ABSTRACT
We have developed a new method for measuring the water vapor permeation through polymers and permeation barrier coatings. A metallic vessel, the gas cell, is filled with water and sealed with the sample. The gas cell is then introduced into an UHV-system and positioned to face a mass spectrometer. The partial pressure of water is taken as measure for the water vapor transmission rate. Using the isotope \(^{20} \text{H}_2 \text{O}\) can improve the sensitivity of the method.

INTRODUCTION
Gas barrier films consisting of a polymer substrate coated with a thin glass layer are used for packaging, ranging from simple food containers to high-end electronic products. Oxygen and water vapor permeation through the packaging material is a main concern as they may influence shelf life and flavor of the products. In high-end products, such as flat panel displays based on organic light emitting diodes, the ingress of even the tiniest amounts of water has to be prevented to avoid deterioration of functionality. To ensure and certify extremely low rates of water vapor transmission rates (WVTR) they have to be accurately measured. The purpose of this paper is to present a new method to measure WVTR with high sensitivity.

EXPERIMENTS
A similar experimental set-up as described in detail in [1] has been used. Figure 1 shows the gas cell, which is a metallic vessel. It is filled with water and subsequently sealed with the sample barrier layer. It is then introduced into an ultra-high vacuum chamber and transferred to face a mass spectrometer. Water vapor permeates through the barrier layer. It then enters the mass spectrometer, which measures the partial pressure of water. The partial pressure of water is proportional to WVTR and can be given in absolute values (g/m²/day) after calibration against samples of known permeability [2].

RESULTS AND DISCUSSION
Figure 2 shows the partial pressure of argon and water as function of time. The gas cell has been filled with water (liquid) and argon (gaseous). As discussed in greater detail in [1] the argon partial pressure decreases as function of time. This decrease, which can be approximated as exponential function for gases with a low critical temperature, is caused by the depletion of argon in the gas cell.
If a water droplet is in the gas cell, the water vapor pressure is determined by the temperature and remains constant as long as there is liquid water left in the gas cell. Figure 2 shows, as expected, a constant partial pressure for water because water vapor is replenished from the water droplet. The partial pressure of water measured by the mass spectrometer is proportional to the WVTR. We have measured both partial pressure (with the mass spectrometer) and WVTR (with MOCON Permatran) of a range of samples with widely varying WVTR. Figure 3 correlates the values obtained by both methods. WVTR rates are shown on the x-axis and the corresponding partial pressure measured for the same material with the mass spectrometer is given on the y-axis. Due to this specific representation data points at very low transmission rates are difficult to represent (aluminium foil is impermeable!). We have included the point for aluminium foil to show the range the method can be applied. In this case we took the lowest available detection limits for both methods as data for the point of aluminium foil in Figure 3. We find a satisfactory correlation between the two methods (Figure 3) and also with literature data [2], which indicates that the mass spectrometer is a suitable instrument to estimate WVTR.

In order to achieve a high sensitivity the background signal in the mass spectrum has to be kept as low as possible. The sensitivity of the mass spectrometric method can be greatly enhanced if a water isotope, which is different from the background water signal of the vacuum chamber, is used. We have found that this can be achieved with the isotope $^{20}\text{H}_2\text{O}$, which was used to generate the data shown in Figure 3.

CONCLUSION

We have presented a new method to measure WVTR with high sensitivity by recording the partial pressure of water, which has permeated through a sample. Partial pressures obtained by the mass spectrometer show a good correlation with absolute values of WVTR as obtained in control experiment using MOCON Permatran equipment. It seems to be beneficial to use water isotopes, which are not contained in the background of the vacuum chamber.

This work was carried out in the Oxford Toppan Centre, which is supported by the Toppan Printing Company.

REFERENCES
