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Progress towards traceable inline measurement of water activity

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Definition – Water activity

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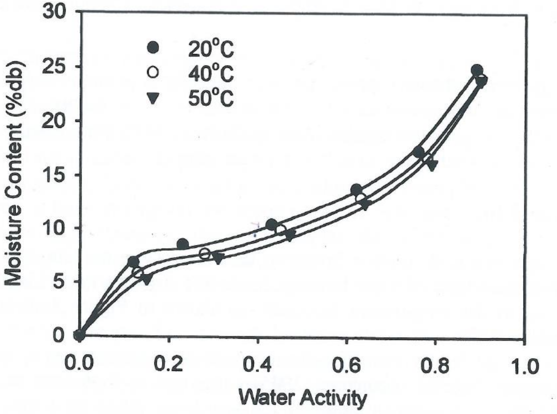
The two concepts we are working on:

Moisture content or here: water content in a sample and
Water activity

- 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 (< 0.6 no growth)
  - There are no such direct correlations to the water content
- Food- and feed designers use the water activity to control the shelf-life or texture of their products.
- **Sorption isotherms** links **water content** 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 may be applied 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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Water content

- On DRY basis

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

- On WET basis

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

The data reported in this presentation are on DRY basis

Project goal: Sorption isotherm, 20 – 70 °C

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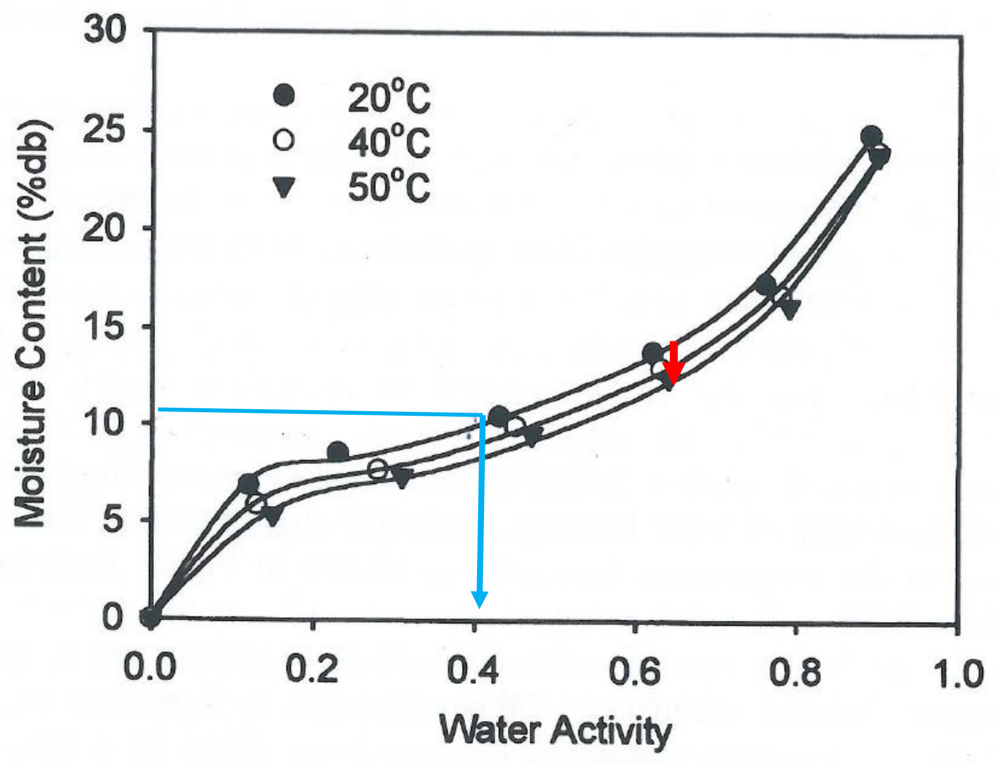
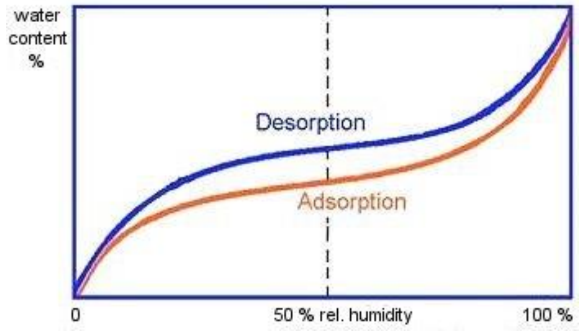


