

Determination of water in various types of coffee and related products



**Conference on Water in Food
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Methods for Determination of Water Content

- **LOD**
- **Micro wave**
- **NMR**
- **GC**
- **Karl Fischer titration**
 - by direct volumetric titration
 - by the indirect method using a KF oven

Our investigations

- **Comparison of results LOD and KF titration for roasted, grinded coffee beans**
- **Comparison of results for direct volumetric titration in order to find out the most suitable procedure**
- **Comparison of results using the KF oven and LOD**
- **Comparison of results investigating three different Coffee extracts**

Content of Coffee Beans according Literature

	Raw	Roasted
Water	8-12%	?%
Sugars	10%	2%
Cellulose	24%	25%
Oils and fats	12%	14%
Chlorogene acid	6,8%	4,5%
Proteines		4%
N-based substances	12%	
Non N-based substances		18%
Derivatives of carbohydrates		30%
Coffeine	1,1-4,5%	1,1-4,5%
Water soluble extracts		24-27%
Ash	4,1%	4,5%



Equation of Karl Fischer Titration

Eugen Scholz postulated



Coffee Extract

We have investigated 3 types of coffee extract:

- **Freeze dried**
- **Agglomerated**
- **Spray-dried**

and found out, that in each case to some degree a side reaction took place.

The side reaction is pH dependend, typically, if an ingredient causes a side reaction with iodine as polyphenols.

After lowering the pH by addition of salicylic acid the side reaction was supressed.

An addition of formamide improved the dissolution of the coffee or extracted its water.

Results Coffee Extracts

Freeze dried (dissolved)
3,17% H₂O secondary reaction visible
2,18% H₂O after addition of salicylic acid

Agglomerated (water extracted)
5,54% H₂O weak secondary reaction visible
5,32% H₂O after addition of salicylic acid

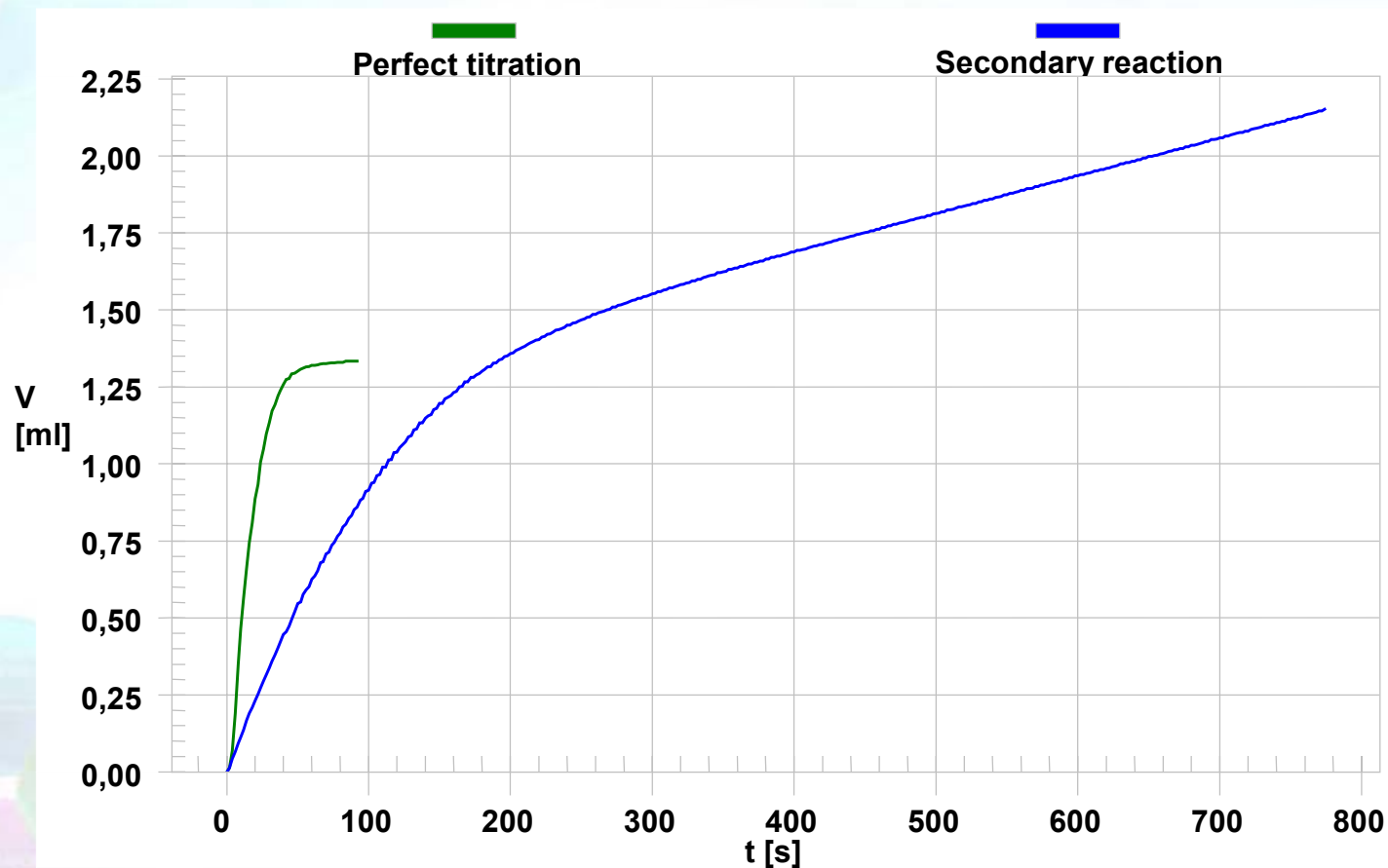
Spray dried (widely dissolved, some flakes left)
strong side reaction, no EP, titration interrupted
5,22% H₂O after addition of salicylic acid

General procedure

40 ml of a methanol-formamide mixture 2:1 and 7g salicylic acid is filled into the titration vessel and titrated to dryness with HYDRANAL[®] Composite 5.

