SARTORIUS

Application Highlight

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Weighing in an Inert Gas Atmosphere

Important Considerations When Transferring Your Sartorius Balances Into an Inert Atmosphere Gas Box and Weighing Samples

Abstract

The weighing performance of Sartorius balances was measured following their transfer into a common gas box flooded with argon atmosphere. This technical note summarizes results of the study, which confirmed the balances operated within specification, and provides recommendations for the transfer and operation of balances in inert atmosphere gas boxes.

Introduction

An inert gas box is a closed environment of approximately 1 m^3 volume that controls the internal atmosphere, preventing degradation of air-sensitive samples by unwanted chemical reactions such as oxidation or hydrolysis. The air in the box is replaced by displacement and dilution with a purified inert gas such as nitrogen or argon, which excludes oxygen and moisture. Inert gas boxes are typically operated at a higher pressure than the surrounding air, called "overpressure," to block oxygen and other contaminations from entering the chamber interior. Most boxes are made of stainless steel and are equipped with a front panel of transparent plastic. Two O-ring gloves are attached to the front and used to operate or manipulate objects inside the box.

General Considerations

Inert gas boxes are equipped with thick butyl gloves and for hygienic purposes, the user wears a pair of gloves made of cotton. This combination of gloves makes use of touch displays very difficult, and scroll and wipe operations can be extremely challenging; a capacitive stylus pen is therefore recommended. Alternatively, the balance display can be installed outside the inner gas box using an extension cable. A foot switch or motion sensor can also be helpful to operate automatic draft shields and initiate actions like printing and taring of the balance.

In general, use of ionizers is not necessary as the inert argon gas prevents the formation of charges on samples and vessels. In contrast, if balances are equipped with a build-in ionizer, it must not be used in an argon atmosphere. Furthermore, the power supply must not be installed in the box as it typically is not certified for operation in argon gas.

For manual note taking, use a pencil inside the inert gas box to avoid evaporation effects and contamination, which may influence measurement results.

A stable, vibration-free installation of the balance is essential. The higher the resolution of the balance, the more attention should be paid to the location of the gas inlet and outlet and the vibrations caused by the box. The balance should not be subject to the gas flow inside the box; ideally, the balance installation location is mechanically decoupled from the box.

With very low humidity, it is recommended to let the transferred balances equilibrate for at least 24 hours in the inert gas atmosphere to prevent measured values from drifting. Significant changes in the humidity can influence balance readings and should be avoided.

Transferring Equipment or Chemicals Into the Inert Gas Box

Since the gas box is flooded by an inert gas atmosphere, equipment and chemicals to be used in the box must be transferred through an air lock into the box interior. The air lock is located on the side of the inert gas box.

The volume in the air lock is evacuated by a rotary pump and then flooded with inert gas. By repeating the process, introduction of moisture or oxygen into the glovebox is minimized. The moisture level and oxygen concentration in the box are continuously monitored by sensors. During introduction of objects through the air lock, small amounts of air and moisture may enter the box. Therefore, a gas purification system is integrated to the inert gas box that cleans the entire atmosphere in the box by circulation. The gas cleaning system usually contains two cleaning stages—a BTS catalyst uses copper compounds that interact with O_2 to form copper oxide, which reacts and removes residual oxygen from the atmosphere. Removal of water is facilitated by a molecular sieve with a pore size of approximately 400 pm, which binds small molecules with high affinity. The oxygen and water levels in a well-maintained inert gas box are both below 1 ppm.

