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Frost-Point Measurement Error Due to a Leak in a Sampling Line

Martti Heinonen · Martin Vilbaste

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Abstract When measuring low dew-point temperatures, a leak from the sampling tubing to ambient may cause a significant error due to back-diffusion of water vapor. In the work reported in this article, the significance of this error was studied experimentally and theoretically. The effects of leaking VCR® and Swagelok® connectors were studied experimentally and analyzed by comparing the results to the outcomes of experiments with actual leak holes. Theoretical calculations with a simplified combined convection–diffusion model were used to evaluate the results. Calculations were successfully carried out to predict the minimum leak flow rate required to prevent any water vapor from back-diffusing into the tubing. It was concluded that, in many cases, maintaining gauge pressure in a sampling line prevents penetration of water vapor into the line. Tiny leaks, however, can be a route for harmful back-diffusing of water vapor. VCR connectors were shown to be more sensitive to leaks compared with Swagelok connectors.

Keywords Back-diffusion · Dew-point temperature · Leak

1 Introduction

A sampling system is usually needed in dew-point temperature measurements. In most chilled-mirror hygrometers, a continuous sampling gas flow passes through the sensor unit. If the gas pressure in the sample tubing is lower than the ambient air pressure, special attention must be focused on preventing leaks. Due to the pressure difference,

M. Heinonen (✉)
Centre for Metrology and Accreditation (MIKES), Tekniikantie 1, P.O. Box 9, 02150 Espoo, Finland
e-mail: martti.heinonen@mikes.fi

M. Vilbaste
University of Tartu, Tartu, Estonia

a leak allows ambient air to enter the sampling tube, causing a direct effect on the measurement result. If the sampling pressure, however, is higher than the ambient air pressure, convective flow at the leak is outwards and the sampling gas escapes the tubing. When measuring dew-point temperatures higher than the ambient humidity level, a small leak in the sampling line causes no detectable error in the measurements if the effect of the pressure drop in the line is taken into account. In a lower humidity range, however, back-diffusion of water vapor may cause an error despite the higher sample gas pressure because the water-vapor pressure in the line is lower than ambient pressure.

Special attention has often been focused on preventing any leakage in sampling lines when measuring low dew-point temperatures [1,2]. However, in earlier studies with chilled-mirror hygrometers [3] and when developing a low frost-point generator [4,5], the authors noticed that the back-diffusion effect of a leak was often negligible if the sample gas pressure was higher than the ambient pressure.

Back-diffusion of water vapor in tubes and pipes has been investigated theoretically and experimentally by several researchers [6–8]. They have searched for optimal combinations of the gas-flow rate, tube length, and diameter when constructing gas delivery systems. These results indicate that water vapor cannot back-diffuse into the system if the bulk flow rate is sufficiently large.

In the work reported in this article, experimental and theoretical approaches were applied in investigating the back-diffusion effect of leaks. In the experiments, the effect of leaks in Swagelok® and VCR® connectors and in a tube wall through artificial round holes were studied. In the theoretical work, a simplified theoretical model was developed to predict the minimum leak flow rate required to prevent water vapor from back-diffusing into the tubing.

2 Leak Through a Drilled Hole

2.1 Investigation Method

When considering gas flow and water-vapor diffusion, the geometry of the flow path is one of the most important factors to be studied. If a leak takes place in an actual tube connector, the flow path of leaking gas is usually unknown and difficult to study. Therefore, the work reported in this article was mainly focused on leaks through holes with different diameters drilled in sampling tubes. One hole was drilled into each tube that could be placed between a chilled-mirror hygrometer and a dew-point generator. The hole was drilled with a laser-based system (see Fig. 1). The stainless-steel tubes with an inner diameter of 4 mm have an electropolished inner surface. Four tubes were studied with hole diameters of 0.05, 0.1, 0.2, and 0.5 mm.

Air with a well-controlled dew-point temperature was supplied by the dew/frost-point generator MDFG [4,5]. An MBW373LX chilled-mirror hygrometer was used to measure the dew-point temperature of air passed through a tube with a leak hole (see Fig. 2). Ambient conditions were measured with capacitive hygrometers calibrated at the MIKES. A PTB220 digital barometer was used to determine the pressure difference between the MDFG saturator and the MBW hygrometer. By analyzing the

