the size of the additional volume E in tube D,[1].

The pipe was then rotated to an upright position with the end pointing downward closed by the fingertip. This was then lowered into a mercury reservoir and the fingertip was removed, exposing the column to the reservoir, which led mercury in the tube to fall to a height of 76 cm, measured from the liquid surface of the vessel. The experiment indicated that the volume left above the mercury column after rotating the tube is actually a vacuum. It showed that the mercury level was independent of the volume above and could be completely filled with water from below.

Two years later in 1646, Torricelli's experiment caught the attention of French scientist Blaise Pascal. Pascal, in turn, repeated the experiment with different types of liquids, he assumed that the the maximum height was precisely inversely proportional to the used liquids density. During a discussion between Pascal and the philosopher Descartes in 1647, they further developed the idea of air-pressure measurements at different altitudes using a Torricellian apparatus. Together with his son-in-law Perier, Pascal tested the experiment on the steep mountain Puy-de-D^ome (about 1500 m). Pierer climbed the mountain with a company of several men and noted the mercury height at different altitudes. From the bottom of the mountain and up to the top, the mercury height difference was 8 centimeters. This attempt was the first successful pressure measurement in history. Unfortunately, Torricelli was not able to enjoy the results achieved by Pascal, which were based on Torricelli's experiment, as he passed away a year earlier [1].

2.2 Vacuum and Its Use

The industrial and scientific significance of vacuum technology has grown during the past 30 years. Vacuum of low-pressure gas, discharge tubes by semiconductors and integrated circuits has forced an even greater request and more strict conditions on vacuum techniques for processing and manufacturing of the devices. This has led to development of number of "dry" vacuum pumps to produce a "clean vacuum" free of hydrocarbons. Vacuum tech-

nology is ubiquitous today and can be found in vastly different fields; the space programs, high-energy accelerators, analytical instruments, freeze-drying of foods and drugs, the manufacture of televisions, incandescent and metal vapor lamps, high-power vacuum and x-ray tubes all require vacuum technology [6].

2.2.1 What is Vacuum?

A vacuum is defined as a diluted gas, or the corresponding state at which its pressure is lower than that of the ambient surrounding atmosphere. Because atmospheric pressure varies over the earth's surface and decreases as altitude above sea level increases, it is not possible to specify a general upper limit for the vacuum range in space, since space continues to infinity.

In order to achieve a vacuum it is necessary to produce a pressure in a vessel that is lower than the surrounding pressure. Due to the Earth's gravity, atmospheric pressure differ with altitude in agreement with the barometric altitude formula

$$p = p_0 \exp\left(-\frac{g\rho_0 h}{p_0}\right), \quad (2.1)$$

where $p_0 = 101.3$ kPa is the atmospheric pressure at sea level and 0°C , $g = 9.81$ m/s^2 is the acceleration due to Earth's gravity, $\rho_0 = 1.293$ kg/m^3 is the density of air at sea level and 0°C , h is altitude height in meters [11].

By using equation 2.1 and solving for $p_{1/2}$ to get at what altitude h the atmospheric pressure will decrease to one half, gives $h = 5.548$ km. This means that the atmospheric pressure will decline by approximately one half every 5.548 km [11].

In a given volume, vacuum can be achieved by removing air and other gases. The air and gases are removed with specific pumps known as vacuum pumps. There are many and very different types of vacuum pumps. Some of them pump away gases, other pumps retains gases or change their shape in the chamber. In all cases, the pumps task is to remove as many and as much of the gas as possible from the vessel [12].

There are different vacuum range and category depending on area of use. The table 2.1 below will illustrate these different pressure ranges and the applications that are used within specific area.

Table 2.1: Different pressure range and some applications [11].

Pressure, mbar	Pressure Range	Application
$10^3 - 10^0$	Low vacuum	Freeze drying-Packing industry, Degassing
$10^0 - 10^{-3}$	Medium vacuum	Incandescent lamp manufacturing
$10^{-3} - 10^{-7}$	High vacuum	Electronic tubes, space simulation
$10^{-7} - 10^{-12}$	Ultra high vacuum	Particle accelerators, Nuclear physics

From the table you can see which vacuum category is needed for a specific application area. More advanced and sensitive products require cleaner environments which means lower vacuum range. Worth mentioning is that the SEA5 measurements requires a vacuum somewhere between the high and ultra-high range.

Chapter 3

Theory

In this chapter the principle of pressure will be presented in detail. The physical term and its description will be analyzed.

3.1 Kinetic theory of gases

For a correct understanding of the concept of gases, mainly at low pressures, it is necessary to reflect on these phenomena from the point of view of the kinetic theory of gases. This theory base actually upon two fundamental assumptions. The first assumption is that matter is made up of very small particles, which in the gaseous state at average temperatures are monatomic or polyatomic molecules and at higher temperatures may be completely separated into atoms or also positive ions and electrons to shape a plasma. Secondly assumption is that the molecules of gas are in constant motion, and this movement is closely related to macroscopic properties known as the temperature and pressure T , P , which characterize the state of the gas in a specified small region [6].

As mentioned in the paragraph above, monatomic particals belong to the so-called single atoms, examples of these atoms are helium He and neon Ne . On the other hand, multi-atomic molecules are called polyatomic molecules, and example of these are, oxy- gen O_2 and carbon dioxide CO_2 particles.

A confined gas consist of a large number of fast moving particles or molecules. As an example, see figure 3.1, consider a rectangle of volume V filled with N gas molecules, each with a mass m and moving with a speed v .

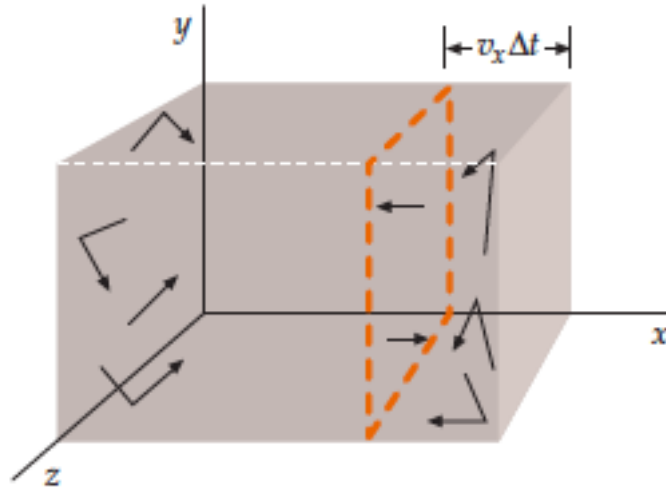


Figure 3.1: Container filled with some gas to illustrate the kinetic gas theory, the arrows illustrate randomly moving molecules [7].

Very large number of molecules are randomly moving at speeds of hundreds of meters per second. These movement causing the molecules to elastic collisions, both with each other and with the walls of the container, these will cause a force exerted by these molecules on the right-hand wall in the figure 3.1, that will lead to the equation below [7].

$$n = \frac{1}{2} \frac{N}{V} |v_x| \Delta t A, \quad (3.1)$$

where n is the number molecules hitting the wall, A is the area of the wall and $|v_x| \Delta t$ is the distance to it. By Newton's second law, the force is the rate of change of momentum of the gas molecules colliding with the wall. Related to motion along the x -axis, the momentum of a molecule is $+mv_x$ before it hits the wall, and $-mv_x$ after an perfect elastic collision with the wall. These will lead to a total magnitude change $2mv_x$ and total change in momentum $|\Delta \vec{p}|$ during a time interval Δt . Simply the entirely momentum change of all molecules will be the total magnitude times total number molecules hitting the wall, that means

$$|\Delta \vec{p}| = 2mv_x n = 2mv_x \left(\frac{1}{2} \frac{N}{V} |v_x| \Delta t A \right), \quad (3.2)$$

the magnitude of the force exerted by the wall on the molecules, and the magnitude of the force exerted by the molecules on the wall, is the ratio $\frac{|\Delta \vec{p}|}{\Delta t}$. The pressure is the magnitude of this force divided by the area A

$$P = \frac{F}{A} = \frac{1}{A} \frac{|\Delta \vec{p}|}{\Delta t} = \frac{N}{V} m v_x^2 \quad (3.3)$$

or simply in terms of kinetic energy can 3.3 be written as

$$PV = 2N \left(\frac{1}{2} m v_x^2 \right) \quad (3.4)$$

All these equations above are valid under ideal conditions, which means we neglect gravity force, air resistance and varying speed. An example of a limitation is the speed (v^2), all the molecules do not move at a constant speed, instead their speed are various from each other. To handle this limitation a average speed $(v^2)_{av}$ need to be calculated and that will lead to the square root of $(v^2)_a$ is referred as the root-mean-square (rms) speed

$$v_{rms} = \sqrt{(v^2)_{av}} = \sqrt{\frac{3RT}{M}} \quad (3.5)$$

where M is the molar mass, T is the absolute temperature and R it the universal gas constant.

In addition to translational kinetic energy, the molecules may also have rotational or vibrational kinetic energy. But in any cases, only the translational kinetic energy is relevant to the calculation of the pressure applied by a gas on the walls of its vessel [7].

3.2 The ideal gas law

Now that we have presented the concept of kinetic energy and its meaning, we are ready for the next important law, the ideal gas law.

The properties of gas sample that have low densities guides to us the definition of the ideal gas temperature scale. Suppose the following scenario, if we compress such a gas while remain its temperature constant, the pressure increases. In the same way, if a gas expands at constant temperature, its pressure decreases. As an estimation, the product of the pressure P and volume of gas sample that has low density is constant at a constant temperature T . This outcome or result was discovered experimentally by Robert Boyle during (1627–1691), and is known as Boyle's law, which follows by the equation 3.6 below

$$PV = constant, \quad (3.6)$$

There is a more common law exists that recreate Boyle's law as a special case. The absolute temperature of a gas example that has low density is proportional to its pressure at a constant volume. In addition to that, the absolute temperature of a gas example that has a low density is proportional to its volume at a constant pressure. This outcome was discovered experimentally during (1746–1823) by Jacques Charles and during (1778–1850) by Joseph Gay-Lussac. By combine these two achieved results, that led to the equation below

$$PV = CT, \quad (3.7)$$

where C is a constant that has positive value. We can see that this constant is proportional to the number of molecules of the gas example by keeping in mind the following. Assume that we have two containers that have exactly the same volumes and each carrying the same quantity of the same kind of gas at the same temperature and pressure. If we then assume the two containers as one system by merging them, we have twice the amount of gas at twice the volume, but still at the same temperature and pressure. We have then doubled the quantity $\frac{PV}{T} = C$ by doubling the amount of gas. We can therefore write C as, a constant k times the number N molecules in the gas, and by the equation below we get then

$$PV = NkT, \quad (3.8)$$

where $C = Nk$. The constant k is called Boltzmann's constant. It is found experimentally to have the same value for any kind of gas. If we have n moles of a substance, then the number of molecules is $N = nN_A$ where N_A is the Avogadro's number

$$PV = nN_A kT = nRT \quad (3.9)$$

where $R = N_A k$ is the universal gas constant for any gases. Finally the equation 3.9 leads to the ideal gas law, which later in chapter 5 is used to derive the ratios by using these above mentioned laws [7].