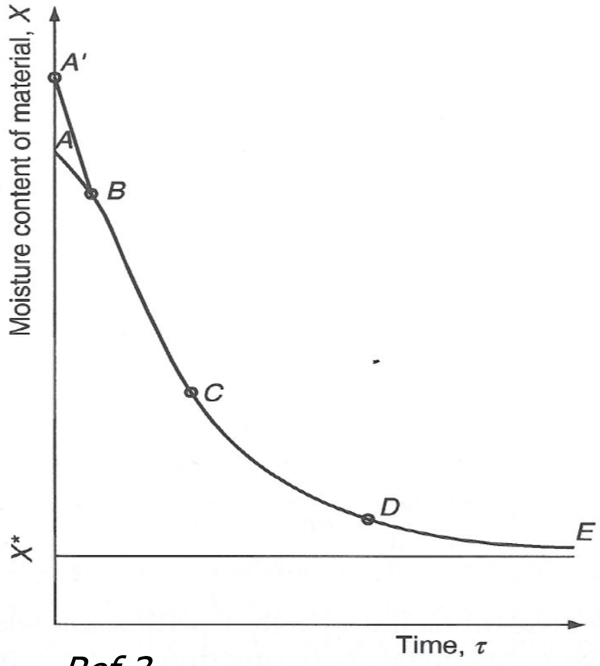
Ref 1



Ref 2

Process of convective drying

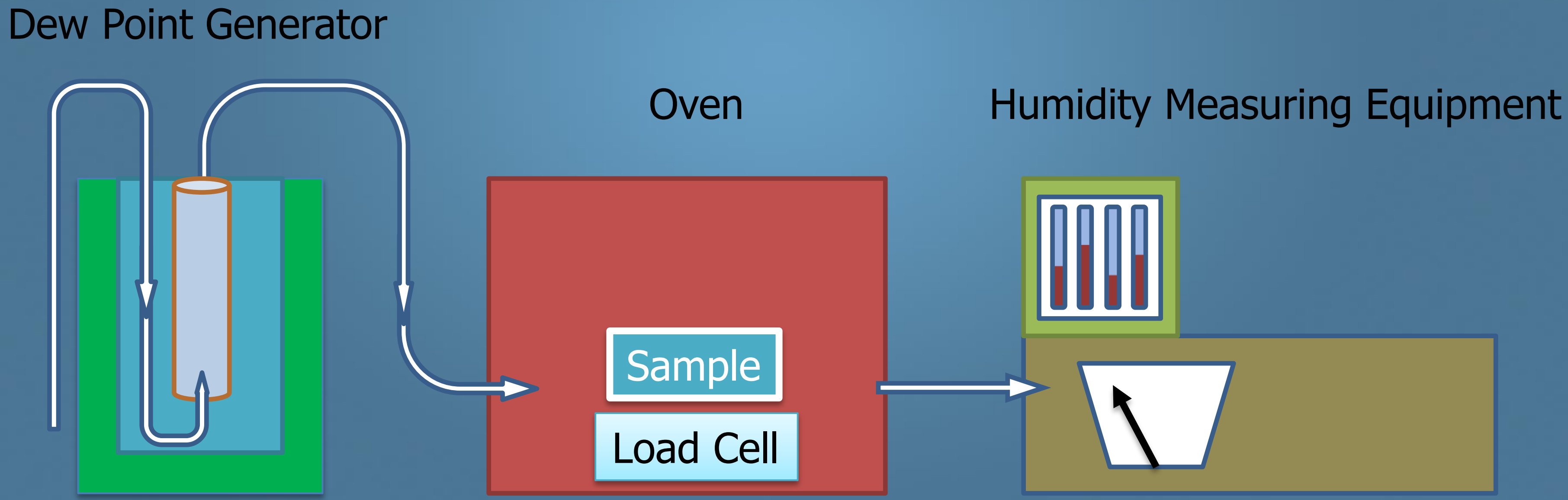
- **A→B:** Getting into equillibrium with air humidity (typically surface water)
- **B→C:** Drying is governed by external conditions (surface is still wet): heat-transfer to the surface of the sample and mass transfer of water from the surface to the ambient media (associated with the term ”free water”).
- **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.