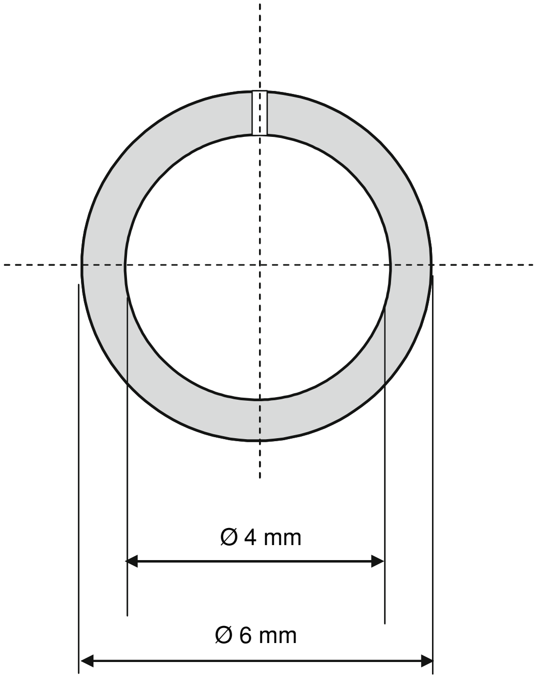


Fig. 1 Cross section of a tube with a hole drilled through its wall

response of the hygrometer to a slightly fluctuating saturation temperature, it was shown that the hygrometer reading follows well even small changes in the actual dew-point temperature (less than 0.05°C).

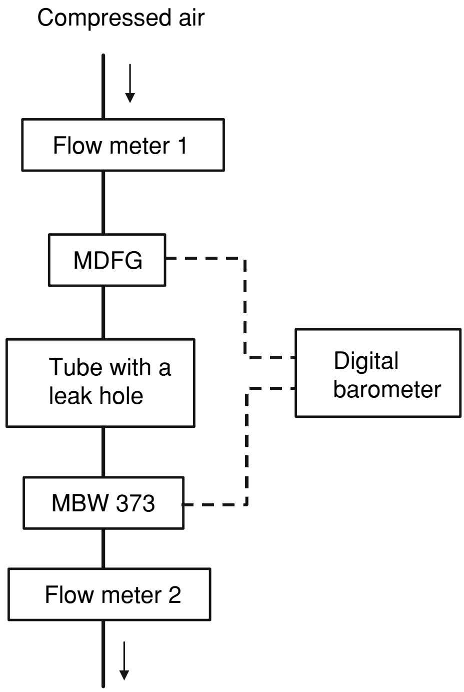
Characteristic leak flow rates through the drilled holes were determined experimentally in the MIKES Flow Laboratory using laminar-flow-element-based calibration equipment. The leak flow rate versus the difference between the sample gas and ambient pressure is shown in Fig. 3. Results for the 0.5 mm hole are not in the figure because the leak flow rate was found to be 400–820 $ml \cdot min^{-1}$.

The measurement setup shown in Fig. 2 was used experimentally to investigate the leak effect. The effect was also studied theoretically using a simplified mass transfer analysis as described in the next section.

2.2 Theoretical Prediction for a Critical Leak Flow Rate

Let us consider a hole drilled into a tube wall. The sample air pressure in the tube is slightly higher than the ambient pressure, causing a leak flow through the hole to ambient. In the case of a relatively small leak, the flow is assumed to be laminar in the hole because the Reynolds number is smaller than 1,000. Although it is well known that the flow profile of a fully developed flow in a hole or tube is parabolic, we carried out the calculation assuming a flat (i.e., uniform) profile. The velocity ($v \geq 0$) is calculated from the measured leak volume flow rate (V_L). It can be shown that the actual maximum flow velocity in the hole is twice the mean flow velocity [9].

Fig. 2 Block diagram of the measurement setup including the dew-point generator MDFG and the chilled-mirror hygrometer MBW 373



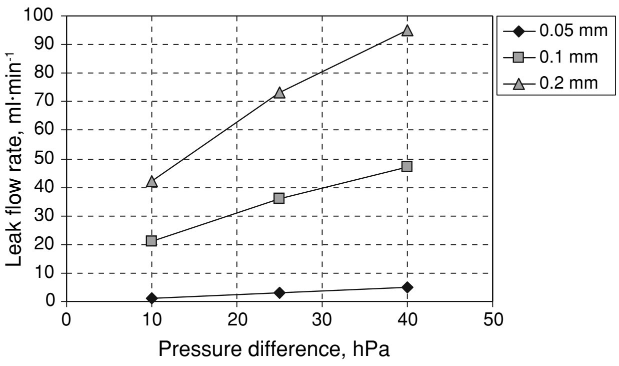


Fig. 3 Measured leak flow rates for leak holes of 0.05, 0.1, and 0.2 mm

Applying Ficks's law of diffusion, we can derive an equation for the molar flux of water vapor in the hole (N_w);

$$N_\mathrm{w} = -D\frac{\partial C_\mathrm{w}}{\partial y} - vC_\mathrm{w} \quad (1)$$

where C_w, D, and y are the molar concentration of water, diffusion coefficient for water vapor in air, and coordinate on the axis pointing from the ambient into the tube (see Fig. 4), respectively. It is assumed that the water-vapor concentration at $y = 0$

