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# Characterisation of a System that Measures Gas Samples Using a Mass Flow Meter

## **Abstract**

### **Characterisation of a system that measures gas samples using a mass flow meter**

#### **Preparatory work for a gravimetrical in situ calibration procedure**

A calibration method has been prepared for a new constructed gas cylinder filling station based on a mass flow measurement technique. The construction is located at AGA Gas AB special gas factory in Lidingö, Stockholm, Sweden. Several investigations have been performed on the system and different parameters such as pressure, gas density, and amount of added gas has been analysed. The calibration method gave a total measurement uncertainty of  $\pm 281$  mg for Nitrogen,  $\pm 368$  mg for Argon and  $\pm 162$  mg for Helium.

The principal results of the investigations are:

1. The mass flow meter error is not proportional to the amount of filled gas.
2. The gas filling system is almost always filling over the nominal value, which bothering leads to deviations from the nominal concentration in a gas mixture.
3. A low flow rate causes larger mass flow meter errors.
4. The deviation from the nominally set value increases with line (input) pressure.
5. The mass flow meter error depends on the gas density.

**Key words:** Mass flow meter, Mass flow measurement, Calibration, Coriolis, AGA, Gas, Pressure, Weighing.

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# 1 Introduction

AGA is one of the world's leading gas companies. The group produces and sells industrial and medical gases in some 40 countries in Europe, the U.S. and Latin America. These operations employ over 10000 people. The Gas Accumulator Company, later to be called AGA was founded in 1904.

AGA's key products are oxygen, nitrogen and argon. They account for half of sales revenues. These gases are produced from air, mostly by means of distillation at very low, cryogenic temperatures. The fuel gases acetylene and propane, as well as hydrogen and carbon dioxide, are other important products. After oxygen, mixtures of nitrous oxide in nitrogen is the most important medical gas.

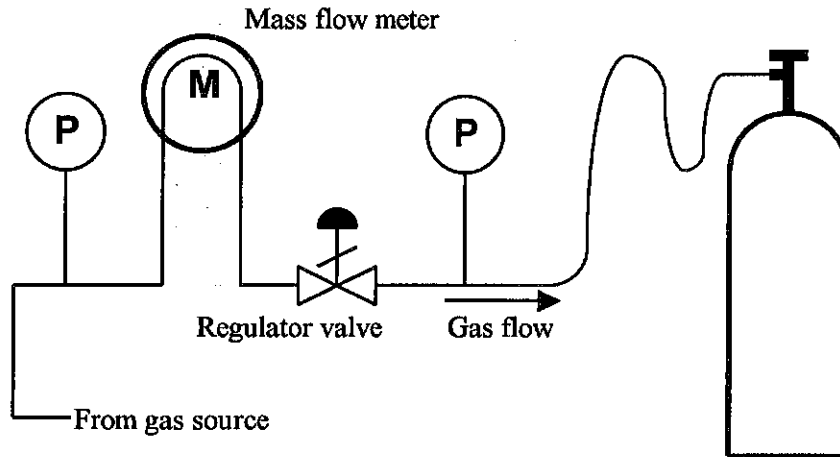
Gases are used in most manufacturing processes. The gas is delivered in tanks or gas cylinders or produced on-site. Quantities vary from thousands of tons of oxygen and nitrogen to small cylinders containing precise mixtures of speciality gases. Industrial gases are used in practically all industries and in most manufacturing processes. Medical gases are consumed in hospitals and clinics, and to an increasing extent within home therapy. Speciality gases, in the form of both high purity gases and special mixtures are used in laboratories, in semiconductor manufacturing, for process control and for the calibration of measuring instruments. These products meet high purity requirements and demands on accuracy and precision.

## 2 Background

The automated gas filling system at the speciality gas factory in Lidingö, Stockholm had to be renewed. The old system was not working satisfyingly and it was a weak link in the production line. The project SPEGAL (SPEcial GAS Lidingö) was formed to utilise new flow measurement techniques in order to fulfil customer's demands on high accuracy and repeatability concerning the mixture of gas components in gas cylinders. The project group decided to use the mass flow measurement technique (coriolis meter), which is a rather new technique, especially when measuring gases under high pressure. One part of the SPEGAL project was to develop and produce a gas-filling panel for separate gas mixtures. Each of these mixtures is occasional and few of them are produced in a large number (more than 25 per year). This panel is called the One-piece panel or the SELMA-panel. AGA aimed to calibrate the SELMA-panel in order to reach traceability for the different gas masses to the Swedish national kilogram. Failure and uncertainty analyses together with a suitable calibration method design were the first steps towards the calibration. For this purpose it was necessary to investigate the influence of some process parameters, such as pressure, gas flow, filling time, gas density etc. An expectation regarding the SELMA-panel was to reach an uncertainty less than 1 molar % at small amounts such as 20 g added gas. Peter Lau at the Swedish National Testing & Research Institute was consulted for this work and in agreement with AGA it was suggested to carry out the task as a bachelor thesis project for some ambitious engineering student.

### 3 The SELMA panel

The SELMA panel is constructed with seven gas entrances and can automatically produce the desired gas mixture. Three of these entrances are intended for the in house gas line system, which supports the factory with often-used gases like Helium, Argon, Carbon dioxide, Nitrogen and Oxygen. The other four entrances can be connected to different gas cylinders that either contain a pure gas or a mixture. A mixture is often used in order to reach a low concentration level. The design in detail is shown in appendix A. Here is a simplified version:



Figur 1.

The SELMA panel is equipped with a mass flow meter from Micro Motion Inc. The sensor and transmitter models are not named in this public report. This mass flow meter is designed for high-pressure liquids and gases and has a maximum flowing range of 55 kg/h (15 g/s). The resolution is 0,001 g/s and 0,01 g for total summations. A regulator valve from Kämmer Ventile GMBH, model 20300 controls the gas flow through the system. A PLC system with an input signal from the mass flow meter is used to control the degree of opening in the regulator valve.

## 4 Investigation of the balance characteristics

The balance is used as a tool when comparing different masses. In this case a gas cylinder which is weighed before and after filling. A balance of manufacturer Mettler-Toledo Multirange ID5 was used during the entire work. The balance is mounted in the ceiling and the gas cylinders were hanged in a hook beneath the balance (see Figure 2). In order to investigate the balance behaviour and characteristics a few tests were made.

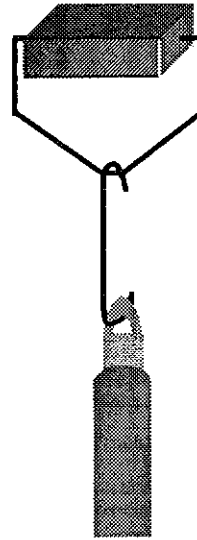


Figure 2.

### 4.1 The balance non-linearity

Knowing that no balance is perfectly linear, a calibration of the balance had to be performed. Since two different gas cylinders with different masses were used in the tests (these are described in chapter 6), the balance was calibrated within two different scale ranges. Several combinations of weights were used during the calibration. These weights had been calibrated by SP - The Swedish National Testing & Research Institute and are traceable to the Swedish national kilogram. All weights are tabulated in table 1.

Weight	mass	Uncertainty at $k=2$ .
	[kg]	[mg]
S10	9,999998	15
A10	10,00006	50
S5	5,000003	7
A5	5,000036	25
A2	2,000013	10
A1	1,000009	5
A0,5	0,5000043	2,5
A0,2	0,2000015	1

Table 1.

The weights designated with "A" belong to AGA Gas AB, the other two were borrowed from SP for this special purpose, which explain the differences in uncertainty. Since the balance is mounted in the ceiling, the calibration weights had to be placed on a special calibration table, which was hanged in the hook (see figure 2). The balance was set to zero when the table was empty. This empty table was also hanging in the hook during all tests and investigations in order to maintain the same weighing range. The results from this calibration series can be seen in detail in appendix B. The results for the lower scale range, which is suited for a 5-L gas cylinder is shown in figure 3. In this range (10-11,5 kg), the balance seems to have a practically constant measurement error. This is very convenient, because each weighing result

is a difference between two weighings (before and after filling). The uncertainty interval that is indicated in figure 3 is a combination of the spread in the weighing series and the uncertainty in mass for the weights.

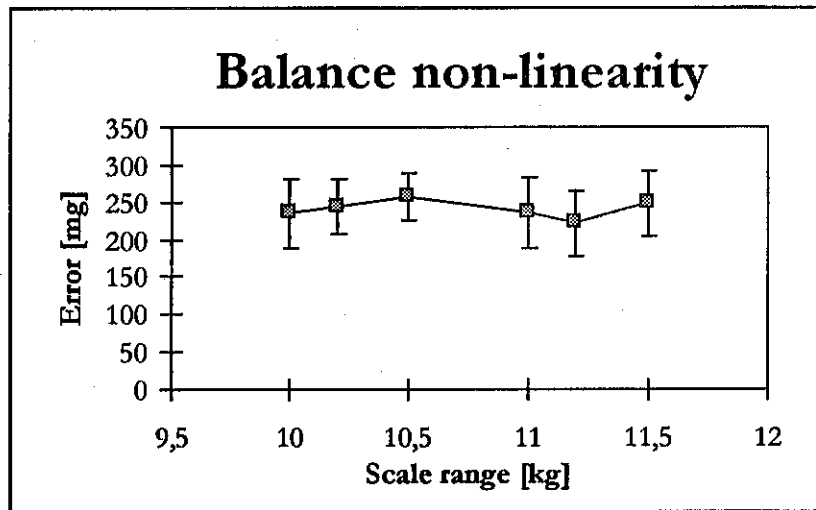


Figure 3.

The second scale range that was calibrated is suited for a 20-L gas cylinder. The result for this range is shown in figure 4. Unfortunately, the balance error is not constant in that part of the range. Thus, the balance has a difference in sensitivity for this particular range. The calibration result reminds of a slope. A linear equation is adjusted to the calibration result, which is used for correction of weighing results later on.

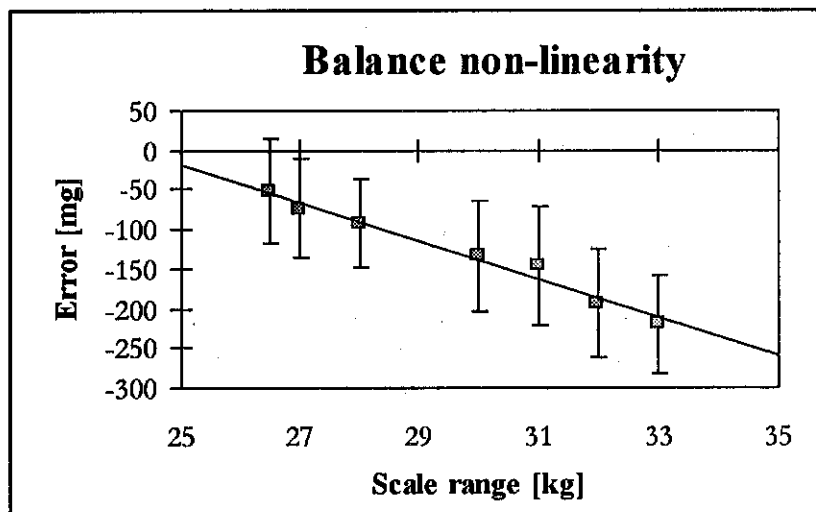


Figure 4.

## 4.2 Balance hysteresis

For a given mass, the weighing result may be different depending on whether the applied load on the balance is increasing or decreasing. This phenomenon is called the "Hystereris effect". Some of the weights tabulated in table 1 were used in order to investigate the balance hysteresis. The result of this test is shown in figure 5. The hysteresis effect is obvious in the

figure. However, the effect will not cause any problem since the gas cylinders neither increase nor decrease in mass during weighing.

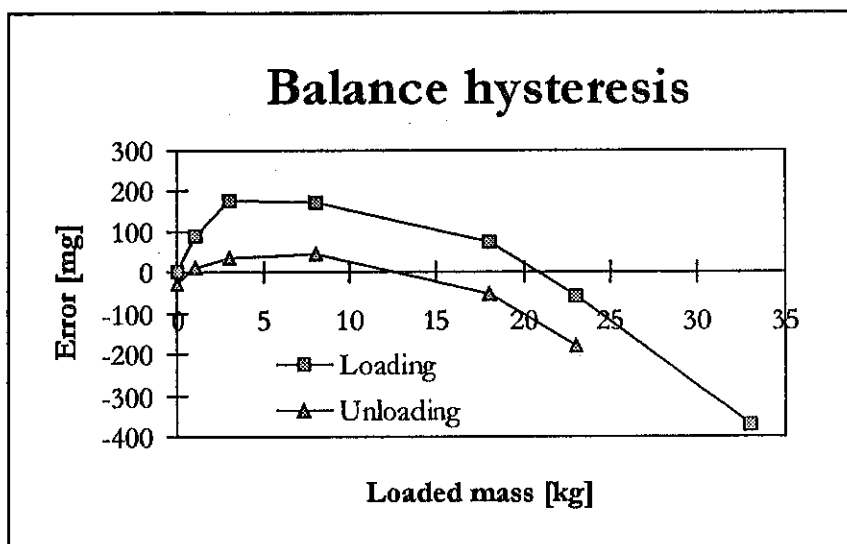


Figure 5.

### 4.3 The effect of a warm cylinder at weighing

When a cylinder is filled, it heats up. This is due to the high pressure in the cylinder. At a normal filling situation, the pressure reaches above 200 bar. Depending on the kind of gas that is filled, the cylinder temperature will rise to about 35 °C. The opposite situation occurs when a cylinder is emptied. A rise in temperature will cause mainly three effects:

- Expansion of the cylinder.
- Desorption of compounds from the cylinder surface.
- Convection

#### 4.3.1 Expansion of cylinder

When the cylinder is heated the volume will increase. This gives rise to an increase in air buoyancy. Because the warm cylinder replaces a larger amount of air, the observed mass will be lower. The difference in volume can be calculated using the following equation:

$$\Delta V = V_0 \cdot (1 + \alpha \cdot \Delta T)^3 - V_0$$

$V_0$  = Volume at  $\Delta T = 0$ .

$\alpha$  = Expansion coefficient.

$\Delta T$  = Difference in temperature compared to room temperature.

For a 20-liter aluminium cylinder heated to a temperature 15 °C above room temperature, the change in buoyancy corresponds to about 31 mg ( $\alpha_{Al} = 2,32 \cdot 10^{-5} \text{ K}^{-1}$ ). There is further a small change in air density around the warm cylinder causing the buoyancy effect to decrease.

#### 4.3.2 Desorption of compounds

The cylinder has in normal room temperature a thin layer of water at the surface. As the temperature increases the water will evaporate to the surrounding. This also results in a

decrease in observed mass. The effect is small, about  $0,2 \mu\text{g}/\text{cm}^3$  and insignificant compared to the balance resolution.