The international SI unit for pressure is set to Pascal (P) after the famous French physicist Blaise Pascal but there are many more units except it, some of these are the following

- mBar
- atm - atmosphere
- Torr

it does not matter which unit you use to calculate the pressure, there are conversion tables that derive the conversion between these above-mentioned units. Many measuring instruments measure under different units and can convert them to the desired one. [12].

Chapter 4

Experimental system

In this chapter, the experimental system SEA5 and the measurement tools of vacuum will be discussed.



Figure 4.1: A: DPI1515, B: Turbo pump, C: Ionization gauge, D: Connection flange, E: Baratron gauge, F: Ruska 7050i, G: Volume V1, H: Valve, I: Volume V2, J: Volume V3 and K: Volume V4

4.1 Static expansion apparatus V

The static expansion apparatus V at RISE, the department of Measurement Technology and the National Meteorology Institute is shown in the figure 4.1 above. The Figure 4.1 illustrates an overview over the entire system SEA5 and its measurement instrument.

The gas inlet into the system starts from the left side of the schematic figure 4.2 below by the regulator DPI515 and the reference sensor Ruska 7050i, which are interconnected, through V1 and the finale pressure is generated in V9. The valves are presented by the letters (A-S), and all the volumes presented by (V1-V9). There are four turbo pumps which located below the valves (K, L, M and N) to evacuate the volumes, and R1-R4 are the usual pumps. Finally the measurements gauges are connected in CP as shown in the schematic figure during the characterization process, both the Baratron and the SRG head. In this case CP stands for the flanges on the containers, see figure 4.1.

Generating lower pressures in the system is based on the simple idea, V1 is about 200 times less than $(V2 + V3)$, and when an expansion occurs from V1 into $(V2 + V3)$, the start pressure is reduced by 200. Then the expansion can continue with the same principle all the way to V9. Furthermore, to describe the calibration process of a meter via SEA5, we can look at the following example. A meter to be calibrated is mounted on the large volume V9. Based on the meter's measuring range, a pressure in V9 is generated by setting a start pressure in the system and calculating the final low pressure with high accuracy in V9 using volume ratios. Let's say for simplicity the meter's measuring range is 0.1-10 mbar. Three measuring points are measured by the meter within the measuring range of the gauge, one at the lower range, one at the middle and the last point at the end, each point with repetition of three, to get an overview of the standard deviation and to see the gauges linearity measuring behavior. The obtained values are compared to SEA5 as a reference to determine how much the gauge deviates, thereafter determining the uncertainty of the gauge. This is the simple idea of gauge calibration, and it indicates how important it's to determine these ratios with high accuracy.

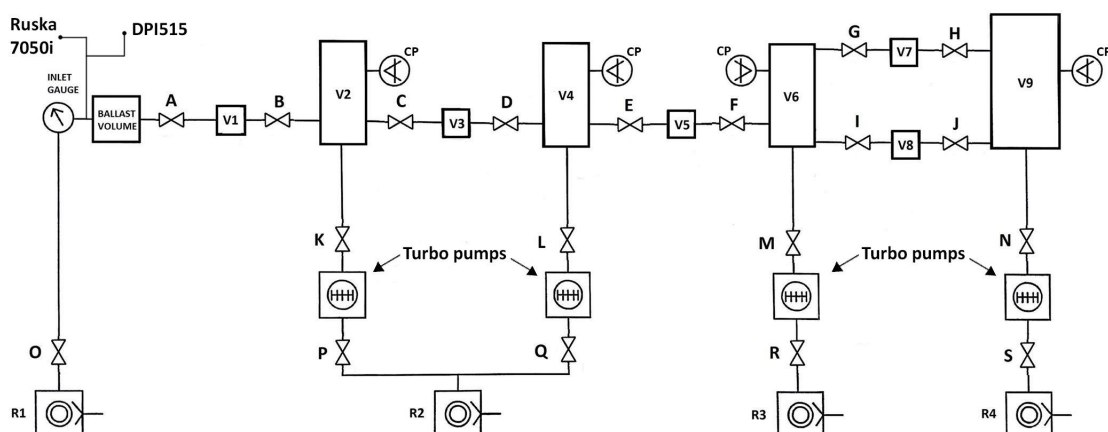


Figure 4.2: Schematic overview of SEA5, gas inlet is from left side through V1 and the finale pressure are generated in the last volume V9. CP: Connection Point

As mentioned during thesis out line 1.4, the system has five specific ratios between the volumes. These are

- R1, the ratio for an expansion from: V1 into (V2 + V3)
- R2, the ratio for an expansion from: V3 into (V4 + V5)
- R3, the ratio for an expansion from: V5 into (V6 + V7 + V8)
- R4n, the ratio for an expansion from: V7 into V9
- R4d, the ratio for an expansion from: V8 into V9

This means that the system has five ratio steps from start (V1) to the end (V9), and the purpose is to determine these ratios by the two methods.

4.2 Instruments for the measurement of pressure

The system is connected to different sensors and measuring instrument. An more detailed description on the SRG head and the Baratron gauge will be covered during the sections below.

There are temperature sensors attached at every volume surface to measure the volume temperature except the V9 which have three. The reason is, V9 has a big surface area and to get a more accurate value it is necessary to calculate the mean value of these three sensors.

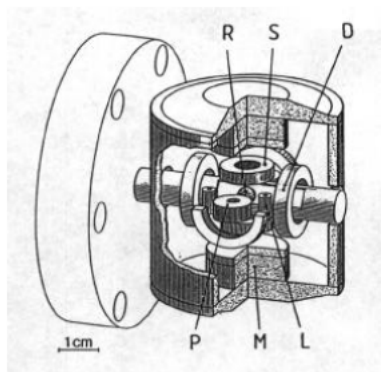
Then there are Ionization gauges that measure the residual drag or known as rest-pressure in the vessel, these gauges are assembled at the big volumes tops (V2, V4, V6 and V9). These sensors are important for the system especially during a baking process, since these give an indication on the residual drag in the containers to avoid any possible risks during baking caused by a increased pressure. The last volumes (V6-V9) are prepared for a baking process, that is necessary to obtain an very low rest-pressure in the containers. By baking the volumes during a cycle of approximately 20 hours and at 150-200 Celsius, that will heats away dust and bubbles from the inside walls of the containers. This procedure will decrease the rest-pressure by 2-3 decades, as best down to 10^{-11} mbar.

Finally the regulator DPI515 and the reference sensor Ruska 7050i are located at the inlet of the system. The DPI515 regulates the desired pressure and Ruska 7050i as a reference measures the pressure and feed it back to the DPI515 to generate the required pressure, that why Ruska 7050i is named as a reference sensor.

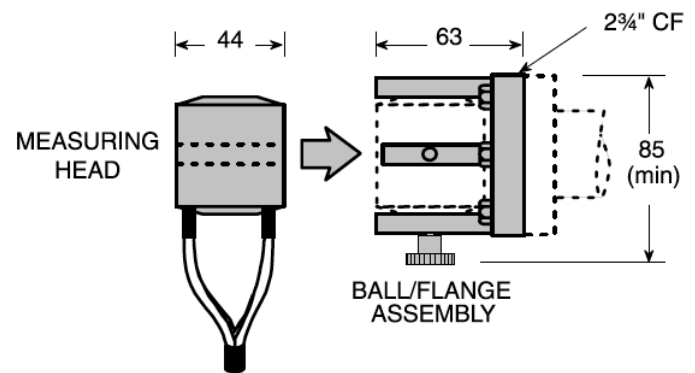
A slightly more detailed description of the two important gauges mainly used during each characterization method will be presented separately below.

4.2.1 MKS spinning rotor gauge

The main part of the spinning rotor gauge is a freely rotating metal sphere caused by a magnetic field in vacuum, which slows down by the friction from gas molecules. The metal sphere is located inside the flange as shown in the figure 4.3b below. The corresponding deacceleration is proportional to the pressure. The measuring range of this gauges is 1×10^{-7} to 1 mbar. The figure 4.3 below shows closely the details of the measurement head.



(a) Details of the SRG head.



(b) The SRG head and the connection house, the flange.

Figure 4.3: A detailed illustration of the SRG head [4].

The figure 4.3a shows the rotor sphere R in the measurement cylinder of the flange. There are two permanent magnets M which create a strong homogeneous magnetic field in the section of the measurement cylinder. While the rotor is in the working position i. e. with its center on the cylinder axis, an unstable vertical position is reached. Stabilization is accomplished by the two opposite coils S which increase the upper and lower field of the permanent magnets as claimed with the deviation of the rotor. The Horizontal deviations of the rotor controls by four coils L . The rotor is driven by a two-phase motor existing of four coils D . And finally the signal pick-up is executed by two coils P which are placed opposite from each other and are switched in series.

All sensors have their limitations and their measuring range. The indicated pressure for the SRG is shown in the 4.4 below for different input pressures. The graph illustrate that the linear region where the SRG measures linearly is approximately up to two 1×10^{-2} mbar, after which the output becomes non-linear and increases the uncertainty in the obtained measurement [4].

