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Progress towards traceable inline measurement of water activity

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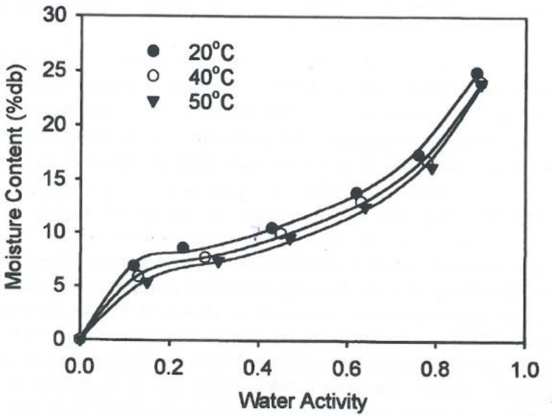
Definition – Water activity

Moisture content: water content in a sample

Water activity: is how easy it is to remove the water

- The **water activity (a_w)** is a measure of how much "free" water there is available for chemical reactions and growth of microorganisms such as bacteria and fungi.
  - Every microorganism has a water activity level below which it cannot grow
  - There are no such direct correlations to the moisture content
- Food- and feed designers use the water activity to control the shelf-life or texture of their products.
- **Sorption isotherms** links **moisture** and **water activity**
  - ***Part of the HIT project***

Aim



Determination of water activity

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In equilibrium

- water activity = relative humidity over the sample

$$a_w = \frac{p}{p_0} = RH$$

Examples

- $a_w = 0 \rightarrow$ "bone dry"
- $a_w = 100\% \rightarrow$ "pure water"

- a_w can be determined indirectly by measuring the relative humidity (RH) above a sample in a temperature stabilised sealed chamber by means of a chilled mirror hygrometer or a relative humidity sensor.

- Depending on the material, the time to reach equilibrium can be very long…

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Sampling →

- Automatic sampling for measurement of water activity

Challenges

- Measurements are **slow**
- Samples are **warm**
- The **volume** of the measurement chamber in commercially available analysers is often **very small**

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Moisture

- On DRY basis

$$\frac{Mass\ of\ water}{Mass\ of\ dry\ sample} \cdot 100\%$$

- On WET basis

$$\frac{Mass\ of\ water}{Mass\ of\ wet\ sample} \cdot 100\%$$

Project goal: Sorption isotherm, 25 – 50 °C

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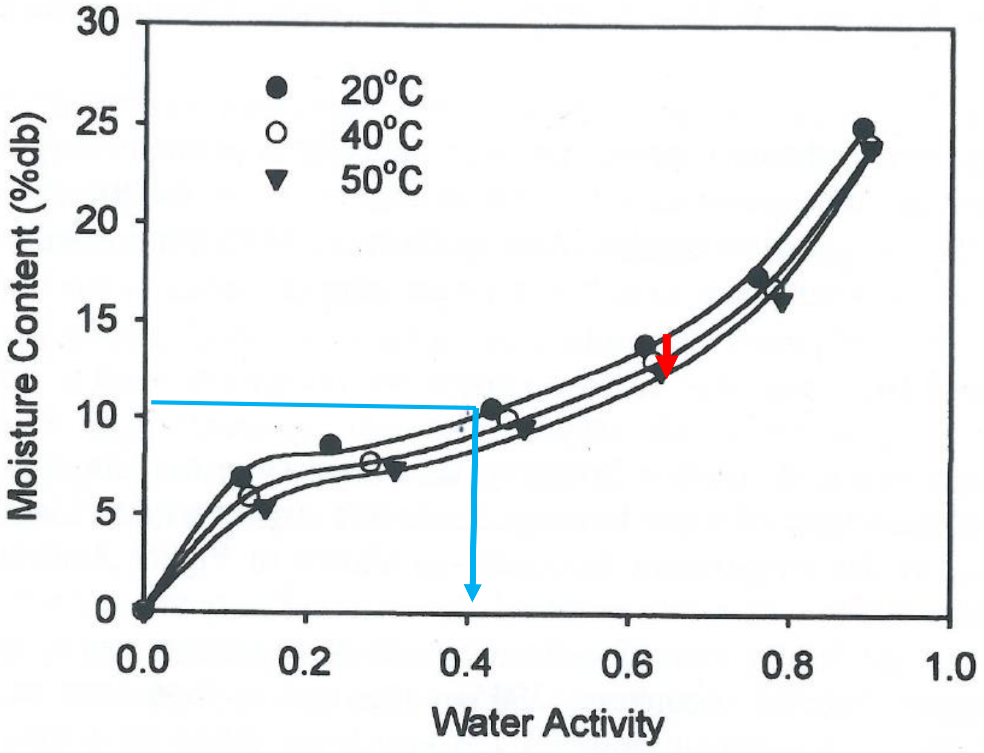
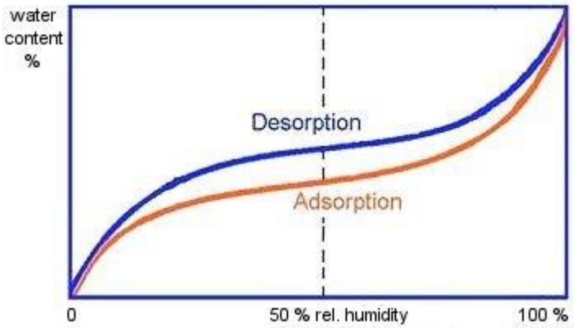