### 4.3.3 Convection

A warm cylinder will transfer the heat to the surrounding air. As the air temperature increases it starts to move in an upward direction. Due to friction against the cylinder surface a force is applied to the cylinder. Hence, the observed mass will be lower. Per Rosby (SP) described the following expression for the effect in his report:

$$\Delta m = -9,2 \cdot 10^{-7} \cdot A \cdot h^{1/4} \cdot \Delta T^q \quad [\text{g}]$$

Where:

- A = Surface area,  $[\text{cm}^2]$
- h = Cylinder height,  $[\text{cm}]$
- $\Delta T$  = Temperature difference,  $[\text{K}]$
- q = 1 (constant)

Suppose a 20-liter cylinder with a diameter of 20 cm and a height of 100 cm, heated  $15^\circ\text{C}$  above room temperature. The result is:  $\Delta m = -0,3 \text{ g}$ . The expression is suited for a weight on a balance scale. In our case the cylinder is suspended and not placed on a scale. For that reason the expression had to be experimentally confirmed.

### 4.3.4 Confirmation of the theoretical model

An evacuated 5-L OTM cylinder was filled with helium and immediately hanged on the balance hook. The cylinder was equipped with three temperature sensors placed according to figure 6. The temperature and the balance indication were noted continuously. These readings can be seen in appendix D. The observed mass increased exponentially as expected, with a total mass difference of  $-0,19 \text{ g}$ . The corresponding value for theoretical model is  $-0,1 \text{ g}$  (with  $h = 50 \text{ cm}$ , diameter =  $15,2 \text{ cm}$  and  $\Delta T = 13,6 \text{ K}$ ). Thus, the theoretical model is now considered to be usable for correction purposes. The observations are plotted in the following diagram, see figure 7.

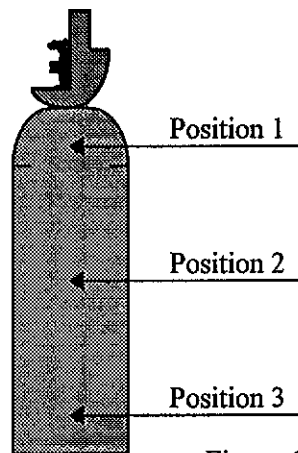


Figure 6.

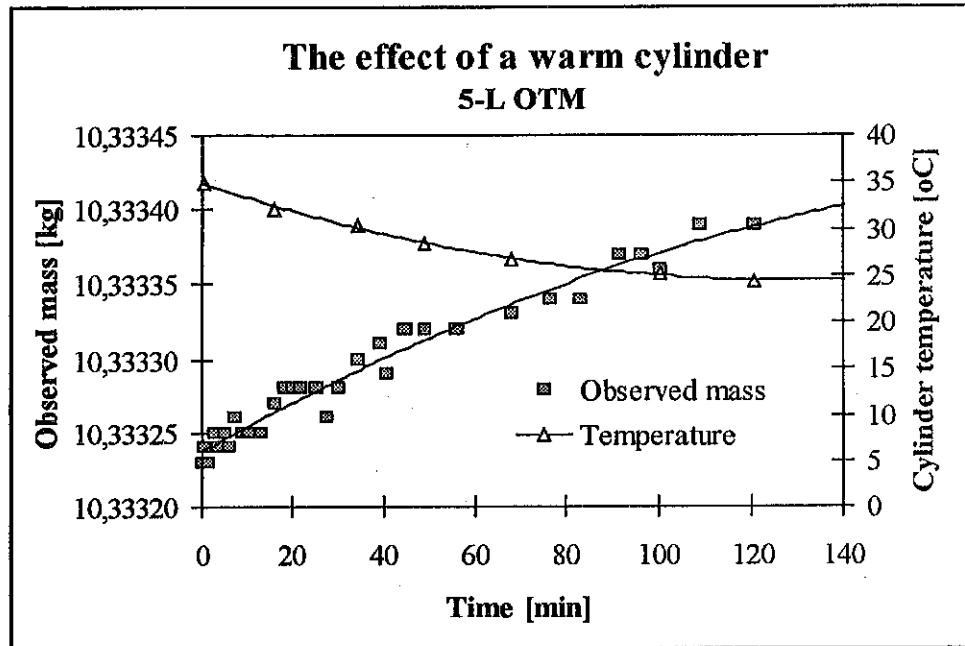


Figure 7.

## 5 Measurement method

During a normal filling situation the mass flow meter measures the total amount of gas passed through the meter during a given time interval. The purpose of this method is to determine the true mass value with a belonging uncertainty and to compare it to the mass flow meter reading and the nominally value. The measurement situation is described in figure 8.

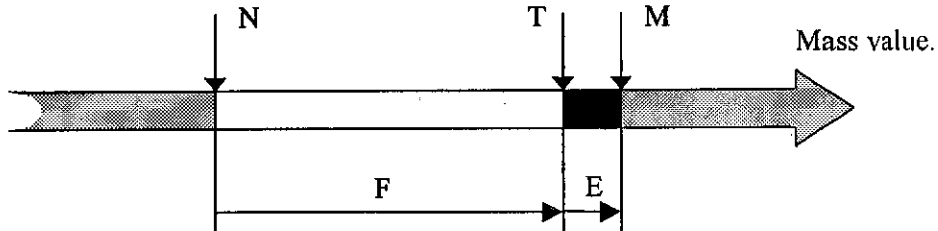


Figure 8.

Where:

- N = Nominal value.
- T = True value.
- M = Mass flow meter reading.
- F =  $N - T$  = The filling error.
- E =  $M - T$  = The mass flow meter error.

### 5.1 Method design

To calibrate the mass flow meter must an independent reference instrument, preferably a balance, record the same amount of gas that has passed through the meter. The crucial problem with this calibration method is to collect all the measured gas and bring it to the balance. There is always some gas left in the pipe work between the mass flow regulator (see  $R_2$  in figure 9) and the cylinder valve. This amount of gas has to be considered. In order to determine it, a second cylinder is used for collecting the remaining gas in the outlet tubing (see  $V_2$  and  $V_3$  in figure 9). A regulator valve ( $R_1$  in figure 9) can reduce the line pressure to the desired level. The purpose of this action is to avoid fluctuations in the line pressure that can cause a significant error, see chapter 4.4.6. For this calibration method an extra valve was installed to the panel ( $R_3$  in figure 9). It prevents the gas in the tubing to escape to the atmosphere while changing from 20L to 5L cylinder.

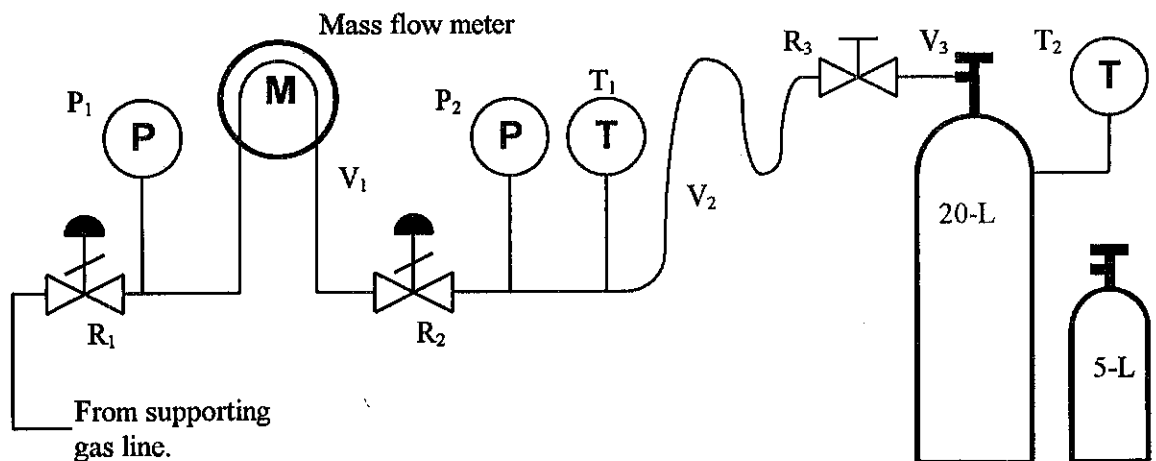


Figure 9.

Parts included in the panel in normal use:

R <sub>2</sub> :	A regulator valve which controls the mass flow. Its input signal comes from the mass flow meter via the PLC system.
P <sub>1</sub> :	Pressure gauge. Measures the reduced line pressure.
P <sub>2</sub> :	Pressure gauge. Measures the cylinder pressure when R <sub>3</sub> is open. When R <sub>3</sub> is closed it measures the pressure in volume V <sub>2</sub> .
V <sub>1</sub> :	Volume between the mass flow meter and the regulator valve R <sub>2</sub> .
V <sub>2</sub> :	Volume between the regulator valve R <sub>2</sub> and the valve R <sub>3</sub> .

Extra parts included in the panel for the calibration method:

R <sub>1</sub> :	Regulator valve, reduces the line pressure to a desired level.
R <sub>3</sub> :	Manual valve.
V <sub>3</sub> :	Volume between the valve R <sub>3</sub> and the cylinder valve.

Temporary measurement fittings for the calibration method:

T <sub>1</sub> :	Temperature sensor attached to the tubing. It indicates the gas temperature in volume V <sub>2</sub> .
T <sub>2</sub> :	Temperature sensor attached to the cylinder wall. it indicates the cylinder temperature during weighing.

A detailed drawing of the SELMA-panel can be seen in appendix A. Two cylinders are used in the investigations, one 20-L OTM and one 5-L OTM. Both of them are aluminium cylinders. The 5-L cylinder is intended for the gas that is remained in the outlet tubing (see V<sub>2</sub> and V<sub>3</sub> in figure 9) after filling the 20-L cylinder.

## 5.2 The constituents of the measurement result

The measurement results are principally a sum of three parts, these are; the weighing result for the 20-L cylinder, the weighing result for the 5-L cylinder and a number of corrections. Each measurement result, T ("true" value) consists of the following parts:

$$T = X20 + B20 + X5 + B5 + R + L - A + P \quad [g]$$

Where: X20 is the weighing result for the 20-L cylinder (difference reading).

B20 is the balance and weighing correction for the 20-L cylinder.

X5 is the weighing result for the 5-L cylinder (difference reading).

B5 is the balance and weighing correction for the 5-L cylinder.

R is a correction for gas that is remained in the panel after filling.

L is correction for gas that is lost to the atmosphere during the method procedure.

A is correction for air that is added to the 5-L cylinder during the method procedure.

P is correction for differences in the line pressure.

All corrections are explained in detail in chapter 5.4. The method is aimed to define two important measures for the panel. If the corresponding mass flow meter reading is designated as M, could the it's error E be expressed as:

$$E = M - T \quad [g]$$

A target or nominal value N given in a recipe defines each amount of gas in a mixture. The filling error F is the deviation from this nominal or desired value:

$$F = N - T \quad [g]$$

### 5.3 General measurement procedure

1. The panel is arranged with additional features according to the layout in figure 9.
2. Air temperature, humidity and pressure in the weighing room are determined before the first weighing.
3. The two cylinders are evacuated and weighed. The temperature of the 20-L cylinder is measured during the weighing.
4. A special calibration recipe is created, for instance 50 g nitrogen and a nominal mass flow of 5 g/s.
5. The 20-L cylinder is attached to the panel and the filling procedure starts.  $R_3$  and the cylinder valve are open.
6. The pressure  $P_1$  is noted at the same time as the regulator  $R_2$  starts to open.
7. When the target mass value is reached and the computer controlled regulator ( $R_2$ ) stops the gas flow, the pressure ( $P_1$ ) is noted again.
8. The cylinder valve and the valve  $R_3$  are closed.
9. The temperature  $T_1$  and the pressure  $P_2$  are noted.
10. The 20-L cylinder is disconnected from the panel.
11. The 5-L cylinder is attached to the panel. The cylinder valve and the valve  $R_3$  are opened for a while to allow pressure equalisation and gas from  $V_2$  and  $V_3$  to enter the cylinder.
12. The cylinder valve and the valve  $R_3$  are closed again.
13. The 5-L cylinder is disconnected from the panel.
14. Both cylinders are carried away to the weighing room, where they are weighed. The temperature of the 20-L cylinder is measured during the weighing.
15. The test procedure is repeated several times in the same manner.
16. Air temperature, pressure and humidity are determined in the weighing room after the last filling and weighing.

### 5.4 Corrections of measurement results

Each measurement result,  $T$  ("true" value) consists of a number of corrections in order to correct for known errors. These six different corrections performed are described in the following.

#### 5.4.1 Balance and weighing correction for the 20-L cylinder, B20

This correction (B20) can be divided into three parts, and these are; balance non-linearity, increased cylinder volume due to the high pressure and convection due to a warm cylinder.

##### 5.4.1.1 Balance non-linearity

The balance characteristics and non-linearity was investigated as described in chapter 4. The calibration results in a linear slope, which can be described with the following equation:

$$e_1 = -24,201 \cdot X + 587,384 \quad [mg]$$

Where  $e_1$  is the error when weighing  $X$  kg (the balance is tared with an empty weight table). When a gas cylinder is weighed both before and after the filling procedure, the error and thus the correction differs due to the fact that the balance error is not constant in this part of the range. The total correction due to the balance non-linearity is therefore:

$$c_1 = (W_A - W_B) \cdot 0,0242007 \quad [g]$$

Where  $W_A$  is the balance reading in kg after the filling procedure and  $W_B$  is the corresponding value before the filling (often an evacuated gas cylinder).

#### 5.4.1.2 Cylinder expansion due to high pressure

According to a report by Roland Johnsson (AGA), the cylinder volume increases 1 ml/bar due to the gas pressure on the cylinder walls. This effect will produce a difference in air buoyancy before and after the gas cylinder is filled. However, the increase in volume applies to a 50-L cylinder and the pressure effect is supposed to be smaller for a 20-L cylinder, about 0,5 ml/bar. If the air density is  $1,2 \text{ kg/m}^3$ , the correction  $c_2$  in air buoyancy correction will be:

$$c_2 = (P_A - P_B) \cdot 0,5 \cdot 10^{-3} \cdot 1,2 \quad [g]$$

Where  $P_A$  is the gas pressure in bar measured after the filling procedure and  $P_B$  is the corresponding value before the filling.

#### 5.4.1.3 Convection due to a warm cylinder

This correction is based on the theoretical model, which is described in chapter 4.4.4. A correction is needed if the temperature of the cylinder differs from the air temperature in the weighing room. If the temperature of both the cylinder and the room is maintained during the gas filling procedure, the correction is zero. The error due to convection is:

$$e_3 = -9,2 \cdot 10^{-7} \cdot A \cdot h^{1/4} \cdot (T_C - T_R) \quad [g]$$

Where:

- $A$  = Surface area,  $[\text{cm}^2]$
- $h$  = Cylinder height,  $[\text{cm}]$
- $T_C$  = Temperature of the gas cylinder,  $[\text{K}]$
- $T_R$  = Temperature in the weighing room,  $[\text{K}]$

Since the gas cylinder is weighed twice (before and after filling) and within a short period, the difference in room temperature is negligible. The correction  $c_3$  due to convection is therefore simplified to the following expression:

$$c_3 = 9,2 \cdot 10^{-7} \cdot A \cdot h^{1/4} \cdot (T_B - T_A) \quad [g]$$

Where  $T_B$  is the temperature in K of the gas cylinder before the gas filling procedure and  $T_A$  is the corresponding temperature afterwards.

