

# A Pumping Method for Assessing Airtightness of Packs – Application to Heated Tobacco Products \*

by

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## SUMMARY

The airtightness of heated tobacco product (HTP) packs is a very important indicator for the product quality and is also of great importance during the conditioning process. A method for evaluation of the airtightness was developed based on the air pressure difference in a constant pumping configuration. The essential feature of this method is that the pressure difference between the inside and the outside of the HTP packs during the deflation process is used to characterize the sealing quality of HTP packs. The detailed setup, the principle as well as the determination procedure are described. The accuracy and the repeatability of the method were assessed, and the effect of airtightness on the conditioning process was also investigated. The developed method is proven to be reliable with a standard deviation less than 0.09 kPa and repeatability less than 0.30 kPa. In addition, it was found that, although the transmission of moisture between HTPs and atmosphere could not be entirely prevented by the packs, airtightness still plays a significant role during the conditioning process, especially if the airtightness was at a relatively low level (e.g., lower than 1.5 kPa under a pumping flow rate of 200 mL/min). The method provides a promising way to assess and

monitor the sealing quality of HTP packs, and it is suggested that the airtightness of the pack should not be lower than 2 kPa under a pumping flow rate of 200 mL/min. [Contrib. Tob. Nicotine Res. 32 (2023) 140–145]

## KEYWORDS

Heated tobacco product (HTP); HTP pack; airtightness; conditioning

## ZUSAMMENFASSUNG

Die Luftdichtheit der Verpackungen von Tabakerhitzern (HTP) ist ein sehr wichtiger Indikator für die Produktqualität und ist auch während des Konditionierungsprozesses von großer Bedeutung. Es wurde eine Methode zur Bewertung der Luftdichtheit entwickelt, die auf dem Luftdruckunterschied in einer konstanten Pumpkonfiguration basiert. Im Wesentlichen geht es bei dieser Methode darum, die während des Entlüftungsvorgangs entstehende Differenz zwischen dem Innen- und Außendruck einer HTP-Verpackung als Indikator für die Dichtleistung zu betrachten.

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Es werden der detaillierte Versuchsaufbau, das Prinzip und die Bestimmungsmethode beschrieben. Die Genauigkeit und Wiederholbarkeit der Methode wurden bewertet, und der Einfluss der Luftdichtheit auf den Konditionierungsprozess untersucht. Mit einer maximalen Standardabweichung von weniger als 0,9 kPa und einer Wiederholbarkeit von weniger als 0,30 kPa erwies sich die entwickelte Methode als zuverlässig. Außerdem wurde festgestellt, dass der Austausch von Feuchtigkeit durch die HTP-Verpackung zwar nicht vollständig verhindert werden kann, die Luftdichtheit während des Konditionierungsprozesses und insbesondere bei relativ niedrigen Werten (z.B. unter 1,5 kPa bei einer Pumpendurchflussrate von 200 mL/min) dennoch eine signifikante Rolle spielt. Die entwickelte Methode bietet eine vielversprechende Möglichkeit zur Evaluierung und Überwachung der Dichtleistung einer HTP-Verpackung. Es wird vorgeschlagen, dass die Luftdichtheit der Packung bei einer Pumpfließrate von 200 mL/min nicht unter 2 kPa liegen sollte. [Contrib. Tob. Nicotine Res. 32 (2023) 140–145]

## RESUME

L'étanchéité à l'air d'un paquet contenant un produit de tabac chauffé constitue un indicateur très pertinent de la qualité dudit produit et se révèle également d'une grande importance lors du processus de conditionnement. Une méthodologie de test de l'étanchéité à l'air fut mise au point sur le principe de la pression négative. En substance, cette méthodologie repose sur le constat que le différentiel créé lors de la mise sous vide entre la pression interne et la pression externe du paquet contenant un produit de tabac chauffé devrait être considéré comme un indicateur de la performance d'étanchéité pour les produits de tabac chauffé. Le protocole précis, à savoir le principe et la procédure de détermination, fut détaillé. La précision ainsi que la répétabilité de la méthode furent évaluées et les effets de l'étanchéité à l'air sur le processus de conditionnement furent aussi analysés. La méthodologie mise au point s'avéra être assez fiable par rapport à l'écart-type le plus élevé pointant à 0,085 et à la répétabilité relevée de 0,28. En outre, il apparut que même si l'échange de molécules d'eau ne pouvait être totalement bloqué par le paquet de produit de tabac chauffé, l'étanchéité à l'air jouait malgré tout un rôle significatif durant le processus de conditionnement, tout particulièrement à un niveau relativement bas (inférieur, par exemple, à 1,5 kPa). La méthodologie mise au point dégage une piste prometteuse en vue de l'évaluation et du contrôle de la performance d'étanchéité d'un paquet de produit de tabac chauffé; suggestion est faite de porter l'étanchéité à l'air du paquet à une valeur équivalente ou supérieure à 2 kPa. [Contrib. Tob. Nicotine Res. 32 (2023) 140–145]