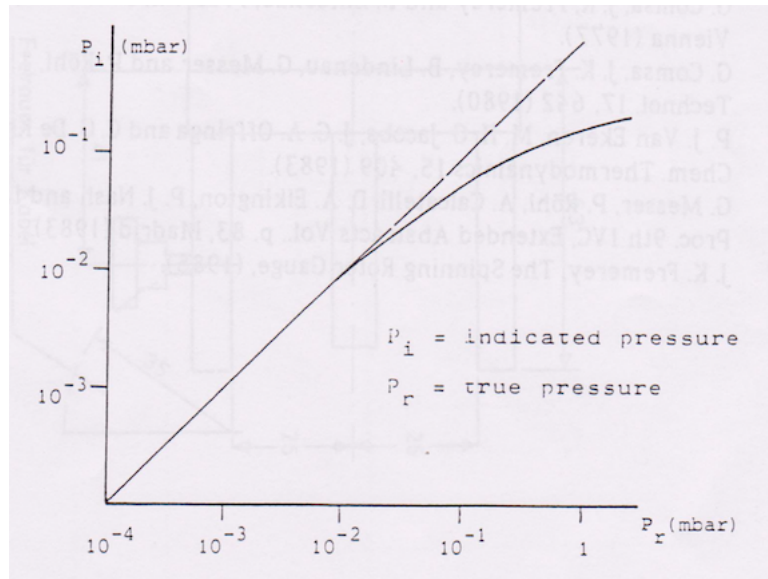


Figure 4.4: The pressure range for SRG gauge and the linearity region [4].

4.2.2 MKS type 690 Baratron

The Baratron 690A is designed for high accuracy pressure measurements between 0-130 mbar with single-ended dual electrode/AC bridge instrumentation that is extremely stable and designed to minimize the influence from temperature changes [5].

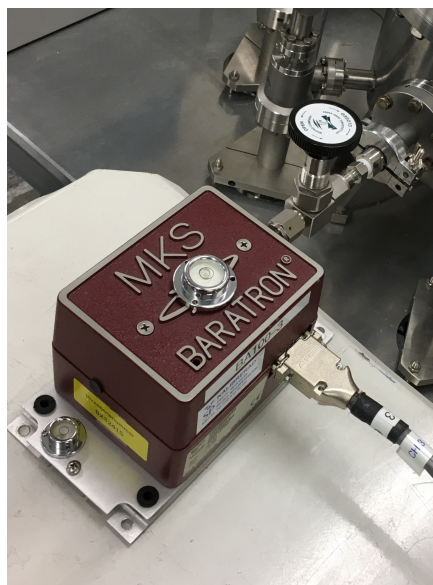


Figure 4.5: MKS Baratron type 690 connected to a vessel during a measurement.

The sensor technique in the baratron is as follows. Changes in pressure/vacuum are determined by measuring the deviation in capacitance between the sensor's diaphragm

and the electrode disk. This capacitance change is converted to a usable output through the built in electronic board .

The MKS type 690 baratron needs a power supply of $\pm 15\text{VDC}$ input power and gives an output of 0-10VDC signal with a linear pressure characteristic. MKS 690 baratron is interfaced with the high accuracy signal conditioner of type MKS 270D to convert the measured signal to a readable pressure, by converting the output signal 0-10VDC from the Baratron to a pressure using a conversion formula. According to calibration certificate, that the pressure becomes linearly above 25 mbar for the used sensor during the characterization process.

This gauge was mainly used during the gas accumulation method for measuring the pressure after the expansion.

Chapter 5

Ratio determination methods

The following chapter will describe the two volume ratio determinations methods used during the project. It will also present an alternative method. During each method the ratio will be derived and discussed, both the benefits and the limitations will be mentioned.

5.1 Introduction to Ratio Determination

The fundamental method used to determine the ratios of SEA5 is based on gas expansion. Consider the following scenario: a small previously evacuated volume is filled with a suitable gas measured at sufficient high pressure, and expanding it into a pre-evacuated larger volume, this will lead to a pressure reduction in the system volume which is calculable from the known ratio of the system's volume. Consider the initial or start pressure in the smaller volume be v and p , and let the big volume V be evacuated by vacuum pumps down to a negligibly low pressure, lower than the used gauge's resolution limit. Finally by opening the valve in between and the volumes we will get, under isothermal circumstances and by applying Boyle's law,

$$pv = P(v + V), \quad (5.1)$$

then by solving for P it leads to

$$P = \frac{pv}{(v + V)},$$

and finally the ratio can be expressed as

$$R = \frac{(v + V)}{v}, \quad (5.2)$$

where P is the low pressure caused the by expansion and R is the required volume ratio of the system. The mentioned method above is based on the Knudsen *Expansion* method [3].

Thus, the expanded pressure P is directly calculable from the initial pressure p and the volume ratio R . To attain large ratios, different techniques are possible, these are,

- A single stage expansion system with volume ratio about $7 * 10^6$
- A multi step system in which the large overall volume ratio is effectively attained by successive expansions through a series of volume pairs, as the case for SEA5.

NPL had a system with five stages each of volume ratio about 10^2 . This is known as the series expansion method. There are, of course, in between methods such as that of Messer whose apparatus at PTB The Natural Metrology institute of Germany has 2 stages of approximate ratios $2 * 10^4$ and $2 * 10^3$ [3]. The big advantage of having several steps compared to single stage is that the first mentioned case has a high accuracy, achieved from the relative low uncertainties in the determination of the volume ratios, in the single stage the estimation of the small volume can be a serious limitation on accuracy caused by the gauge's resolution.

5.2 Gas accumulation

By using single stage system the uncertainty of the small volume will normally limit the systems accuracy, caused by the gauges. This limitation can be overcome by multi stage systems with separate stages of moderate volume ratio. Meaning, by measuring the pressures in every vessel before and after an expansion, but this solution still has the limitation on the lower pressure. There is a better trick to overcome this obstacle. The big benefit of this method is that the ratio is determined during exactly state, and this will also take into account any changes related to the valve volumes.

Successive expansion came as a solution as there was a limitation on pressure gauges measuring at low pressure. The generated low pressure after expansion from a small volume into larger volume was difficult to measure with good accuracy, limited by the gauge resolution. The trick was then, to build up the low pressure in the big volume as well as the high pressure re-established in the small one, and repeating the procedure until reaching a high enough pressure in the big volume to be measured with a high accuracy.

To understand the method let us see the following scenario. The determination of a volume ratio let say of 100 and with a initial pressure in the small volume 100 kPa. After the first expansion, the valve in between is slowly closed without affecting the pressure distribution in the volumes, and the initial pressure re-established. By repeating the procedure once again, it will generate approximately a pressure of 2 kPa in the big volume. This procedure can be repeated until desired pressure is reached in the larger volume to be measured with a better accuracy. The procedure of this method known as the Successive Expansion method. Volume ratio by the mentioned method can be determined using the equation below 5.3

$$R = \frac{1}{(1 - (1 - \frac{P_n}{p})^{1/n})}, \quad (5.3)$$

where P_n is the lower pressure in the large volume after n expansions, p is the initial high pressure in the small volume and n is the n^{th} expansion [3].

So far the ratio expression above is valid under ideal conditions without taken into account any deviations on the system or on the gas, such as temperature etc. There are some

necessary corrections steps needed to be made on the ratio value to get a more accurate or correct ratio. The ratio needs to be corrected for some factors which affects the ratio, those are

- Correction for temperature drift
- Correction for temperature gradient
- Correction for departure from ideal gas law
- Correction for the gauge and connection volumes

5.2.1 Correction for temperature drift

To make a correction for the temperature drift, assume that the container or vessels walls are increasing in temperature via a steady dT kelvin between the successive expansions. Now by reflecting on that after the second expansion, about half the mass of the gas will be increased in temperature dT kelvins and the noticed pressure should be $dT/2T$ higher than the case for ideal condition. By keeping this in mind, after n expansion the pressure should be approximately $(n - 1)dT/2T$ higher. Then, it could be expressed by letting T_1 and T_n be the temperature of the large vessel at the first and n^{th} expansion. This assumption will lead to the following expression,

$$dT = \frac{(T_n - T_1)}{(n - 1)},$$

and the pressure increase therefore becomes:

$$\frac{(T_n - T_1)}{(2T_1)},$$

i.e. to correct for temperature drift, values of P_n must be reduced by a factor

$$\frac{(T_n - T_1)}{(2T_1)},$$

To present this correction in terms of R , we can see from equation 5.3 that for $P_n/p \ll 1$, $R \approx np/P_n$. Thus, a correction which reduces P increases R in the same proportion, thereby the necessary correction factor for R is then,

$$1 + \frac{(T_n - T_1)}{(2T_1)} \quad (5.4)$$

5.2.2 Correction for temperature gradient

As mentioned earlier the equation 5.3 is valid under isothermal circumstances, but in reality there will be a small temperature gradient throughout the vessels. By assuming that the temperature for the big volume is T_V kelvins and that for the small volume T_v kelvins. The maintained pressure after n expansion will deviate from the ideal situation by T_v/T_V . Since the large volume surface is the big and the dominant one, which will effect the correction most, the correction factor for the expanded pressure is T_v/T_V , and for the volume ratio R is,

$$\frac{T_V}{T_v} \quad (5.5)$$

5.2.3 Correction for deviation from ideal gas law

The temperature correction terms and the derivation of the equation 5.3 is valid for the ideal gas equation $PV/T = \text{constant}$. But there are well-known departures from this ideal equation which are small for the used gas nitrogen during the characterization at room temperatures and moderate pressure. Bearing this in mind we can now introduce the thermodynamic state of gas equation,

$$\frac{PV_m}{R_0T} = 1 + \frac{B}{V_m}, \quad (5.6)$$

where V_m is the molar volume, and B is the second virial coefficient, which for a given gas is a function of temperature only [3]. This value are attached in the appendix for nitrogen and some other gases. By re-arranging the equation above, the expanded gas must be corrected by a correction factor,

$$1 - B\left(\frac{1}{V_m} - \frac{1}{v_m}\right),$$

where v_m and V_m are the molar volumes before and after expansion. Since the ratios that we are trying to decide are greater than 50, the term $1/V_m$ can be neglected by a small error, since the term is related to the large volume. By taking that in mind the derivation of the equation 5.3 should be corrected by a correction factor,

$$(1 - B/v_m) \quad (5.7)$$

5.2.4 Correction for the gauge and connection volumes

So far, we have measured the ratio including the volume of baratron v_g and its connection, which means the measured ratio is $(V + v + v_g)/v$. The desired volume ratio is therefore $(V + v)/v$, which means it is necessary to evaluate the term v_g/v from the measured ratio. A comfortable method to calculate the term v_g/v is by direct expansion. The method is performed by the following steps, first pressurize the large volume and the connected baratron gauge up to the gauges upper limit. Then, by closing the valve between the container and pressure gauge, the next step is evacuate the large volume completely, and lastly the valve is re-opened and the expanded gas is measured by the gauge its self or by some auxiliary reference gauge, for example the SRG gauge [3].