#### 5.4.1.4 Sum of the balance and weighing correction (20-L)

The three corrections caused by non-linearity, expansion and convection give the total correction B20 according to the following expression:

$$B20 = c_1 + c_2 + c_3 \quad [g]$$

#### 5.4.2 Balance and weighing correction for the 5-L cylinder, B5

The same corrections could be performed for the 5-L cylinder. However, the 5-L cylinder is only intended for small amounts of gas. This means that the convection effect and the expansion are negligible under these circumstances. Due to the small amount of gas, the used weighing range is narrow. Hence, the non-linearity correction is omitted for the 5-L cylinder.

$$B5 < 0,01 \quad [g]$$

### 5.4.3 Correction for gas that is remained in the panel after filling, R

Although most of the gas is collected in the two gas cylinders, some gas is left in the outlet tubing. This amount of gas is also measured by the mass flow meter; thus a correction is necessary. The correction R can be derived from the proportion between the volume of the outlet tubing ( $V_2+V_3$ ) and the 5-L cylinder volume ( $V_{5L}$ ), see figure 9.

$$R = \frac{X5 \cdot (V_2 + V_3)}{V_{5L}} \quad [g]$$

Where X5 is the difference in weight between the empty and the filled gas cylinder. The volume  $V_2$  is about 75 ml according to an earlier investigation. The small volume  $V_3$  between regulator valve  $R_3$  and the cylinder valve was measured to be 4,8 ml.

### 5.4.4 Correction for gas that is lost to the atmosphere during the method procedure, L

When the 20-L cylinder is changed to the 5-L cylinder, some gas escapes from the volume  $V_3$  to the surrounding air. This amount of gas can not be neglected if the cylinder pressure is high. As mentioned before the volume  $V_3$  is about 4,8 ml. The correction L is calculated using the ideal gas law according to the following expression:

$$L = \frac{k \cdot M \cdot P \cdot V_3}{R \cdot T} \quad [g]$$

Where k is a correction constant due to non-ideal circumstances, M is the molar weight for the actual gas, P is the pressure, R is the molar gas constant and T is the gas temperature (The temperature sensor  $T_1$  gives an indication of the gas temperature).

### 5.4.5 Correction for air that is added to the 5-L cylinder during the method procedure, A

The volume  $V_3$  is filled with air instead of gas when changing from the 20-L cylinder to the 5-L cylinder. This amount of air is not measured by the mass flow meter, therefore a negative correction has to be applied. The correction is relatively small due to the atmospheric pressure. If the density of air is assumed to be  $1,2 \text{ kg/m}^3$ , the correction A will be:

$$A = V_3 \cdot \rho \quad [g]$$

Where  $\rho$  is the density of air at atmospheric pressure.

### 5.4.6 Correction for differences in the line pressure, P

The volume  $V_1$  in figure 9 is filled with gas when the filling procedure begins. When the regulator valve  $R_2$  starts to open the software program also starts to measure the gas that is flowing through the meter. In other words, the gas in volume  $V_1$  is never measured but it certainly is transferred to the cylinder that is attached to the filling panel. On the other hand, when the regulator valve finally closes at the end of the filling procedure the situation is the opposite. The volume  $V_1$  is now filled with gas that is measured but who never will enter the

cylinder. These two effects cancel each other and the error is zero, assuming the same line pressure at the beginning and at the end of the procedure. But in reality the pressure differs and a correction is needed. The correction  $P$  is calculated using the ideal gas law according to the following expression:

$$P = \frac{k \cdot M \cdot V_1}{R \cdot T} \cdot (P_B - P_A) \quad [g]$$

Where  $k$  is a correction constant due to non-ideal circumstances,  $M$  is the mole weight for the actual gas,  $R$  is the molar gas constant and  $T$  is the gas temperature (The temperature is assumed to be 293 K).  $P_B$  is the line pressure when the regulator valve starts to open.  $P_A$  is the line pressure when the valve is closed.

## 5.5 Measurement uncertainty

The method uses a balance, which is calibrated (see chapter 4) with a combination of weights with known mass. These weights are calibrated at SP - The Swedish National Testing & Research Institute and are traceable to the Swedish national kilogram.

Each measurement value (designated as "true" value) has a belonging uncertainty interval. This interval consists of several uncertainty components. Each uncertainty component is evaluated either as a type A or type B evaluation. The type A evaluation is based on statistical methods like the standard deviation etc. A type B evaluation is a judgement based on experiences with the equipment. The uncertainty of the method can be compared to the analysis uncertainty using a gas chromatograph (GC) or a similar analytical instrument.

### 5.5.1 Uncertainty in weighing of gas in the 20-L cylinder

The gas mass is determined by weighing the cylinder before and after every filling. At each weighing occasion, the weighings are repeated three times to give a standard deviation. A typical value is  $s_{20} = 40$  mg. In order to give the result from three values a more statistical weight, the standard deviation is multiplied with a Student's  $t$ -factor with 2 degrees of freedom (number of weighings ( $n$ ) - 1). The type A uncertainty component is:

$$u_{x_{20b}} = \frac{t \cdot s_{20}}{\sqrt{n}} = \frac{1,32 \cdot 40}{\sqrt{3}} = \pm 30,5 \text{ mg}$$

Where  $t$  is the Student distribution coefficient with 2 degrees of freedom at confidence level one sigma ( $k = 1$ ). The  $t$  value are found in Chatfield 1983, page 141. Since the weighings are carried out both before and after the filling, the total uncertainty for the mass difference is the quadratic sum of two equal components (at confidence level  $1\sigma$ ):

$$u_{x_{20}} = \sqrt{30,5^2 + 30,5^2} = \sqrt{2} \cdot 30,5 = \pm 43 \text{ mg}$$

### 5.5.2 Uncertainty in weighing of gas in the 5-L cylinder

The weighing procedure for the 5-L cylinder is similar to the procedure for the 20-L cylinder. The weighing is repeated three times before and after the filling. The standard deviation is somewhat smaller compared to that of the 20-L cylinder. This is probably due to that the 5-L cylinder can be smoothly hanged beneath the balance manually whereas the 20-L cylinder had

to be handled with the help of a special lifting device. A normal standard deviation for the 5-cylinder is  $s_{x5} = 25$  mg. The type A uncertainty with 2 degrees of freedom and  $n = 3$  is:

$$u_{x5b} = \frac{t \cdot s_5}{\sqrt{n}} = \frac{1,32 \cdot 25}{\sqrt{3}} = \pm 19,1 \text{ mg}$$

Since the weighings are carried out both before and after the filling, the total uncertainty for the mass difference is the quadratic sum of two equal components (at confidence level  $1\sigma$ ):

$$u_{x5} = \sqrt{19,1^2 + 19,1^2} = \sqrt{2} \cdot 19,1 = \pm 27 \text{ mg}$$

### 5.5.3 Uncertainties in the balance and the weighing correction

The balance and the weighing corrections (B20) made on the test results consists of three parts; balance non-linearity, increased cylinder volume due to the high pressure and convection due to a warm cylinder.

The balance non-linearity was investigated in advance, see chapter 4. The corrections performed still have an uncertainty of about 70 mg at  $k = 2$ , see sector 4.1. This gives an uncertainty component  $u_1$  at  $k=1$ :

$$u_1 = \frac{1}{2} \cdot 70 = \pm 35 \text{ mg}$$

The volume of the cylinder is increasing due to the pressure. This causes a difference in air buoyancy. The cylinder volume increases 1 ml/bar according to Roland Johnsson (AGA). This applies to a 50-L cylinder and the pressure effect is supposed to be smaller for a 20-L cylinder, about 0,5 ml/bar with an assumed uncertainty of  $\pm 0,25$  ml/bar. If the air density is  $1,2 \text{ kg/m}^3$  and the difference in cylinder pressure is 150 bar, the uncertainty  $u_2$  in air buoyancy correction will be:

$$u_2 = \frac{0,25 \cdot 150 \cdot 1,2}{\sqrt{3}} = 26 \text{ mg}$$

A type B evaluation with a rectangular distribution ( $1/\sqrt{3}$ ) is used ( $k=1$ ).

The temperature of the 20-L cylinder is measured during weighing and a correction for the thermal convection effect is made according to the formula described in sector 5.4.1.3. The uncertainty in this correction depends on uncertainties in the convection model itself, uncertainties in the temperature determination etc. Here, the uncertainty is expressed as a temperature interval  $\pm 3$  K. A type B evaluation with a rectangular distribution gives the uncertainty  $u_3$  at  $k=1$ :

$$u_3 = \frac{9,2 \cdot 10^{-7} \cdot 7100 \cdot 100^{1/4} \cdot 3 \cdot 10^3}{\sqrt{3}} = \pm 36 \text{ mg}$$

These three correction uncertainties are combined as a quadratic sum at  $k=1$  according to:

$$u_{B20} = \sqrt{u_1^2 + u_2^2 + u_3^2} = \sqrt{35^2 + 26^2 + 36^2} = \pm 57 \text{ mg}$$

### 5.5.4 Uncertainty in the correction for gas that is left in the tubing

Even though the 5-L cylinder accepts most of the gas in the tubing (see  $V_2$  and  $V_3$  in figure 9), a small part is left. This amount of gas (R) is calculated according to:

$$R = \frac{X5 \cdot (V_2 + V_3)}{V_{5L}} \quad \text{Where } V_{5L} \text{ is the volume of the 5L cylinder.}$$

The uncertainty in this calculation is depending on the uncertainties in the volume of the cylinder, the volume of the outlet tubing and the dispersion in the weighing. These uncertainties are at  $k=1$ :

Gas mass in 5-L cylinder:  $X5 = 18 \cdot 10^3 \pm 27 \text{ mg}$

(18 g is a maximum value, see appendix E.)

Volume  $V_2$  and  $V_3$ :  $V_{2,3} = 79,8 \pm 2,5 \text{ ml}$

Volume of 5-L cylinder:  $V_{5L} = 5000 \pm 10 \text{ ml}$

The uncertainty in the correction R given above is expressed as:

$$u_R = \sqrt{\left(\frac{\partial R}{\partial X5} \cdot u_{X5}\right)^2 + \left(\frac{\partial R}{\partial V_{2,3}} \cdot u_{V_{2,3}}\right)^2 + \left(\frac{\partial R}{\partial V_{5L}} \cdot u_{V_{5L}}\right)^2} \quad \text{this gives:}$$

$$u_R = \sqrt{0,16 + 81 + 0,29} = \pm 9,03 \text{ mg} \quad (\text{at } k=1)$$

### 5.5.5 Uncertainty in the correction for gas that is lost to the atmosphere

All gas in the volume  $V_3$  (see figure 9) is measured by the mass flow meter but is lost to the atmosphere when the 20-L cylinder is disconnected from the panel. The correction is calculated according to:

$$L = \frac{X20 \cdot V_3}{V_{20L}} \quad \text{Where } V_{20L} \text{ is the volume of the 20L cylinder.}$$

The uncertainty in this calculation is depending on the uncertainties in the volume of the cylinder, the volume  $V_3$  and the dispersion in the weighing. These uncertainties are at  $k=1$ :

Gas mass in 20-L cylinder:  $X20 = 1397 \cdot 10^3 \pm 43 \text{ mg}$

(1397 g is a maximum value, see appendix E.)

Volume  $V_3$ :  $V_3 = 4,8 \pm 0,25 \text{ ml}$

Volume of 20-L cylinder:  $V_{20L} = 20000 \pm 15 \text{ ml}$

The uncertainty in the correction L is expressed as:

$$u_L = \sqrt{\left(\frac{\partial L}{\partial X_{20}} \cdot u_{X_{20}}\right)^2 + \left(\frac{\partial L}{\partial V_3} \cdot u_{V_3}\right)^2 + \left(\frac{\partial L}{\partial V_{20L}} \cdot u_{V_{20L}}\right)^2} \quad \text{this gives :}$$

$$u_L = \sqrt{1,1 \cdot 10^{-4} + 304,9 + 0,063} = \pm 17,5 \text{ mg} \quad (\text{at } k=1)$$

### 5.5.6 Uncertainty in the correction for air that is added to the 5-L cylinder

When the 5-L cylinder is attached to the panel, the volume  $V_3$  is filled with air at atmospheric conditions. The uncertainty in this correction is due to the uncertainty in the volume  $V_3$ . If the air density is supposed to be  $1,2 \text{ kg/m}^3$  and the volume  $V_3$  is  $4,8 \pm 0,25 \text{ ml}$ , the uncertainty in the correction A will be:

$$u_A = \frac{0,25 \cdot 1,2}{\sqrt{3}} = \pm 0,173 \text{ mg}$$

A type B evaluation with a rectangular distribution ( $1/\sqrt{3}$ ) at used ( $k=1$ ).

### 5.5.7 Uncertainty in the correction due to different line pressures at start and stop

This correction has a relative large uncertainty, which is due to the uncertainties in the pressure readings. Unfortunately, the pressure is not stable during the filling procedure and the pressure value is a result of an instantaneous reading. The pressure is generally more unstable at the end of the filling. The uncertainty of the pressure reading could have been better if the regulator system had been more intelligent. The uncertainty of the start and stop pressure is estimated to  $\pm 1,5 \text{ bar}$  and  $\pm 2,5 \text{ bar}$  respectively. The volume  $V_1$  (see figure 9) is estimated to about  $60 \text{ ml}$ . The pressure uncertainty in bar can be converted to mass by the Ideal gas law. Here, the uncertainty is calculated for nitrogen with a rectangular distribution at  $k=1$ :

$$u_{\text{start}, N_2} = \frac{28,0134 \cdot 1,5 \cdot 10^5 \cdot 60 \cdot 10^{-6} \cdot 10^3}{8,31451 \cdot 293,15 \cdot \sqrt{3}} = \pm 59,7 \text{ mg}$$

$$u_{\text{stop}, N_2} = \frac{28,0134 \cdot 2,5 \cdot 10^5 \cdot 60 \cdot 10^{-6} \cdot 10^3}{8,31451 \cdot 293,15 \cdot \sqrt{3}} = \pm 99,5 \text{ mg}$$

$$u_{P, N_2} = \sqrt{59,7^2 + 99,5^2} = \pm 116 \text{ mg}$$

Calculated uncertainty for argon:

$$u_{\text{start}, Ar} = \frac{39,948 \cdot 1,5 \cdot 10^5 \cdot 60 \cdot 10^{-6} \cdot 10^3}{8,31451 \cdot 293,15 \cdot \sqrt{3}} = \pm 85,2 \text{ mg}$$

$$u_{stop, Ar} = \frac{39,948 \cdot 2,5 \cdot 10^5 \cdot 60 \cdot 10^{-6} \cdot 10^3}{8,31451 \cdot 293,15 \cdot \sqrt{3}} = \pm 141,9 mg$$

$$u_{P, Ar} = \sqrt{85,2^2 + 141,9^2} = \pm 166 mg$$

Calculated uncertainty for helium:

$$u_{start, He} = \frac{4,003 \cdot 1,5 \cdot 10^5 \cdot 60 \cdot 10^{-6} \cdot 10^3}{8,31451 \cdot 293,15 \cdot \sqrt{3}} = \pm 8,5 mg$$

$$u_{stop, He} = \frac{4,003 \cdot 2,5 \cdot 10^5 \cdot 60 \cdot 10^{-6} \cdot 10^3}{8,31451 \cdot 293,15 \cdot \sqrt{3}} = \pm 14,2 mg$$

$$u_{P, He} = \sqrt{8,5^2 + 14,2^2} = \pm 17 mg$$

Compared to other uncertainty contributions the one from the line pressure variation is dominating. Further more it is dependent on the gas. As a consequence the total uncertainty has to be specified separately for each gas.