Test Specimens and the Environment

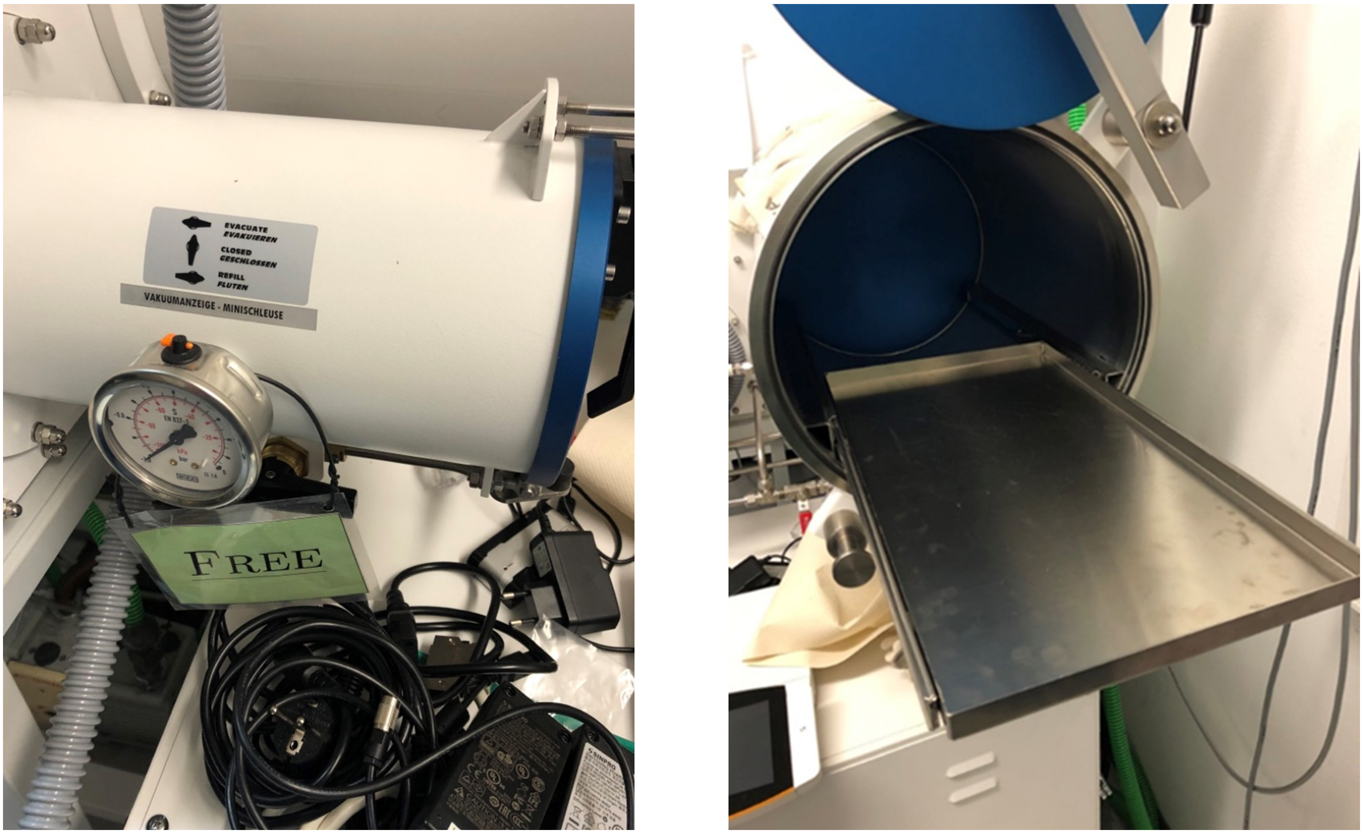
In this study, an MBRAUN inert gas workstation LABmaster Pro with UNIlab plus SP/DP gas purifier and MOD box MB20/MB200 G was installed at the Institute for Inorganic Chemistry, University of Göttingen, Germany.

Figure 1
Inert Gas Box MBRAUN LABmaster Pro

Note. Sartorius Cubis® MSA225S and Cubis® II MCA10.6 balances were transferred into the gas box and subjected to various measurements under the argon atmosphere.

Figure 2

A. *Small Air Lock With 150 mm Diameter* B. *Large Air Lock With 390 mm Diameter*



Note. Balances were transferred into the box via the large air lock and small accessories via the small air lock.

Transferring Balances Into the Inert Gas Box

The LABmaster pro is equipped with small and large air locks (antechambers) and pressure gauges. The workstation has a mini antechamber with a diameter of 150 mm for transferring small tools and samples into the box and a large antechamber with a diameter of 390 mm for transferring larger equipment. The box was flooded with argon 5.0 and operated under light overpressure of 20 mbar.