## 1. INTRODUCTION

Heated tobacco products (HTPs), also known as heat-not-burn products, are believed to be a modified-risk tobacco product (1–3), since the levels of harmful chemicals in the generated aerosols are significantly reduced compared to

cigarette smoke. However, due to the special product characteristics (i.e., presence of considerable amounts of glycerol and propylene glycol within the processed tobacco), HTPs are much more likely affected by the ambient relative humidity, leading to an influence on the results of determined aerosol collected mass (ACM). In addition, unlike conventional cigarettes, packs are required to be unopened during the conditioning of HTPs (4, 5). Therefore, the airtightness of the HTP packs plays a more significant role than it does on cigarette packs, and thus there is a need to be able to measure this important property.

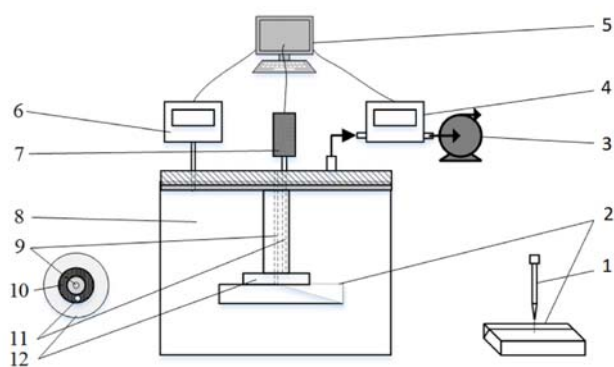
Assessment of the airtightness of food packaging and of packaging in the pharmaceutical industry was thoroughly investigated (6–10). Several methods were applied, including an inflation method, a water immersion method, and a differential-pressure method. However, most of the developed methods can only be used qualitatively, and are not suitable for the packaging techniques used in the cigarette industry. In addition, a few studies have been conducted on the airtightness of cigarette packs (11–13). So far, it seems that no investigation has been published on how to evaluate the airtightness of HTP packs.

Therefore, in this paper, in order to assess the airtightness of HTP packs, a method based on the air pressure difference in a constant pumping configuration was applied. The detailed setup, the principle as well as the determination procedure will be described. Furthermore, the precision of the method and the effect of airtightness on the conditioning of HTPs were investigated. Besides, a minimum requirement for the airtightness of HTP packs are suggested based on the conditioning experiments using the proposed method.

## 2. MATERIALS AND PRINCIPLE

### 2.1 Setup and parameters

The experimental setup for the assessment of airtightness for HTP packs used in this work is shown in Figure 1. The setup includes a program control system (controlling the procedure of assessment, including the punching position, pumping flow rate, sucking the pack and reading the differential pressure gauge), a punching device, a testing chamber (cylinder shape: 130 mm diameter and 90 mm height) as well as a sucker (20 mm diameter, with two holes connecting the ventilation channel and the vacuum channel, respectively) connected to the bottom of the chamber's lid. The chamber's lid was used to seal the chamber with an O-ring. The diameter of punching device was set between 2 mm and 3 mm and the inner diameter of the ventilation channel (3 mm diameter and 25 mm length) should be not smaller than the diameter of the punching device. Moreover, the flow rate of deflation pump was set to be 200 mL/min with a tolerance of 5 mL/min. The range of differential pressure gauge was (0–10) kPa with a resolution of 0.01 kPa and an accuracy of 0.02 kPa. The range could be adjusted based on the real test situation. For clarification, all assessments were conducted in the environment specified in ISO 3402 (14).