### 5.5.8 The total method uncertainty

The total expanded method uncertainty at  $k=2$  is the quadratic sum according to the following expression:

$$u_{TOT} = 2 \cdot \sqrt{u_{X20}^2 + u_{X5}^2 + u_{B20}^2 + u_R^2 + u_L^2 + u_A^2 + u_P^2}$$

The total method uncertainty for nitrogen measurements:

$$u_{TOT, N_2} = 2 \cdot \sqrt{43^2 + 27^2 + 57^2 + 9^2 + 18^2 + 0,2^2 + 116^2} = \pm 281 mg$$

The total method uncertainty for argon measurements:

$$u_{TOT, Ar} = 2 \cdot \sqrt{43^2 + 27^2 + 57^2 + 9^2 + 18^2 + 0,2^2 + 166^2} = \pm 368 mg$$

The total method uncertainty for helium measurements:

$$u_{TOT, He} = 2 \cdot \sqrt{43^2 + 27^2 + 57^2 + 9^2 + 18^2 + 0,2^2 + 17^2} = \pm 162 mg$$

## 6 Experimental investigations

It is not possible to determine one error of a flow meter. The working conditions have normally large impact on the behaviour of a mass flow meter. Therefore, three investigations in all were carried out on the filling panel. These were:

- Test 1 - The effect of the cylinder pressure and the amount of added gas.
- Test 2 - The line pressure effect.
- Test 3 - The gas density effect.

The testing conditions were chosen to simulate a normal filling situation. All tests are described in this chapter.

### 6.1 Test 1 - The effect of the cylinder pressure and the amount of added gas

The first investigation had two main purposes. The first was to find out how the increasing cylinder pressure affects the mass flow measurements. The SELMA-panel is often used for filling gas mixtures with several components. Thus, the first gas component faces an evacuated cylinder while the following components enter at increasing cylinder pressures changing from 0 to about 200 bar. An evacuated cylinder makes the gas to rush into the cylinder resulting in a higher mass flow rate. If the cylinder has a high pressure the filling time is longer and mass flow rate lower.

The second purpose of this investigation was to find out if and how the mass measurements are affected by the amount of gas. Is the relative error larger for a small amount of added gas than for a large one? An expectation in the SELMA-project at AGA was to reach an uncertainty less than 1 % at small amounts such as 20 g of added gas.

#### 6.1.1 Test plan

A cylinder is filled adding gas in six separate steps to a high pressure. These six steps are repeated in several series. At the beginning the cylinder is evacuated and after the sixth adding the cylinder has reached a pressure of about 200 bar. Two different amounts of gas are added, 50 g and 1400 g using two different flow rates. When 50 g are added, the nominal mass flow is adjusted to 5 g/s in order to simulate real filling conditions. If 1400 g are added, a mass flow of 15 g/s is chosen. Nitrogen is used throughout the test. The method is designed according to the layout given in figure 9.

Adding nr:	Nominal amount [g]	Series 1, results [g]	Series 2, results [g]	Series 3, results [g]
1	50	T <sub>11</sub>	T <sub>21</sub>	T <sub>31</sub>
2	1400	T <sub>12</sub>	T <sub>22</sub>	T <sub>32</sub>
3	50	T <sub>13</sub>	T <sub>23</sub>	T <sub>33</sub>
4	1400	T <sub>14</sub>	T <sub>24</sub>	T <sub>34</sub>
5	50	T <sub>15</sub>	T <sub>25</sub>	T <sub>35</sub>
6	1400	T <sub>16</sub>	T <sub>26</sub>	T <sub>36</sub>

Table 2.

### 6.1.2 Test procedure

1. The panel is arranged according to the layout in figure 9.
2. Air temperature, humidity and pressure in the weighing room are determined.
3. The two cylinders are evacuated and weighed. The temperature of the 20-L cylinder is measured during the weighing procedure.
4. A recipe is created, for instance 50 g nitrogen and 5 g/s mass flow for the first adding.
5. The 20-L cylinder is attached to the panel and the filling procedure starts.  $R_3$  and the cylinder valve are open.
6. The pressure  $P_1$  is noted at the same time as the regulator  $R_2$  starts to open.
7. When the target mass value is reached and the regulator ( $R_2$ ) stops the gas flow, the pressure ( $P_1$ ) is noted again.
8. The cylinder valve and the valve  $R_3$  are closed.
9. The temperature  $T_1$  and the pressure  $P_2$  are noted.
10. The 20-L cylinder is disconnected from the panel.
11. The 5-L cylinder is attached to the panel. The cylinder valve and the valve  $R_3$  are opened for a while.
12. The cylinder valve and the valve  $R_3$  are closed again.
13. The 5-L cylinder is disconnected from the panel.
14. Both cylinders are carried away to the weighing room, where they are weighed. The temperature of the 20-L cylinder is measured during the weighing.
15. For each adding the test procedure is repeated from point 3 to this point five times. The 20-L cylinder is not evacuated until a new series starts.
16. Air temperature, pressure and humidity are determined in the weighing room after the sixth adding.

### 6.1.3 Test results

The following table shows the results from the three series. The nominal mass  $N$  is the target value, which is given, in the filling recipe. The meter reading  $M_{ji}$  is the indication from the mass flow meter. All meter readings are collected from the belonging filling reports. The "True" values are calculated according to the method in chapter 5. The uncertainty in these values is  $\pm 281$  mg at  $k=2$ , see 5.5.8. More detailed tables can be found in appendix E.

Adding nr:	N		Series 1		Series 2		Series 3	
	Nominal mass	Cylinder pressure at start	$M_{1i}$	$T_{1i}$	$M_{2i}$	$T_{2i}$	$M_{3i}$	$T_{3i}$
			Meter reading	True value	Meter reading	True value	Meter reading	True value
			[g]	[g]	[g]	[g]	[g]	[g]
1	50	~0	51,8	51,76	50,4	49,69	51,6	51,94
2	1400	2,3	1403	1402,94	1401,3	1401,49	1401	1400,94
3	50	63,9	51,7	51,47	51,5	51,52	53,1	53,31
4	1400	65,1	1402,4	1402,41	1402	1401,67	1401,5	1400,86
5	50	131,1	50,9	50,00	50,7	50,83	52,9	52,38
6	1400	129,8	(1401,4)	(1398,47)	1400,9	1400,18	1400,7	1399,95

Table 3.

The most interesting information in this test can be seen in table 4. It shows the mass flow meter error E and the filling error F. The last two columns to the right contain average values for the three series.

Adding nr:	N	Cylinder pressure at start [bar]	Series 1		Series 2		Series 3		Average, all series	
	Nominal mass		E <sub>1i</sub>	F <sub>1i</sub>	E <sub>2i</sub>	F <sub>2i</sub>	E <sub>3i</sub>	F <sub>3i</sub>		
	[g]		Meter error [g]	Filling error [g]	Meter error [g]	Filling error [g]	Meter error [g]	Filling error [g]	Meter error [g]	Filling error [g]
1	50	~0	0,04	-1,76	0,71	0,31	-0,34	-1,94	0,14	-1,13
2	1400	2,3	0,06	-2,94	-0,19	-1,49	0,06	-0,94	-0,02	-1,79
3	50	63,9	0,23	-1,47	-0,02	-1,52	-0,21	-3,31	0,00	-2,10
4	1400	65,1	-0,01	-2,41	0,33	-1,67	0,64	-0,86	0,32	-1,65
5	50	131,1	0,90	0,00	-0,13	-0,83	0,52	-2,38	0,43	-1,07
6	1400	129,8	(2,93)	(1,53)	0,72	-0,18	0,75	0,05	0,74	-0,07

Table 4.

At the end of adding 6 in the first series (values in brackets), the line pressure was too low compared to the cylinder pressure. The filling procedure therefore stopped automatically. The regulator valve was then adjusted to about 215 bar and the adding continued. The large error is most likely due to the extra start and stop procedure. The values E<sub>16</sub> and F<sub>16</sub> are therefore excluded.

Tables 3 and 4 are summarised and shown in figure 10 for 50g and figure 11 for 1400g gas samples.

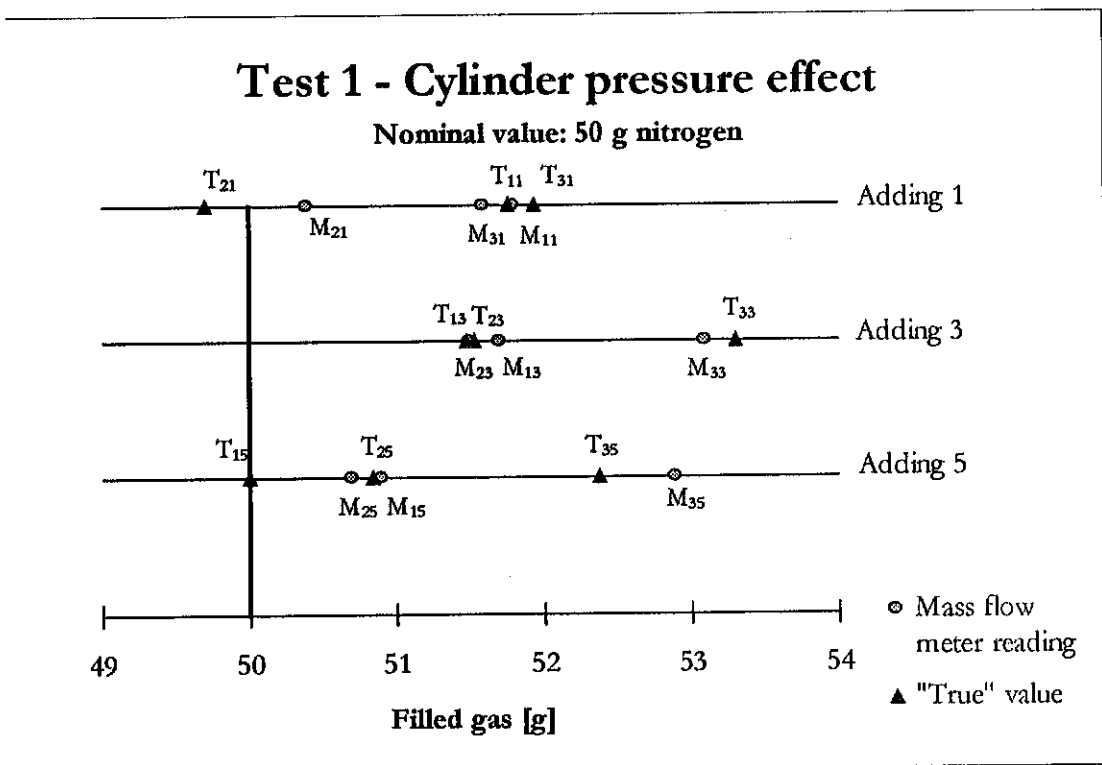


Figure 10.

## Test 1 - Cylinder pressure effect

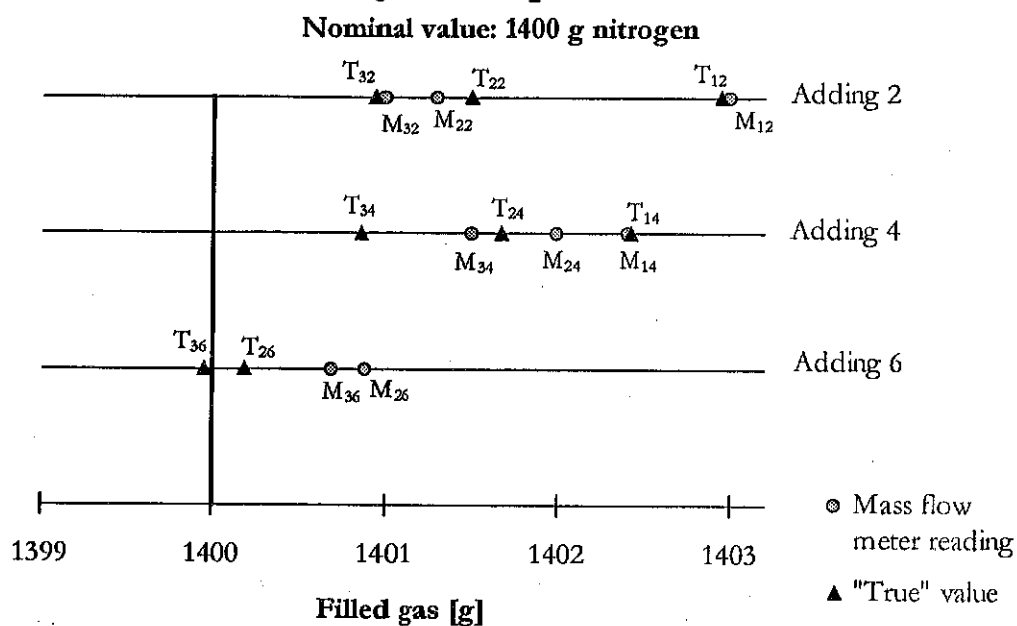


Figure 11.

### 6.1.4 Conclusions

Several conclusions can be drawn from the test results. The conclusions could have been even more numerous if the test was repeated more than three series. The number of series was limited to three due to time limits.

- There are no large differences in meter error between the two amounts of added gas (50 and 1400 g nitrogen). In other words, the mass flow meter error is not proportional to the amount of filled gas, see table 4. Observe that the mass flow rate differs for the two amounts of added gas.
- The SELMA-panel is almost always filling over the nominal value. This is not astonishing as the valve closes when the nominal value is read. An extreme value is 53,31 g, i.e. 3,31 g too much. This can lead to large deviations from the nominal concentration in a gas mixture. Ex: If a 1% gas mixture is wanted, with 50 g as a nominal value for the first component (total weight 5 kg):

$$\text{Example : } \frac{53,31}{53,31 + 4950} \cdot 100 \approx 1,065\%$$

$$\text{The error in \% : } \frac{0,01065 - 0,01}{0,01} \cdot 100 = 6,5\%$$

The relative error can be large for small amounts of added gas. It is hard to reach a relative error below 1% if the component is smaller than 350g.