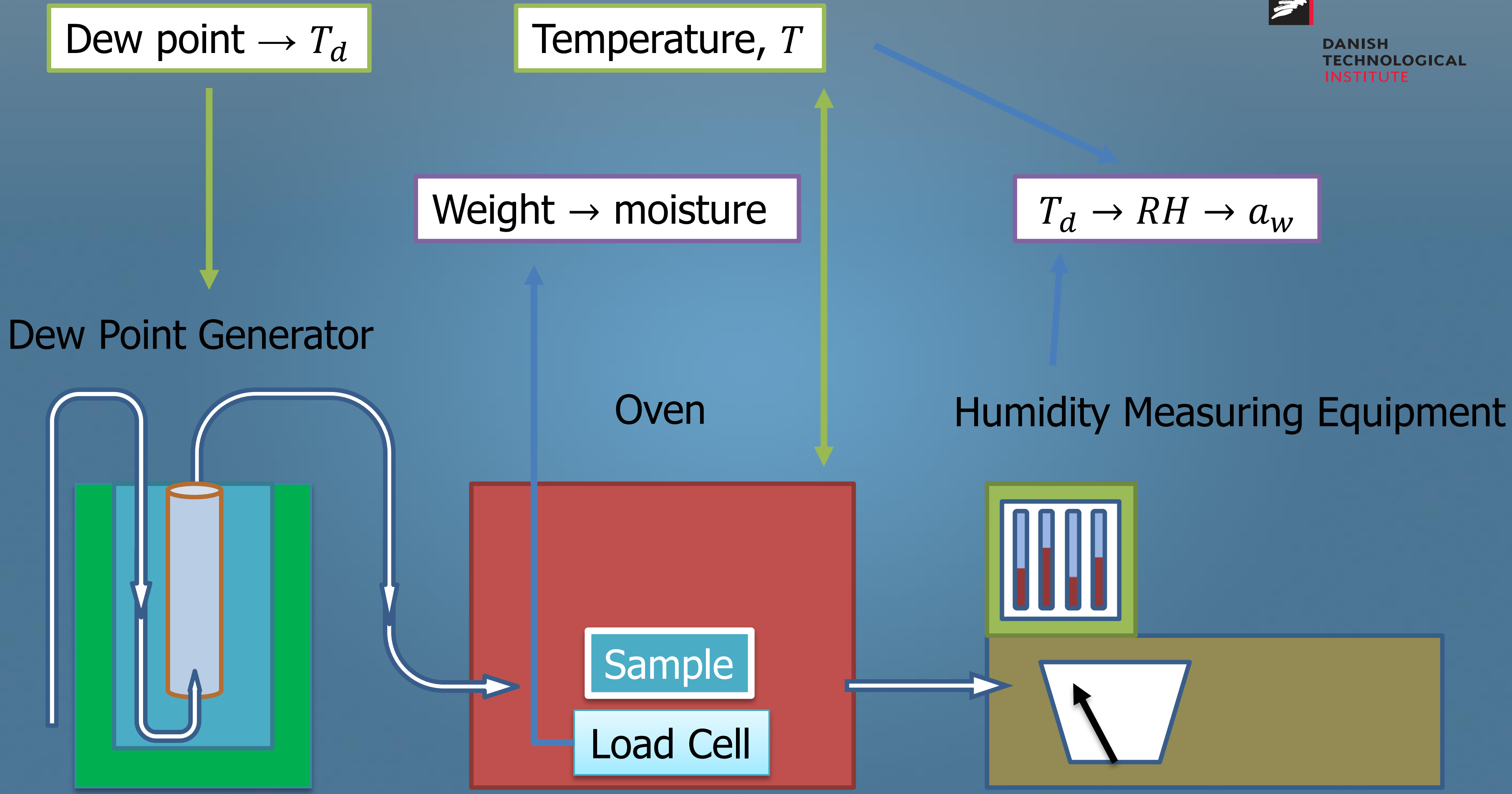


Ref 3

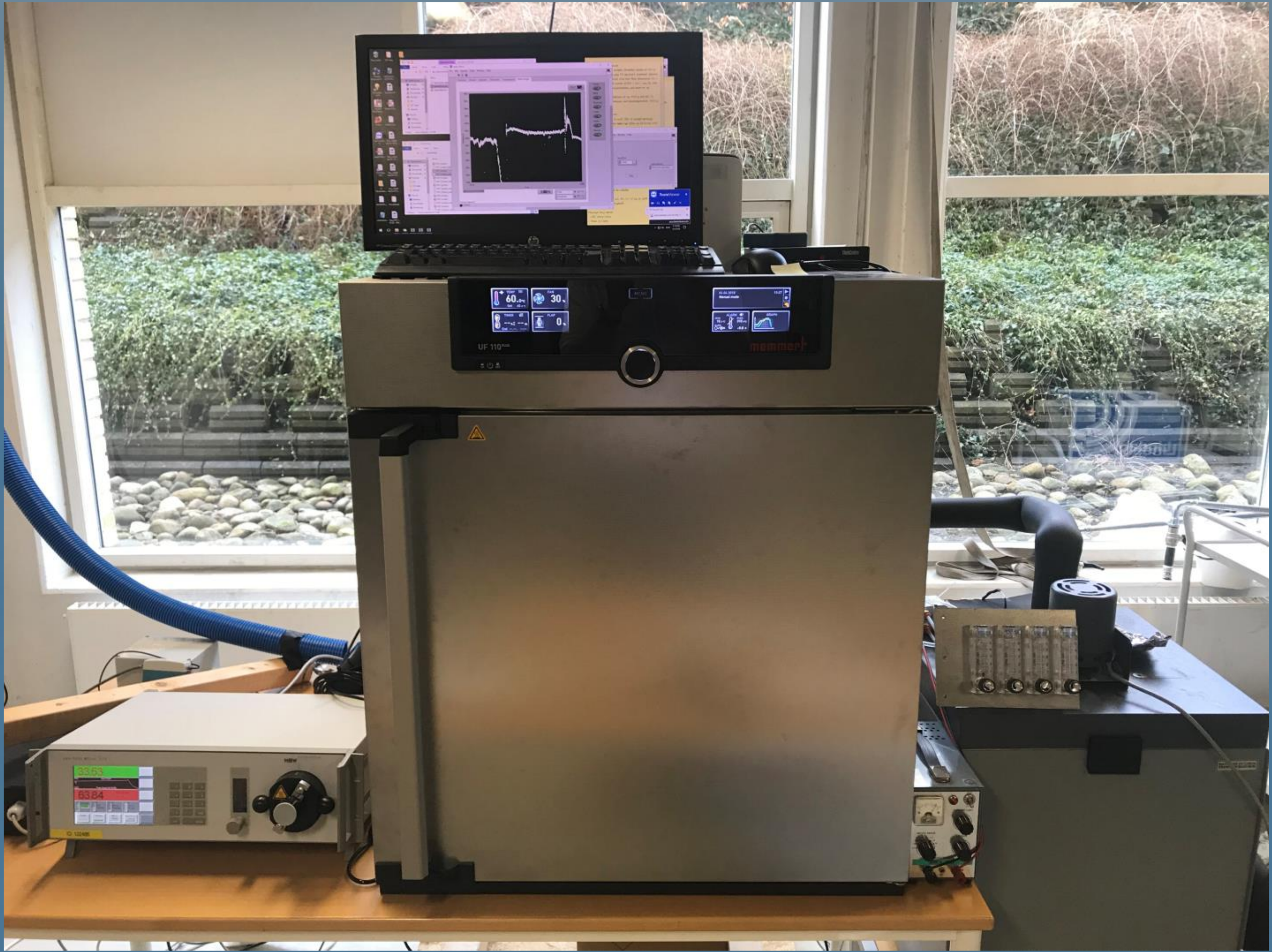
Sketch of setup

- NB: Traceable measurements





Dew point → T_d
Temperature, T
Weight → moisture
$T_d \rightarrow RH \rightarrow a_w$
Dew Point Generator
Oven
Humidity Measuring Equipment
Sample
Load Cell



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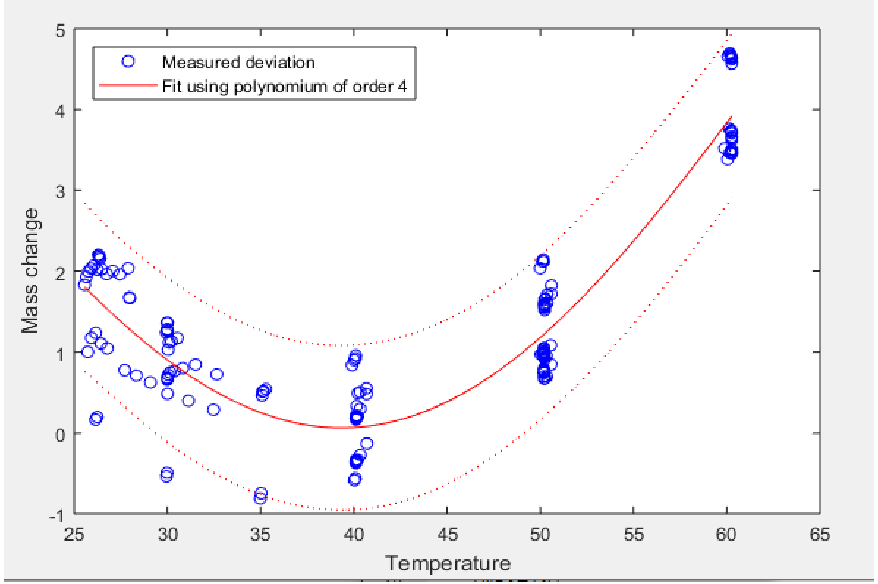
Challenge 1: Load cell in oven