Ref 1



Ref 2

Process of convective drying

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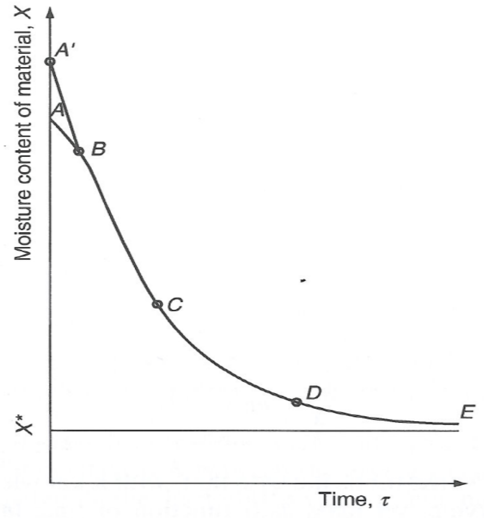
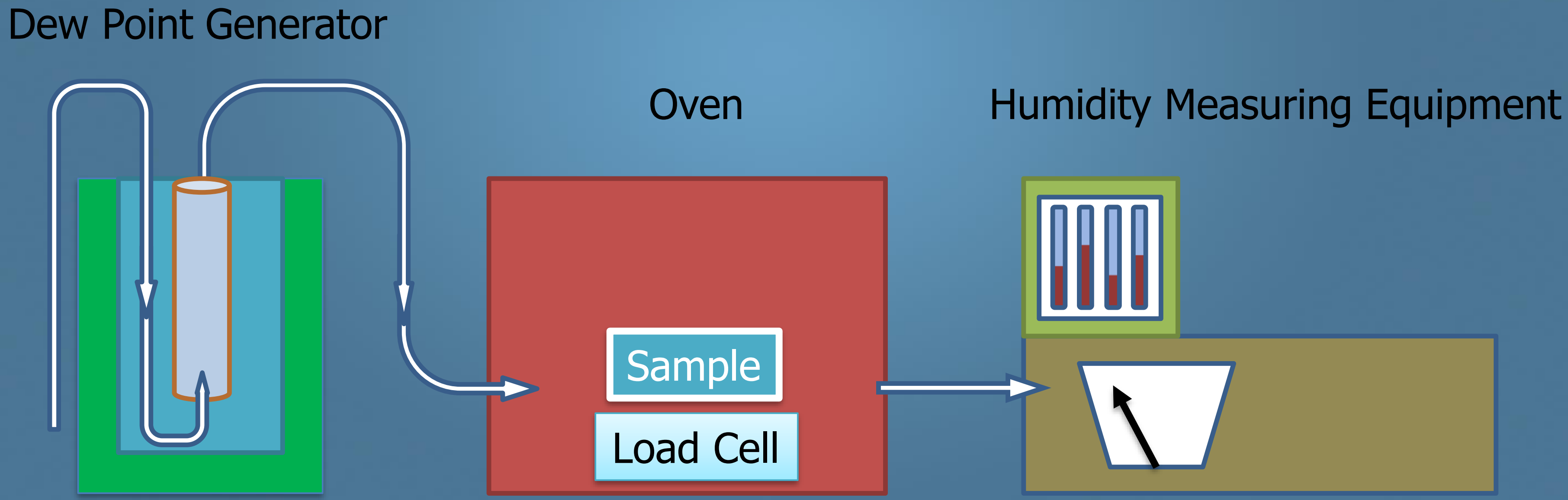
- **A→B:** Getting into equillibrium with air humidity (typically surface water)
- **B→C:** Drying is governed by external conditions (surface is still wet): heat-transfer to the surface of the sample and mass transfer of water from the surface to the ambient media (associated with the term ”free water”).
- **C→E:** Drying is governed by internal transport conditions in the product: internal moisture transfer and heat-conduction, leading to decreasing evaporation rate
- When we sample from the production line we do not know the water content, if there is free water on the surface or if the evaporation rate is governed by moisture mass transfer inside the product.