**Figure 1: Structure of an airtightness assessing system.**

1) punching device; 2) pack of HTPs; 3) deflation pump; 4) gas flowmeter; 5) program control system; 6) differential pressure gauge; 7) vacuum generator; 8) testing chamber; 9) ventilation channel; 10) vacuum area; 11) vacuum channel; 12) sucker.

## 2.2 Principle

The HTP pack was punched to create a ventilation hole and sealed tightly by a sucker mounted on the inside of the testing chamber lid connected to a vacuum generator. The inside of the pack was connected to the outside atmosphere via the punched hole and the ventilation channel. Then the chamber was sealed and continuously deflated at a stable flow rate of 200 mL/min, until the pressure difference between the inside of the chamber and atmosphere maintained steady for a certain period of time. The final pressure difference was recorded as a reflection of the airtightness of the HTP pack. Higher pressure difference indicates a better airtightness of the HTP pack.

## 3. PROCEDURE

The checking the absence of leakage and the calibration of pressure were conducted before assessment of airtightness. For checking the absence of leakage, the chamber was sealed and evacuated with an air flow of 200 mL/min. When the pressure difference between inside and outside of the chamber reached 10 kPa, the evacuation process was stopped. It was considered as no leakage as long as less than 0.01 kPa of pressure difference was detected for 3 s. For the pressure calibration, the sealed chamber was gradually evacuated, during which the differential pressure gauge for assessment was calibrated using a specially certified pressure gauge at 2 kPa, 4 kPa, 6 kPa, 8 kPa and 10 kPa, respectively. The difference between the two pressure gauges should be less than 0.01 kPa.

The dimensional information of the HTP pack was previously provided to the system before initiating the determination process. This was done to make sure that the HTP pack was punched on the correct position (geometric center of the pack), making the method suitable for different types of HTP packs.

After submitting the size information, the pack was punched to create a ventilation hole for connecting the inside of the pack to the ambient atmosphere. Then the punched pack was sealed tightly by a sucker mounted on

the inside of the chamber's lid with a vacuum generator. The ventilation channel of the sucker was directly connected to the hole of the pack.

Subsequently, the testing chamber was sealed with a gasket and a deflation process was applied with a continuous air flow of 200 mL/min. The pressure difference between the inside of the chamber and the ambient atmosphere was constantly measured by the differential pressure gauge (resolution of 0.01 kPa) every 0.1 s. The pressure difference was recorded until less than 0.01 kPa of pressure difference was detected for 3 s and then considered as the final result. All obtained results were expressed in kPa. Then the chamber was opened and the pack was removed from the sucker.

## 4. RESULTS AND DISCUSSIONS

### 4.1 Precision

The accuracy of this method was assessed using the transfer standard of permeable disks with a series of pressure differences from 0.1 kPa to 8.1 kPa, which was similar to the one used for air permeability measurement according to ISO 2965 (15). Each disk was measured 10 times by a single operator with the same equipment. All procedures were included except for the punching step. The results are given in Table 1. As can be observed, the highest standard deviation of 0.025 kPa and repeatability of 0.07 kPa was obtained for the disk of lowest permeability (corresponding to a pressure difference of 8.08 kPa on average). Furthermore, three different HTP pack samples were tested, using two randomly selected packs per sample. Each individual pack was continuously tested 10 times, and the results are shown in Table 2. All procedures were applied for the repeatability tests, including the punching step. It can be seen that the standard deviation ranges from 0.019 kPa to 0.085 kPa, while the repeatability is from 0.06 kPa to 0.28 kPa. These results demonstrate that the developed method is valid to assess the airtightness of the pack for HTPs products.