- Mass of chamber: 5000 g
- Operates up to 85 ºC
- Challenge: Repeatability
- Uncertainty: ±1 g $(k = 2)$



Samples so far

(Milk powder, clots at high temperature)

1. Hazelnuts (ground, ~300 g)
2. Dog feed (500 g)
3. Hazelnuts (whole, ~500 g)
4. *Cat feed (from production line)*

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Challenge 2: Slow rate

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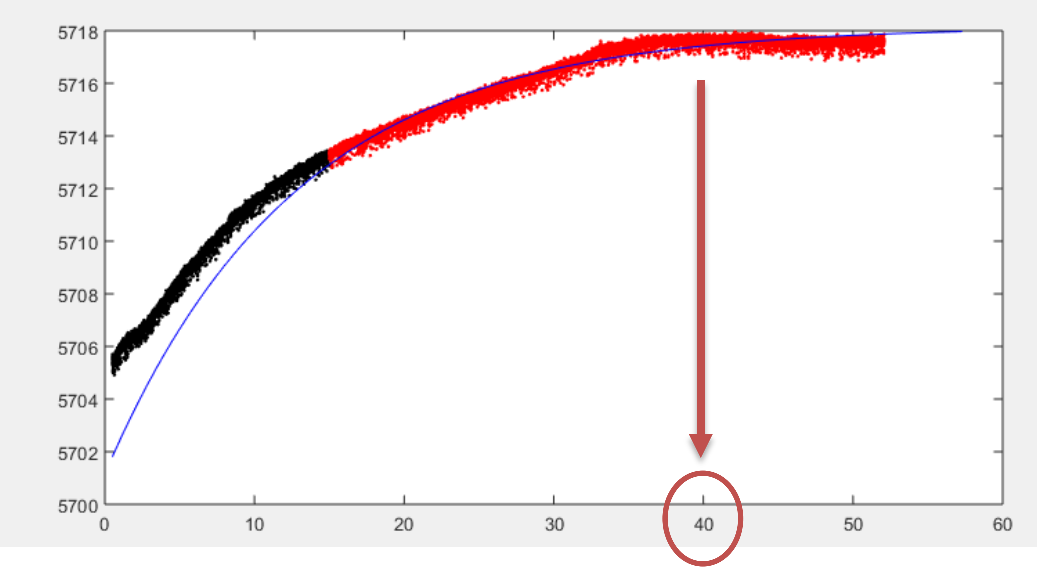
- Equilibrium

$$a_w = RH$$

- 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)

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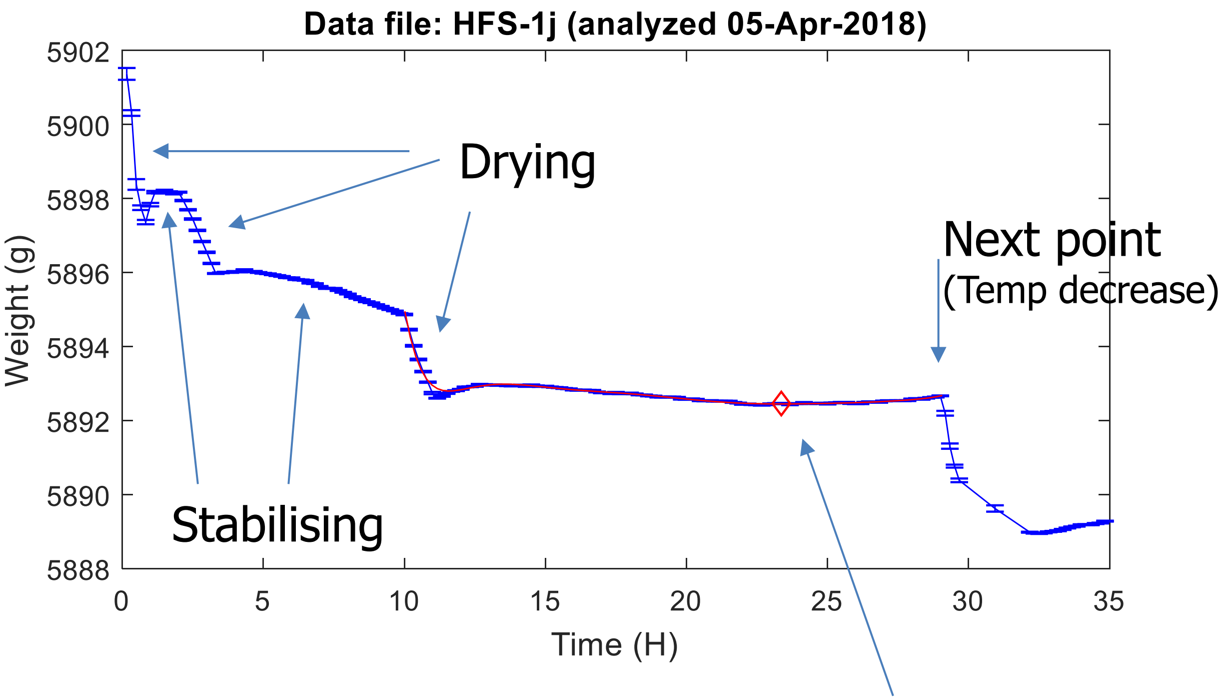
Challenge 2: Slow rate (cont.)