5.3 SRG Spinning Rotor Gauge

The second method which will be presented here is so called SRG method, which also based on expansion technique. The pressures before and after the expansion are used for directly determination of the expansion ratio, which means an expansion from the small volume v into the large one V is executed, the first DCR1 value is taken. Then by isolating the small volume v and evacuating the large volume V down to its resolution limit, thereafter the second expansion from the small volume into the large one is performed

again, and the second DCR2 value is measured. The same SRG head is used to measure the pressure both before and after the gas expansion. The ratio can simply be expressed by the equation below,

$$R = \frac{DCR1}{DCR2}, \quad (5.8)$$

where the *DCR* is the deceleration rate of the rotor of the SRG which corresponds to a pressure reading. The most beneficial aspect of the method is its ease of use, it requires only the the measured pressure before and after an expansion to calculate the ratio. But the method has its limitations too. The problem of using a SRG for low expansion ratio measurements is that the useful linear range of the SRG is relatively small. At pressures above about 1 Pa the deceleration rate of the rotor becomes nonlinear with pressure due to viscosity effects and at low pressures of a few mPa the accuracy of the reading is affected by the offset fluctuations of the SRG, as mentioned earlier in the report [2].

Since we have been within the gauges linearity region we only need to correct the ratio *R* for temperature gradient. It is the same correction factor as for the case of successive expansion as mentioned in the section 5.2.2, the correction factor of the ratio *R* is then,

$$R_{corr} = \left(\frac{DCR1}{DCR2}\right) * \left(\frac{T_v}{T_v}\right) \quad (5.9)$$

5.4 Alternative method

There are more possible methods besides gas accumulation and SRG methods. The most natural method of determining the volume ratio of an expansion stage is to measure the volume of each container separately. It can be achieved either by dimensional determination by filling the container with a suitable liquid with known density and weighing it. During ideal conditions this method has a high accuracy, often better than 1 ppm .

But a limitation of this method is that the form complexity of the container and the valves can trap air bubbles, which can lead to an uncertainty in the volume determination. Distilled water is mostly used during this method as a reference liquid, since the density at the measured temperature is well known and will therefore not contribute significantly to the uncertainty. This method is, however, not a favorite among vacuum scientist, since they avoid to fill their systems with water, which is very hard to remove the water from the system afterwards.

5.5 Software implementation

Now that the measuring instruments, SEA5 and the different volume ratio determination methods have been presented and the actual implementation in LabView will be followed during this section.

The remaining step is to interconnect all these instruments with each other via a monitoring control program that can enable a user to perform automated characterization for the vacuum system. There are several different monitoring and control programs available at the market, but the one RISE is using and has upgraded its system to is a control program developed in LabView.

Before going into the details a short presentation of LabView will follow. "LabView is a graphical programming software, which facilitates the working, enables programming of hardware for testing, measurement, control, analyzing data, presenting results, and distributing systems". Worth to mention for the reader that LabView is developed by the US software company National Instruments. During the next section, a more detailed description of the software implementation will be presented.

5.5.1 Interface

The major part of this thesis has been applied to software implementation where the two volume ratio determination methods gas accumulation and SRG were implemented in the control program as an addition to the existing code. The goal was to be able to measure and present the measurement data in an efficient and transparent way. Therefore, it was necessary to get into the existing LabView interface and afterwards add the measurement routines to follow the red thread in the graphical environment software.

The graphical programming language LabView differs from the traditional programming languages such as Java and C++. The biggest difference is that programming logic is based on the graphical interface, that is, building up its sequence using graphic components or elements on the "Front Panel" window where all the graphical elements are located and the obtained results. For each element that placed on the front panel, such as table, button, string and so on, its logical symbol appears under "Block Diagram". The Block Diagram window in LabView is where all the created elements symbols appears, and as well all programming is performed during this window. LabView has different execution structures in order to meet the logical statements of a certain problem formulation. A "Case Structure" is similar to if, else or switch statements in text based programming languages. There are more structures, such as "For", "While", "Flat sequence" and so on. Flat sequence is interesting. This structure makes it possible to perform a task at a time before executing the next state, this is important tool since LabView compilation occurs parallel and differs from the text-based languages. It is a way to have a control over the sequences. This structure was used throughout the programming section. In addition to structures, LabView also has "functions" which are necessary for type conversion use, as an example from integer to string. There are functions which can be used during a communication process with the switched-on instruments, in our case, the measurement instruments. Those mentioned structures and functions are some of the LabViews library tools which were used during the programming part.

Before starting an measurement sequence, the system parameter tab must be filled in order to make the necessary corrections for the measuring instrument PPI 7050i and MKS baratron. These parameters are derived from a calibration certificate that indicates how much these measurements differ from the actual measured pressure, a figure can be found in the appendix B.1.

5.5.2 Gas accumulation and SRG implementation

The figure B.3 in the appendix illustrates the gas accumulation method and its implemented code sequence, but in the figure 5.1 below an overview of the code sequence is shown by flowchart, the flowchart is valid for the both methods.

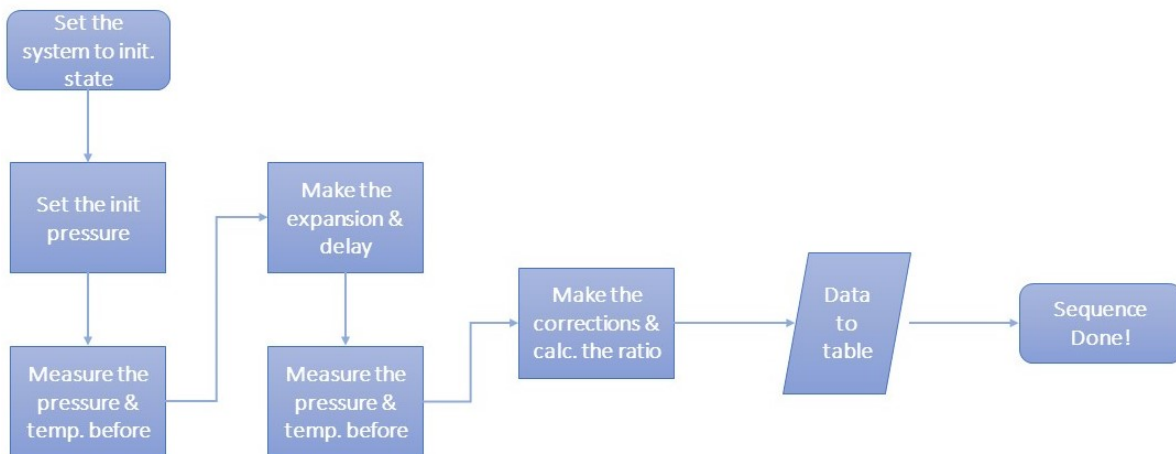


Figure 5.1: Flowchart of gas accumulation and SRG sequence.

The entire sequence is structured with "While", "Case Structure", "For" and "flat Sequence" to be able to execute one frame at a time in order to meet the desired logic in the methods. Starting by setting the whole system to initial state, namely setting all valves to initial state before pressurizing the system. Afterward measuring the the high pressure and its temperature, later on making the expansion between the volumes and waiting until the pressure distribution stabilize in the system, thereafter measuring the low pressure and its temperature, the next step, making all the mentioned corrections on the methods during this process before collecting all data to a table. The table can be saved to a file for a further analysis, for example, in Excel or in Matlab. The sequence follow the methods as described in 5.2 and 5.3. For further details, see the appendix B, there follows figures over the front panel and code sequences for R1 implementation.

Chapter 6

Result and discussion

During this final chapter, the achieved result will be presented, and as closer the chapter will have a outlook, discussion and a conclusion.

6.1 Result

The achieved results will be presented during this section. The ratios shall be compared with the existing old ones, which RISE carried out by NPL programed characterization routine in the old control software. Starting randomly by presenting R3 and showing the different correction steps for the ratio's mean value achieved by the gas accumulation method.

The R3 measurement consists of 22 measuring data (the whole data sheet is available under appendix A.5) and the obtained ratio R3 is *195,565*, the ratio was corrected afterwards for measurements uncertainty caused by the measuring gauge MKS baratron and PPI 7050i. This correction is necessary, to ensure that the obtained values are correct, the correction is done by the corrections parameters obtained by the calibrations certificate. The ratio corrected to *196,879*.

Now this ratio will be corrected for the different cases as mentioned in chapter 5. The ratio after correction for temperature drift and using the correction equation 5.4 leads to *196,867*, next step is to correct for temperature gradient by equation 5.5 which leads to *197,003*, thereafter next correction to make is the correction for deviation from ideal gas law by equation 5.7 and that leads to *197,050* and finely the last correction to make is the correction for the gauge and connection volumes by the subsection 5.2.4 and the ratio ends up at *197,043*. Summering them all under the table 6.1 below, we get.

Table 6.1: R3 by gas accumulation and the corrections.

Old ratio	197,081
Uncorrected ratio	195,565
Corrected for measuring uncertainty	196,879
Corrected for temperature drift	196,867
Corrected for temperature gradient	197,003
Corrected for deviation from ideal gas law	197,050
Corrected for Volume and gauge corr.	197,043

The SRG measurement of R3 consists 20 cycles (the whole measuring data sheet is available under appendix A.6), the table 6.2 below shows the obtained result for R3 and its corrections. The only correction made on the SRG mean value is the correction for temperature gradient by the equation 5.9 as mentioned earlier.

Table 6.2: R3 by SRG and the corrections.

Old ratio	197,081
Uncorrected ratio	193,066
Corrected for temperature gradient	193,086

By following the same procedure above and the correction steps, the remaining ratios can be derived. The tables 6.3 and 6.4 below shows all the remaining ratios by their mean corrected values and their standard deviation.

Table 6.3: All the gas accumulation ratios presented by their mean corrected values and the standard deviation.

Ratio	Old value	Gas accumulation	Standard deviation
R1	196,714	196,478	0,030
R2	197,607	197,630	0,046
R3	197,081	197,043	0,030
R4n	295,766	297,775	0,160
R4d	99,131	99,948	0,028

Table 6.4: All the SRG ratios presented by their mean corrected values and the standard deviation.