**Table 1. Accuracy results obtained with air permeable disks.**

| Disk                 | 1<br>(kPa) | 2<br>(kPa) | 3<br>(kPa) | 4<br>(kPa) | 5<br>(kPa) |
|----------------------|------------|------------|------------|------------|------------|
| 1                    | 0.07       | 1.11       | 3.25       | 5.13       | 8.04       |
| 2                    | 0.08       | 1.11       | 3.27       | 5.13       | 8.05       |
| 3                    | 0.07       | 1.11       | 3.27       | 5.14       | 8.06       |
| 4                    | 0.06       | 1.11       | 3.27       | 5.13       | 8.07       |
| 5                    | 0.07       | 1.11       | 3.27       | 5.13       | 8.08       |
| 6                    | 0.05       | 1.12       | 3.27       | 5.13       | 8.06       |
| 7                    | 0.07       | 1.11       | 3.27       | 5.14       | 8.08       |
| 8                    | 0.07       | 1.11       | 3.27       | 5.14       | 8.10       |
| 9                    | 0.07       | 1.11       | 3.27       | 5.12       | 8.11       |
| 10                   | 0.07       | 1.12       | 3.26       | 5.14       | 8.11       |
| Mean                 | 0.07       | 1.11       | 3.27       | 5.13       | 8.08       |
| SD                   | 0.008      | 0.004      | 0.007      | 0.011      | 0.025      |
| <i>r</i>             | 0.030      | 0.010      | 0.020      | 0.040      | 0.070      |
| <i>c<sub>v</sub></i> | 0.118      | 0.004      | 0.002      | 0.002      | 0.003      |

SD: standard deviation; *r*: repeatability; *c<sub>v</sub>*: coefficient of variation.

**Table 2. Results obtained with HTP packs.**

| HTP packs            | Sample C1<br>(kPa) | Sample C2<br>(kPa) | Sample M1<br>(kPa) | Sample M2<br>(kPa) | Sample T1<br>(kPa) | Sample T2<br>(kPa) |
|----------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| 1                    | 6.26               | 3.56               | 1.40               | 2.33               | 4.20               | 3.69               |
| 2                    | 6.14               | 3.56               | 1.39               | 2.35               | 4.18               | 3.54               |
| 3                    | 6.15               | 3.55               | 1.37               | 2.37               | 4.18               | 3.54               |
| 4                    | 6.06               | 3.54               | 1.40               | 2.39               | 4.17               | 3.57               |
| 5                    | 6.04               | 3.54               | 1.40               | 2.40               | 4.21               | 3.46               |
| 6                    | 6.03               | 3.52               | 1.40               | 2.42               | 4.30               | 3.46               |
| 7                    | 6.04               | 3.53               | 1.39               | 2.44               | 4.33               | 3.64               |
| 8                    | 6.01               | 3.53               | 1.42               | 2.42               | 4.34               | 3.57               |
| 9                    | 5.98               | 3.52               | 1.33               | 2.44               | 4.38               | 3.51               |
| 10                   | 6.02               | 3.50               | 1.36               | 2.45               | 4.38               | 3.42               |
| Mean                 | 6.07               | 3.54               | 1.39               | 2.40               | 4.27               | 3.54               |
| SD                   | 0.085              | 0.019              | 0.026              | 0.041              | 0.087              | 0.083              |
| <i>r</i>             | 0.280              | 0.060              | 0.090              | 0.120              | 0.210              | 0.270              |
| <i>c<sub>v</sub></i> | 0.014              | 0.005              | 0.019              | 0.017              | 0.020              | 0.023              |

SD: standard deviation; *r*: repeatability; *c<sub>v</sub>*: coefficient of variation.

#### 4.2 Effect of airtightness on the conditioning of HTPs

The effect of airtightness on the conditioning process was also investigated by monitoring the mass change during a conditioning process. The same HTP samples (Sample C, Sample M and Sample T) were selected for testing, and the conditioning duration was set to 10 days. The test atmosphere was specified as in ISO 3402 (14). The mass of each individual pack was recorded every 4 min, and the airtightness of each pack was assessed after the conditioning process. The airtightness and the mass increase of samples C, M and T are shown in Figure 2. The sample mass increase for the tested samples after 10 days of conditioning is shown in Figure 3. As shown in Figure 2, a continuous increase of sample mass was observed for all tested samples during the 10 days of conditioning. This suggests that, no matter how well the HTP packs are sealed, the transmission of moisture between HTPs and atmosphere cannot be entirely prevented by the packaging under such conditions. This might be attributed to the presence of considerable amounts of glycerol and propylene glycol in the tobacco substrate, which are highly hydrophilic. Furthermore, as can be seen from Figure 2a, when the airtightness was higher than 2.48 kPa, similar gradients of mass increase were observed. After 10 days of conditioning, a mass increase of approximately 0.023 g was found for the sample C with airtightness of 2.48 kPa, 3.65 kPa, 6.20 kPa and 7.37 kPa (Figure 2a). On the other hand, when the airtightness was at a relatively low level (i.e., 0.12 kPa, 0.63 kPa and 1.24 kPa), a severe raise was shown. The mass increase of 0.035 g, 0.041 g and 0.081 g was observed for the sample with the airtightness of 1.23 kPa, 0.63 kPa and 0.12 kPa, respectively (Figure 2a). Moreover, similar results were observed for Sample M and Sample T, respectively (Figures 1b, 1c and Figures 2b, 2c). As long as the pressure difference during airtightness assessment was higher than 2 kPa, no obvious difference of mass increase was detected when improving the airtightness of the pack. While the airtightness was lower than 1.5 kPa, poorer airtightness led to more rapid mass increase during the