- The average mass value for adding 1,3 and 5 was 51,4 g. The corresponding value for adding 2,4 and 6 was 1401,3 g. Accordingly, the nominal value can be reduced with about 1,3 g to reach a better result (applies only to nitrogen). The result for adding 6 is lower in comparison with the others. This is presumably due to the high counter pressure in the cylinder. Since the filling error is negative and the mass flow meter error often is positive, these two errors fortunately counteract each other. This is shown in figure 12.

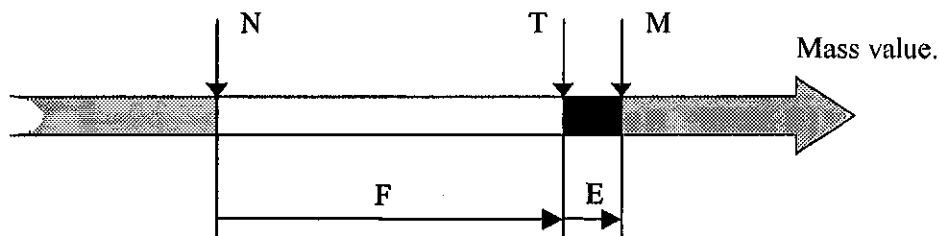


Figure 11.

Where: N = Nominal value.  
 T = True value.  
 M = Mass flow meter reading.  
 $F = N - T$  = The filling error.  
 $E = M - T$  = The mass flow meter error.

- The pressure in the cylinder affects the result. If the cylinder pressure is high, the mass flow rate will be lower. A low flow rate causes larger mass flow meter errors. The mass flow meter has its best measuring performance at maximum flow rate, 15g/s. The cylinder pressure effect can be seen in figure 13 and 14. An evacuated cylinder seems also to affect the measuring result negatively. It is important to relate the measurement errors to the measurement uncertainty. The test series should be repeated several times in order to draw a conclusion that is more established.

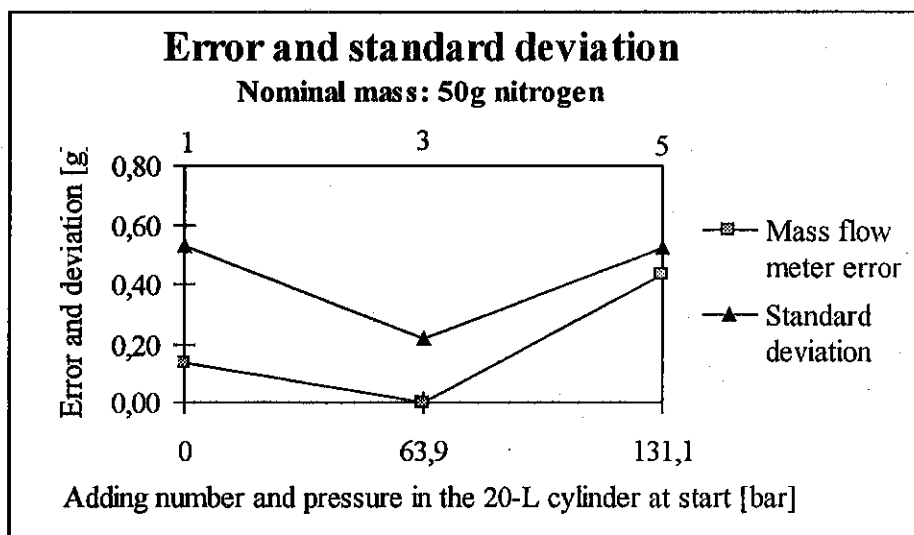


Figure 13.

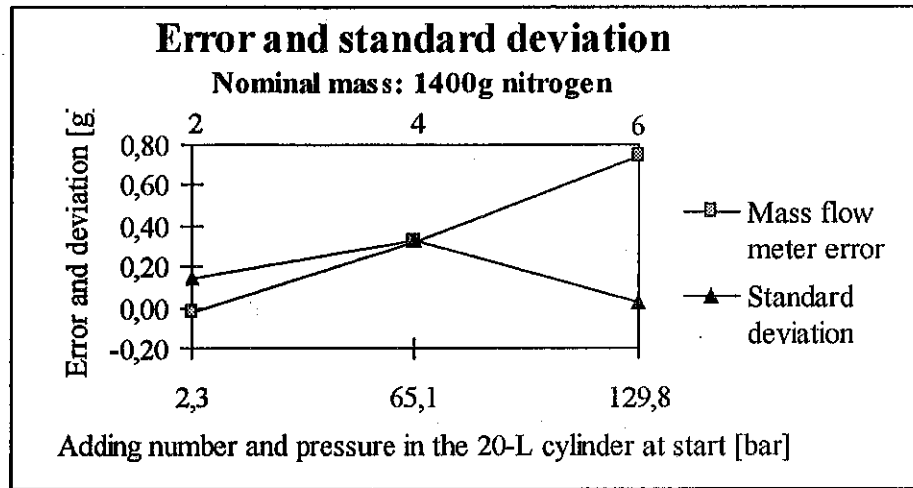


Figure 14.

## 6.2 Test 2 - Line pressure test

The gas providing system feeds the filling panel with gas with a very high pressure. This line pressure is often about 250 bar in normal operation. The purpose of this investigation was to find out how this high pressure affects the filling performance. Presumably, both the mass flow meter and the regulator valve are affected.

### 6.2.1 Test plan

A 20-L OTM cylinder is filled with 500 g nitrogen at six different increasing line pressures. These pressures are: 40, 80, 120, 160, 200 and 240 bar. The procedure can be repeated in several series, in this case only once due to lack of time. The line pressure is reduced to the desired level by a regulator valve,  $R_1$  in figure 9. Disregarding the different pressures and the amount of filled gas, the test procedure is the same as in test 1. Both cylinders are evacuated before each filling. A nominal (and maximum) mass flow rate of 15 g/s was selected in the filling recipe. Again a 5-L OTM cylinder is used to collect the remaining gas in volume  $V_2$  for correction purposes.

Filling nr:	Reduced line pressure [bar]	Series 1, results [g]	Series 2, results [g]
1	40	$T_{11}$	$T_{21}$
2	80	$T_{12}$	$T_{22}$
3	120	$T_{13}$	$T_{23}$
4	160	$T_{14}$	$T_{24}$
5	200	$T_{15}$	$T_{25}$
6	240	$T_{16}$	$T_{26}$

Table 5.

### 6.2.2 Test results

Table 6 shows the results from both series 1 and 2. The line pressures are average values from series 1 and 2. The meter readings are the indication from the mass flow meter and are

collected from the belonging filling reports. The "True" values are calculated according to the method in chapter 5. The uncertainty in these values are  $\pm 281$  mg at  $k=2$ , see 5.5.8. More detailed tables can be found in appendix F.

Filling nr:	Line pressure [bar]	Series 1		Series 2	
		$M_{1i}$	$T_{1i}$	$M_{2i}$	$T_{2i}$
		Meter reading	True value	Meter reading	True value
		[g]	[g]	[g]	[g]
1	39,9	500,5	499,96	500,6	499,99
2	83,8	501,0	500,91	500,4	499,87
3	124,5	500,1	500,15	500,5	500,73
4	168,0	501,6	501,51	500,4	500,23
5	208,3	500,4	500,89	500,1	499,85
6	246,5	503,8	503,37	502,1	501,79

Table 6.

Next table shows the mass flow meter error and the filling error for the two series. The last two columns to the right contain average values for series 1 and 2.

Filling nr:	Line pressure [bar]	Series 1		Series 2		Average, both series	
		$E_{1i}$	$F_{1i}$	$E_{2i}$	$F_{2i}$		
		Meter error	Filling error	Meter error	Filling error	Meter error	Filling error
		[g]	[g]	[g]	[g]	[g]	[g]
1	39,9	0,54	0,04	0,61	0,01	0,57	0,02
2	83,8	0,09	-0,91	0,53	0,13	0,31	-0,39
3	124,5	-0,05	-0,15	-0,23	-0,73	-0,14	-0,44
4	168,0	0,09	-1,51	0,17	-0,23	0,13	-0,87
5	208,3	-0,49	-0,89	0,25	0,15	-0,12	-0,37
6	246,5	0,43	-3,37	0,31	-1,79	0,37	-2,58

Table 7.

### 6.2.3 Conclusions

- The performance of the mass flow meter seems to be best in the line pressure range 120 - 200 bar. The results are summarised and displayed in figure 15. A low line pressure gives a low mass flow rate, causing longer filling time and larger errors. When the line pressure is high (250 bar), the error seems to increase. It is possible that the meter is affected of the high pressure, which leads to a change in the measuring performance.

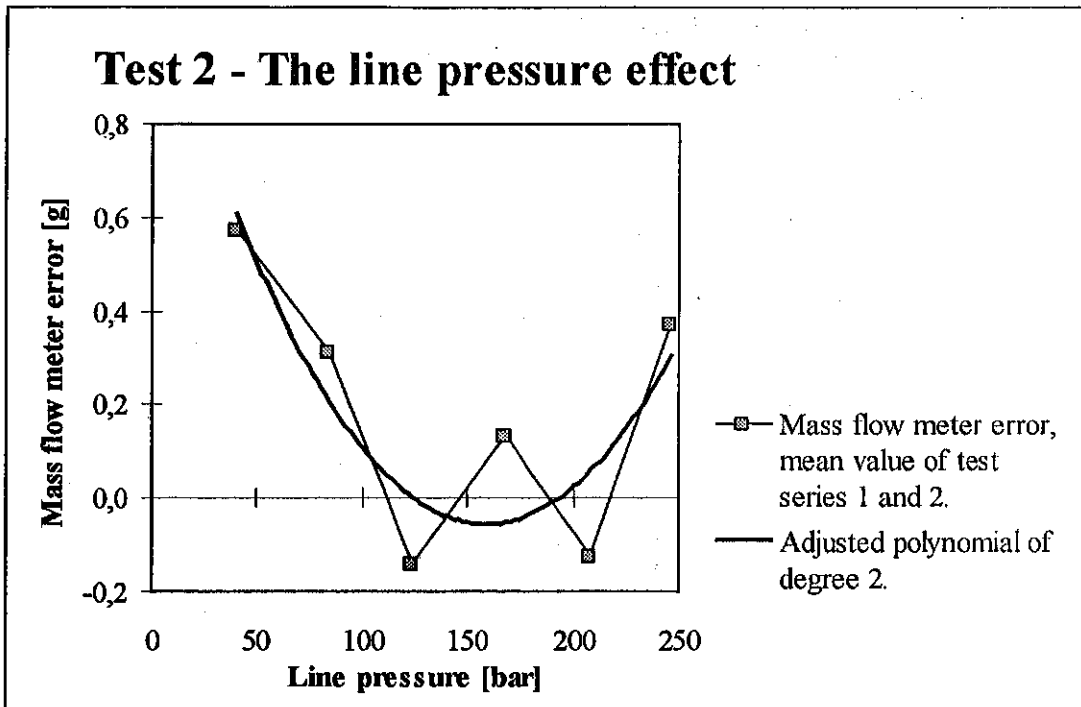


Figure 15.

- The filling error is increasing with increasing line pressure. This is most probably due to the slowness of the regulator valve. It takes a short while until the valve has closed after the mass flow meter indicates the nominal mass value. During this short moment a certain amount of gas pass through the valve. This amount of gas is larger if the line pressure is high, which leads to a result over the nominal mass value. Figure 16 shows the average filling errors for series 1 and 2 at different line pressures.

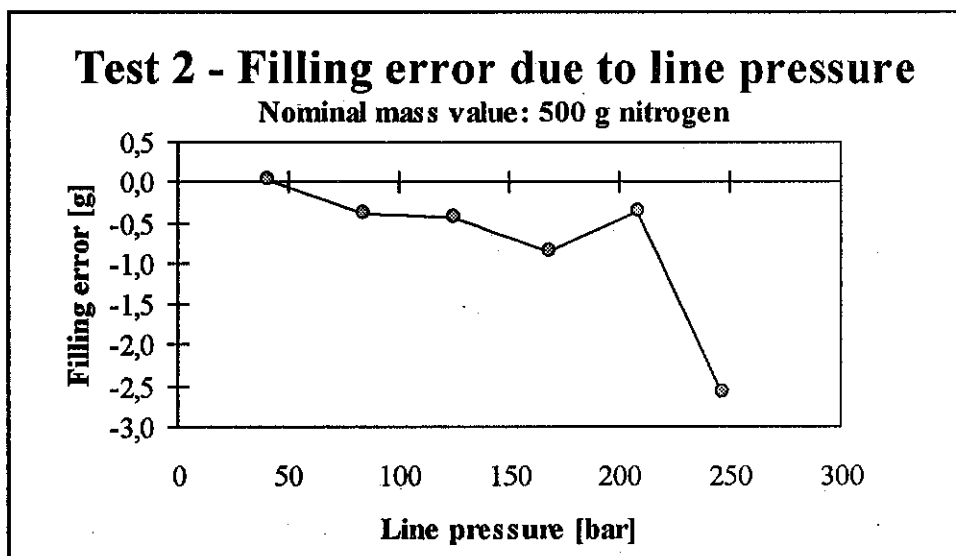


Figure 16.

### 6.3 Test 3 - Gas density test

The filling panel is used for several gases and especially constructed to produce gas mixtures of well defined constituents. These gases vary with regard to density and molecular weight.

The density of argon and carbon dioxide is about ten times higher than the density of helium. The purpose of this test was to find out if the gas density affects the mass flow meter performance. Table 8 shows some frequently used gases in AGA's special gas mixtures.

Gas	Molecular weight	Ideal density at 15 °C, 1 bar
	[g/mole]	[kg/m <sup>3</sup> ]
Nitrogen	28,0134	1,1693
Argon	39,948	1,6674
Helium	4,003	0,1671
Carbon dioxide	44,01	1,8370
Oxygen	31,9988	1,3356
Sulphur dioxide	64,06	2,6738
Propane	44,096	1,8406

Table 8.

The values are collected from AGA Gas AB, "QA - Handbook, molecular weight and density".

### 6.3.1 Test plan

Three gases are used in the test; Nitrogen, helium and argon. Each gas is filled three times with 500 g as a nominal mass value. The line pressure is adjusted to about 210 bar. The maximum mass flow rate is selected to 15 g/s in the recipe specification.

Filling nr:	Nominal mass value	Nitrogen, results	Helium, results	Argon, results
	[g]	[g]	[g]	[g]
1	500	T <sub>N1</sub>	T <sub>H1</sub>	T <sub>A1</sub>
2	500	T <sub>N2</sub>	T <sub>H2</sub>	T <sub>A2</sub>
3	500	T <sub>N3</sub>	T <sub>H3</sub>	T <sub>A3</sub>

Table 9.

