TRACEABLE CALIBRATIONS FOR WATER VAPOUR FLUX INSTRUMENTS

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Abstract: Water vapour flux instruments are used for the determination of transepidermal water loss (TEWL). TEWL is the rate of water loss through skin and is an important indicator of skin barrier function.

The comparison of data recorded by different TEWL instruments is difficult due to the lack of a clear validated calibration method. Current state of the art membrane-based calibration techniques are based on an ASTM standard E96-00 – Standard Test Methods for Water Vapour Transmission of Materials but this method is shown to be unsuitable for the calibration of TEWL devices.

Two recent UK Department of Trade and Industry projects have gone some considerable way towards developing a traceable alternative calibration method. This approach to calibration consists of a 1 µl droplet of water dispensed into the base of a specially designed calibration cap and the flux time-series curve is then logged until the droplet has fully evaporated. A calibration factor can be derived from the relationship between the mass of water dispensed and the area under the flux time-series curve. A series of calibration caps of different sizes provide different water vapour flux rates over the range of interest.

The approach and detail of the calibration method, and some preliminary results are reported. By implementing a harmonized and traceable calibration, disparity between results from several different TEWL instruments was approximately halved in the range around 15 g m⁻² h⁻¹.

Key words: water vapour, flux, relative humidity, TEWL.

1. BACKGROUND

Transepidermal water loss (TEWL) can be defined in terms of water vapour transmission rate (WVTR) or flux. WVTR can be given as the steady water vapour flow in unit time through unit area of a body.

\[ WVTR = \frac{G}{At} \] (1)

Where \( G \) is the mass of water vapour, \( t \) is time and \( A \) is area.

The measurement of TEWL gives important information regarding the barrier properties of the skin and can be used to detect damage (often invisible) as a result of exposure to solvents, for example. Instruments designed for the measurement of TEWL are used widely in fields such as occupational health, medical research, defence and for new product trials in the cosmetics industry.

TEWL is evaluated by measuring the water vapour loss from the skin surface using instruments incorporating relative humidity sensors. Healthy skin exhibits water loss typically in the range from 10 g m⁻² h⁻¹ to 50 g m⁻² h⁻¹ or more, depending on where on the body the measurements are made.

The instruments to measure TEWL already exist, but due to the lack of a clear validated calibration method it is difficult to have confidence in any results obtained. Each manufacturer uses their own method of calibration, so the comparability of results is impaired. In practice, users can find that different TEWL instruments may disagree by up to 50 % for readings of the same condition.

Figure 1: A photograph of the four instrument types involved in the water vapour flux traceable calibration project.

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3 The TEWL Calibration Project is sponsored by the UK Department of Trade and Industry. Project partners are: - EnviroDerm Services (C L Packham and H E Packham), London South Bank University (R E Imhof, H E Packham and P Xiao), UK National Physical Laboratory (S A Bell, R M Gee and M Stevens), Biox Systems (E P Berg, R E Imhof and P Xiao), Dstl Ports Down (R P Chilcott & C H Dalton), Gillette UK (A Stevens & N Weston), Beira University (M de Jesus), Delfin Technologies (A Immonen and J Nuutinen), Courage and Khazaka Electronic (G Khazaka) and Skinos (I Suzuki).
The aim of the project was to provide a validated calibration method with a clear route of traceability to national standards for all types of TEWL devices currently commercially available.

Four instrument manufacturers have made instruments available for the project and these are pictured in Figure 1. The measurement techniques are similar for each instrument but each utilises a slightly different method. All the instruments employ one or two relative humidity sensors to measure or estimate the vapour pressure at two points in the measurement chamber. From this information a vapour pressure differential and hence a transmission rate or flux can be determined.

There are four main types of TEWL device: the open chamber, the continuous flow chamber, the condenser chamber and the closed unventilated chamber.

All the instruments use cylindrical measurement heads of internal diameter between 7 mm and 12 mm, which are open at the end applied to skin.