Ratio	Old value	SRG	Standard deviation
R1	196,714	185,702	0,403
R2	197,607	193,976	0,296
R3	197,081	193,046	0,051
R4n	295,766	293,450	0,023
R4d	99,131	98,687	0,065

6.2 Discussion and Outlook

The system is designed to have the ratios of 100, 200 and 300 as a starting point by NPL. According to the results above, the gas accumulation method yielded a more stable values during characterization versus SRG. This has to do with the meter's measuring range and the structure of the method, which means that the system is not evacuated during the characterization sequence. However, this means that the system doesn't need to be emptied in between the expansions. On the other hand, SRG requires an such action, which causes limitations. Firstly, a long evacuate time between the expansions for pumping out, secondly the meter is very sensitive at such low pressures, which again requires long time to be stabilized. With that said, the obtained ratio by the gas accumulation is in agreement with the system's constructed ratio, and even with old one carried out by RISE 2008. This is not so surprising, since the old characterization routine RISE had used the gas accumulation method.

To reflect over the result, the diagram below 6.1 shows the ratio R3 obtained by the methods. Clearly, the corrected value of the gas accumulation method converge towards the old value achieved by RISE 2008. Worth to mention, according the calibration certificate for the MKS Baratron, the meter will measure strictly linear after 25 mbar, in order to eliminate this limitation, the first six measuring data excluded from the plot as seen in the diagram.

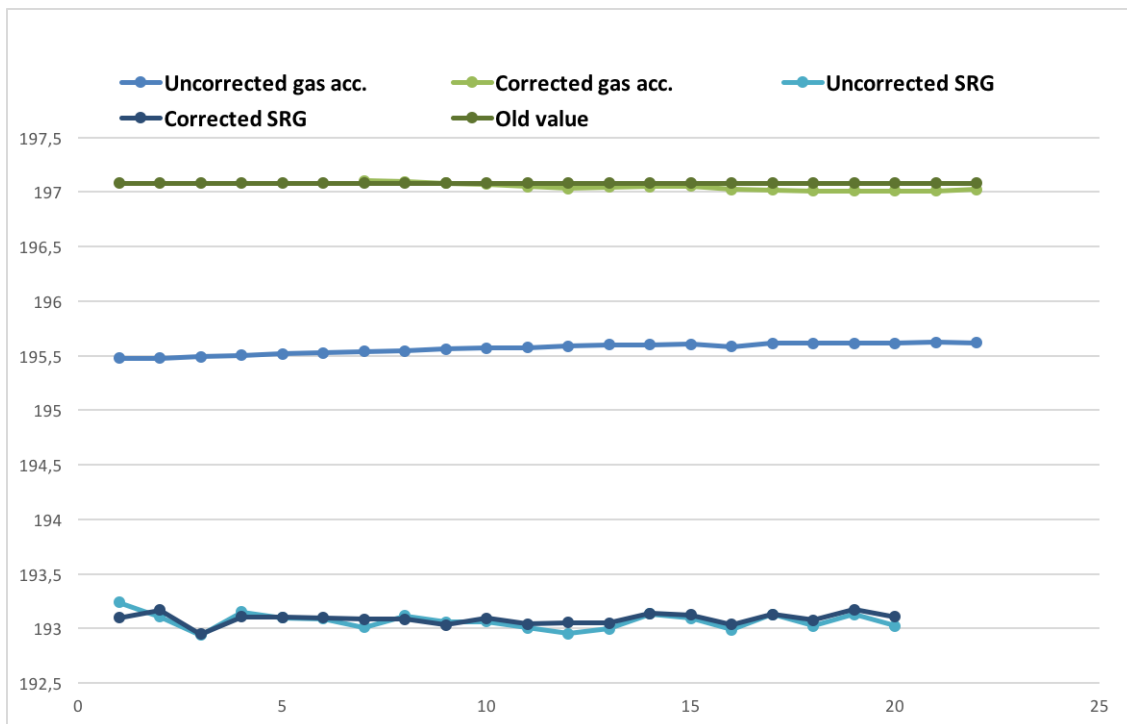


Figure 6.1: Gas accumulation and SRG diagram of the obtained data during characterization process for R3.

The reason that SRG didn't deliver the expected result is due to the system's residual drag pressure in the volumes, especially the R1 and R2, since the volumes V2 and V4

couldn't be baked because there wasn't equipment for that purpose. This contributed measurement errors caused by the residual drag, it clearly can be seen on the table 6.4 above, where the first two ratios have a significantly higher standard deviation than the remaining three, since that part of the system could be baked before measurement. As mentioned at the beginning of the report, the SRG method is time-consuming, this agreed well, because it took almost 15 hours to characterize the R3 with SRG method and approximately 2 hours with the gas accumulation. This is without taking in account the time for the baking process. To characterize the entire system will take a week with the SRG method.

The project offers several challenges which must be addressed before the characterization can be performed. During the characterization process several obstacles appeared. The biggest issue concerns the control valves. All valves in SEA5 are controlled by a Siemens S7 PLC. Certain critical valves are implemented in the PLC software to be 'soft closed'; this means that there are two pneumatic valves controllers connected in series. The first one restricts the pneumatic pressure which causes the valve to close slowly, while the second one actuates without restriction ten seconds after the first to make sure the valve is fully closed. This is necessary to get an even gas distribution on either side of the valve and a correct initial pressure reading before an expansion. In the original configuration the system used this soft close technique on valves A, C, E, G and I, but in order to reliably implement the SRG method, valves B, D, F, H and J would also need the same setup. This requires a reconfiguration of the PLC and more relays need to be added to the system's relay board, which requires quite a bit of practical work.

Another issue which has to be resolved relates to the bake-out mechanism of the system. As a safety measure, this procedure requires continuous monitoring of the residual gas pressure in system volumes V6 and V9, see figure 4.1. In case of a system failure and sudden increase in pressure the bake-out procedure would automatically turn off. Each volume is equipped with a ionization gauge for this purpose, but they are in a non-operational state. This also require hands-on work and troubleshooting to fix.

6.3 Conclusion

In conclusion, according to the achieved values, both their accuracy and ease of use, the gas accumulation gave a significantly better ratios than the SRG method. Based on that, RISE should characterized the SEA5 by this method. However, the reason that R4n and R4d ratios showed slightly higher ratios than the old ones were due to the pressure measurement before expansion, namely, the initial pressure measurement wasn't stable as in the previous cases. It can be especially seen on R4n, where its standard deviation value over the entire measurement sequence is higher compared to the rest in table 6.3. This limitation can be overcome by a second Baratron at V6. Measuring the initial pressure at V6 instead of Ruska 7050i, which will lead to significantly more stable initial pressure reading.

The SRG method can be improved if the first part of the system (V1-V4) could be baked to achieve lower residual pressures in the volumes, and increasing the pumping time in between the expansions to stabilize the measuring state of the meter.

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Appendices

Appendix A

Appendix

A.1 Gas accumulation data for R1

A.2 SRG data for R1

A.3 Gas accumulation data for R2

A.4 SRG data for R2

A.5 Gas accumulation data for R3

A.6 SRG data for R3

A.7 Gas accumulation data for R4n

A.8 SRG data for R4n

A.9 Gas accumulation data for R4d

A.10 SRG data for R4d

Table A.1: Gas accumulation data for R1, uncorrected

Iteration, n	Init pressure	Press. Before	Temp. Tv1	Temp. Tv2	Press. after	Ratio
1	998,502	-0,001	23,25	23	5,112	196,439
2	999,392	5,113	23,28	22,96	10,204	196,461
3	999,695	10,206	23,29	23	15,272	196,473
4	999,868	15,274	23,29	22,98	20,314	196,468
5	999,936	20,316	23,26	22,98	25,330	196,483
6	999,971	25,334	23,36	22,97	30,321	196,411
7	999,929	30,325	23,29	22,96	35,285	196,443
8	999,960	35,290	23,3	22,97	40,224	196,447
9	999,987	40,229	23,28	22,99	45,137	196,473
10	999,963	45,143	23,3	22,94	50,024	196,444
11	1000,196	50,032	23,3	22,96	54,888	196,494
12	1000,253	54,896	23,32	22,97	59,727	196,497
13	1000,297	59,735	23,32	22,96	64,541	196,504
14	1000,242	64,551	23,36	22,94	69,329	196,466
15	1000,063	69,338	23,33	22,95	74,091	196,460
16	999,937	74,100	23,31	22,97	78,829	196,463
17	999,907	78,840	23,3	22,94	83,542	196,461
18	999,922	83,554	23,29	22,94	88,233	196,475
19	1000,010	88,245	23,28	22,96	92,901	196,508
20	1000,170	92,913	23,31	22,95	97,544	196,520
21	1000,252	97,558	23,31	22,95	102,167	196,533
22	1000,236	102,180	23,31	22,93	106,766	196,522
23	1000,173	106,781	23,33	22,94	111,343	196,493
24	1000,155	111,360	23,32	22,93	115,899	196,485
25	1000,033	115,915	23,3	22,94	120,434	196,464

