Measuring Water Content in Raisins

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Aim of this Work

Development of a methodology for the fast and accurate measurement of the absolute amount of water in raisins

- Production departments
- Collection points
- Storage places
- Quality control processes
- Packaging processes

Water in Raisins

Water content – key parameter to the quality of raisins

- Nutritional value and flavor
- Degradation processes

Two major problems in measuring water content in raisin

- Complex mixture of sugars and volatile substances
- Outer semi permeable membrane

Official Methods for Measuring Water in Raisins

- Moisture in Dried Fruits (22.013)

1. Weighing of the sample (5 g)
2. Mixing with asbestos (2 g)
3. Moisten with hot water
4. 6 h **oven drying** at 70 ^{o}C under vacuum (P < 100 mm Hg)

Total analysis time at least 7 hrs

Low accuracy and reproducibility

Official Methods for Measuring Water in Raisins

- Dried food moisture tester meter (22.014)

1. Grinding of the sample (3 times)
2. Cooling of the sample
3. Packing ground sample into a bakelite cylinder with fingers
4. Measurement of sample conductivity with two electrodes

Low accuracy and reproducibility

Requirements for the New Method

- Fast measurement (less than 5 min)
- High Accuracy (> +/- 0.5%)
- Applicable to field measurements
- Elimination of dangerous reagents
- No need for skilled personnel
- Low cost

Methods for Measuring Water Content

- Thermogravimetric Methods
  - Oven drying
  - Infrared drying
  - Halogen drying
  - Microwave drying

  - Low accuracy and reproducibility
  - Possible decomposition of the sample

Methods for Measuring Water Content

- Chemical Methods
  - Redox titration (Karl Fischer)
  - Calcium carbide method

    $CaC_2 + 2H_2O \rightarrow Ca(OH)_2 + C_2H_2$

  - Use of dangerous reagents
  - Need for skilled personnel

Methods for Measuring Water Content

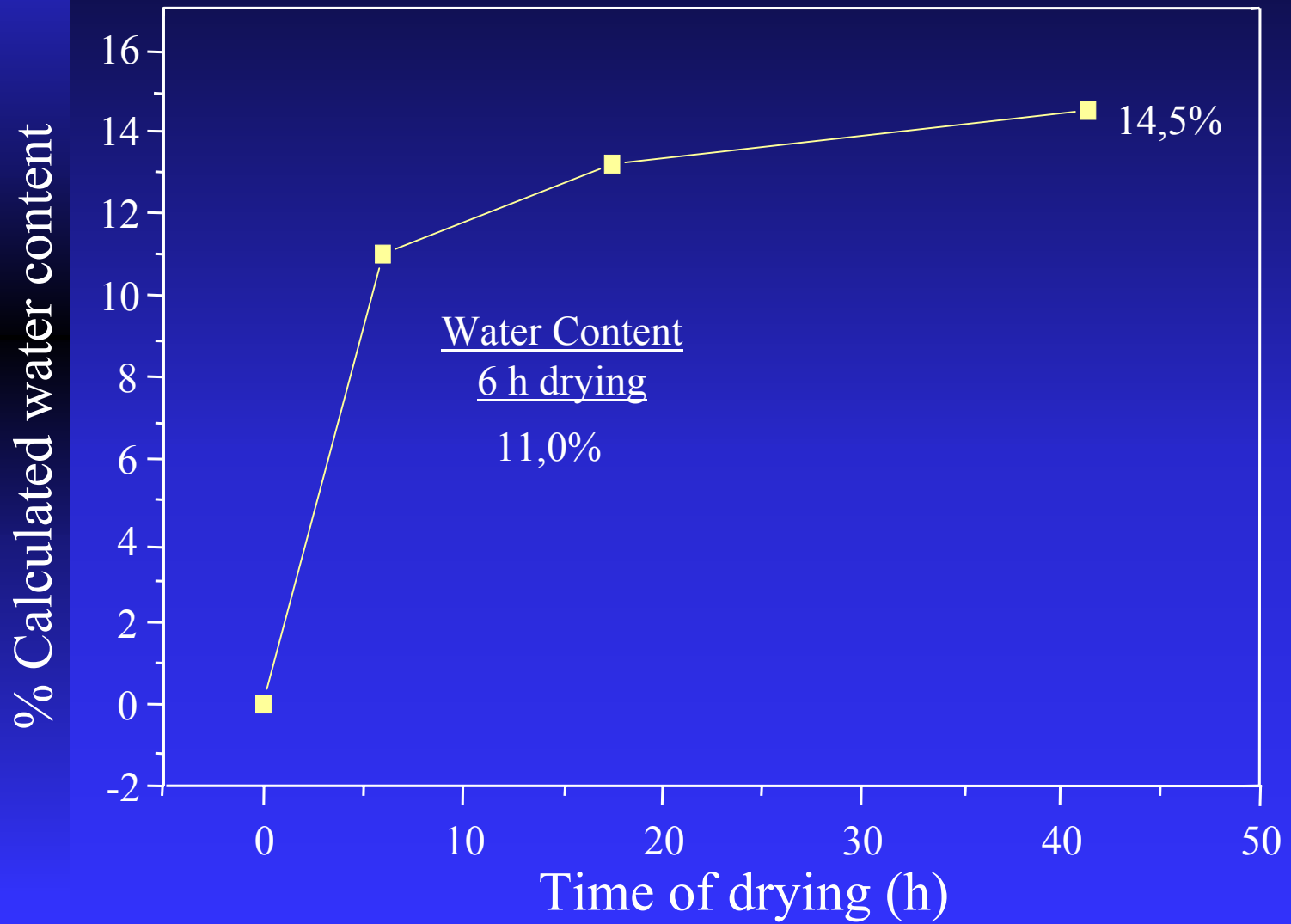
- Spectroscopic Methods
  - NIR spectroscopy
  - Microwave spectroscopy
  - NMR spectroscopy