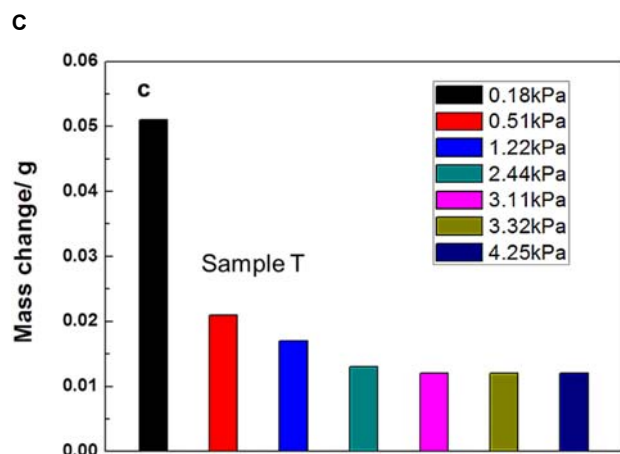
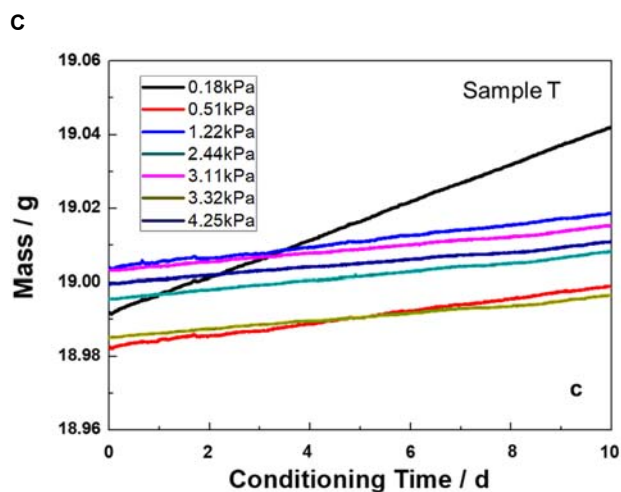
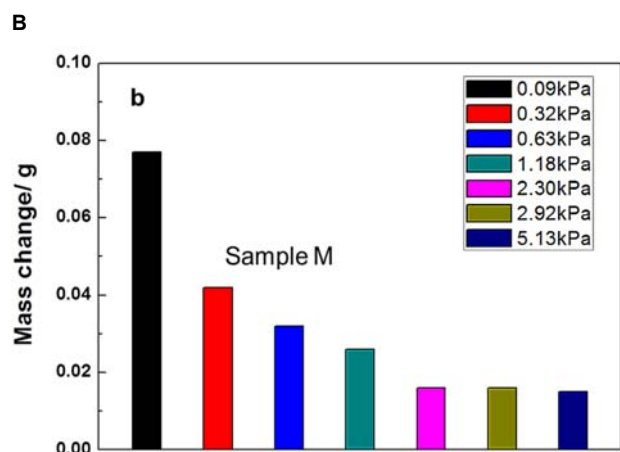
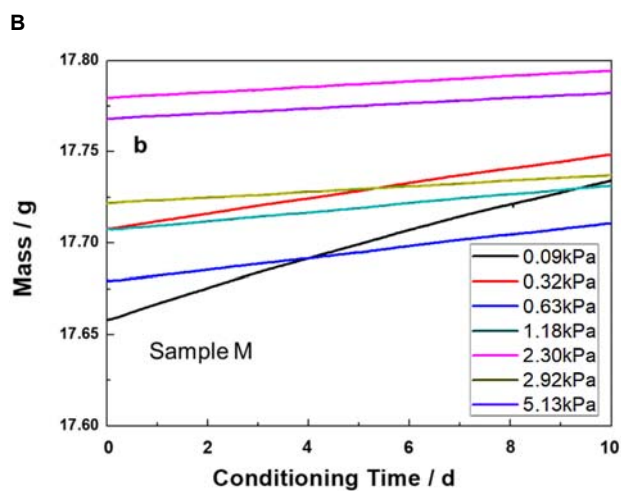
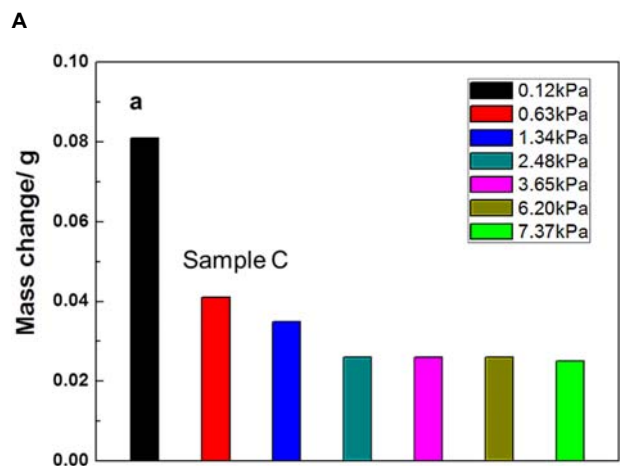
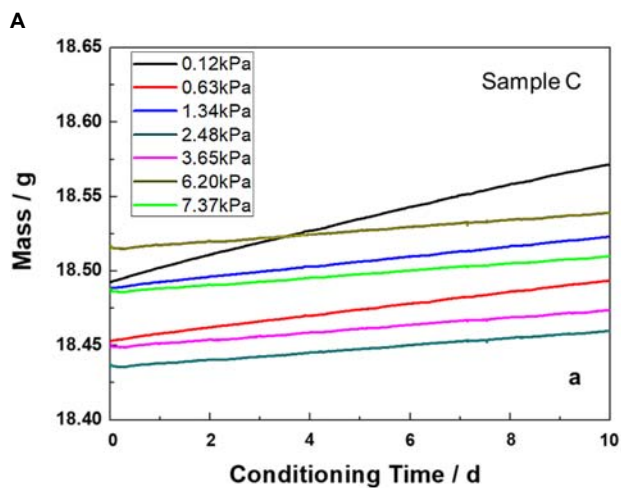
10 days of conditioning. It is important to mention that the increase of the sample mass was mainly attributed to the adsorption of water, which could influence the result of the determined aerosol collected mass (ACM). Therefore, it is deemed that the airtightness of HTPs' pack plays a significant role during the conditioning process, for which certain level of quality, i.e., no lower than 2 kPa, is recommended under the conditions of the test.