~~Solution 3: Small sample~~

Large samples required for representative sampling in food and feed production

Measuring procedure (i) (25 – 70 °C)



Advantages

- Fairly robust
- Reasonably fast

Challenges

- Adjustment by hand required
- Require significant moisture change

Strategy: at T = const. -> adjust T_d until Weight = const. $\left(\frac{\partial W}{\partial T_d} = 0\right)$

Recirculation

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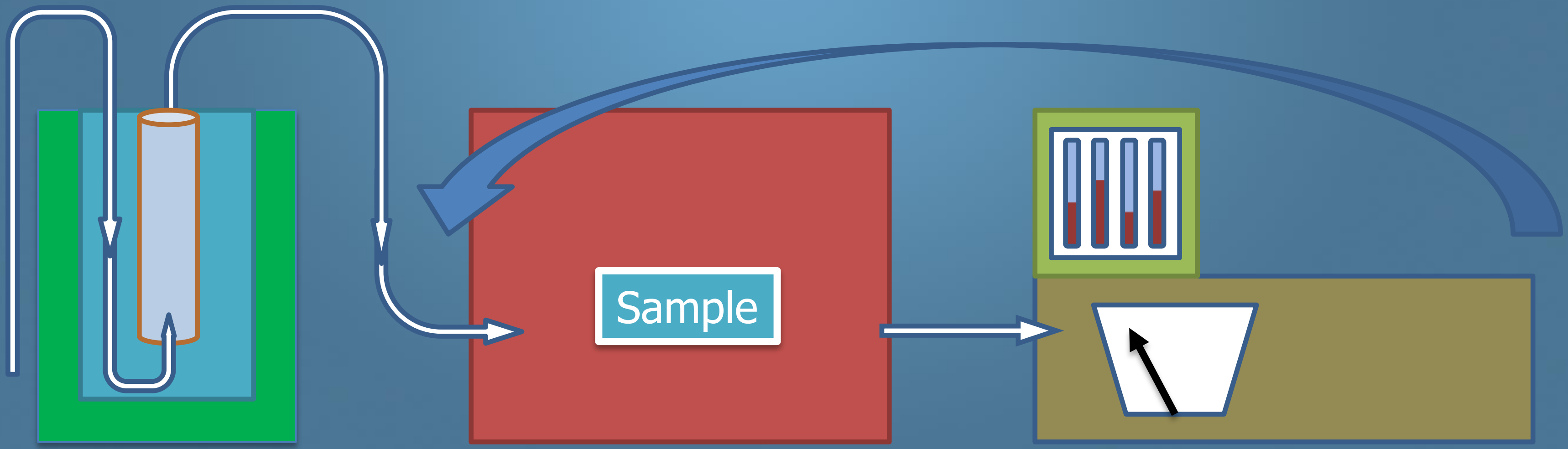
- Method: Gas output feeded into gas input
- Principle: RH of surrounding air reach equilibrium
- Effective for large sample / small gas volume



Measuring procedure (ii) (30 – 50 °C)

→ *Recirculation of gas for a_w measurement combined with predictive algoritm.*

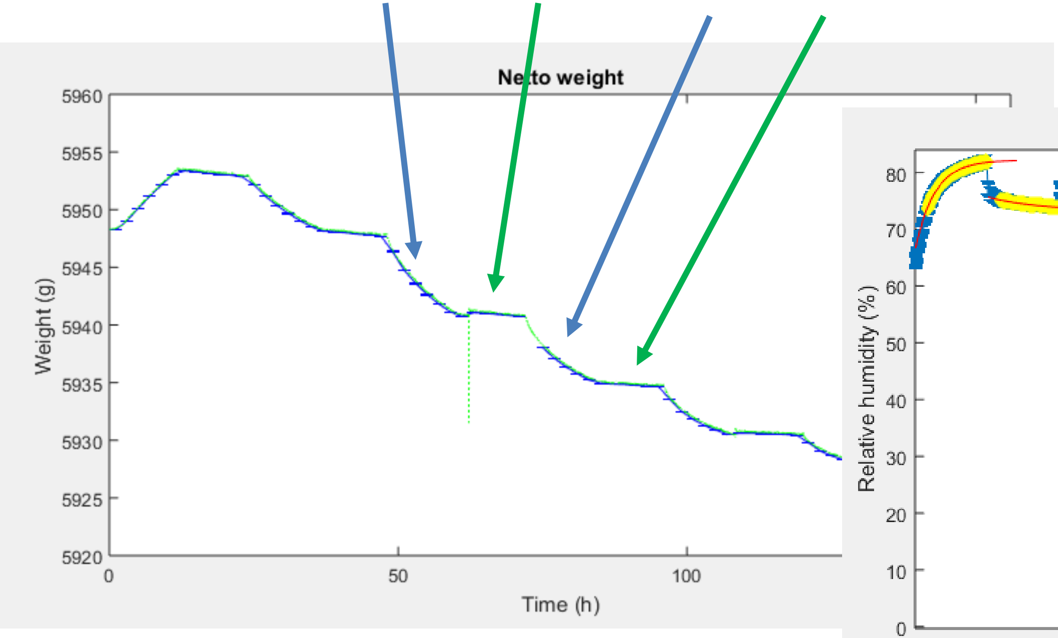
Sequence: Dry – Recycle – Dry – Recycle – …