Table A.2: SRG data for R1, uncorrected

Iteration, n	DCR1	Temp. Tv1	DCR2	Temp. Tv2	Ratio	Offset before
1	2,678E-04	23,16	2,387E-06	22,91	112,1146	9,390E-07
2	2,683E-04	23,33	2,377E-06	22,88	112,7167	9,406E-07
3	2,683E-04	23,26	2,375E-06	22,83	112,8045	9,405E-07
4	2,684E-04	23,25	2,376E-06	22,82	112,7974	9,400E-07
5	2,685E-04	23,2	2,374E-06	22,87	112,9726	9,401E-07
6	2,685E-04	23,17	2,377E-06	22,91	112,8615	9,401E-07
7	2,686E-04	23,18	2,379E-06	22,87	112,787	9,401E-07
8	2,687E-04	23,1	2,378E-06	22,85	112,8919	9,396E-07
9	2,685E-04	23,16	2,378E-06	22,91	112,8079	9,404E-07
10	2,685E-04	23,14	2,378E-06	22,97	112,8563	9,399E-07
11	2,685E-04	23,13	2,379E-06	23,06	112,8322	9,396E-07
12	2,686E-04	23,13	2,381E-06	23,06	112,79	9,396E-07
13	2,686E-04	23,12	2,380E-06	23,03	112,8055	9,400E-07
14	2,685E-04	23,15	2,380E-06	22,96	112,7601	9,394E-07
15	2,685E-04	23,11	2,381E-06	22,95	112,7294	9,397E-07
16	2,685E-04	23,15	2,380E-06	22,94	112,712	9,400E-07
17	2,684E-04	23,14	2,380E-06	22,92	112,6872	9,396E-07
18	2,684E-04	23,2	2,381E-06	22,89	112,6482	9,394E-07
19	2,684E-04	23,23	2,380E-06	22,91	112,625	9,390E-07
20	2,683E-04	23,26	2,382E-06	23	112,5549	9,398E-07
21	2,678E-04	23,16	2,387E-06	22,91	112,1146	9,390E-07
22	2,683E-04	23,33	2,377E-06	22,88	112,7167	9,406E-07
23	2,683E-04	23,26	2,375E-06	22,83	112,8045	9,405E-07
24	2,684E-04	23,25	2,376E-06	22,82	112,7974	9,400E-07
25	2,685E-04	23,2	2,374E-06	22,87	112,9726	9,401E-07
26	2,685E-04	23,17	2,377E-06	22,91	112,8615	9,401E-07
27	2,686E-04	23,18	2,379E-06	22,87	112,787	9,401E-07
28	2,687E-04	23,1	2,378E-06	22,85	112,8919	9,396E-07
29	2,685E-04	23,16	2,378E-06	22,91	112,8079	9,404E-07
30	2,685E-04	23,14	2,378E-06	22,97	112,8563	9,399E-07
31	2,685E-04	23,13	2,379E-06	23,06	112,8322	9,396E-07
32	2,686E-04	23,13	2,381E-06	23,06	112,79	9,396E-07
33	2,686E-04	23,12	2,380E-06	23,03	112,8055	9,400E-07
34	2,685E-04	23,15	2,380E-06	22,96	112,7601	9,394E-07
35	2,685E-04	23,11	2,381E-06	22,95	112,7294	9,397E-07
36	2,685E-04	23,15	2,380E-06	22,94	112,712	9,400E-07
37	2,684E-04	23,14	2,380E-06	22,92	112,6872	9,396E-07
38	2,684E-04	23,2	2,381E-06	22,89	112,6482	9,394E-07
39	2,684E-04	23,23	2,380E-06	22,91	112,625	9,390E-07
40	2,683E-04	23,26	2,382E-06	23	112,5549	9,398E-07

Table A.3: Gas accumulation data for R2, uncorrected

Iteration, n	Init pressure	Press. before	Temp. Tv3	Temp. Tv4	Press. after	Ratio
1	999,672	-1,229E-03	22,43	22,44	5,093E+00	196,276
2	999,526	5,094E+00	22,46	22,45	1,016E+01	196,244
3	999,547	1,016E+01	22,47	22,45	1,520E+01	196,248
4	999,551	1,520E+01	22,48	22,45	2,022E+01	196,253
5	999,544	2,022E+01	22,48	22,42	2,521E+01	196,258
6	999,592	2,521E+01	22,47	22,42	3,017E+01	196,271
7	999,586	3,018E+01	22,47	22,41	3,511E+01	196,275
8	999,627	3,511E+01	22,47	22,42	4,002E+01	196,286
9	999,627	4,003E+01	22,49	22,41	4,491E+01	196,291
10	999,637	4,492E+01	22,5	22,43	4,977E+01	196,299
11	999,601	4,978E+01	22,49	22,41	5,461E+01	196,302
12	999,610	5,462E+01	22,49	22,41	5,942E+01	196,312
13	999,706	5,943E+01	22,48	22,39	6,421E+01	196,339
14	999,665	6,422E+01	22,47	22,38	6,897E+01	196,336
15	999,687	6,898E+01	22,47	22,4	7,371E+01	196,344
16	999,626	7,372E+01	22,47	22,38	7,842E+01	196,338
17	999,610	7,843E+01	22,48	22,4	8,311E+01	196,338
18	999,660	8,313E+01	22,47	22,39	8,778E+01	196,351
19	999,634	8,779E+01	22,47	22,4	9,242E+01	196,345
20	999,638	9,244E+01	22,47	22,41	9,705E+01	196,345
21	999,627	9,706E+01	22,48	22,38	1,016E+02	196,342
22	999,635	1,017E+02	22,48	22,37	1,062E+02	196,342
23	999,695	1,062E+02	22,47	22,38	1,108E+02	196,348
24	999,650	1,108E+02	22,49	22,34	1,153E+02	196,317
25	999,663	1,153E+02	22,5	22,37	1,198E+02	196,325

Table A.4: SRG data for R2, uncorrected

Iteration, n	DCR1	Temp. Tv3	DCR2	Temp. Tv4	Ratio	Offset before
1	2,263E-04	22,8	2,326E-06	23,14	194,835	1,164E-06
2	2,318E-04	22,71	2,356E-06	22,73	194,504	1,167E-06
3	2,318E-04	22,71	2,357E-06	22,63	194,422	1,168E-06
4	2,323E-04	22,63	2,359E-06	22,5	194,351	1,169E-06
5	2,323E-04	22,6	2,360E-06	22,49	194,269	1,169E-06
6	2,319E-04	22,6	2,358E-06	22,48	194,236	1,170E-06
7	2,320E-04	22,58	2,359E-06	22,46	194,198	1,170E-06
8	2,324E-04	22,62	2,361E-06	22,54	194,117	1,171E-06
9	2,319E-04	22,63	2,359E-06	22,49	194,170	1,171E-06
10	2,319E-04	22,58	2,358E-06	22,44	194,211	1,171E-06
11	2,322E-04	22,54	2,361E-06	22,39	194,097	1,171E-06
12	2,316E-04	22,5	2,358E-06	22,37	194,115	1,171E-06
13	2,314E-04	22,48	2,357E-06	22,35	194,094	1,171E-06
14	2,325E-04	22,46	2,363E-06	22,33	194,006	1,171E-06
15	2,331E-04	22,44	2,366E-06	22,32	194,045	1,171E-06
16	2,313E-04	22,44	2,358E-06	22,31	193,853	1,171E-06
17	2,314E-04	22,43	2,357E-06	22,3	193,995	1,171E-06
18	2,315E-04	22,42	2,358E-06	22,29	193,939	1,171E-06
19	2,317E-04	22,4	2,360E-06	22,27	193,832	1,171E-06
20	2,318E-04	22,4	2,360E-06	22,27	193,875	1,171E-06
21	2,324E-04	22,42	2,363E-06	22,3	193,840	1,171E-06
22	2,311E-04	22,45	2,356E-06	22,36	193,898	1,171E-06
23	2,312E-04	22,53	2,358E-06	22,43	193,777	1,171E-06
24	2,318E-04	22,62	2,361E-06	22,49	193,776	1,172E-06
25	2,316E-04	22,66	2,360E-06	22,59	193,713	1,172E-06
26	2,315E-04	22,63	2,359E-06	22,59	193,768	1,172E-06
27	2,320E-04	22,61	2,362E-06	22,55	193,701	1,171E-06
28	2,317E-04	22,64	2,359E-06	22,54	193,887	1,170E-06
29	2,320E-04	22,66	2,362E-06	22,55	193,750	1,171E-06
30	2,313E-04	22,66	2,357E-06	22,5	193,878	1,170E-06

Table A.5: Gas accumulation data for R3, uncorrected

Iteration, n	Init pressure	Press. before	Temp. Tv5	Temp. Tv6	Press. after	Ratio
1	999,880	0,000	22,490000	23,120000	5,115	195,476
2	999,709	5,115	22,500000	23,040000	10,202	195,475
3	999,702	10,203	22,470000	22,980000	15,263	195,490
4	999,701	15,265	22,470000	22,920000	20,298	195,502
5	999,740	20,300	22,460000	22,880000	25,306	195,518
6	999,726	25,309	22,440000	22,830000	30,289	195,526
7	999,753	30,292	22,450000	22,790000	35,245	195,540
8	999,726	35,249	22,430000	22,760000	40,176	195,545
9	999,761	40,180	22,420000	22,720000	45,081	195,559
10	999,775	45,086	22,420000	22,700000	49,961	195,570
11	999,744	49,967	22,420000	22,670000	54,815	195,573
12	999,748	54,822	22,450000	22,660000	59,644	195,588
13	999,788	59,651	22,430000	22,640000	64,448	195,598
14	999,782	64,457	22,420000	22,630000	69,229	195,600
15	999,774	69,238	22,410000	22,620000	73,984	195,604
16	999,782	73,995	22,440000	22,600000	78,716	195,584
17	999,781	78,727	22,450000	22,600000	83,424	195,611
18	999,780	83,435	22,460000	22,590000	88,108	195,613
19	999,771	88,119	22,450000	22,580000	92,767	195,614
20	999,752	92,778	22,440000	22,570000	97,402	195,614
21	999,819	97,416	22,460000	22,570000	102,017	195,622
22	999,812	102,030	22,440000	22,570000	106,608	195,619

Table A.6: SRG data for R3, uncorrected

Iteration, n	DCR1	Temp. Tv5	DCR2	Temp. Tv6	Ratio	Offset before
1	2,460E-04	22,52	2,272E-06	22,74	193,2371	1,004E-06
2	2,461E-04	22,51	2,272E-06	22,62	193,1047	1,002E-06
3	2,461E-04	22,52	2,273E-06	22,57	192,938	1,002E-06
4	2,462E-04	22,51	2,272E-06	22,55	193,1517	1,003E-06
5	2,462E-04	22,54	2,272E-06	22,54	193,0985	1,002E-06
6	2,461E-04	22,53	2,272E-06	22,53	193,0901	1,002E-06
7	2,461E-04	22,52	2,272E-06	22,54	193,0071	1,002E-06
8	2,461E-04	22,51	2,272E-06	22,53	193,1124	1,002E-06
9	2,461E-04	22,53	2,272E-06	22,53	193,0584	1,002E-06
10	2,462E-04	22,51	2,272E-06	22,53	193,0609	1,002E-06
11	2,463E-04	22,47	2,273E-06	22,52	193,0022	1,002E-06
12	2,463E-04	22,45	2,273E-06	22,51	192,9497	1,002E-06
13	2,462E-04	22,46	2,273E-06	22,51	192,9944	1,002E-06
14	2,462E-04	22,5	2,272E-06	22,5	193,13	1,002E-06
15	2,462E-04	22,48	2,272E-06	22,5	193,0918	1,002E-06
16	2,462E-04	22,49	2,272E-06	22,49	192,985	1,002E-06
17	2,462E-04	22,49	2,272E-06	22,48	193,1295	1,002E-06
18	2,462E-04	22,51	2,272E-06	22,49	193,0228	1,002E-06
19	2,462E-04	22,49	2,271E-06	22,48	193,1297	1,002E-06
20	2,462E-04	22,49	2,272E-06	22,49	193,0235	1,002E-06