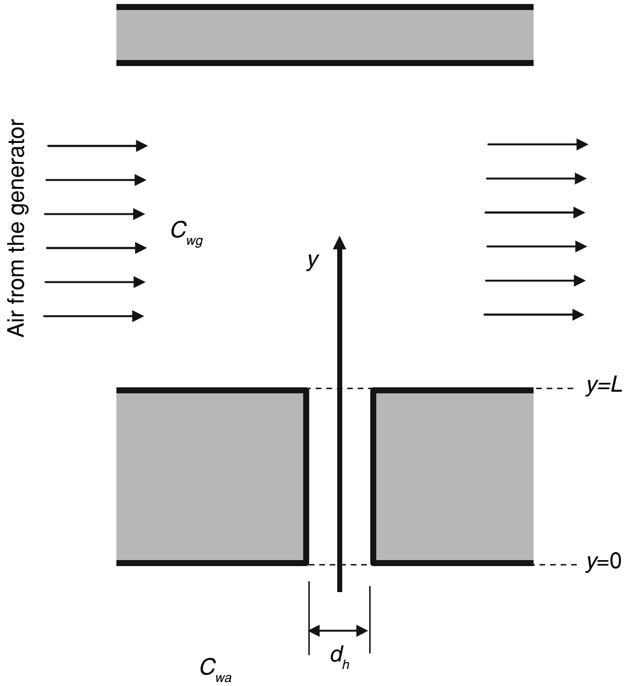


Fig. 4 Drawing of the leaking hole

equals the ambient concentration , i.e., $C_{\mathrm{w}}(0) = C_{\mathrm{wa}}$. On the inner surface ($y = L$), the concentration is assumed to be equal to the concentration of gas from the dew-point generator, i.e., $C_{\mathrm{w}}(L) = C_{\mathrm{wg}}$.

Due to conservation of mass, the diffusion rate is constant along the axis. Thus, the motion of water vapor in the hole can be described by the following differential equation:

$$D\frac{\partial^2 C_{\mathrm{w}}}{\partial y^2} + v\frac{\partial C_{\mathrm{w}}}{\partial y} = 0 \tag{2}$$

This can be solved analytically with the given boundary conditions. Combining the solution with Eq. 1, we get

$$m_{\mathrm{w}} = \frac{1}{4}\pi\ d_{\mathrm{h}}^2 N_{\mathrm{w}} M_{\mathrm{w}} = V_{\mathrm{L}} M_{\mathrm{w}} \left[\frac{C_{\mathrm{wL}} - C_{\mathrm{wa}}}{\exp\left(\frac{-4\ V_{\mathrm{L}} L}{\pi\ d_{\mathrm{h}}^2 D}\right) - 1} - C_{\mathrm{wa}} \right] \tag{3}$$

where m_{w} and d_{h} are the mass flow rate of water through the hole and the hole diameter, respectively. By setting $N_{\mathrm{w}} = 0$, we can estimate the minimum flow rate required to

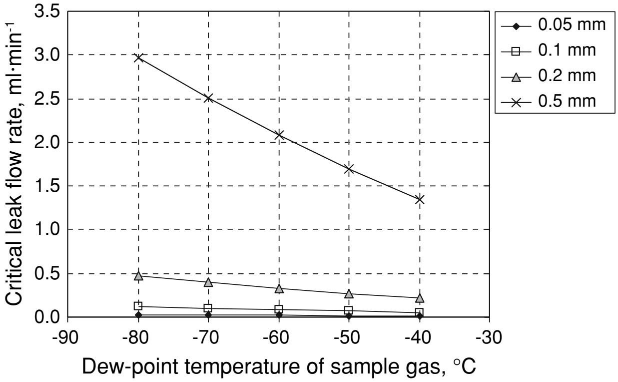


Fig. 5 Calculated critical leak flow-rate values for each hole with dew-point temperatures of −80°C to −40°C. Dew-point temperature of ambient air at room temperature was 9°C

prevent the back-diffusion of water into the tube;

$$V_{\mathrm{c}} = \frac{-\pi \ d_{\mathrm{h}}^{2} D}{4L} \ln\left(\frac{C_{\mathrm{wL}}}{C_{\mathrm{wa}}}\right) \tag{4}$$