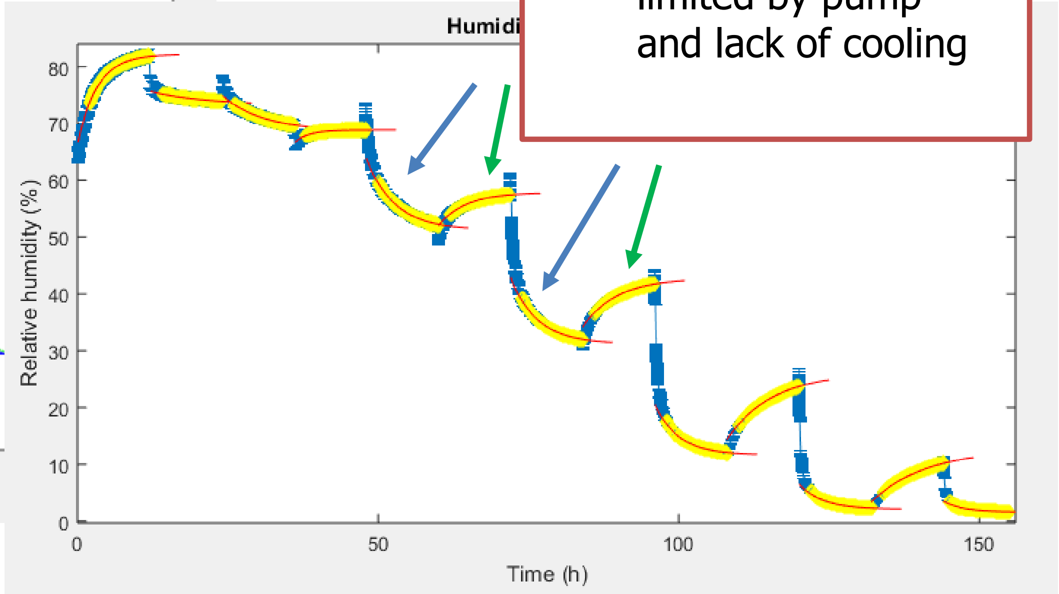
Advantages

- Robust
- Easy
- Economical (water)
- $M\ vs.T$

Challenge

- Temperature range limited by pump and lack of cooling

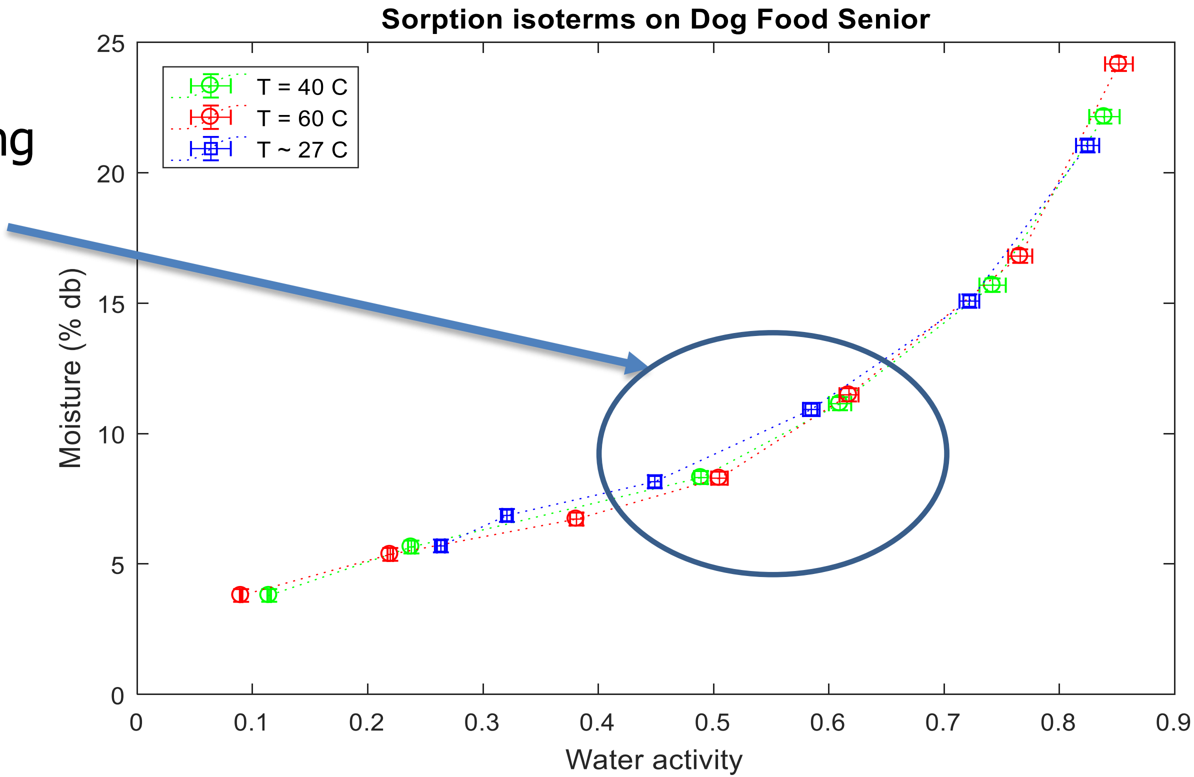




Sample: Dog Food – Senior

Proc. (i)

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



Curve fitting

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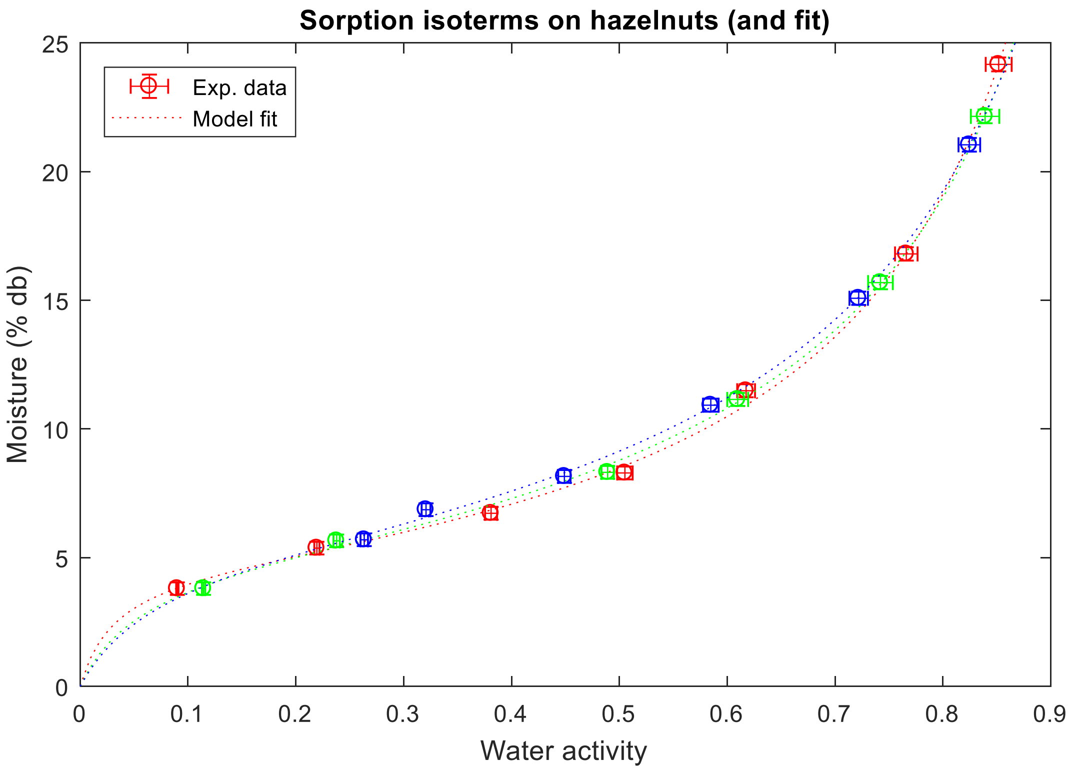
- Model