The test procedure is similar to the procedure that is described for test 1. A 5-L cylinder is used for the gas that is left in the tubing. Both cylinders are weighed before and after each filling.

### 6.3.2 Test results

The following table shows the test results. The "True" values are calculated according to the method which is described in chapter 5. The measurement uncertainty (at k=2) for nitrogen is ±281 mg, for helium ±162 mg and for argon ±368 mg.

Filling nr:	N	Nitrogen		Helium		Argon	
		M <sub>Ni</sub>	T <sub>Ni</sub>	M <sub>Hi</sub>	T <sub>Hi</sub>	M <sub>Ai</sub>	T <sub>Ai</sub>
	Nominal mass	Meter reading	True value	Meter reading	True value	Meter reading	True value
	[g]	[g]	[g]	[g]	[g]	[g]	[g]
1	500	500,1	499,85	500,8	499,81	501,4	500,56
2	500	502,5	502,34	500,6	499,73	500,6	500,29
3	500	502,7	502,59	500,8	500,08	501,4	501,23

Table 10.

Filling nr:	N	Nitrogen		Helium		Argon	
		$E_{Ni}$	$F_{Ni}$	$E_{Hi}$	$F_{Hi}$	$E_{Ai}$	$F_{Ai}$
	Nominal mass [g]	Meter error [g]	Filling error [g]	Meter error [g]	Filling error [g]	Meter error [g]	Filling error [g]
1	500	0,25	0,15	0,99	0,19	0,84	-0,56
2	500	0,16	-2,34	0,87	0,27	0,31	-0,29
3	500	0,11	-2,59	0,72	-0,08	0,17	-1,23
Average value:		0,18	-1,59	0,86	0,13	0,44	-0,69

Table 11.

Table 10 and 11 is summarised and displayed in figure 17.

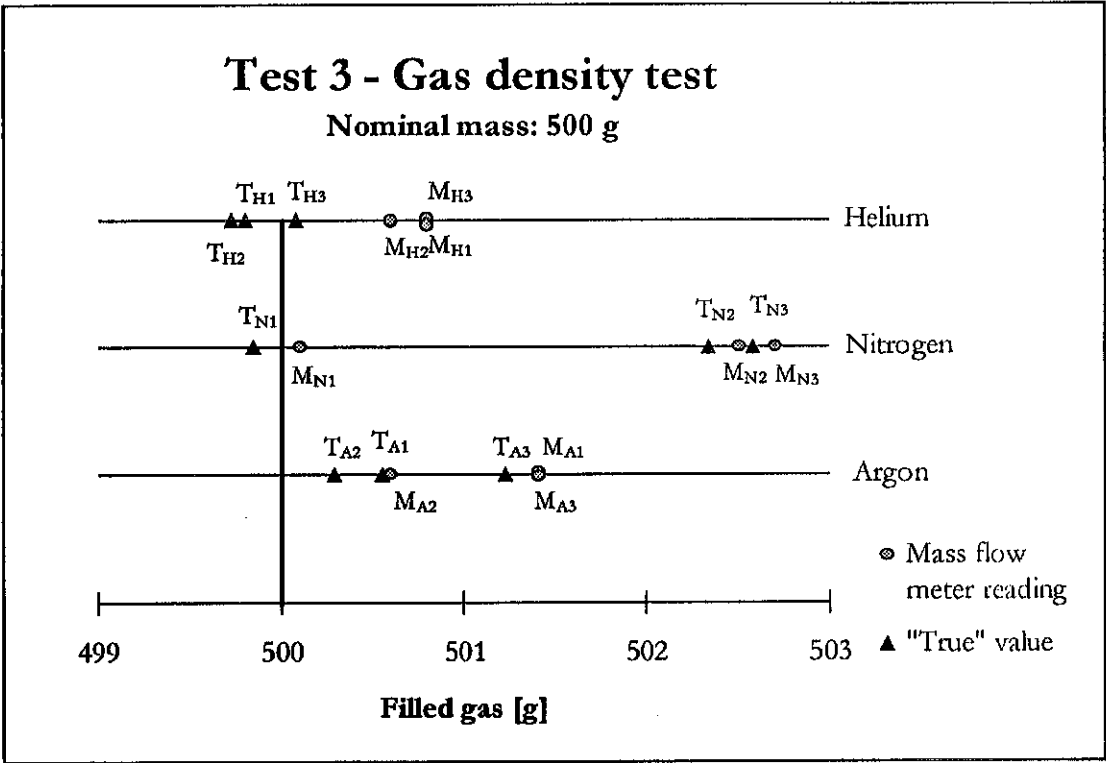


Figure 17.

6.3.3 Conclusions

- The mass flow meter error is smallest when nitrogen is filled, a bit larger for argon and the largest error is found when the cylinder is filled with helium gas. When the cylinder is filled with helium it reaches a high-pressure level, about 175 bar. This could be one of the reasons to the relative large error. The pressure levels for nitrogen and argon are much lower in comparison.

- The situation is the opposite if the filling errors are compared. The error is largest for nitrogen and smallest for helium. A cylinder that is filled with helium is not over filled to the same extent, this is due to the cylinder pressure.

## 7 Evaluation and possible improvements

The performance of the SELMA-panel is very satisfying in many ways. The construction is flexible and robust. The mass flow meter, which is manufactured by Micro Motion Inc., is working at high pressures and is measuring the mass flow with high accuracy, despite large differences in gas density. The weak link in this panel is undoubtedly the regulator system, which has an input signal coming from the mass flow meter. An improvement can also be made on the software. The purpose of all improvements is to:

1. Reduce measurement error.
2. Correct for measurement error.
3. Reduce filling error.
4. Correct for filling error in the recipe.

### 7.1 Improvements on the panel construction

Some improvements can be made on the panel construction. All of them concern the reduction of "dead" volume. Tube lengths can be shortened which helps to reduce errors and uncertainty. Figure 18 shows the SELMA-panel as it is working in normal use.

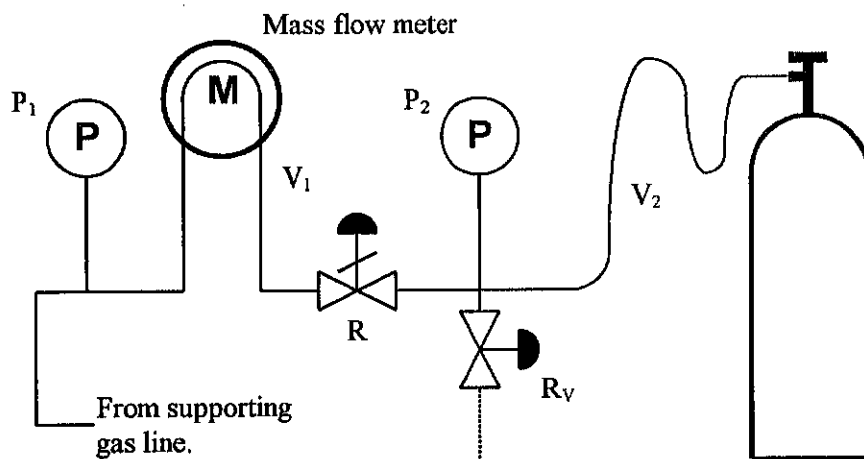


Figure 18.

The regulator valve  $R$  may well be placed immediately after the mass flow meter, the volume  $V_1$  is in that way reduced and the error due to different pressures at start and stop is also reduced. This problem is described earlier in sector 5.4.6. The interior of the valve makes a large part of the volume  $V_2$ ; a valve with a different design would be better. The situation is the same for volume  $V_2$ ; a decrease in tube length leads to reduced errors. The smaller volume  $V_2$  the smaller is the filling error for the last component in a normal filling situation, since the volume  $V_2$  contains gas that will not be transferred to the cylinder. This applies only to the last component. The tubing to the pressure gauge  $P_2$  is too long. It is possible that this tube part retain gas from the first component, since volume  $V_2$  is under pressure during the entire filling procedure.

## 7.2 Improvements on the regulator system

The regulator valve controls the gas flow through the mass flow meter to the cylinder. It is important to maintain a gas flow, which is below the limit for the meter capacity. On the other hand, a low gas flow results in larger measurement errors. Figure 19 shows a normal filling procedure with respect to the gas flow rate and the filling time. This graph is not based on measurement values but on experiences during the investigations.

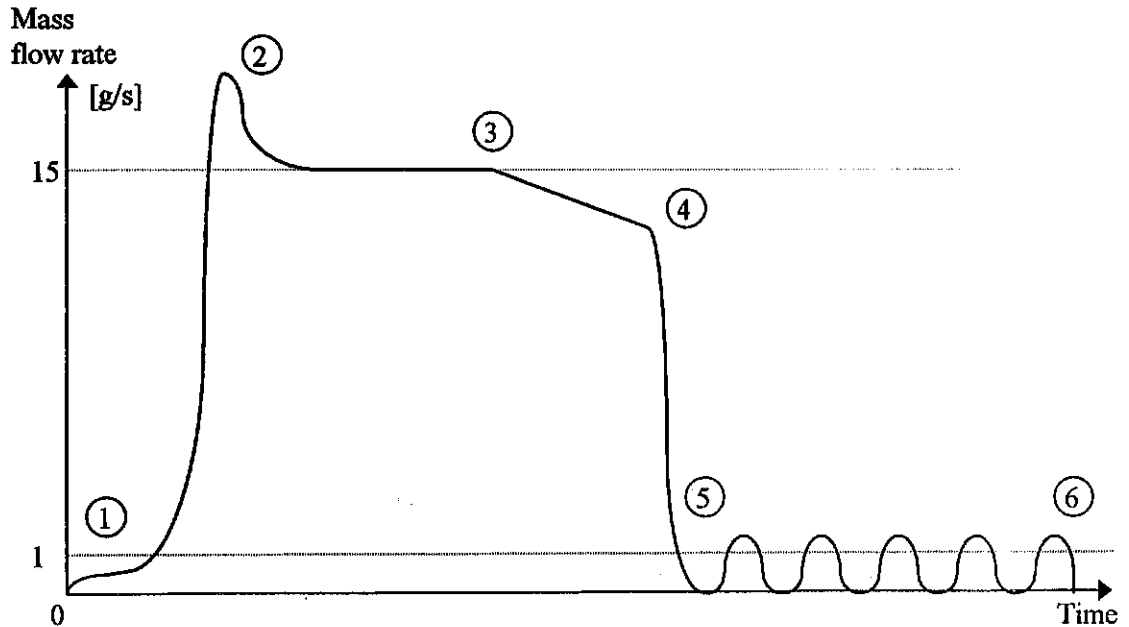


Figure 19.

1. The regulator opens slowly and the gas flow is low for a short time.
2. The recommended mass flow rate is sometimes exceeded. This occurs for example when the line pressure is high and the cylinder is evacuated.
3. If the difference between the line and the cylinder pressure is too small, the regulator reaches its opening maximum and the mass flow rate starts to fall.
4. The regulator valve is closing in order to prevent the panel from filling over the nominal value.
5. The regulator starts to oscillate and the mass flow rate fluctuates from zero to about three gram per second. This gives undoubtedly rise to an increase in measurement error.
6. The mass flow meter indicates that the nominal mass value is reached and the regulator valve is closing. Due to the slowness of the regulator valve some amount of gas passes through the valve after the nominal value is reached. The size of this amount of gas depends on the actually mass flow rate, which varies due to the oscillation.

A better regulator system is needed in order to prevent the mass flow meter from measuring gas at very low flow rates. There are many alternative regulation techniques to solve this problem; for example the traditional PID-regulator. Figure 20 shows the desired filling procedure with respect to the gas flow rate and the filling time.

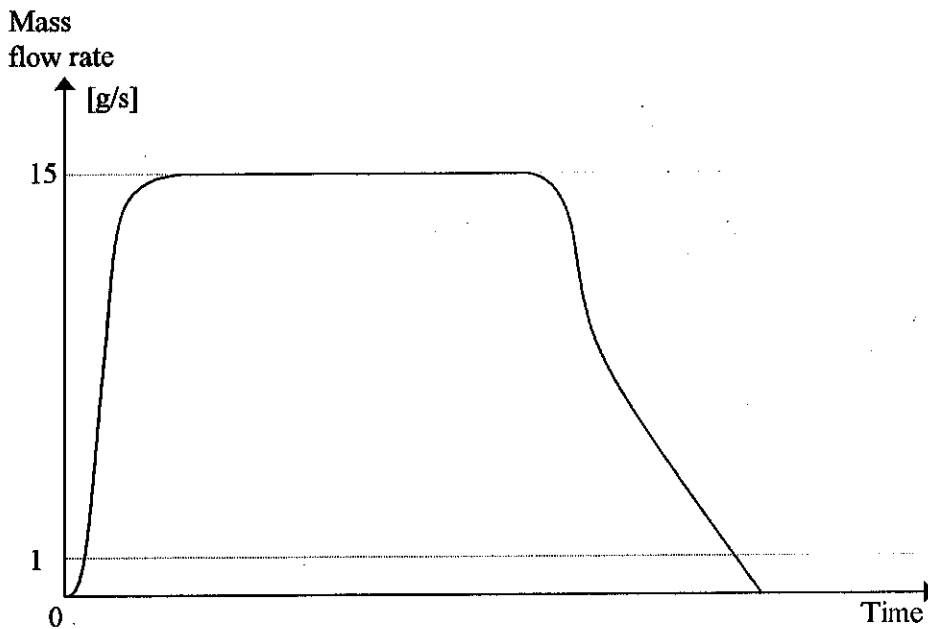


Figure 20.

### 7.3 Software related improvements

The SELMA-panel is controlled by a PLC-system, which interacts with a database containing all gas mixture recipes and their specifications. These recipes need to be scrutinised, cause some of them contain round off errors. Unfortunately, some of these errors are of the magnitude 1 molar %. The filling results can be printed out as a report, which shows each gas component result in terms of gram. These results are represented with only one decimal digit, which is an unfortunately round off from the original number. The mass flow meter gives at least two decimal digits. The volume  $V_1$  (see figure 18) is filled with gas just a second before the regulator starts to open. This situation makes it difficult to read the starting pressure in volume  $V_1$ , which gives rise to large uncertainties in the calibration method (see part 5.4.6). A suggestion is to introduce a time delay (about 10s) before the regulator starts to open. During this time delay, the gas pressure is stabilised and readable.