In this article, the minimum flow rate is called the critical leak flow rate. Equation 4 was applied to calculate the critical flow-rate values for the leak holes described above. Calculations were carried out for different dew-point temperatures of the sample gas. Normal laboratory ambient conditions were assumed for the surrounding air (p = 101, 600 Pa, $t_{\mathrm{d}} = 9$ °C, $t = 23$°C). The results in Fig. 5 show that the critical leak flow rate is always less than 3 ml · min^{-1} and mostly less than 0.5 ml · min^{-1}. If we compare these results with those in Fig. 3, we can conclude that water vapor can back-diffuse into the sampling tube only if the sample gas pressure is almost equal to the ambient pressure. This is, however, rarely the case because of flow resistance in the dew-point hygrometer and in the flow meter/control after the hygrometer.

2.3 Evaluating the Critical Flow-Rate Calculations

In order to evaluate the results presented in the previous section, experiments with the tube with the 0.5 mm leak hole were carried out using the setup shown in Fig. 2. Since the sample gas pressure could not be controlled satisfactorily in the gauge pressure range below 1 kPa, the tube with the leak hole was located in a measurement cell as shown in Fig. 6. Air escapes from the sampling tube through the hole to the cell and then through a thermal mass flow controller (MFC) to ambient. Air in the cell is humidified by evaporation from the water surface on the bottom and from a wet sleeve wrapped around the tube. The sampling flow rate through the MBW373 hygrometer is controlled with another MFC. Both MFCs have been calibrated in the MIKES Flow Laboratory. Due to the cell, it was possible to investigate effects of very small leak flows at elevated sample gas pressure.

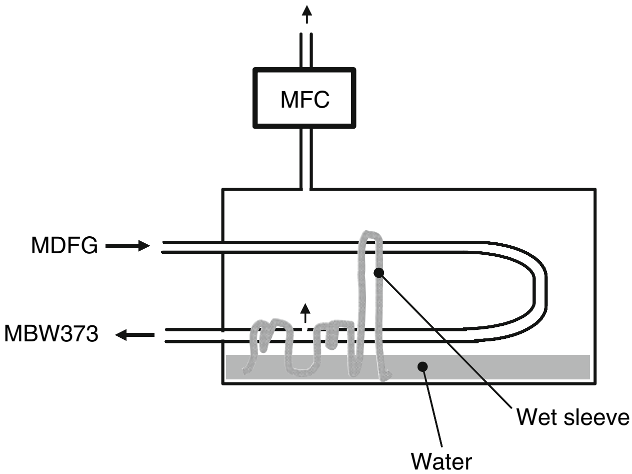


Fig. 6 Tube with 0.5 mm leak hole in a measurement cell: MFC = thermal mass-flow controller

Table 1 Uncertainty budget for the experimental critical flow rate

Component	Estimate	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution ($ml \cdot min^{-1}$)
Detected critical flow rate	$2\,ml \cdot min^{-1}$	$0.23\,ml \cdot min^{-1}$	1	0.2
Detection error in td change	0°C	0.03°C	$0.3\,ml \cdot min^{-1} \cdot °C^{-1}$	0.0
MFC calibration	$0\,ml \cdot min^{-1}$	$0.05\,ml \cdot min^{-1}$	1	0.1
Error in flow measurement	$0\,ml \cdot min^{-1}$	$0.29\,ml \cdot min^{-1}$	1	0.3

$u = 0.4\,ml \cdot min^{-1}$
$U = 0.7\,ml \cdot min^{-1}$

Measurements were carried out over about 200 h at the dew-point temperature of −80°C and absolute pressure of 122 kPa. The leak flow rate was increased from 0 to $4\,ml \cdot min^{-1}$ in seven steps. After increasing the indicated flow rate from 1.5 to $2.3\,ml \cdot min^{-1}$, changes in leak flow rate did not have any effect on the hygrometer reading. The experimental value for the critical flow rate was estimated to $(2 \pm 1)\,ml \cdot min^{-1}$. The corresponding calculated value is $(3 \pm 1)\,ml \cdot min^{-1}$. Thus, the agreement between theoretical and experimental results is fairly good.

Errors in detecting the critical flow rate and measuring the flow rate values were included in the uncertainty estimation for the experimental result (see Table 1). The uncertainty of the dimensions of the leak hole dominates the uncertainty budget for the theoretical critical flow rate value as shown in Table 2.