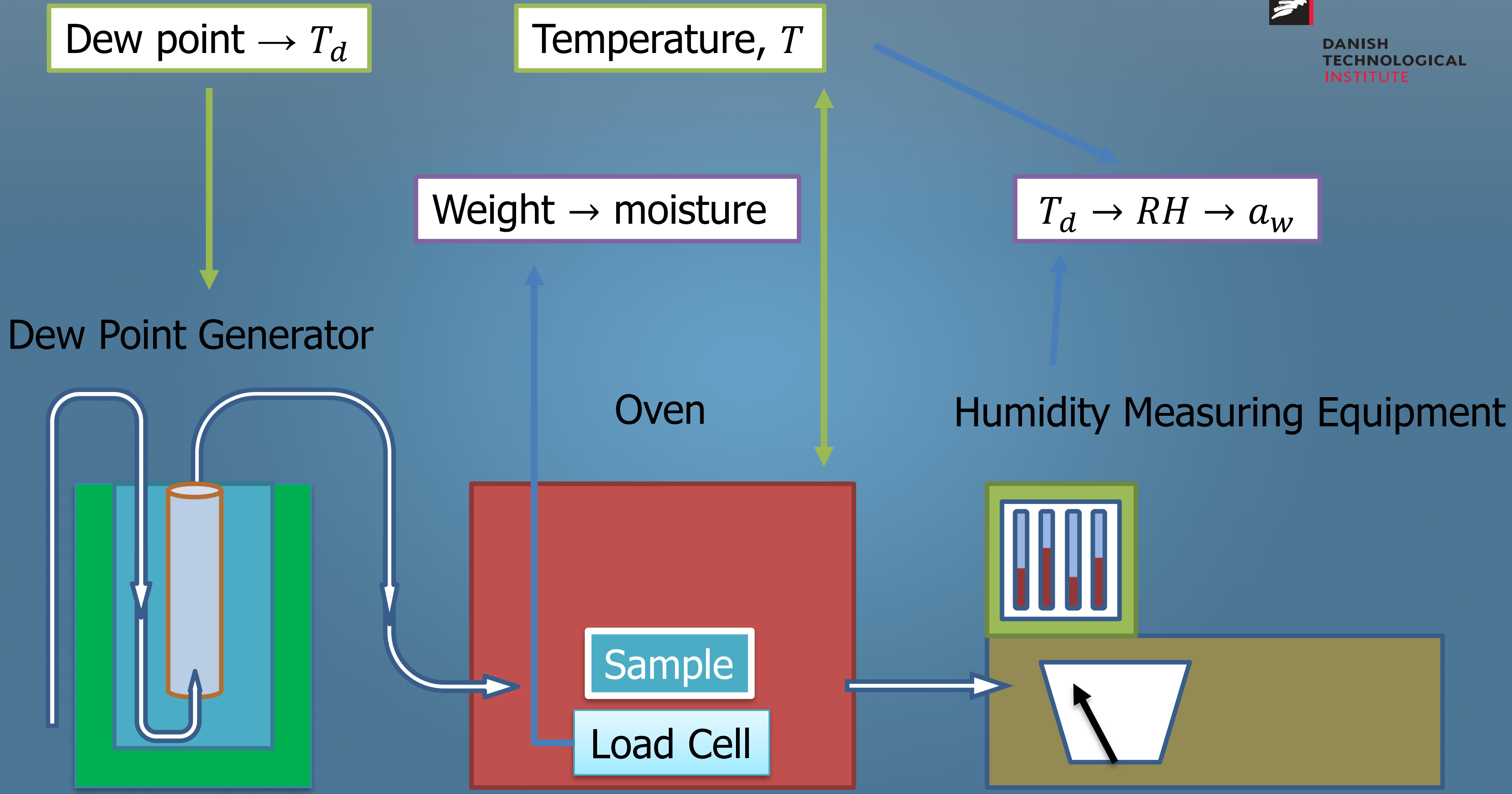


Ref 3

Sketch of setup

- NB: Traceable measurements





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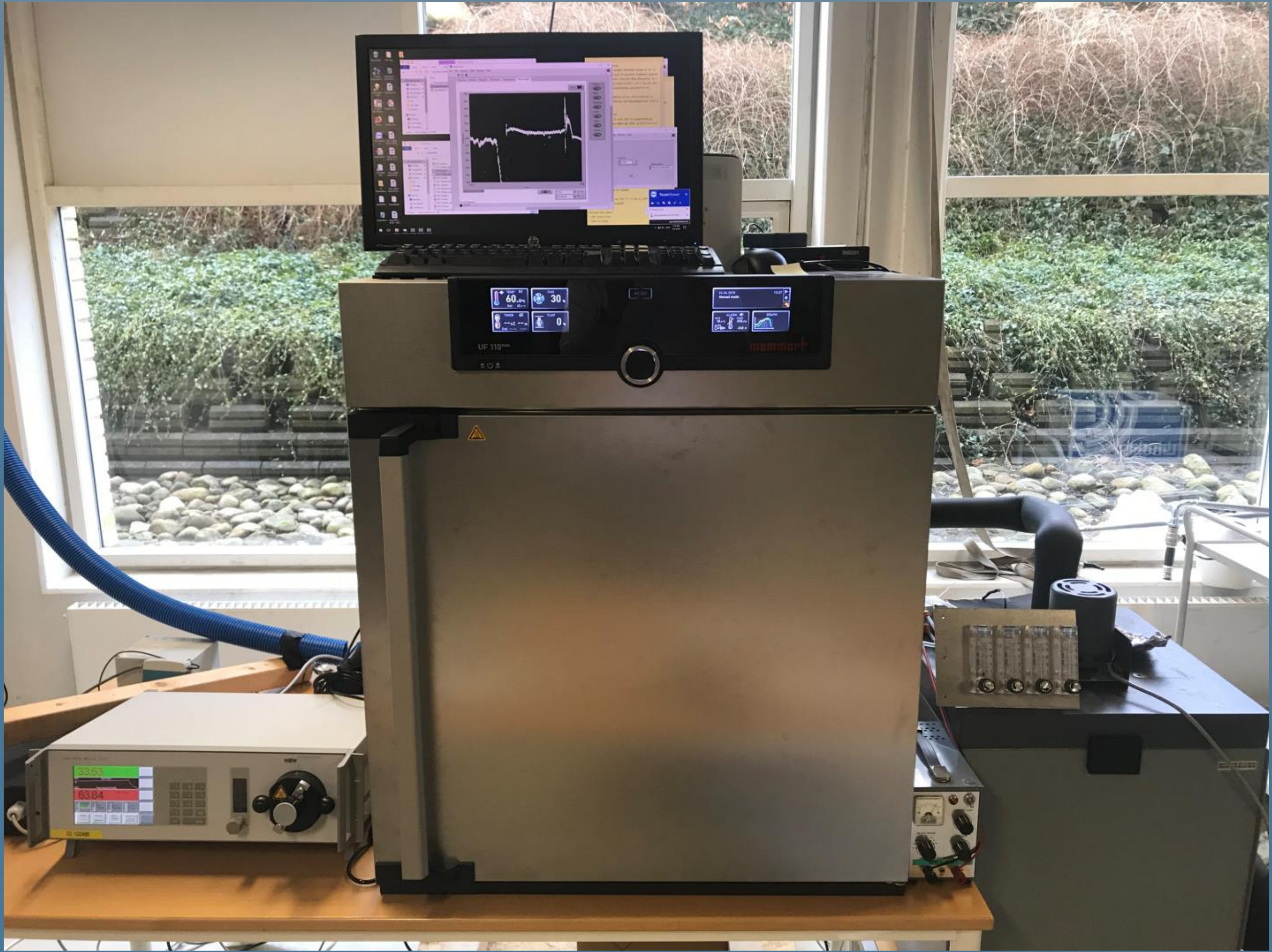