Type (a) shown in Figure 2 is the “open chamber” instrument [1] and is open at both ends. The difference between two relative humidity values at different heights in the chamber is used to estimate the water vapour pressure gradient, and hence water vapour flux.

Type (b) in Figure 2 is a “differential deduction ventilation method” or “continuous flow method” instrument where the chamber is flushed with ambient air at a controlled flow rate. Tubing to and from the sensor head, is connected to a pump, and the inlet and outlet relative humidities are measured. The relative humidity of the ambient air is measured as the reference, then a small amount of this air is pumped along the tube to the measurement head and back via the second relative humidity sensor. The change in humidity corresponds to the water loss from the skin being measured.

Type (c) shown in Figure 2 is a “condenser-chamber” instrument [2] featuring a relative humidity sensor, plus a peltier-cooled surface at the far end of the chamber (held at a constant temperature of -13.4 °C). The respective relative humidity and dew-point temperature at the two locations are used to derive the vapour pressure gradient, and hence the water vapour flux. In this case the cold condensing surface is a sink for water vapour allowing steady-state flux to be measured continuously.

Type (d) shown in Figure 2 is a “closed unventilated” chamber [3] (without an outlet or sink for water vapour) and uses a slightly different method to the other three instrument types. A single relative humidity sensor measures the rate at which the closed space fills with water vapour diffusing from the skin. The instrument makes relatively fast measurements and operates by evaluating the gradient of the relative humidity sensor response curve (shown in Figure 3). The product of this gradient and a calibration factor stored on the microprocessor gives the value of TEWL.

![Figure 3: A diagram showing the relative humidity sensor response curve of the closed unventilated instrument. Courtesy of Delfin Technologies Ltd, Finland](image)

All of the instruments perform internal calculations on the output from the relative humidity sensor or sensors. This is then processed with “factory” calibration factors and results in values of water vapour flux expressed in g m⁻² h⁻¹ or as in the case of the continuous flow method instrument in µg cm⁻² min⁻¹. All except the closed unventilated instrument provide continuous measurement with varying degrees of data logging incorporated (however, this particular instrument involved in this work has been modified by the manufacturer to give a continuous output from the relative humidity sensor).

2. EXISTING MEMBRANE BASED CALIBRATION TECHNIQUES

Existing calibration methods for water vapour flux instruments are based on a method of membrane permeance measurement. A container of water is used whose opening is sealed by a semi-permeable membrane. The mean flux of water vapour escaping through this membrane can be determined from measurements of the container weight loss, time and area of the membrane. The instruments can then be calibrated against this “constant” vapour source.

Of the four instruments taking part in this collaborative work three of the instruments (the open chamber, the continuous flow chamber and the closed unventilated instrument) use this method to provide an initial “factory”
calibration. The fourth instrument, the condenser-chamber instrument utilises a version of the droplet method of calibration reported here [4].

ASTM standard E96-00 – Standard Test Methods for Water Vapour transmission of Materials [5] specifies a standard set of test methods for characterising materials in terms of water vapour transmission. It is this standard that formed the basis for the start of our work. If a specific membrane or series of membranes could be characterised in terms of WVTR they could be used to provide reference values of flux. These calibration artifacts could then form the basis for a traceable calibration of the TEWL devices.

The standard contains two main methods for the characterisation of a membrane - a “wet cup” method involving water as the vapour source and a “dry cup” method involving a desiccant such as anhydrous calcium chloride as a vapour sink. The standard was originally developed for characterising materials for the building industry and plastic films - materials with quite low permeability. To obtain a weight change large enough to reliably measure could take many hours and indeed days in some cases. In contrast, to generate the flux rates required by the TEWL instruments high permeability membranes were required.

2.1 Practical obstacles to the membrane technique

ASTM E96-00 states that the relative humidity at the vapour source is 100 % provided the air space above the water is no more than 25 mm. This can be said to be true in the boundary layer at the water surface, and it may be true at the membrane surface if we consider membranes with a low permeability, but in the case of the work being described here much higher flux rates are needed. It was discovered that the relative humidity at the membrane could be as low as 70 % depending on the distance between the water surface and the membrane. The relative humidity at the membrane surface can never be maintained at a high value due to the ongoing depletion of the boundary layer.