Table A.7: Gas accumulation data for R4n, corrected

Iteration, n	Init pressure	Press. Before	Temp. Tv7	Temp. Tv9	Press. after	Ratio
1	999,818	8,989E-04	22,06	22,043	3,379E+00	297,885
2	999,862	3,379E+00	22,1	22,063	6,745E+00	297,882
3	999,870	6,746E+00	22,15	22,087	1,010E+01	297,852
4	999,901	1,010E+01	22,17	22,113	1,344E+01	297,859
5	999,866	1,345E+01	22,19	22,123	1,678E+01	297,842
6	999,879	1,678E+01	22,23	22,140	2,010E+01	297,817
7	999,888	2,010E+01	22,25	22,160	2,341E+01	297,814
8	999,853	2,341E+01	22,27	22,187	2,671E+01	297,800
9	999,887	2,671E+01	22,28	22,193	3,000E+01	297,807
10	999,885	3,000E+01	22,31	22,203	3,327E+01	297,785
11	999,918	3,327E+01	22,31	22,220	3,654E+01	297,808
12	999,903	3,654E+01	22,33	22,230	3,979E+01	297,793
13	999,858	3,979E+01	22,34	22,253	4,304E+01	297,784
14	999,907	4,304E+01	22,35	22,260	4,627E+01	297,798
15	999,897	4,627E+01	22,36	22,270	4,949E+01	297,793
16	999,903	4,949E+01	22,38	22,280	5,270E+01	297,785
17	999,923	5,270E+01	22,38	22,303	5,590E+01	297,807
18	999,890	5,590E+01	22,39	22,307	5,909E+01	297,793
19	999,924	5,909E+01	22,41	22,333	6,227E+01	297,801
20	999,891	6,227E+01	22,41	22,330	6,544E+01	297,793
21	999,873	6,544E+01	22,44	22,343	6,859E+01	297,764
22	999,885	6,860E+01	22,45	22,353	7,174E+01	297,765
23	999,886	7,174E+01	22,45	22,377	7,488E+01	297,778
24	999,947	7,488E+01	22,46	22,373	7,800E+01	297,789
25	999,842	7,801E+01	22,46	22,393	8,112E+01	297,768
26	999,987	8,112E+01	22,48	22,403	8,422E+01	297,798
27	999,881	8,423E+01	22,5	22,420	8,732E+01	297,751
28	999,839	8,732E+01	22,49	22,407	9,040E+01	297,747
29	999,896	9,040E+01	22,49	22,417	9,347E+01	297,772
30	999,908	9,348E+01	22,5	22,420	9,654E+01	297,769
31	999,924	9,654E+01	22,5	22,423	9,959E+01	297,776
32	999,874	9,959E+01	22,52	22,420	1,026E+02	297,740
33	999,921	1,026E+02	22,52	22,430	1,057E+02	297,757
34	999,909	1,057E+02	22,51	22,430	1,087E+02	297,761
35	999,909	1,087E+02	22,52	22,427	1,117E+02	297,748
36	999,895	1,117E+02	22,52	22,423	1,147E+02	297,737
37	999,889	1,147E+02	22,51	22,437	1,177E+02	297,745
38	999,864	1,177E+02	22,52	22,440	1,207E+02	297,719

Table A.8: SRG data for R4n, uncorrected

Iteration, n	DCR1	Temp. Tv7	DCR2	Temp. Tv9	Ratio	Offset before
1	2,390E-04	22,22	1,812E-06	22,267	293,465	1,001E-06
2	2,390E-04	22,22	1,812E-06	22,270	293,524	1,001E-06
3	2,391E-04	22,23	1,812E-06	22,267	293,623	1,001E-06
4	2,391E-04	22,22	1,814E-06	22,263	292,638	1,000E-06
5	2,391E-04	22,22	1,813E-06	22,257	293,225	1,001E-06
6	2,391E-04	22,22	1,812E-06	22,247	293,622	1,001E-06
7	2,391E-04	22,23	1,813E-06	22,263	293,190	1,000E-06
8	2,391E-04	22,23	1,813E-06	22,260	293,383	1,001E-06
9	2,391E-04	22,22	1,812E-06	22,260	293,456	1,000E-06
10	2,391E-04	22,21	1,812E-06	22,260	293,420	1,000E-06
11	2,391E-04	22,21	1,812E-06	22,250	293,394	1,001E-06
12	2,392E-04	22,22	1,812E-06	22,263	293,408	1,001E-06
13	2,390E-04	22,25	1,812E-06	22,283	293,561	1,001E-06
14	2,390E-04	22,26	1,812E-06	22,297	293,427	1,001E-06
15	2,390E-04	22,26	1,812E-06	22,297	293,524	1,001E-06
16	2,390E-04	22,27	1,812E-06	22,300	293,356	1,001E-06
17	2,390E-04	22,26	1,812E-06	22,297	293,645	1,001E-06
18	2,390E-04	22,26	1,812E-06	22,293	293,331	1,001E-06
19	2,391E-04	22,25	1,812E-06	22,287	293,562	1,001E-06
20	2,391E-04	22,24	1,812E-06	22,283	293,419	1,001E-06
21	2,391E-04	22,23	1,812E-06	22,263	293,467	1,001E-06
22	2,391E-04	22,24	1,812E-06	22,273	293,177	1,000E-06
23	2,391E-04	22,24	1,812E-06	22,303	293,671	1,001E-06
24	2,391E-04	22,23	1,812E-06	22,277	293,538	1,001E-06
25	2,391E-04	22,24	1,813E-06	22,287	293,236	1,001E-06

Table A.9: Gas accumulation data for R4d, corrected

Iteration, n	Init pressure	Press. before	Temp. Tv8	Temp. Tv9	Press. after	Ratio
1	499,883	8,408E-04	22,37	22,540	5,041E+00	99,869
2	499,891	5,041E+00	22,41	22,530	1,003E+01	99,863
3	499,882	1,003E+01	22,41	22,517	1,497E+01	99,867
4	499,882	1,497E+01	22,4	22,503	1,986E+01	99,876
5	499,887	1,986E+01	22,4	22,497	2,470E+01	99,882
6	499,899	2,469E+01	22,4	22,473	2,948E+01	99,888
7	499,886	2,948E+01	22,39	22,460	3,423E+01	99,894
8	499,897	3,422E+01	22,37	22,457	3,892E+01	99,910
9	499,892	3,892E+01	22,38	22,433	4,356E+01	99,909
10	499,893	4,356E+01	22,36	22,420	4,816E+01	99,921
11	499,899	4,816E+01	22,35	22,413	5,271E+01	99,932
12	499,899	5,271E+01	22,35	22,403	5,722E+01	99,937
13	499,898	5,722E+01	22,34	22,400	6,168E+01	99,946
14	499,897	6,168E+01	22,34	22,387	6,609E+01	99,950
15	499,898	6,609E+01	22,33	22,380	7,046E+01	99,959
16	499,899	7,046E+01	22,33	22,373	7,479E+01	99,963
17	499,891	7,479E+01	22,33	22,377	7,907E+01	99,966
18	499,890	7,907E+01	22,33	22,377	8,331E+01	99,969
19	499,905	8,332E+01	22,34	22,377	8,751E+01	99,971
20	499,902	8,751E+01	22,34	22,373	9,167E+01	99,972
21	499,901	9,167E+01	22,35	22,377	9,579E+01	99,971
22	499,901	9,579E+01	22,33	22,373	9,986E+01	99,978
23	499,902	9,986E+01	22,34	22,373	1,039E+02	99,975
24	499,895	1,039E+02	22,34	22,373	1,079E+02	99,973
25	499,899	1,079E+02	22,33	22,373	1,118E+02	99,975

Table A.10: SRG data for R4d, uncorrected

Iteration, n	DCR1	Temp. Tv8	DCR2	Temp. Tv9	Ratio	Offset before
1	2,381E-04	22,41	3,406E-06	22,587	98,533	1,000E-06
2	2,380E-04	22,36	3,405E-06	22,557	98,606	1,001E-06
3	2,380E-04	22,35	3,404E-06	22,530	98,634	1,001E-06
4	2,379E-04	22,33	3,404E-06	22,520	98,610	1,001E-06
5	2,379E-04	22,31	3,402E-06	22,497	98,642	1,001E-06
6	2,379E-04	22,29	3,403E-06	22,483	98,610	1,000E-06
7	2,379E-04	22,28	3,410E-06	22,467	98,327	1,001E-06
8	2,379E-04	22,27	3,403E-06	22,453	98,626	1,001E-06
9	2,379E-04	22,29	3,402E-06	22,457	98,642	1,001E-06
10	2,379E-04	22,26	3,402E-06	22,440	98,626	1,000E-06
11	2,379E-04	22,31	3,402E-06	22,477	98,626	1,000E-06
12	2,379E-04	22,34	3,402E-06	22,480	98,658	1,001E-06
13	2,379E-04	22,34	3,401E-06	22,490	98,671	1,001E-06
14	2,379E-04	22,39	3,401E-06	22,463	98,659	1,001E-06
15	2,379E-04	22,42	3,401E-06	22,450	98,683	1,001E-06
16	2,379E-04	22,4	3,401E-06	22,453	98,691	1,001E-06
17	2,379E-04	22,41	3,401E-06	22,430	98,704	1,001E-06
18	2,379E-04	22,42	3,400E-06	22,440	98,732	1,001E-06
19	2,379E-04	22,42	3,401E-06	22,443	98,695	1,001E-06
20	2,379E-04	22,38	3,402E-06	22,463	98,699	1,001E-06
21	2,379E-04	22,37	3,402E-06	22,463	98,683	1,001E-06
22	2,380E-04	22,36	3,403E-06	22,467	98,651	1,001E-06
23	2,380E-04	22,34	3,402E-06	22,477	98,688	1,001E-06
24	2,379E-04	22,34	3,401E-06	22,443	98,704	1,001E-06
25	2,379E-04	22,32	3,401E-06	22,453	98,716	1,001E-06