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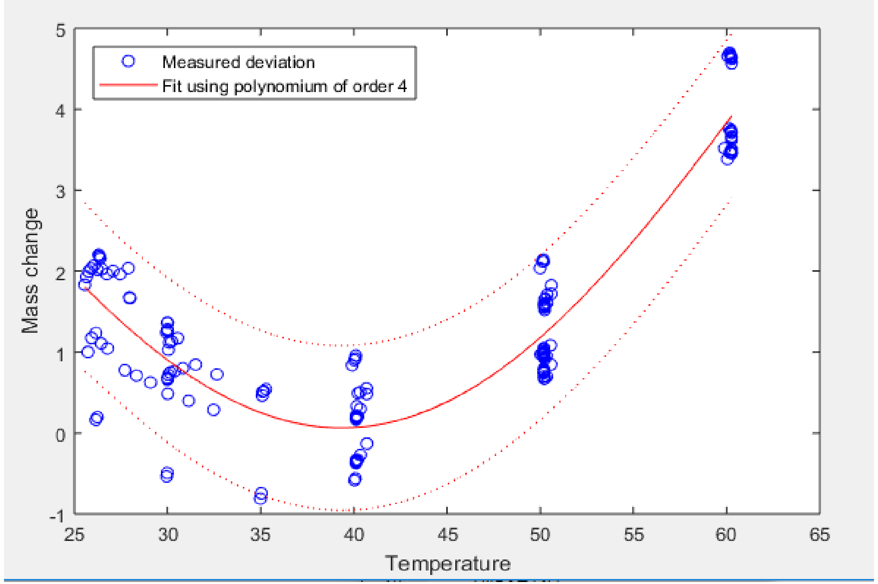
Challenge 1: Load cell in oven

- Mass of chamber: 5000 g
- Target uncertainty: ±1 g ($k = 2$)



Samples so far

(Milk powder, clots at high temperature)