It was decided to attempt a different approach to ASTM E96-00 to characterise a membrane in terms of WVTR. In the diagram in Figure 4, the candidate material is clamped separating the small sample chamber in two. On one side of the sample chamber was a continuous flow of dry air and on the other a continuous flow of air of a known dew point. Both sides of the “membrane sandwich” were continuously monitored using optical dew point hygrometers. The moisture gain of the dry air corresponded to the water vapour transmission through the membrane.

A sensitivity analysis was developed corresponding to a change in water vapour flux of 0.5 g m⁻² h⁻¹. This is the quoted accuracy of the open chamber instrument when operating in ambient conditions where the relative humidity is ≥ 30 %. For the purposes of the sensitivity analysis it has been taken as the limiting resolution, which must be achieved by any calibration apparatus. To meet this criterion, conditions of the dry side of the “membrane sandwich” would need to be measured to 0.05 °C dew point. In the long term it would mean only standards laboratories would have the measurement capability of reproducing this technique. This was not considered a satisfactory basis for disseminating traceability.

Figure 4: A diagram of an alternative approach to ASTM E96-00 – Standard Test Methods for Water Vapour transmission of Materials

2.2 Theoretical obstacles to the membrane technique

ASTM E96-00 stipulates that the flow speed of the ambient air should be maintained between 0.02 m s⁻¹ and 0.3 m s⁻¹. However, variable air movement causes variation in relative humidity above the membrane, which in turn leads to non-constant flux. Modeling has shown that at a water vapour flux level of 15 g m⁻² h⁻¹ the difference in calibration error between the two air velocity limits could be as much as 25 %. The ASTM E96-00 wet cup method does not provide a constant water evaporation device.

The premise of any membrane-based calibration is that the flux density measured during the characterisation of a membrane is equal to the flux density measured by the instrument when under calibration on the same membrane. However, modeling the water vapour flux densities for all the components of the system as shown in Figure 5, it has been found that the WVTR through the membrane to the ambient atmosphere may not be the same as that entering the measurement head. The electrical analogy given in Figure 5 illustrates this potential flaw. Each region of the system can be thought of as having an “effective resistance” to the vapour diffusion (denoted by R₁ to R₃ each corresponding to that numbered part of the system).

Figure 5: A diagram of an electrical analogy showing the effect of the introduction of a measurement head.
The water vapour flux can be identified with current (I), splitting into two distinct branches I_A and I_B) the difference in vapour pressures at x_A and x_B can be identified with potential difference (p_A and p_B respectively).

One solution for this problem would be to reduce the membrane area to match the instrument sampling area. This was found to be impractical, because of the prohibitively long time required to obtain a measurable weight change.

3. THE DROPLET METHOD OF CALIBRATION

Traceability of the new calibration method is through a calibrated precision volumetric syringe. The syringe is a positive displacement 1 µl syringe with an adjustable mechanical end stop, and a digital indicator resolving 0.005 µl. Traceability of measurement is obtained by calibration of the dispensed volume of the syringe at an accredited laboratory, traceable to national standards of mass and length.

A calibration adaptor has been made for each instrument type. The adapter allows a standard series of calibration “caps” or “wells” to be fitted (pictured in Figure 6). A 1 µl droplet of water is dispensed into the base of a calibration cap. The calibration cap is then screwed on to the adapter and the output from the instrument under test is logged until the droplet has evaporated.

The calibration wells of different depths were used to provide a range of water vapour flux rates, so that the TEWL instruments could be calibrated at several values in the range of operation. The wells are made of PTFE, for non-hygroscopicity. The well depths range from nominally zero and 64 mm. The longer diffusion path lengths result in slower diffusion from droplet to instrument, and hence to lower flux rates. Higher flux rates were achieved by applying controlled heating to the base of the well. Time taken for the evaporation of a 1 µl droplet ranged from 5 minutes to 1 hour. A sectional drawing of a calibration cap can be seen in Figure 7; the distance, A, corresponds to the well depth. The purpose of the indent is to guide the syringe to make sure the droplet is deposited centrally, and is approximately 0.5 mm in depth. The droplet is not shown to scale but is nominally 1.1 mm in diameter.