  - Low accuracy for non homogenous samples
  - Expensive equipment needed

Methods for Measuring Water Content

- Other Methods
  - Electrical conductivity measurement
  - Distillation
  - Water activity sensors

Oven Drying
(70 °C, under vacuum)



Karl Fischer Titration

$$I_2 + 2H_2O + SO_2 \longrightarrow 2HI + H_2SO_4$$

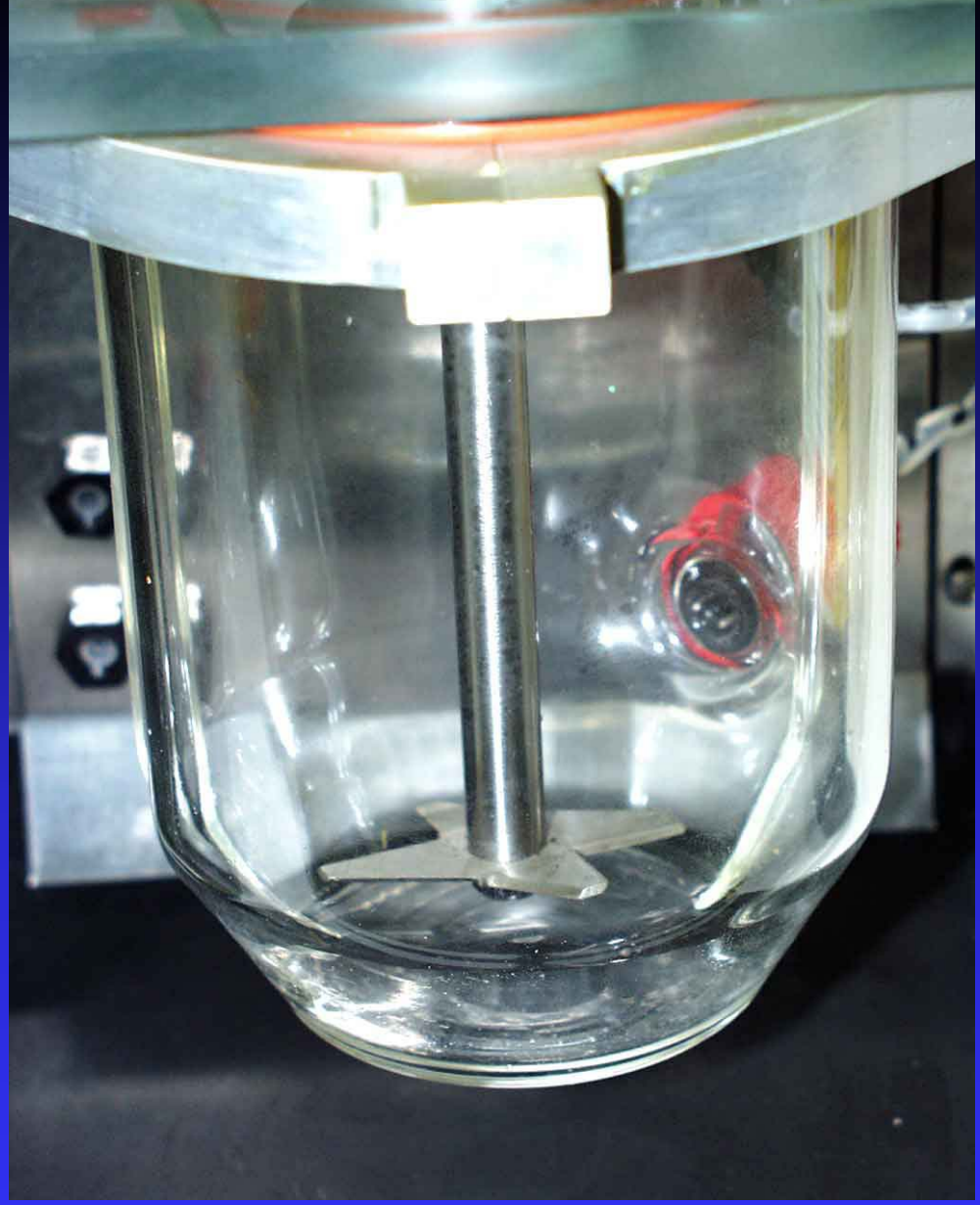
- Volumetric Karl Fischer Titration
- Coulometric Karl Fischer Titration

$$m = \frac{qM}{nF}$$ Faraday's Law

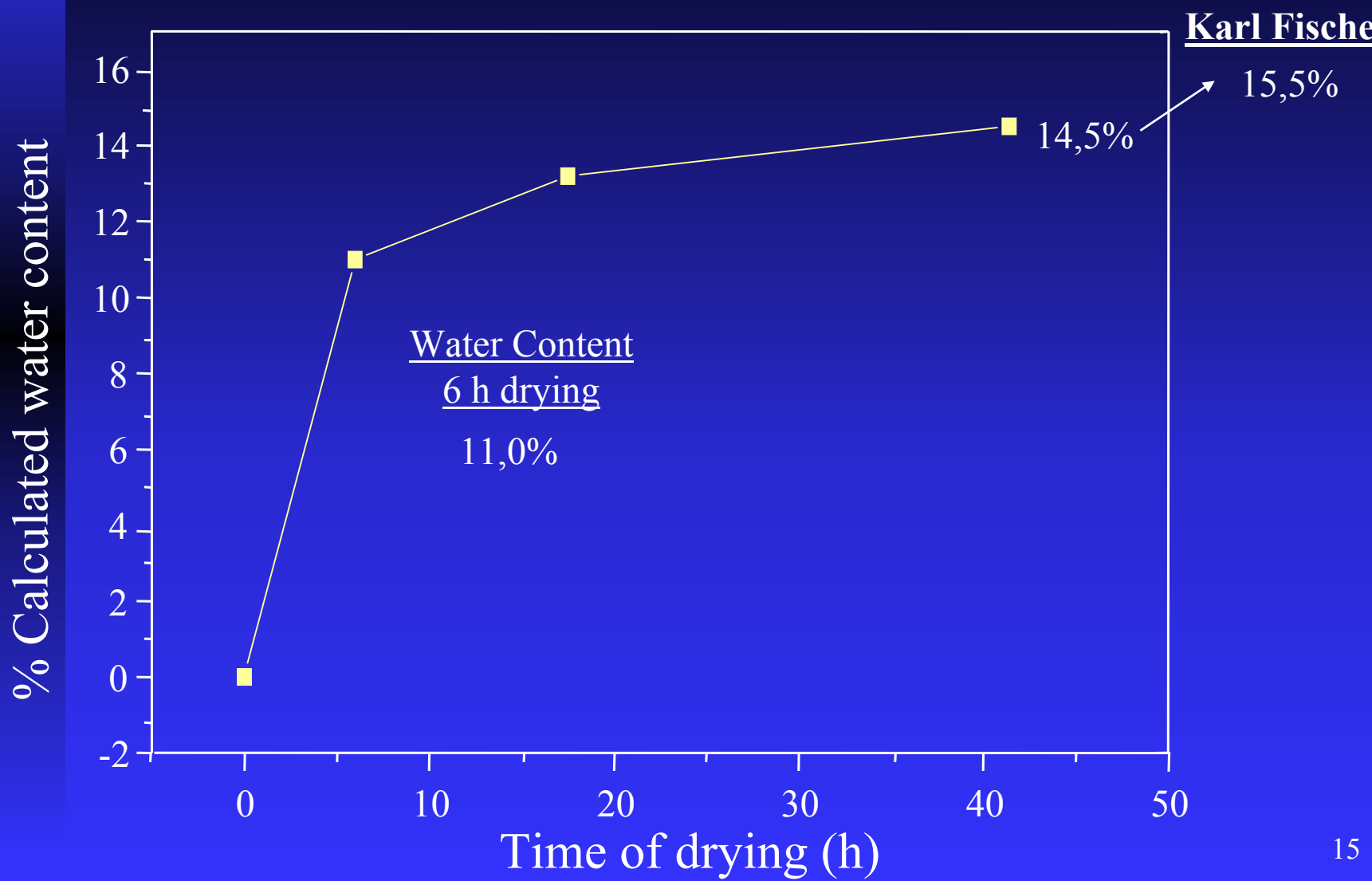
Applying Karl Fischer Titration to the measurement of water in raisins