1. Hazelnuts (~300 g)
2. Dog food (500 g)

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Data for ground Hazelnuts

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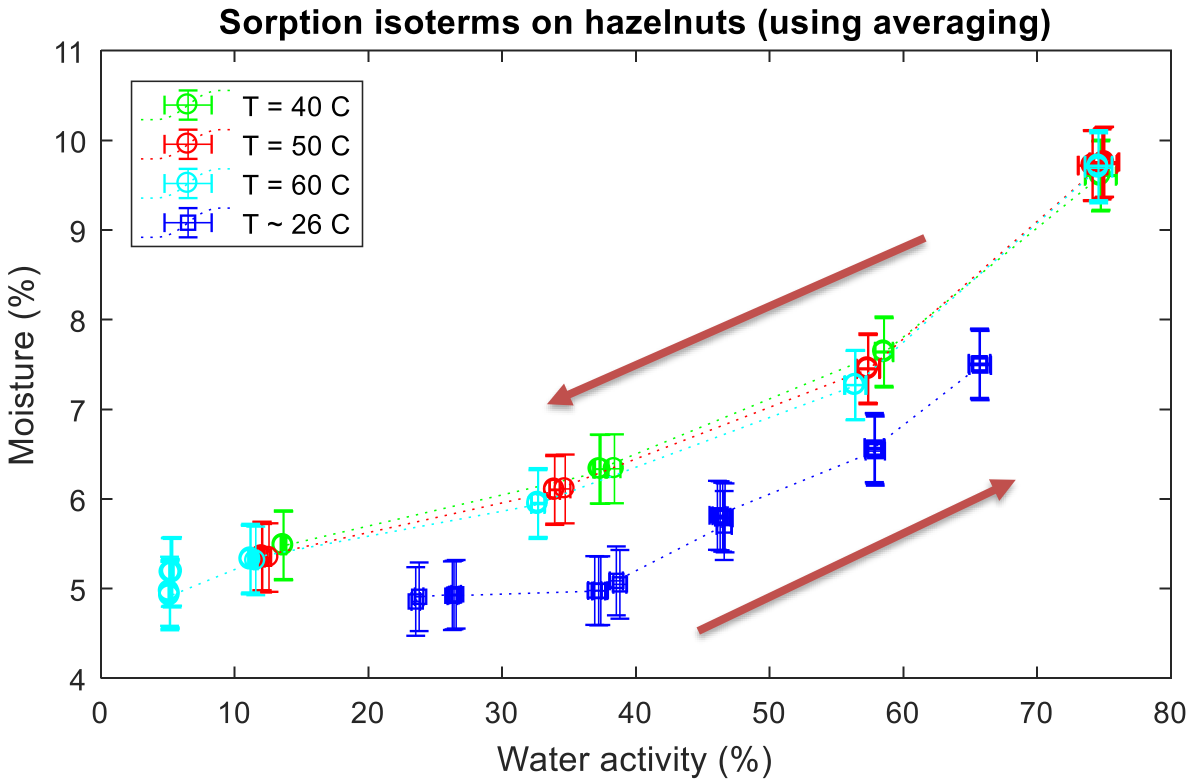

- *Preliminary data*
- *Temperature effect*
  - *Small 40 – 60 °C*
- *Data for 26 °C deviates*
  - Hysteresis ?
  - Not in equilibrium ?

Challenge 2: Slow rate

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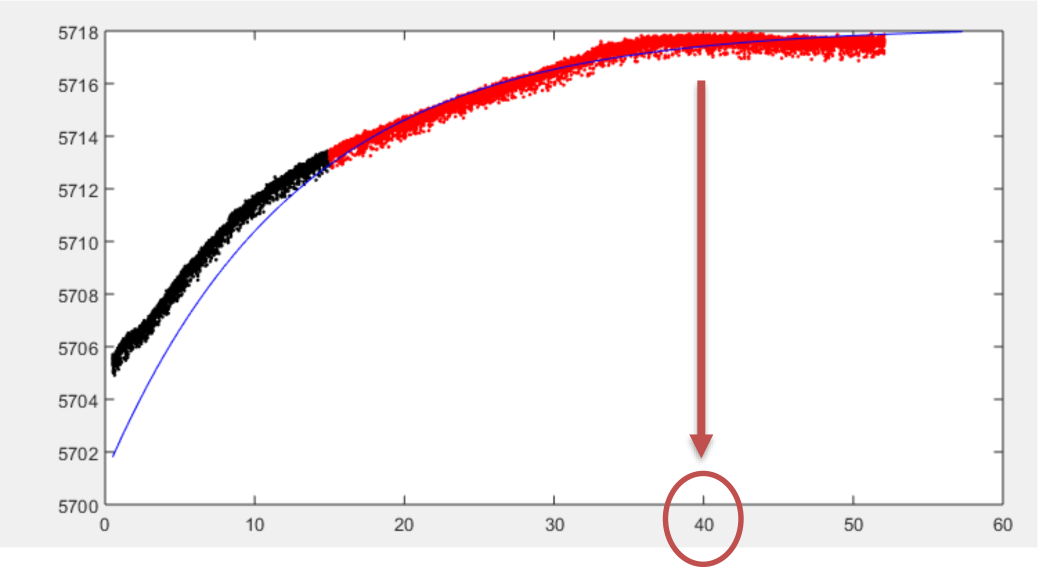
- Equilibrium

$$a_w = RH$$

- Example (→ figure)
  - Weight (g) vs. Time (h)
  - T = 60 °C
  - Humidity change < 1.5%
  - 40 hours
  - → *one point in sorption isotherm*



Sample: Ground hazelnuts (300 g)

Challenge 2: Slow rate (cont.)