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

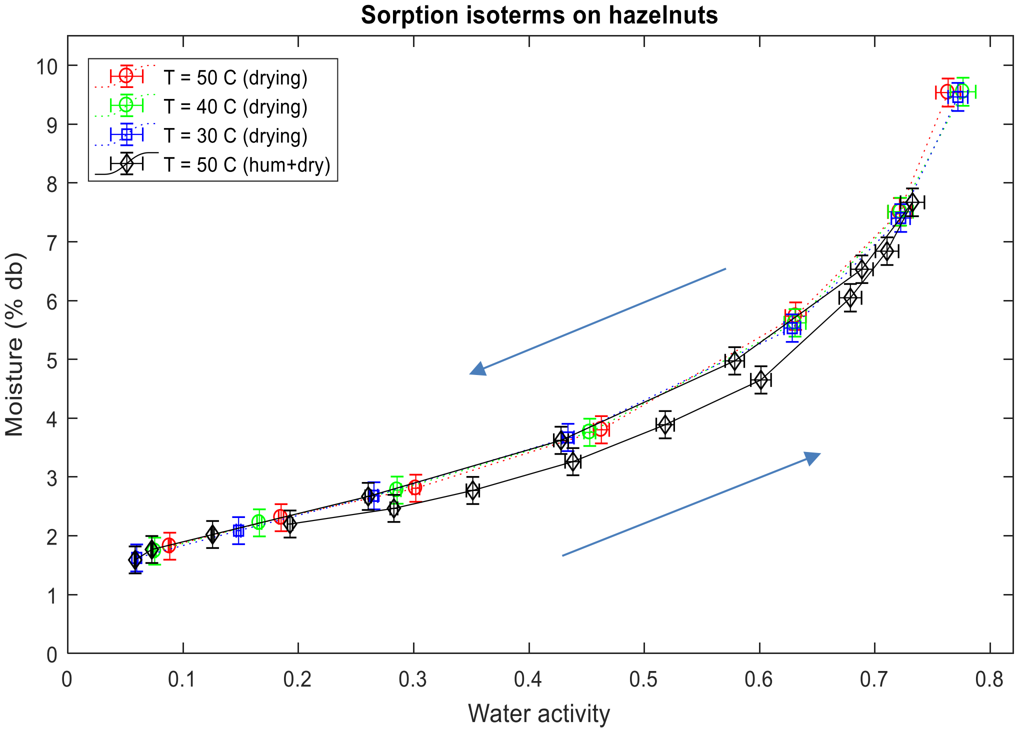
- Based on *Hailwood and Robin Model* (GAB)



Sample: Whole hazelnuts

Proc. (ii)

- Improved procedure
  - Large sample
  - LoD: Loss-on-Drying
  - Recirculation
- Temperature effect small
- Water content smaller (compared to dog food)
- Hysteresis



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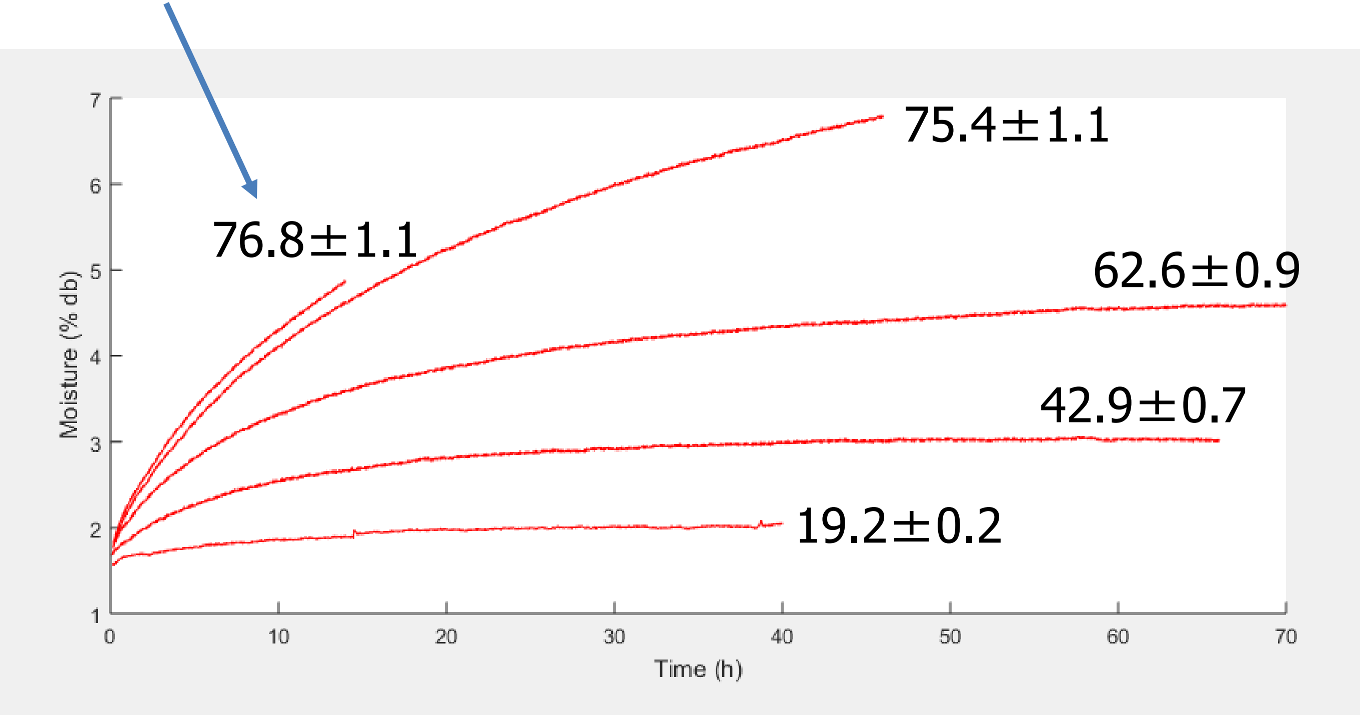
Water content (moisture) vs. Time