- Incomplete extraction of the water of raisins
- Absorption of moisture from the environment
- Non-stable end point of the titration

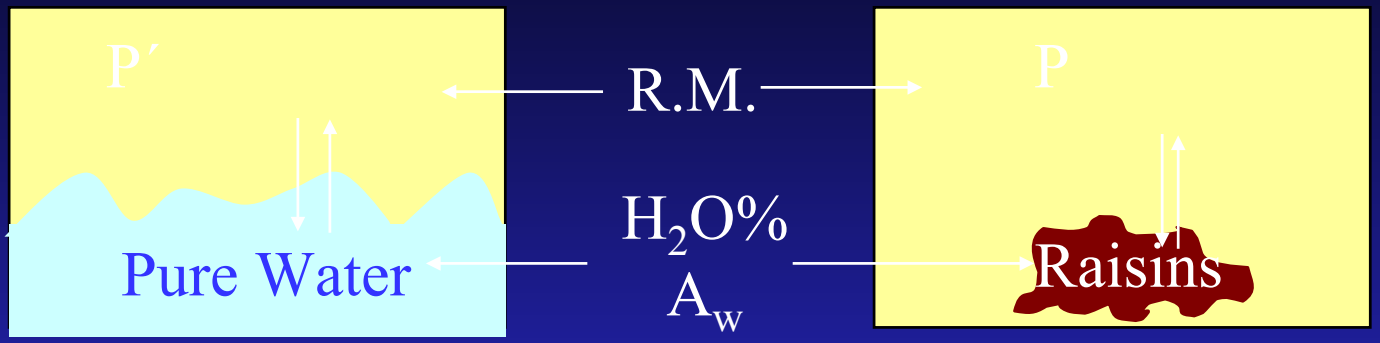
Karl Fisher Titrator (THERMO ORION)



Oven Drying vs. Automatic Karl Fischer Titration



Principle of Operation of Water Activity Sensors



Relative Moisture (R.M.) $\quad R.M.\% = \left(\frac{x}{x'}\right)_T x100 = \left(\frac{P}{P'}\right)_T x100$