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**The construction design is not published in this public report.**

Calibration of the balance

Temperature	Before	After
	21,35	21,57
	oC	
Pressure	1015	1015
	mbar	
Air humidity	37	38
	%	

weight	mass [kg]	uncertainty	
		k=2 [mg]	k=1 [mg]
S10	9,999998	15	7,5
A10	10,00006	50	25
S5	5,000003	7	3,5
A5	5,000036	25	12,5
A2	2,000013	10	5
A1	1,000009	5	2,5
A0,5	0,5000043	2,5	1,25
A0,2	0,2000015	1	0,5

mass	weight combination	unloaded	reading 1	unloaded	reading 2	unloaded	reading 3	mean	std error [mg]	balance error [mg]	uncertainty, k=2 [mg]
10	S10	0,00001	10,00021	-0,00001	10,00024	-0,00001	10,00024	10,00023	29	235	47
10,2	S10+A0,2	0,00002	10,20024	0,00000	10,20025	-0,00001	10,20025	10,20024	21	244	36
10,5	S10+A0,5	0,00000	10,50024	0,00000	10,50027	0,00001	10,50028	10,50026	17	258	31
11	S10+A1	0,00000	11,00021	0,00001	11,00027	0,00001	11,00027	11,00024	29	236	47
11,2	S10+A1+A0,2	0,00001	11,20026	0,00000	11,20020	-0,00001	11,20023	11,20023	26	221	44
11,5	S10+A1+A0,5	0,00001	11,50030	0,00000	11,50025	0,00000	11,50024	11,50026	26	249	44
26,5	S10+A10+S5+A1+A0,5	0,00000	26,50000	0,00001	26,50003	-0,00001	26,50004	26,50002	25	-51	66
27	S10+A10+S5+A2	0,00001	27,00001	0,00000	26,99998	0,00000	27,00002	27,00000	20	-74	62
28	S10+A10+S5+A2+A1	-0,00001	27,99997	0,00000	28,00000	0,00000	27,99999	27,99999	10	-93	56
30	S10+A10+S5+A5	0,00000	29,99995	0,00000	29,99995	0,00001	30,00000	29,99996	23	-134	69
31	S10+A10+S5+A5+A1	0,00000	30,99996	-0,00001	30,99998	0,00000	30,99993	30,99996	30	-146	75
32	S10+A10+S5+A5+A2	0,00001	31,99990	0,00002	31,99995	0,00000	31,99993	31,99992	23	-193	69
33	S10+A10+S5+A5+A2+A1	-0,00001	32,99990	0,00000	32,99989	0,00000	32,99990	32,99990	10	-219	62

## Balance hysteresis

Nominal mass	Weight combination	Loading [kg]	Unloading [kg]	Balance Error, loading [mg]	Balance Error, unloading [mg]
0	-	0,00000	-0,00003	0	-30
1	A1	1,00010	1,00002	91	11
3	A1+A2	3,00020	3,00006	178	38
8	A1+A3+S5	8,00020	8,00007	175	45
18	A1+A3+S5+S10	18,00010	17,99997	77	-53
23	A1+A3+S5+S10+A5	23,00000	22,99988	-59	-179
33	A1+A3+S5+S10+A5+A10	32,99975		-369	

	Before	After	
Temperature	21,47	21,8	°C
Pressure	1008	1008	mbar
Air humidity	48	49	%

weights	mass [kg]	uncertainty	
		k=2 [mg]	k=1 [mg]
S10	9,999998	15	7,5
A10	10,00006	50	25
S5	5,000003	7	3,5
A5	5,000036	25	12,5
A2	2,000013	10	5
A1	1,000009	5	2,5

The effect of a warm cylinder

Time	Observed mass
[min]	[kg]
0,00	10,33323
0,50	10,33324
1,42	10,33323
2,47	10,33325
3,58	10,33324
4,77	10,33325
5,70	10,33324
7,22	10,33326
8,80	10,33325
10,05	10,33325
12,68	10,33325
15,97	10,33327
18,03	10,33328
19,68	10,33328
21,73	10,33328
25,30	10,33328
27,47	10,33326
29,87	10,33328
34,32	10,33330
39,17	10,33331
40,83	10,33329
44,75	10,33332
49,23	10,33332
56,12	10,33332
67,95	10,33333
76,43	10,33334
82,92	10,33334
91,58	10,33337
96,20	10,33337
100,05	10,33336
108,62	10,33339
120,45	10,33339

Time	Bottle temperature [°C]			Average
[min]	Pos. 1	Pos. 2	Pos. 3	[°C]
0,62	33,33	36,00	35,23	34,85
15,62	31,40	32,41	32,58	32,13
33,62	30,59	30,66	29,80	30,35
48,62	27,81	28,53	28,60	28,31
68,62	26,43	26,75	26,97	26,72
98,62	24,83	25,22	25,33	25,13
120,62	24,11	24,44	24,50	24,35

The cylinder was weighed the following day  
(1279 minutes later) with these results:

Observed mass	
[kg]	
1	10,33342
2	10,33344
3	10,33344
Mean	10,33343

Room temperature: 21,23 °C

# Results test 1, series 1

Designation	N <sub>li</sub>	M <sub>li</sub>	X20 <sub>li</sub>	B20 <sub>li</sub>	C5 <sub>li</sub>	P <sub>li</sub>	T <sub>li</sub>	E <sub>li</sub>
Description	Nominal mass	Mass flow meter reading	Gas in 20-L cylinder	Balance and weighing correction	Calculated mass in tubing	Different pressure at start and stop	"True" mass	Mass flow meter error
unit	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]
Adding 1	50	51,8	51,36	0,00	0,18	0,21	51,76	0,04
Adding 2	1400	1403	1396,49	0,14	6,38	-0,07	1402,94	0,06
Adding 3	50	51,7	44,78	0,00	6,41	0,28	51,47	0,23
Adding 4	1400	1402,4	1389,33	0,16	12,85	0,07	1402,41	-0,01
Adding 5	50	50,9	37,51	-0,01	12,63	-0,14	50,00	0,90
Adding 6	1400	1401,4	1380,12	0,17	18,82	-0,64	1398,47	2,93

Results test 1, series 2

Designation	N <sub>2i</sub>	M <sub>2i</sub>	X20 <sub>2i</sub>	B20 <sub>2i</sub>	X5 <sub>2i</sub>	B5 <sub>2i</sub>	R <sub>2i</sub>	L <sub>2i</sub>	A <sub>2i</sub>	P <sub>2i</sub>	T <sub>2i</sub>	E <sub>2i</sub>
Description	Nominal mass	Mass flow meter reading	Gas in 20-L cylinder	Balance and weighing correction	Gas in 5-L cylinder	Balance and weighing correction	Gas remained in tubes	Gas lost to atmosphere	Air added to 5-L cylinder	Different pressure at start and stop	"True" mass	Mass flow meter error
unit	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]
Adding 1	50	50,4	49,68	-0,03	0,17	<0,01	0,00	0,01	0,006	-0,14	49,69	0,71
Adding 2	1400	1401,3	1394,65	0,11	6,05	<0,01	0,09	0,38	0,006	0,21	1401,49	-0,19
Adding 3	50	51,5	44,55	-0,02	6,03	<0,01	0,09	0,37	0,006	0,50	51,52	-0,02
Adding 4	1400	1402	1388,70	0,14	12,04	<0,01	0,18	0,76	0,006	-0,14	1401,67	0,33
Adding 5	50	50,7	37,58	-0,02	11,72	<0,01	0,18	0,74	0,006	0,64	50,83	-0,13
Adding 6	1400	1400,9	1380,83	0,14	17,71	<0,01	0,27	1,16	0,006	0,07	1400,18	0,72

Results test 1, series 3

Designation	N <sub>3i</sub>	M <sub>3i</sub>	X20 <sub>3i</sub>	B20 <sub>3i</sub>	C5 <sub>3i</sub>	P <sub>3i</sub>	T <sub>3i</sub>	E <sub>3i</sub>
Description	Nominal mass	Mass flow meter reading	Gas in 20-L cylinder	Balance and weighing correction	Calculated mass in tubing	Different pressure at start and stop	"True" mass	Mass flow meter error
unit	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]
Adding 1	50	51,6	51,28	-0,02	0,18	0,50	51,94	-0,34
Adding 2	1400	1401	1394,17	0,13	6,43	0,21	1400,94	0,06
Adding 3	50	53,1	46,26	-0,02	6,50	0,57	53,31	-0,21
Adding 4	1400	1401,5	1387,84	0,14	12,88	0,00	1400,86	0,64
Adding 5	50	52,9	39,73	-0,03	12,75	-0,07	52,38	0,52
Adding 6	1400	1400,7	1380,61	0,15	19,25	-0,07	1399,95	0,75

## Results test 2, series 1

Designation	LP <sub>ii</sub>	M <sub>ii</sub>	X20 <sub>ii</sub>	B20 <sub>ii</sub>	X5 <sub>ii</sub>	B5 <sub>ii</sub>	R <sub>ii</sub>	L <sub>ii</sub>	A <sub>ii</sub>	P <sub>ii</sub>	T <sub>ii</sub>	E <sub>ii</sub>
Description	Line pressure	Mass flow meter reading	Gas in 20-L cylinder	Balance and weighing correction	Gas in 5-L cylinder	Balance and weighing correction	Gas remained in tubing	Gas lost to atmosphere	Air added to 5-L cylinder	Different pressure at start and stop	"True" mass	Mass flow meter error
unit	[bar]	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]	[g]
Filling 1	41,3	500,5	497,68	0,06	1,98	<0,01	0,03	0,12	0,006	0,11	499,96	0,54
Filling 2	85,0	501,0	498,59	0,06	1,97	<0,01	0,03	0,12	0,006	0,14	500,91	0,09
Filling 3	126,5	500,1	497,99	0,06	2,03	<0,01	0,03	0,12	0,006	-0,07	500,15	-0,05
Filling 4	168,0	501,6	499,44	0,05	2,02	<0,01	0,03	0,12	0,006	-0,14	501,51	0,09
Filling 5	208,0	500,4	498,38	0,06	2,03	<0,01	0,03	0,12	0,006	0,28	500,89	-0,49
Filling 6	245,5	503,8	501,24	0,04	2,01	<0,01	0,03	0,12	0,006	-0,07	503,37	0,43

Results test 2, series 2

Designation	LP <sub>2i</sub>	M <sub>2i</sub>	X20 <sub>2i</sub>	B20 <sub>2i</sub>	R <sub>2i</sub>	P <sub>2i</sub>	T <sub>2i</sub>	E <sub>2i</sub>
Description	Line pressure	Mass flow meter reading	Gas in 20-L cylinder	Balance and weighing correction	Gas remained in tubing	Different pressure at start and stop	"True" mass	Mass flow meter error
unit	[bar]	[g]	[g]	[g]	[g]	[g]	[g]	[g]
Filling 1	38,5	500,6	497,72	0,05	2,15	0,07	499,99	0,61
Filling 2	82,5	500,4	497,72	0,07	2,15	-0,07	499,87	0,53
Filling 3	122,5	500,5	498,45	0,06	2,15	0,07	500,73	-0,23
Filling 4	168,0	500,4	498,16	0,06	2,15	-0,14	500,23	0,17
Filling 5	208,5	500,1	497,72	0,05	2,15	-0,07	499,85	0,25
Filling 6	247,5	502,1	499,66	0,05	2,15	-0,07	501,79	0,31

Results test 3, nitrogen

Designation	LP <sub>Ni</sub>	M <sub>Ni</sub>	X20 <sub>Ni</sub>	B20 <sub>Ni</sub>	R <sub>Ni</sub>	P <sub>Ni</sub>	T <sub>Ni</sub>	E <sub>Ni</sub>
Description	Line pressure	Mass flow meter reading	Gas in 20-L cylinder	Balance and weighing correction	Gas remained in tubing	Different pressure at start and stop	"True" mass	Mass flow meter error
unit	[bar]	[g]	[g]	[g]	[g]	[g]	[g]	[g]
Filling 1	208,5	500,1	497,72	0,05	2,15	-0,07	499,85	0,25
Filling 2	208,5	502,5	500,20	0,06	2,15	-0,07	502,34	0,16
Filling 3	208,5	502,7	500,45	0,06	2,15	-0,07	502,59	0,11

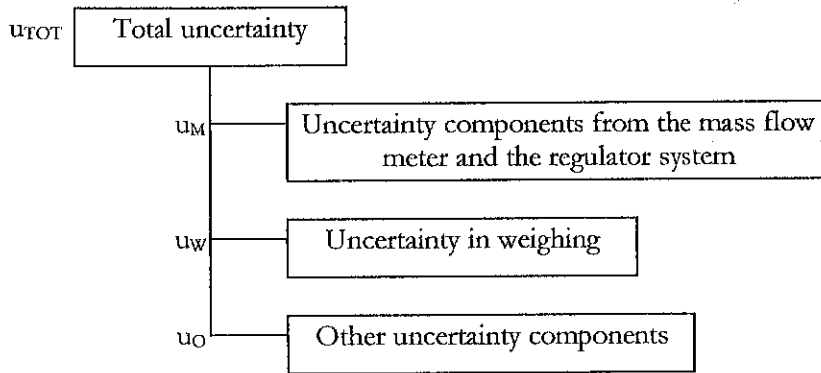
Results test 3, helium

Designation	LP <sub>Hi</sub>	M <sub>Hi</sub>	X20 <sub>Hi</sub>	B20 <sub>Hi</sub>	R <sub>Hi</sub>	P <sub>Hi</sub>	T <sub>Hi</sub>	E <sub>Hi</sub>
Description	Line pressure	Mass flow meter reading	Gas in 20-L cylinder	Balance and weighing correction	Gas remained in tubing	Different pressure at start and stop	"True" mass	Mass flow meter error
unit	[bar]	[g]	[g]	[g]	[g]	[g]	[g]	[g]
Filling 1	202,5	500,8	497,25	0,32	2,19	0,05	499,81	0,99
Filling 2	204,0	500,6	497,18	0,33	2,20	0,02	499,73	0,87
Filling 3	202,0	500,8	497,51	0,32	2,20	0,04	500,08	0,72