Table 1

	Balances	Accessories
Maximum underpressure in air lock	-400 mbar	-1,000 mbar
Evacuation pressure in air lock	600 mbar	Near to 0 mbar
Time for evacuation	1 min	1 min
Settling time	2 to 5 min	1 min
Time to flood air lock	1 min	1 min
Number of cycles	Minimum 10	Minimum 3
Time for transfer	Minimum 40 min	Minimum 9 min

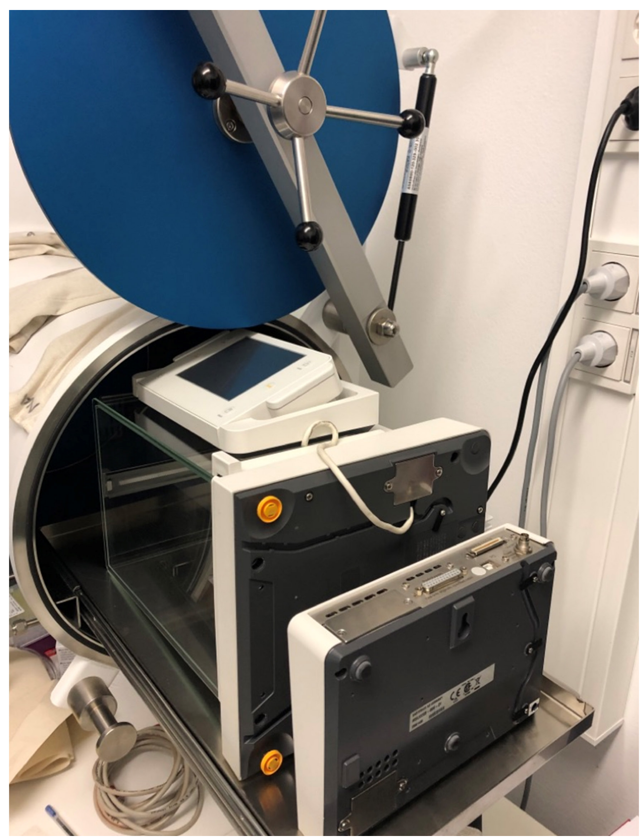
The air locks were repeatedly evacuated and flooded with argon during the transfer of the technical equipment and accessories. For the large air lock, the chamber was evacuated and flooded with argon at least 10 times; for the small air lock, at least 3 times. The minimum time needed for the transfer was 40 minutes for the large air lock and 9 minutes for the small air lock.

A critical factor is the settling time, which depends on the type, material, and shape of the item to be transferred into the box. To make sure the gas atmosphere is well mixed, and the gas enters the interior of the instruments, the settling time for balances was at least 5 minutes. For simple items like weights, cables, etc., a settling time of 1 minute was applied.

For transfer of balances, the pressure must not be too low in order to avoid damages to electronic parts. The underpressure in the air lock was not higher than -400 mbar, and therefore the resulting effective pressure not lower than 600 mbar. Because the air lock could not be evacuated to pressure values less than 600 mbar, the procedure was repeated at least 10 times to ensure saturation with argon and prevent oxygen and moisture from entering the box interior. For the small air lock, the number of cycles could be reduced to 3 since the volume could be completely evacuated and the effective pressure was near zero. The exchange of air against argon was much faster compared to the transfer of pressure-sensitive balances.

Figure 3
Transfer of Balance Model MCA10.6

Figure 4
Transfer of Balance Model MSA225



Weighing Performance

Prior to measurement in the argon atmosphere, the low humidity and density of argon must be considered; argon has a much lower oxygen and humidity compared to air.

Table 2

Gas	Argon 4.0 or Better (99.99%)	Air
Concentration O_2	< 5 ppm	209,430 ppm
Concentration H_2O	< 10 ppm	0–30,000 ppm
Density at 25° C	1.630 kg/m³	1.184 kg/m³
Density at 0° C	1.784 kg/m³	1.250 kg/m³

If the balance is not properly equilibrated following transfer into the inert gas box, the difference in humidity between air and argon can lead to drift of measurements. The release of moisture sticking to the surface of components can induce drift effects at the unloaded balance. To avoid taring before each measurement and compensate for possible drift, an equilibration time of at least 24 hours is highly recommended before beginning the weighing process.