Activity of Water (A_w) $\quad \mu_{(P,T)} - \mu'_{(P,T)} = RT\log A_w$

$$A_w \cong \frac{R.M.\%}{100} = \left(\frac{P}{P'}\right)_T$$

Water Activity Sensors

- Selective for water
- High accuracy
- Fast response
- Easy to use
- Low cost

- Need for calibration
- Thermodynamic equilibrium necessary


rotronic
A 2
HYGROMER®
74.0%RH
18.1°C
HOLD
ON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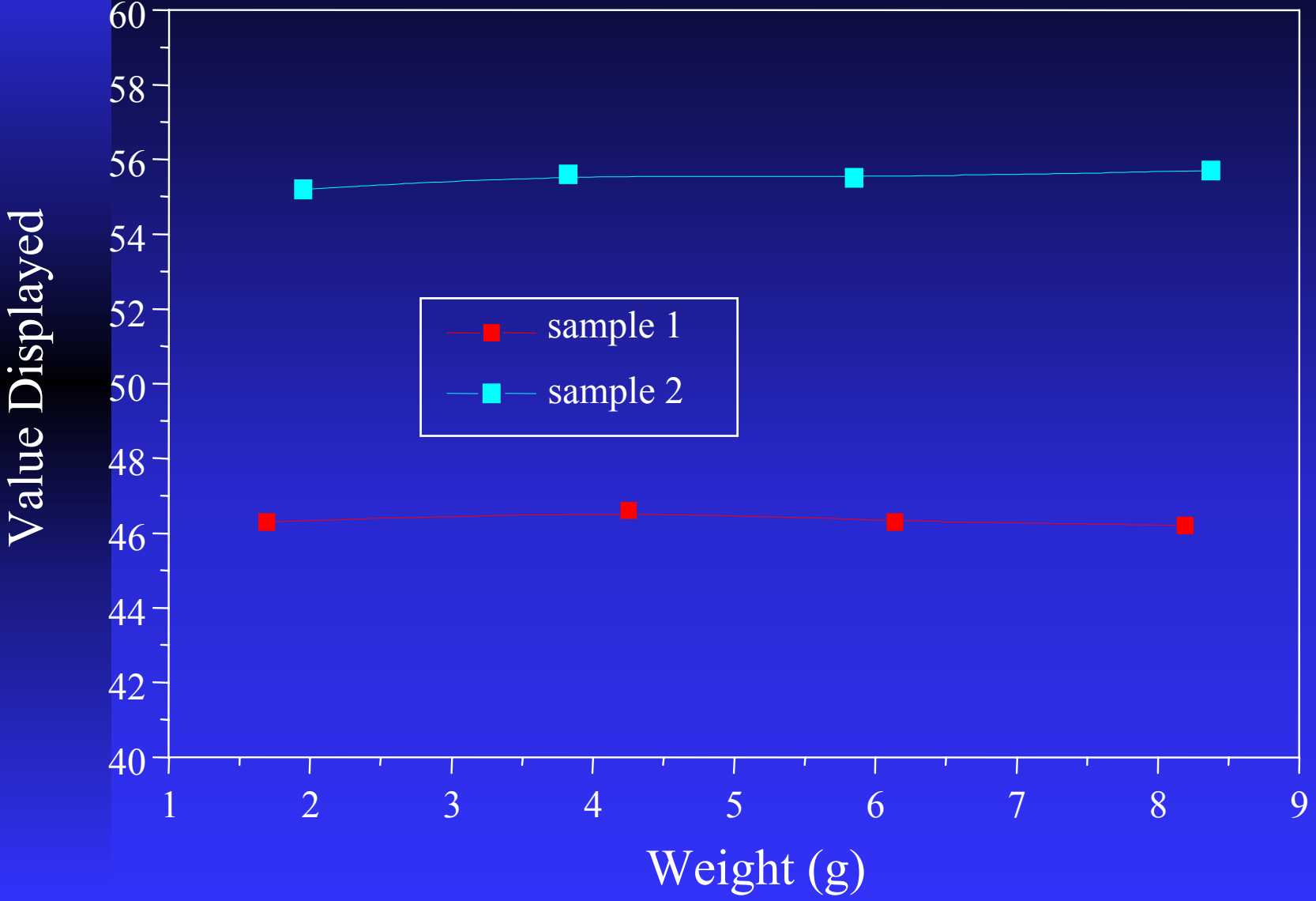
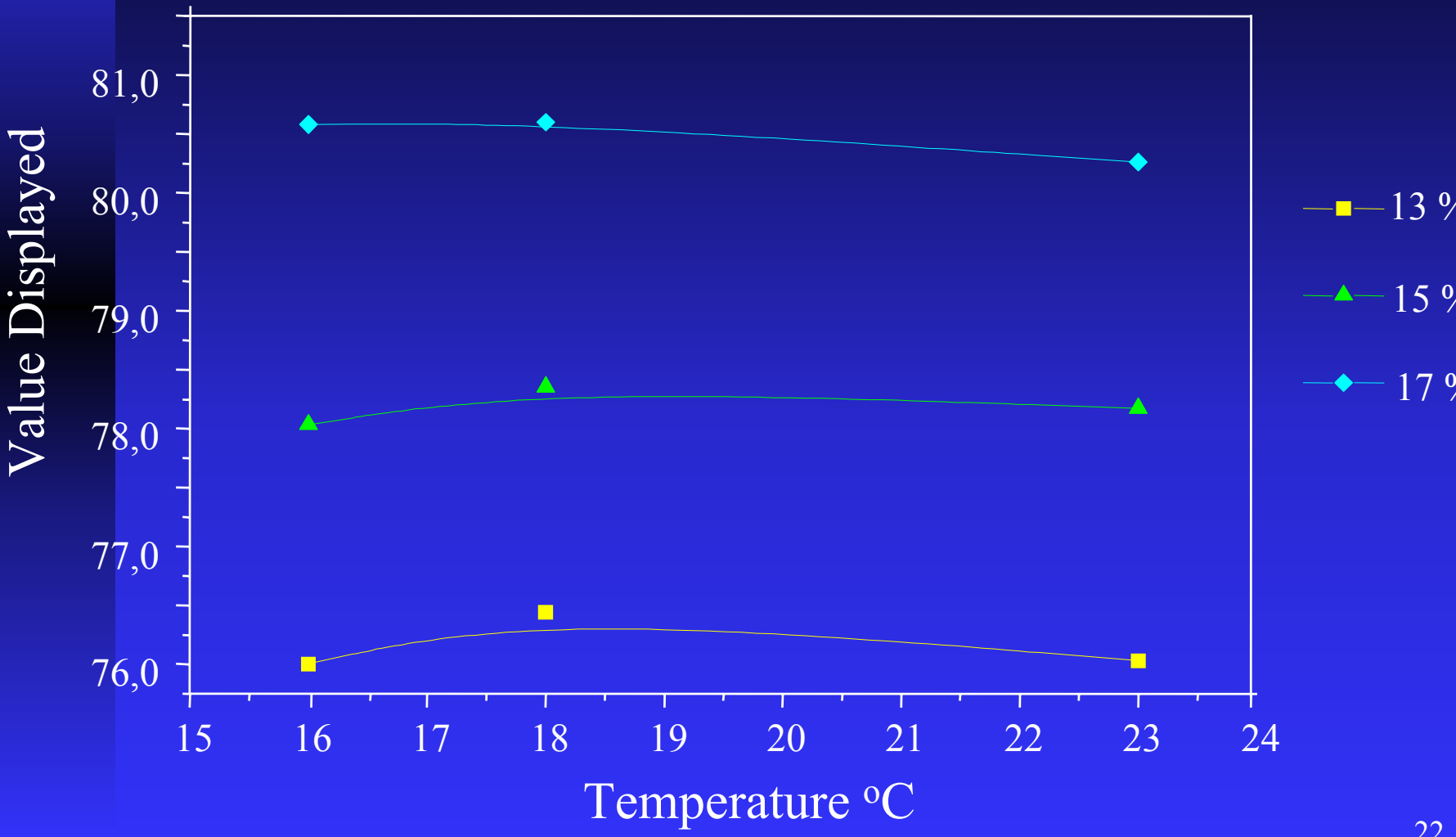
CE