Solution 1: Intelligent sequencing

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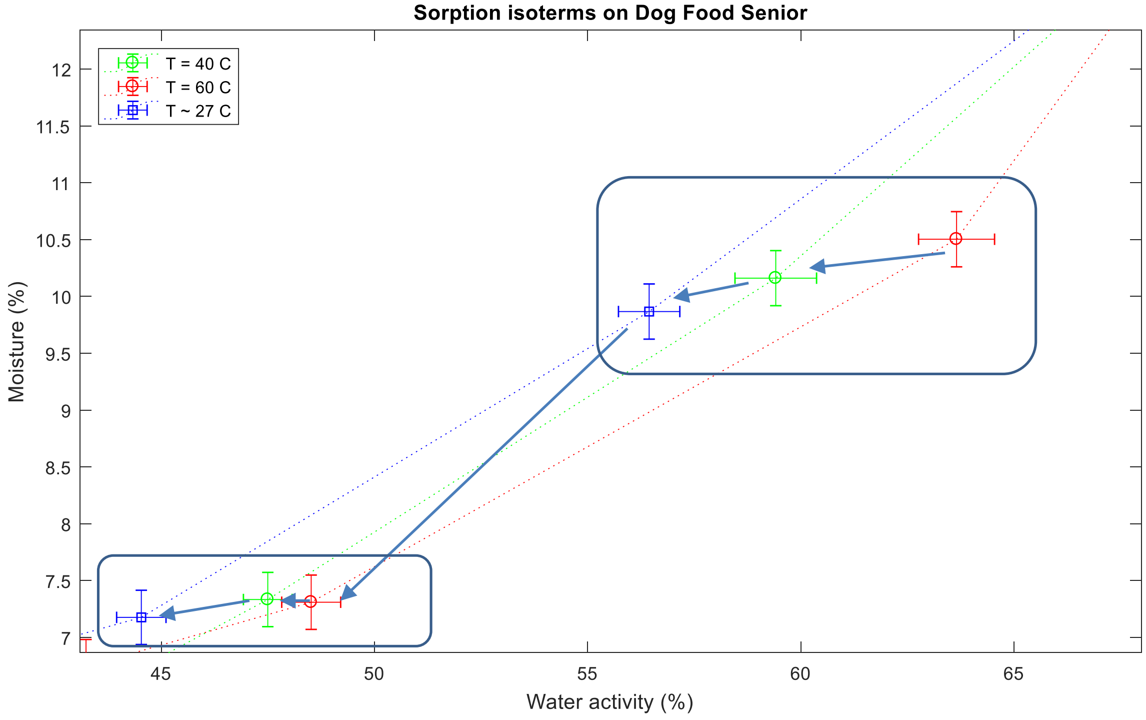

Challenge 2: Slow rate (cont.)

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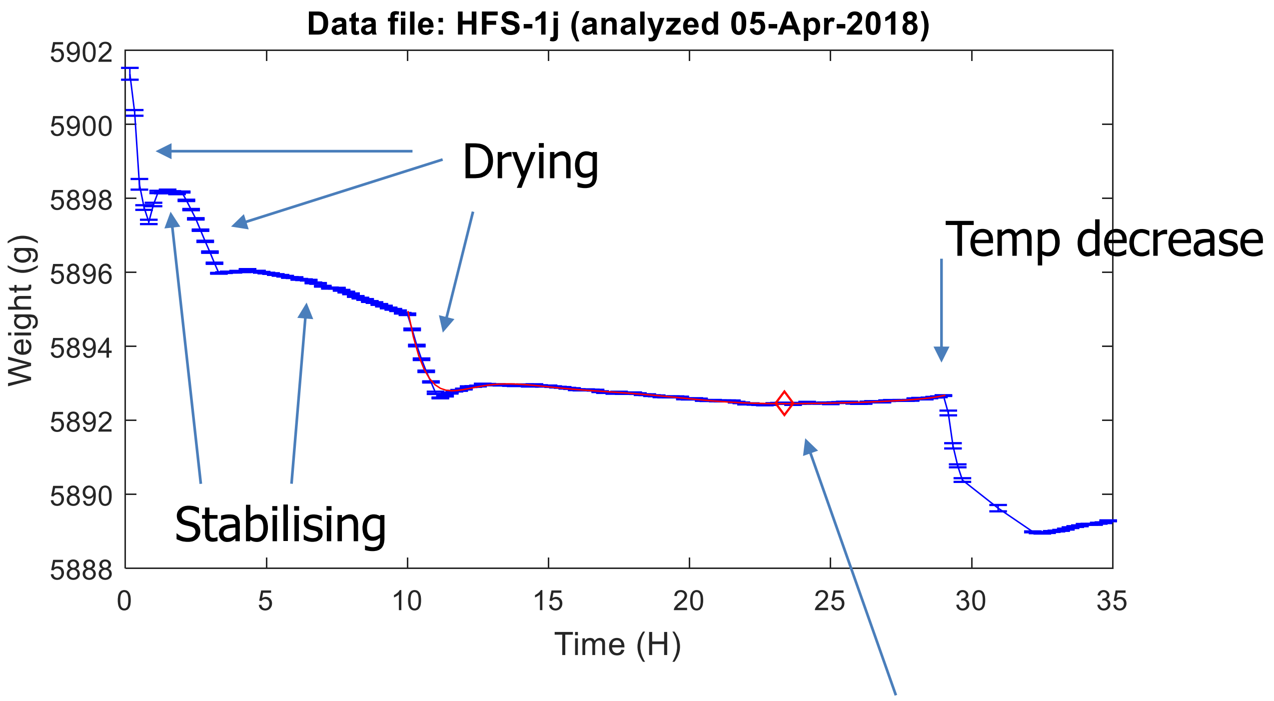
~~Solution 3: Small sample~~