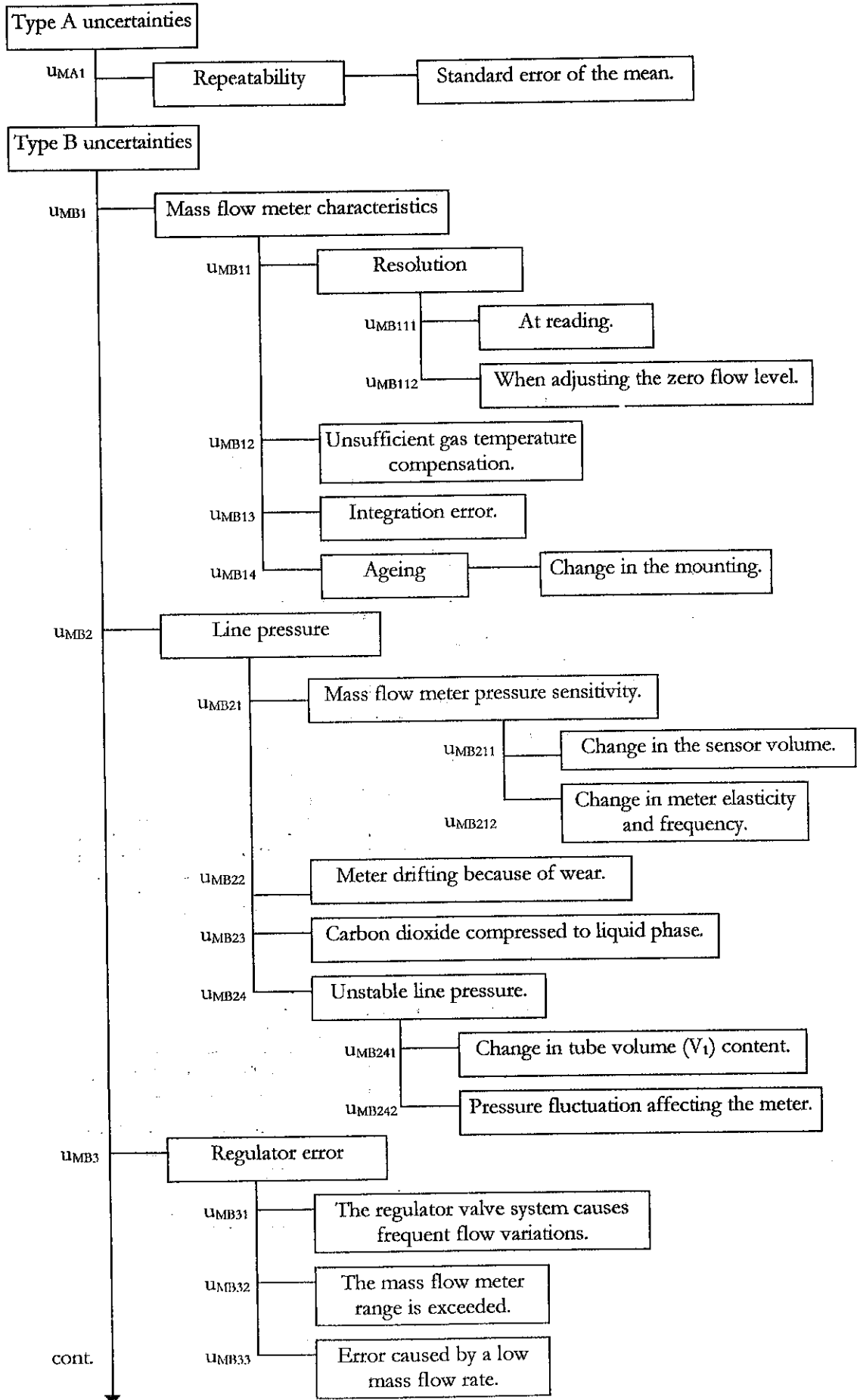
Results test 3, argon

Designation	LP <sub>Ai</sub>	M <sub>Ai</sub>	X20 <sub>Ai</sub>	B20 <sub>Ai</sub>	R <sub>Ai</sub>	P <sub>Ai</sub>	T <sub>Ai</sub>	E <sub>Ai</sub>
Description	Line pressure	Mass flow meter reading	Gas in 20-L cylinder	Balance and weighing correction	Gas remained in tubing	Different pressure at start and stop	"True" mass	Mass flow meter error
unit	[bar]	[g]	[g]	[g]	[g]	[g]	[g]	[g]
Filling 1	210,0	501,4	498,55	0,03	2,18	-0,20	500,56	0,84
Filling 2	209,5	500,6	498,20	0,04	2,15	-0,10	500,29	0,31
Filling 3	209,0	501,4	499,02	0,04	2,17	0,00	501,23	0,17

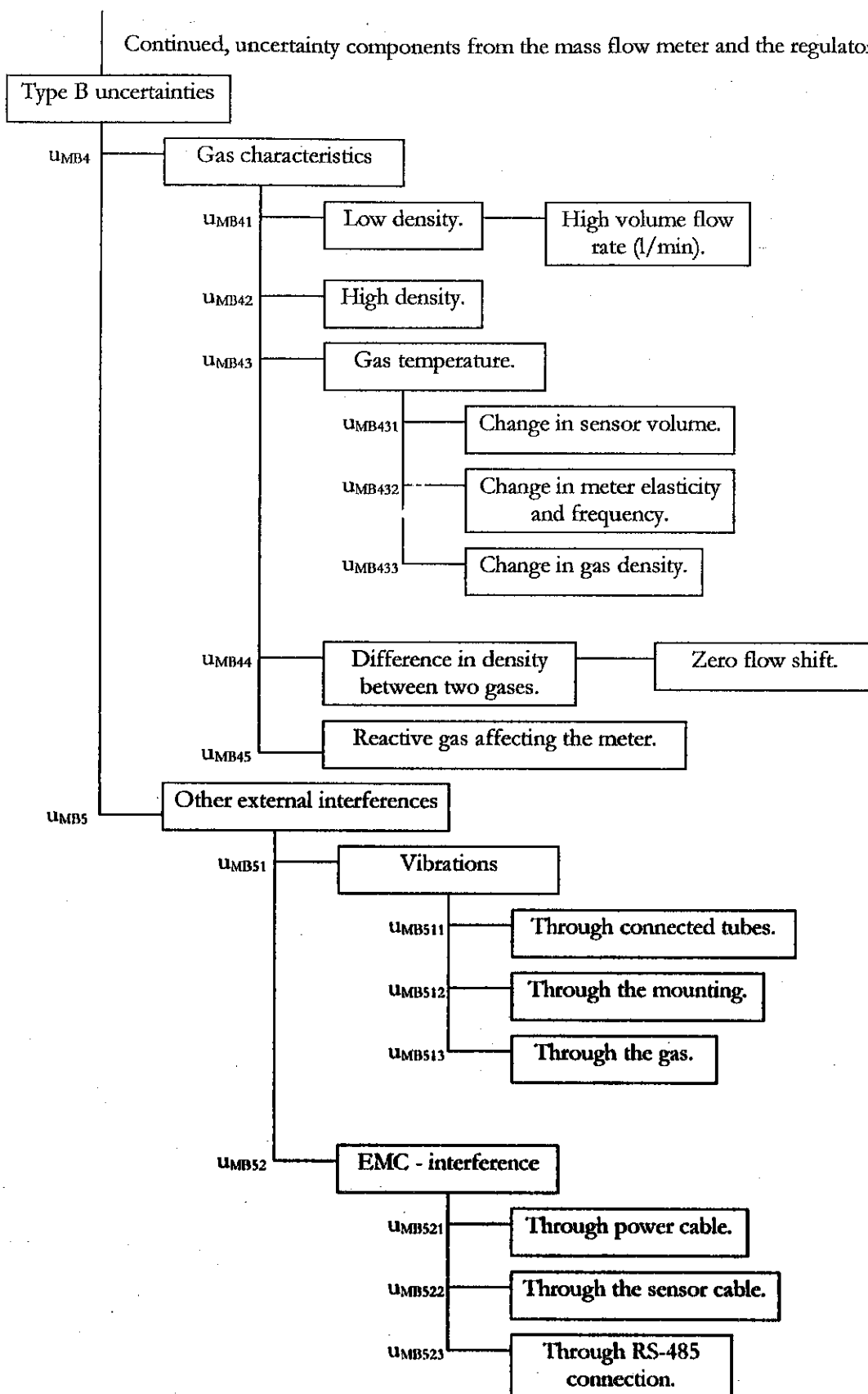
## Scheme of possible uncertainty and error contributions

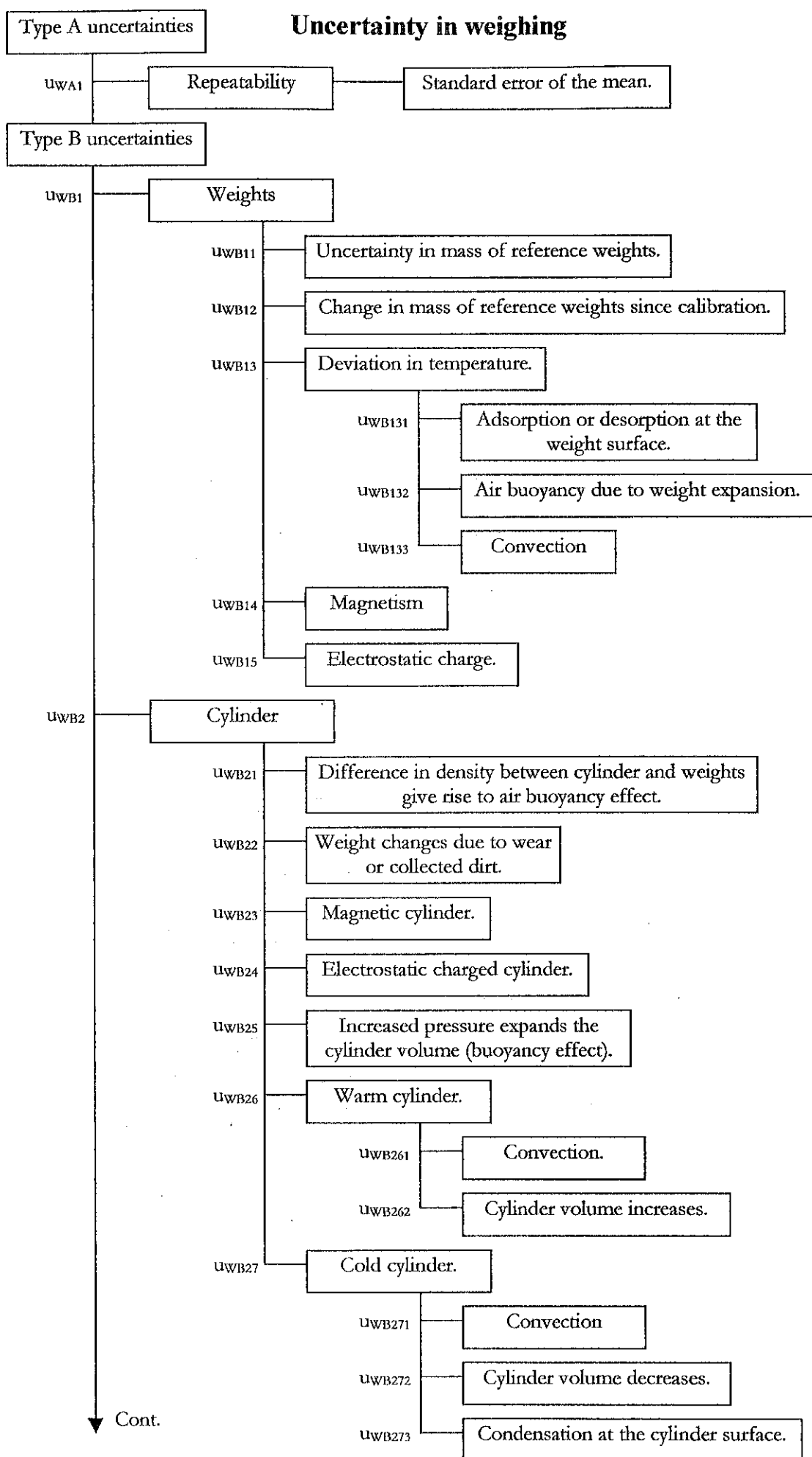


## Uncertainty components from the mass flow meter and the regulator system

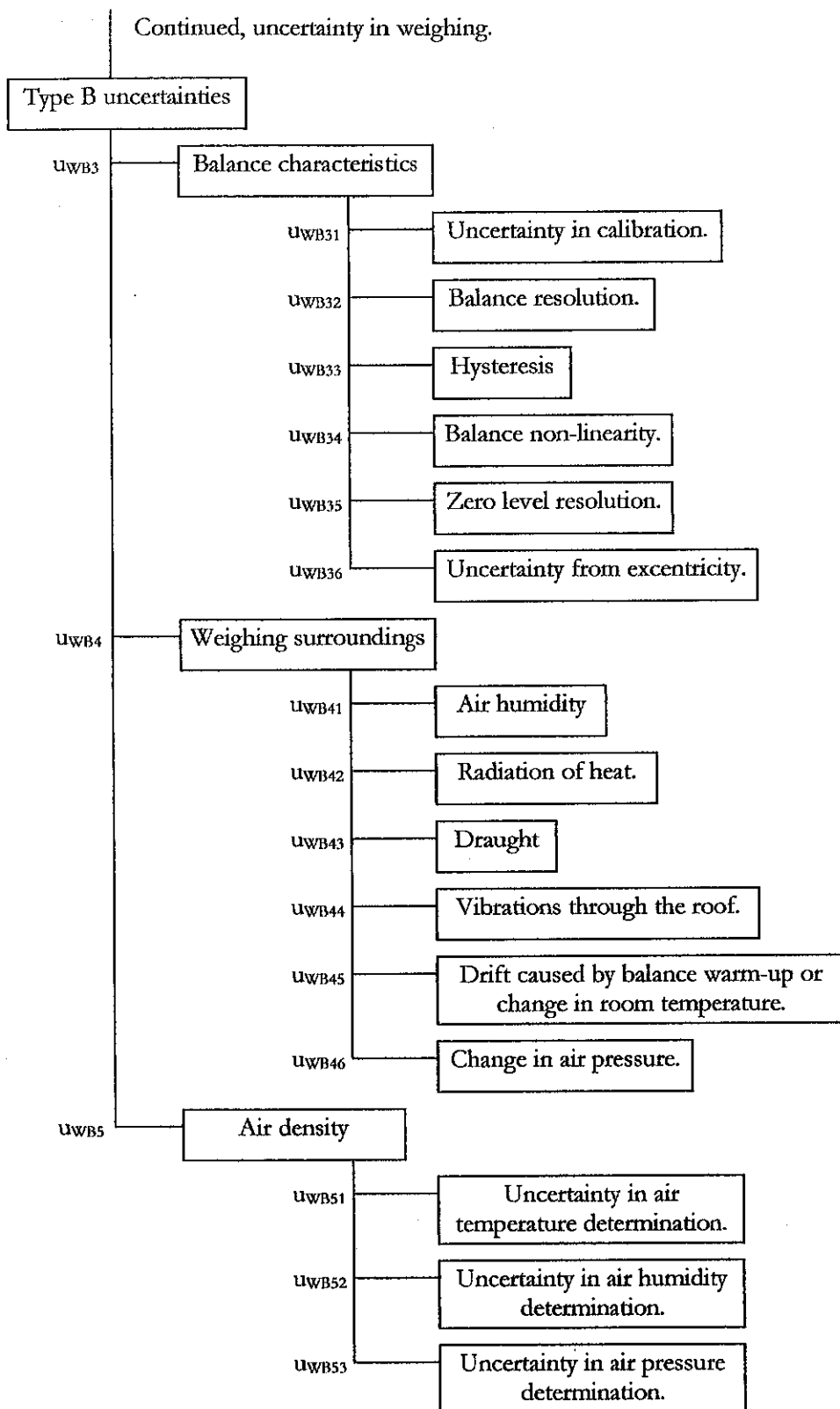


Continued, uncertainty components from the mass flow meter and the regulator system.





Continued, uncertainty in weighing.



## Other uncertainty components