The flux time-series curve is logged until the droplet has fully evaporated. The area under the flux time-series curve is then evaluated and with knowledge of the cross sectional area of the measurement head in question, a mass of water in milligrams can be calculated. At 20 °C a 1 µl droplet has a mass of 0.9982 mg. Therefore the amount of water detected by the instrument under test can be linked to the original delivered mass of water to define a calibration factor for the instrument under calibration.

![Figure 6: A photograph showing the water vapour flux instrument calibration adapters, calibration caps and the precision calibrated syringe.](image)

![Figure 7: A sectional drawing showing a water vapour flux instrument attached to a calibration adapter fitted to a calibration well.](image)

3.1 Droplet method calibration results

![Figure 8: A graph showing the results of the droplet method of calibration for the open chamber instrument. The x-axis gives time in seconds and the y-axis gives the water vapour flux level in g m⁻² h⁻¹.](image)

Figure 8 is a graph showing the results of the droplet method of calibration for the open chamber. Tables 1, 2 and 3 show the calibration results obtained for each instrument except for the closed unventilated instrument. A method for implementing the droplet method calibration approach is still being developed for this instrument type. The results shown in Figure 8 show a series of plateaus – each representing a level of flux in g m⁻² h⁻¹ at which the instrument in question has been calibrated. In this case the...
results shown were obtained using the open chamber instrument.

Table 1 – Open chamber instrument droplet calibration results

<table>
<thead>
<tr>
<th>Applied Droplet Mass</th>
<th>Indicated Flux</th>
<th>Calculated Mass of Water From Indicated Flux</th>
<th>Calculated Calibration Factor to be Applied</th>
</tr>
</thead>
<tbody>
<tr>
<td>mg</td>
<td>g m⁻² h⁻¹</td>
<td>mg</td>
<td></td>
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<tr>
<td>0.9977</td>
<td>7</td>
<td>0.693</td>
<td>1.440</td>
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<td>0.9951</td>
<td>19</td>
<td>0.866</td>
<td>1.149</td>
</tr>
<tr>
<td>0.9875</td>
<td>30</td>
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</tr>
<tr>
<td>0.9827</td>
<td>47</td>
<td>0.688</td>
<td>1.428</td>
</tr>
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</table>

Table 2 – Condenser chamber instrument droplet calibration results

<table>
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<tr>
<th>Applied Droplet Mass</th>
<th>Indicated Flux</th>
<th>Calculated Mass of Water From Indicated Flux</th>
<th>Calculated Calibration Factor to be Applied</th>
</tr>
</thead>
<tbody>
<tr>
<td>mg</td>
<td>g m⁻² h⁻¹</td>
<td>mg</td>
<td></td>
</tr>
<tr>
<td>0.9977</td>
<td>34</td>
<td>1.066</td>
<td>0.936</td>
</tr>
<tr>
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<td>0.930</td>
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Table 3 – Continuous flow instrument droplet calibration results

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<th>Applied Droplet Mass</th>
<th>Indicated Flux</th>
<th>Calculated Mass of Water From Indicated Flux</th>
<th>Calculated Calibration Factor to be Applied</th>
</tr>
</thead>
<tbody>
<tr>
<td>mg</td>
<td>g m⁻² h⁻¹</td>
<td>mg</td>
<td></td>
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<td>0.9951</td>
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<td>0.435</td>
<td>2.287</td>
</tr>
<tr>
<td>0.9875</td>
<td>17</td>
<td>0.398</td>
<td>2.482</td>
</tr>
<tr>
<td>0.9875</td>
<td>35</td>
<td>0.437</td>
<td>2.259</td>
</tr>
</tbody>
</table>

The curves shown are typical of those obtained when calibrating any of the instruments using the droplet method of calibration. The area under the curve has been evaluated by numerical integration and the amount of water registered by each instrument is given in the results tables, which follow. From this mass of water in milligrams, a calibration factor can be obtained allowing a correction to be applied when the instruments are in use.