It should be noted that the difference in density cannot be compensated by equilibration. Relative effects on the measured weight value can be excluded by the combination of internal adjustment and external calibration. Nevertheless, the much higher density of argon compared to air will lead to a significant buoyancy effect, which impacts the absolute weigh value. The buoyancy effect can be calculated by the following equation according to DIN EN ISO 1183:

$$m = m_w * \frac{1 - \frac{p_a}{p_c}}{1 - \frac{p_a}{p}}$$

m = Sample mass (true mass)
m_w = Balance reading (apparent mass)
p_a = Air density during weighing (kg/m^3)
P_c = Reference density (kg/m^3)
P = Sample density (kg/m^3)

Using the permissible density tolerances for weights according to OIML R111 and a typical air density value of 1.2 kg/m^3, the buoyancy effect leads to the following calculated values.

Table 3
Adjustment Error With Permissible Density Range for Weights According to OIML R111

Adjustment Weight	Minimum Permissible Density	Maximum Permissible Density
1 g	-0.0579 mg	-0.0479 mg
100 g	-5.797 mg	-5.702 mg

The adjustment error can be compensated by buoyancy correction. The Cubis® MSA and Cubis® II MCA balances used in this experiment offer software solutions for air buoyancy correction. The software relies on the formula shown above to calculate the buoyancy effect for any reading. The apparent mass is corrected by the buoyancy effect and the true mass is displayed.

In addition to the high density and low moisture content, argon also has a much lower heat conductivity than air.

Table 4
*Heat Conductivity W/(m*K)*

	Temperature	
	0° C	25° C
Air	0.0242	0.026
Argon	0.0179	0.0178

When subjected to argon atmosphere, the heat generated by the balances' electronics could potentially have an influence on the weighing results. The temperature inside the balances was monitored by internal sensors. When operated for 3 hours and at a temperature of approximately 28° C in the inert gas box, the temperature inside the housing of model MSA225 increased by a maximum of 2.07° C and model MCA10.6 by a maximum of 0.17° C. Balances are typically designed for use between 0° C and 40° C. The small temperature increase was compensated by taring and internal adjustment and did not cause the balances to operate out of specification. Only if the temperature in the inert box approaches 40° C must users take care not to operate the balances out of their specifications.

The balances transferred into the inert gas box and equilibrated to the ambient conditions were evaluated for long-term performance. To determine the influence of the lowered humidity and thermal conductivity and raised atmosphere density, absolute values such as the adjustment accuracy and correctness were measured in the argon atmosphere. Some factors, such as linearity and standard deviation, are relative values and, as expected, there were no differences between the values, whether they were measured in air or argon. The balance sensitivity was measured in experiments of 70 minutes (MCA10.6) or 80 minutes (MSA225) either with or without a load. The load was changed every minute. From the measurement values, the standard deviation and linearity were calculated.

Table 5
MSA225

	Without Load	With Load
Standard deviation	0.036 mg	0.019 mg
Linearity	0.05 mg	

Table 6
MCA10.6

	Without Load	With Load
Standard deviation	0.002 mg	0.0007 mg
Linearity	0.004 mg	

Note. For the Cubis® II MCA10.6 balance, the linearity deviation is specified with 0.004 mg tolerance and 0.003 mg for model MSA225 with ± 0.1 mg.

Results

The results of this study demonstrate that these balances operate within specifications in an argon atmosphere if:

- General considerations are followed
- The transfer is conducted as described above
- The equilibration time is sufficient
- Safety features like the internal adjustment are used
- The buoyancy effect is compensated

The most significant challenges observed in this study were the need to conduct experiments using the thick butyl gloves in combination with the limited space in the inert box. From the technical perspective, the influence of the argon atmosphere is manageable and reliable weighing results can be achieved.

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