#### 5. CONCLUSION

A method for assessing the airtightness was developed based on the air pressure difference in a constant pumping configuration. The main idea of this method is that the internal and external pressure difference of HTPs' pack during the deflation process could be considered to reflect its sealing performance of HTPs. The detailed setup, principle as well as the testing procedure was described. The accuracy as well as the repeatability of the method was assessed, and the effect of airtightness on the conditioning process was also investigated. The developed method is proven to be reliable with respect to the highest standard deviation of 0.085 and repeatability of 0.28. In addition, it was found that, despite the exchange of water molecules cannot be entirely prevented by the package of HTPs, airtightness still plays a significant role on the reduction of moisture transmission during the conditioning process, especially at the relatively low level (e.g., lower than 1.5 kPa). The developed method provides a promising way to assess and monitor the sealing quality of HTP packs, and it is recommended that pressure difference should be higher than 2 kPa under the conditions of the test.

#### SUPPLEMENTARY INFORMATION

This publication contains supplementary material available at <https://sciendo.com/article/10.2478/cttr-2023-0017>.



**Figure 2: The airtightness as well as the sample mass during 10 days of conditioning at 22 °C, 60% relative humidity.**  
A) Sample C; B) Sample M; C) Sample T.

**Figure 3: The airtightness as well as the mass change after 10 days of conditioning at 22 °C, 60% relative humidity.**  
A) Sample C; B) Sample M; C) Sample T.

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