Large samples required for representative sampling in food and feed production

Measuring procedure



Adjusting: $RH = a_w$ (equilibrium $\rightarrow$ constant Weight)

Sample: Dog Food - Senior

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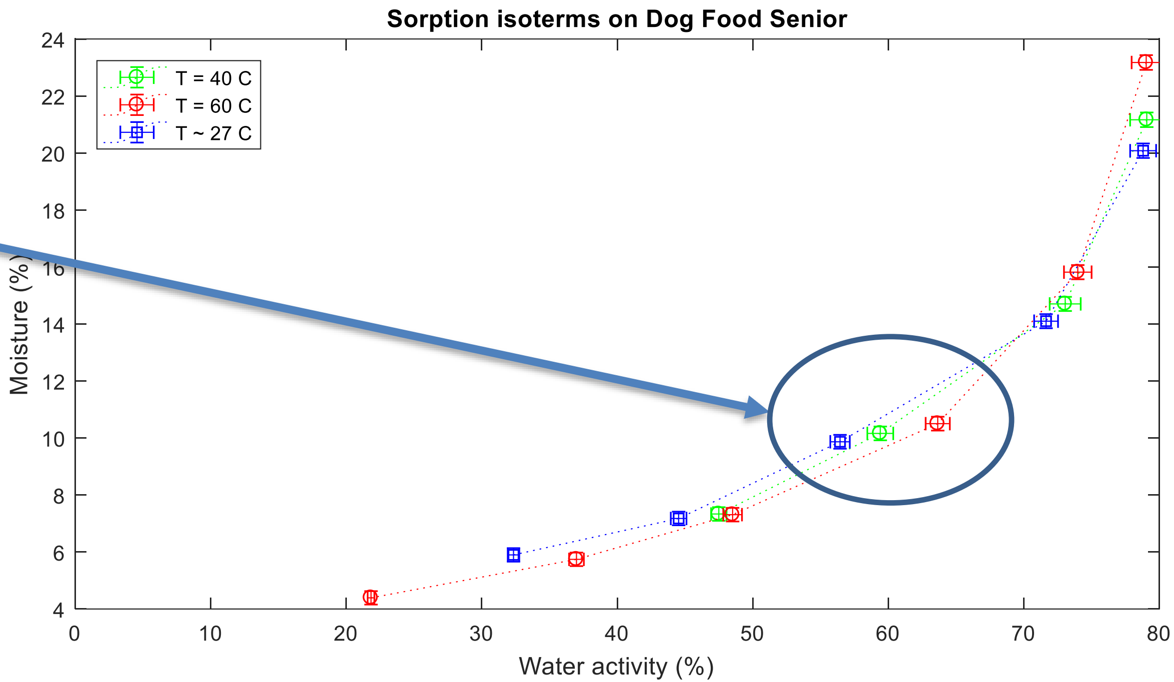
- Improved procedure
  - Large sample
  - LoD: Loss-on-Drying
- Temperature effect