- Needed for calibration of model (UNICLAM)
- A2.2.1 / A2.2.2 / A2.2.3
  - T = 50 °C
  - Relative humidity of air input indicated



- Results of the numerical model in transient conditions
- Pressure diffusion coefficient calibrated by using present data

Source: Gino Cortellessa (Uniclam)

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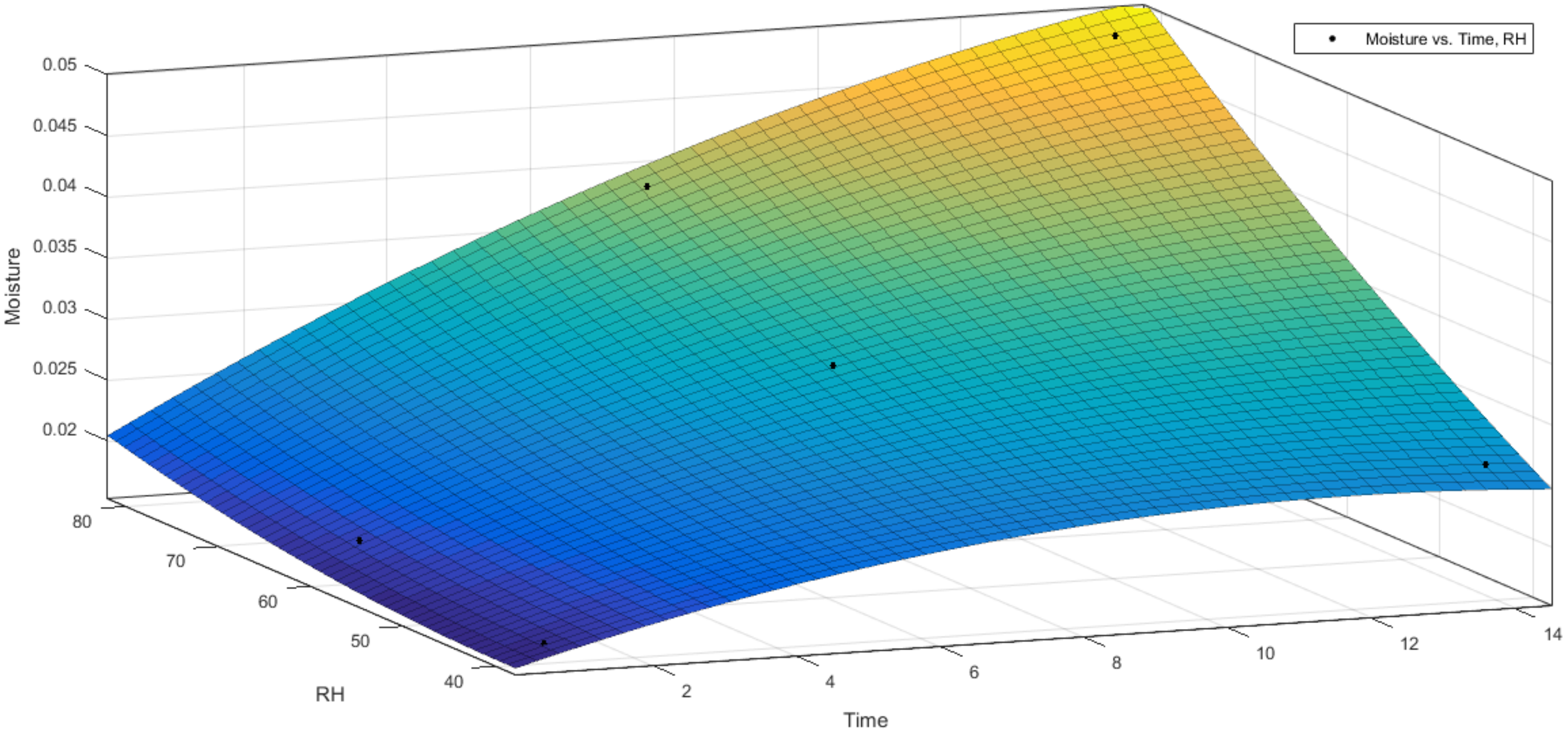

Conclusion

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- New setup for measuring sorption isotherms
  - Temperature: ≤ 70 °C
- Sample size: 300 – 1000 g
  - Allows for representative sampling at food / feed production
- SI Traceable (through choice of method and calibration of sensors)
- Uncertainty ($k = 2 \rightarrow 95\%$ confidence)
  - Weight: ±1 g
  - Water content: ±0.25% @ 10% (for sample ~ 500 g)
  - Water activity: better than ±1% @ $a_w = 80$ %

Left for HIT-project step

- Reporting
- Inline sampling at production line

Your contact:

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References

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- 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