The open chamber is susceptible to changes in ambient conditions and can display a “noisy” output. Ambient air currents and localised changes in ambient temperature and humidity can have an impact. The calibration runs shown in Figure 8 were completed in a three-sided enclosure to minimise this effect.

The plateaus generated are seen to tail off slightly towards the end of a calibration run. This is an effect due to the well depth and the inability of the remaining droplet to maintain the flux level. Towards the end of curve D a step change in the plateau can be seen. This is thought to be due to the secondary evaporation of a smear, which would have occurred on the delivery of the original droplet.

All these effects have been evaluated for the uncertainty analysis (see Section 4).

It should be noted that the data in Tables 1, 2 and 3 are only applicable to the instruments actually calibrated.

4. UNCERTAINTIES

An uncertainty analysis has been carried out taking into account aspects concerned with the calibration of TEWL devices [6]. The uncertainty analysis is consistent with ISO/IEC 17025 and with the ISO Guide to the Expression of Uncertainty in Measurement.

The expanded uncertainty of calibration has been calculated to be approximately 7 % of reading at 15 g m⁻² h (differing slightly for the different instrument types). As flux rates increase the expanded uncertainty of calibration decreases.

5. FINAL RESULTS

![Figure 9: Volunteer Measurements with no droplet calibration correction.](image1)

![Figure 10: Volunteer measurements with the droplet calibration correction applied.](image2)

To obtain the results given in Figures 9 and 10 a series of TEWL measurements were taken from six volunteers using (in the same sequence for each volunteer) the open chamber instrument, followed by the continuous flow instrument and
finally the condenser chamber instrument. The measurements were completed on the back of the non-dominant hand after acclimatisation in an air-conditioned office environment.

The results shown in Figure 9 are uncorrected for the droplet method of calibration. It can be seen that each instrument follows a trend from volunteer to volunteer but that there is poor agreement instrument to instrument.

Figure 10 shows the same data with corrections applied using the droplet method of calibration. The error bars correspond to the calculated expanded uncertainty of calibration for each measurement. In reality the expanded uncertainty should include components for the instruments in use as well as for the calibration. For the six volunteers the corrected measurements taken now agree to within the expanded uncertainty of calibration for four of the six for the condenser chamber instrument and the continuous flow instrument. There is agreement for all three of the instruments to within the expanded uncertainty shown for three of the six volunteers.

To quantify the improvement in the data; the pooled standard deviation of the difference from the individual means was calculated. For the uncorrected data this standard deviation was 5.95 g m\(^{-2}\) h\(^{-1}\). For the data with the droplet calibration correction applied the standard deviation was calculated to be 2.69 g m\(^{-2}\) h\(^{-1}\), a better than 50% reduction in disparity.

6. CONCLUSIONS

An alternative calibration method for water vapour flux instruments has been developed. A known amount of water is dispersed into the base of a specially designed calibration cap. This is then fitted to the instrument under test. The flux time-series curve is logged until the droplet has fully evaporated. The area under the curve is evaluated and with knowledge of the cross sectional area of the measurement head a mass of water in milligrams can be calculated. By relating this amount of water to the amount deposited a calibration correction is obtained. By altering the distance between the probe and droplet and by manipulating the temperature of the surface on to which the droplet is deposited different rates of flux can be generated. It is therefore possible to calibrate an instrument across a range of water vapour flux rates.

While valid for many types of TEWL instrument, this calibration method does not yet support the calibration of devices whose sampling time is short compared to the time taken for a water droplet to evaporate. Further work is in progress to find a way of applying this calibration approach to fast-reading non-continuous TEWL sensors.

Agreement between the open chamber, condenser chamber and the continuous flow chamber has been obtained to approximately the calculated expanded uncertainty of calibration. However, some work still remains; a series of field trials is planned for the near future and aims to practically demonstrate the improvement due to consistent calibration and support the preliminary results published here.

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