Table 2 Uncertainty budget for the theoretical critical flow rate obtained with Eq. 4

	Estimate	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution ($ml \cdot min^{-1}$)
p	122,200 Pa	2,887 Pa	-2×10^{-8} ml · min^{-1} · Pa^{-1}	-6×10^{-5}
t	23.0°C	1.7 °C	0.024 ml · min^{-1} ·°C^{-1}	0.04
t_{da}	23.0°C	1.7°C	0.018 ml · min^{-1} ·°C^{-1}	0.03
L	1 mm	0.06 mm	−3.6 ml · min^{-1} · mm^{-1}	−0.21
d_h	0.5 mm	0.03 mm	16.0 ml · min^{-1} · mm^{-1}	0.46
t_{dL}	−79.0°C	0.23°C	−0.08 ml · min^{-1} ·°C^{-1}	−0.02

The uncertainty of the molar water-vapor concentration is determined from uncertainties in pressure (p), temperature (t), and dew-point-temperature measurements (t_d)
$u = 0.5\,ml \cdot min^{-1}$
$U = 1.0\,ml \cdot min^{-1}$

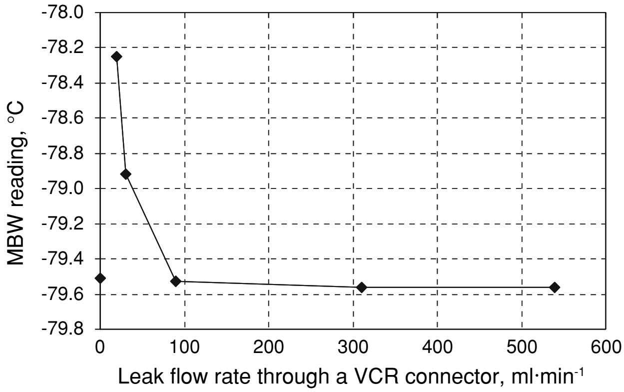


Fig. 7 Effect of leak in a VCR connector on the hygrometer reading

3 Leaks in Actual Connectors

The effect of leaks in actual connectors was studied experimentally by loosening a connector between the generator and hygrometer and monitoring changes in dew-point and flow-rate readings. The flow rate through the hygrometer was kept constant.

Measurements were done with leak flow rates of 100, 240, and 340 ml · min^{-1} through a 6 mm Swagelok connector. The dew-point temperature of air supplied by the generator was −80°C. Increasing the leak had no effect on the measured dew-point temperature. With a VCR connector, we studied leak flow rates from 20 to 540 ml · min^{-1} at about the same dew-point temperature. In this case, a clear leak dependence was observed, as shown in Fig. 7. When the leak flow rate was larger than 100 ml · min^{-1}, the back-diffusion of water vapor did not have any effect on the hygrometer reading. At a dew-point temperature of −60°C, no effect due to back-diffusion was observed.

Leaks through the connectors and the leak-hole tubes were also compared directly to each other. This was done with the setup shown in Fig. 2. At first, the sample air was allowed to escape through the leak hole. Then, the hole was closed with aluminum tape and the same leak was applied through the inlet VCR connector of the hygrometer by loosening the connector. Flow rates and pressure were controlled to ensure the same leak flow rates and sample gas flow rates through the hygrometer in both cases. The leak tubes with 0.1 and 0.5 mm holes were used. The leak flow rates were 30 and 540 $ml \cdot min^{-1}$, respectively. In both cases, no back-diffusion effect was detected. According to the theoretical calculations for the leak tubes, the leak flows were clearly larger than the critical leak flow-rate values.

4 Conclusion

In this work, the effect of water vapor back-diffusion in leaks was studied theoretically and experimentally. The investigation was focused in the dew-point temperature range above −80°C, corresponding to a mixing ratio range larger than $0.3\,\mu g \cdot g^{-1}$. The results show that penetration of water vapor into a sampling line can effectively be prevented by maintaining gauge pressure in the line. Tiny leaks, however, seem to be potential routes for diffusing water vapor.

Experiments with Swagelok and VCR connectors show that the back-diffusion of water vapor is more probable in the latter. This is explained by differences in the geometry. Although equilibrium between the water-vapor concentration on the surface and in the gas is achieved faster in a VCR connector, the diffusion path in the case of a leak is longer in a Swagelok connector and the connector works as a nozzle.

The presented theoretical method was successfully applied in analyzing leaks in artificial holes, but not in actual connectors. Further studies are needed to improve the method and to analyze tiny leaks. In particular, the effect of actual flow profile in the hole should be investigated because the gas velocity close to the wall of the hole is lower than the mean velocity.

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