Appendix B

Appendix

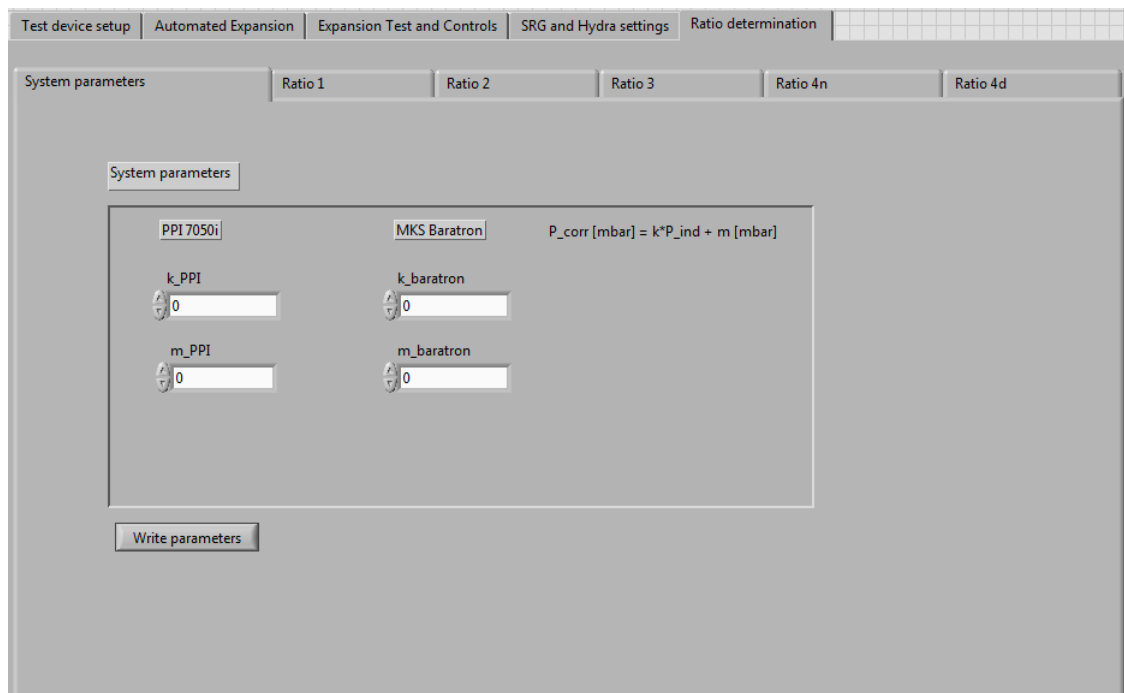


Figure B.1: System parameter figure of the LabView interface, presented by a tab control.

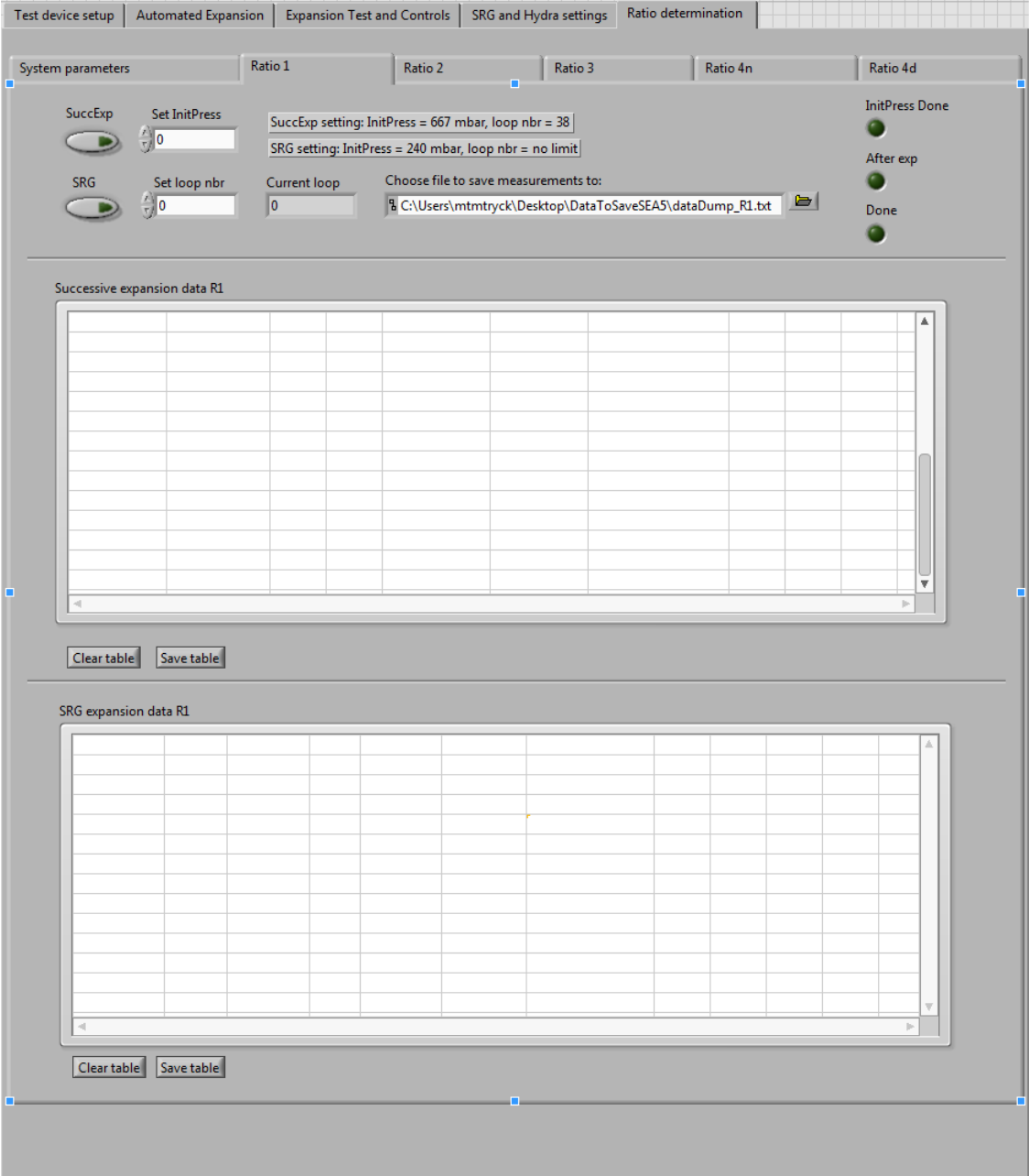
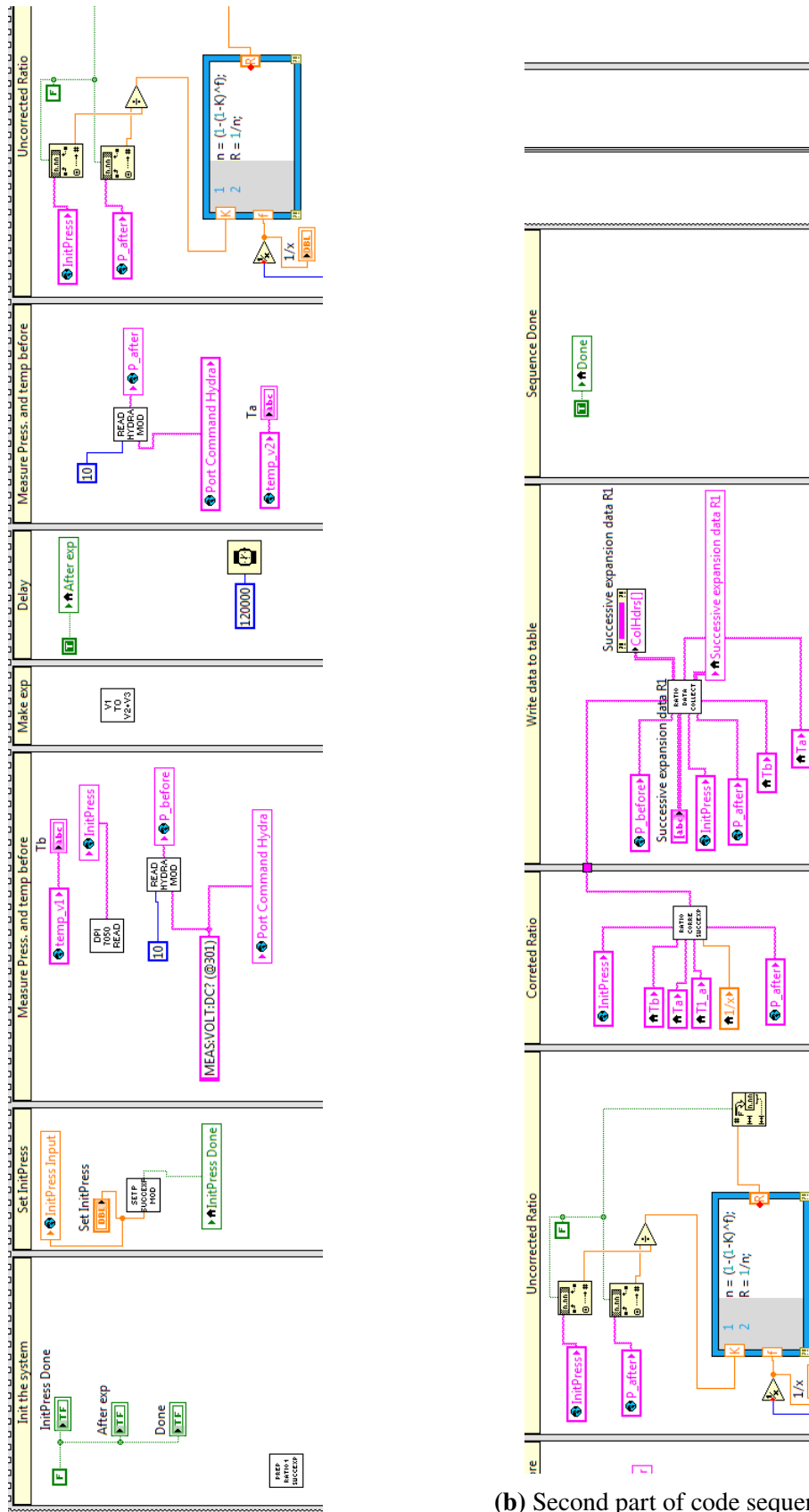


Figure B.2: Measurement and collection tab figure of the Lab-View interface for R1 ratio



(a) First part of code sequence, starts from bottom

(b) Second part of code sequence.

Figure B.3: Code sequence for R1 by gas accumulation.