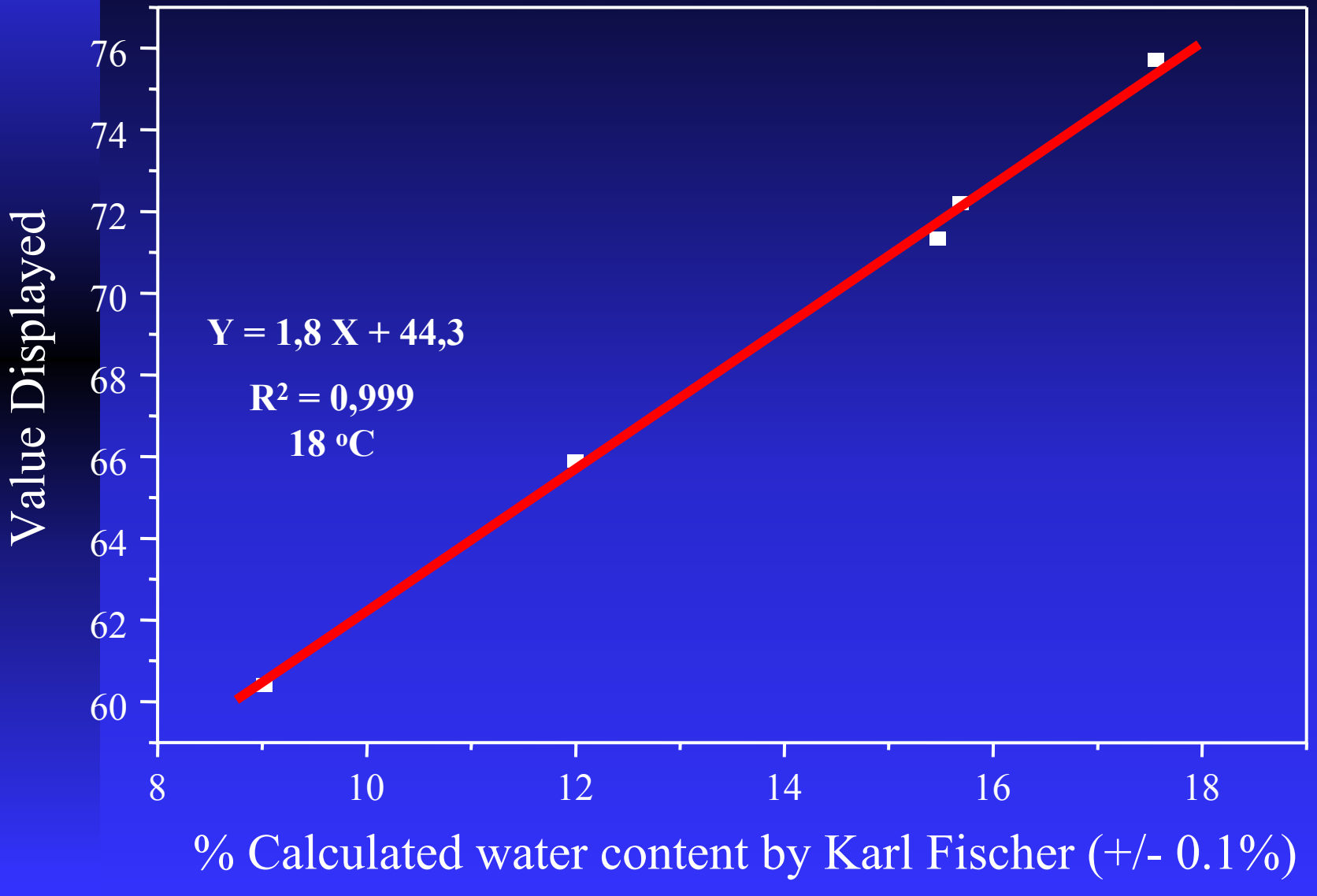
Effect of Sample Weight



Effect of Temperature



Calibration Curve



Conclusions

Advantages of method developed

- No need for sample weighing
- Measurement in 5 min
- Accuracy > +/- 0,5%

Disadvantages of method developed

- Need for calibration curve with standard samples
- Display in relative units
- Need for correlation graph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