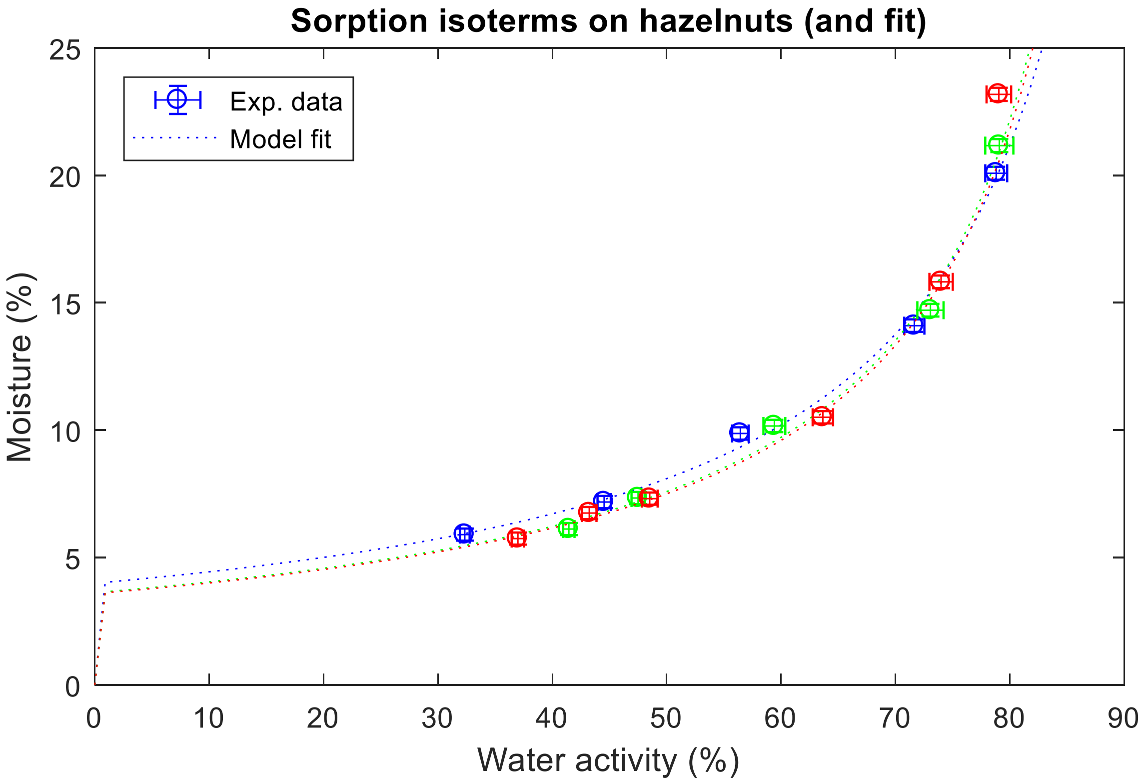


Curve fitting

- Model

$$M = \frac{a_w}{A + B \cdot a_w - C \cdot a_w^2}$$

- Based on *Hailwood and Robin Model*



Outline

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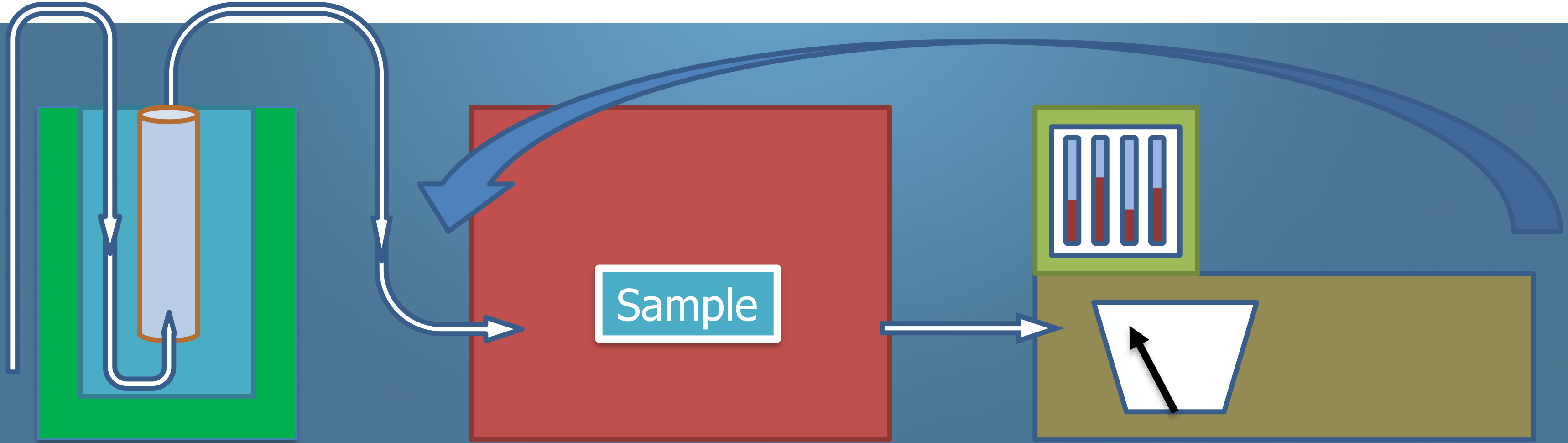
- Method: Recirculation (50 °C)
  - Speed
  - Accuracy
  - Samples from process industry
- Investigations
  - Dynamics (time dependence)
  - Production samples



Conclusion

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- New setup for measuring sorption isotherms
  - Temperature: < 85 °C
- Sample size: 300 – 1000 g
  - Allows for representative sampling at food / feed production
- Traceable through method and calibration of sensors
- Uncertainty ($k = 2 \rightarrow 95\%$ confidence)
  - Weight: ±1 g
  - Moisture: ±0.25% @ 10% (for sample ~ 500 g)
  - Water activity: better than ±1% @ $a_w = 80\ \%$

Next step

- Inline sampling at production line

References

- Ref 1: Bell, L.N., and Labuza, T.P.: "Practical Aspects of Moisture Sorption Isotherm Measurement and Use". 2nd Edition AACC Eagan Press, Eagan, MN 2000
- Ref 2: Tietke, H. W.: ”Studien zu den Möglichkeiten der Senkung von Warenverlusten an feuchteempfindlichen Lebensmitteln unter besonderer Berücksichtigung des Sorptionsverhaltens hygroskopischer Lebensmittel und des notwendigen Verpackungseinsatzes” [Studies into the possible methods for reducing cargo losses in moisture-sensitive foodstuffs with particular reference to the sorption behavior of hygroscopic foodstuffs and the necessary use of packaging], Thesis, Karl-Marx-Univ., Leipzig 1967
- Ref 3: Mujumdar, A. S.: ”Handbook of Industrial Drying” 2nd Edition, CRC Press 2014