Approx. 0,5g sample is then exactly weighed in by difference and its water content titrated with HYDRANAL[®] Composite 5.

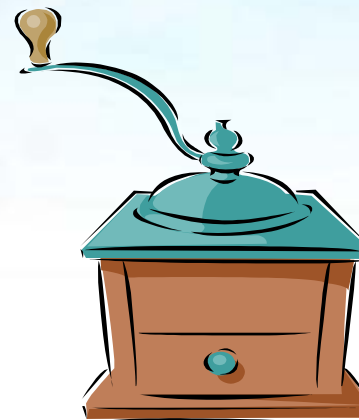
Secondary reaction - performance visible by a titration curve



Coffee Industrial Grinded

We analyzed it

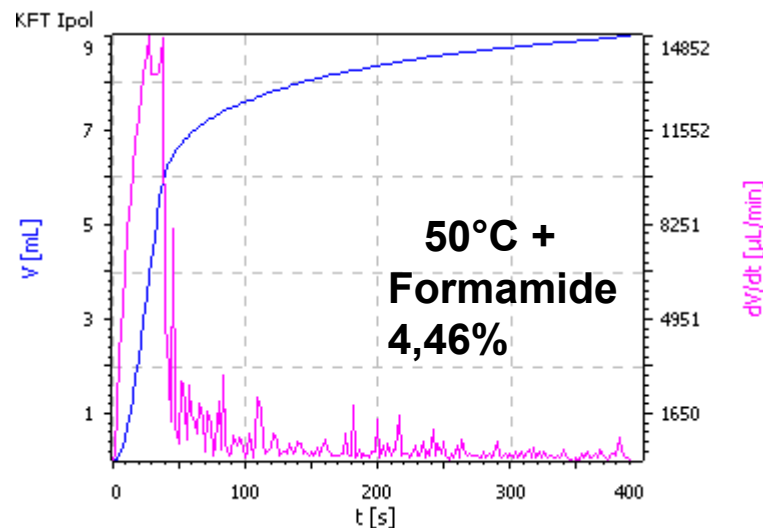
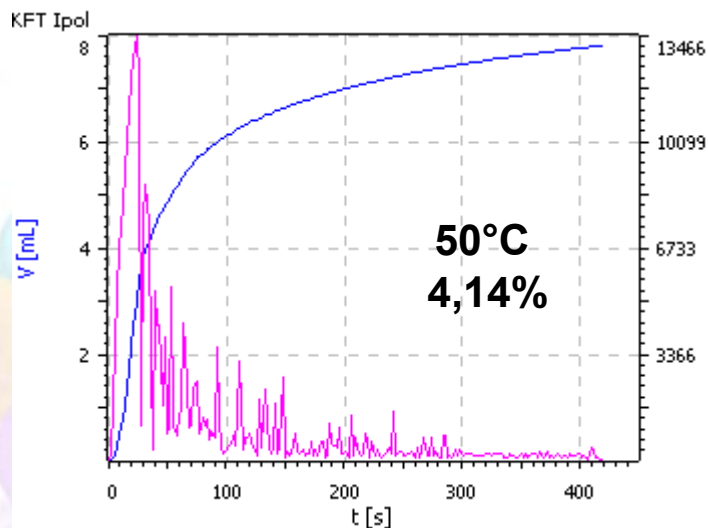
- 1. At 50°C in 40 mL Methanol**
- 2. At 50°C in 20 mL Methanol + 20ml Formamide 1:1**
- 3. At 50°C in 20mL Methanol + 20mL Formamide 1:1 + 7g salicylic acid**
- 4. In 40 mL boiling Methanol**
- 5. In a KF Oven**



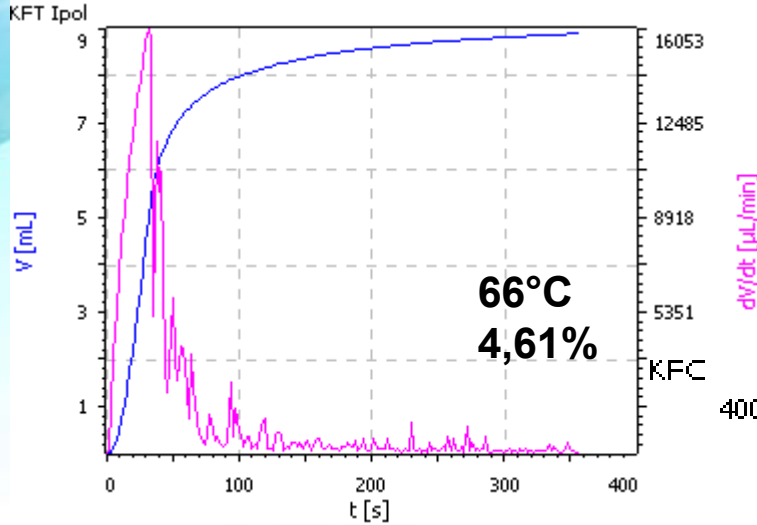
Coffee Industrial Grinded Results



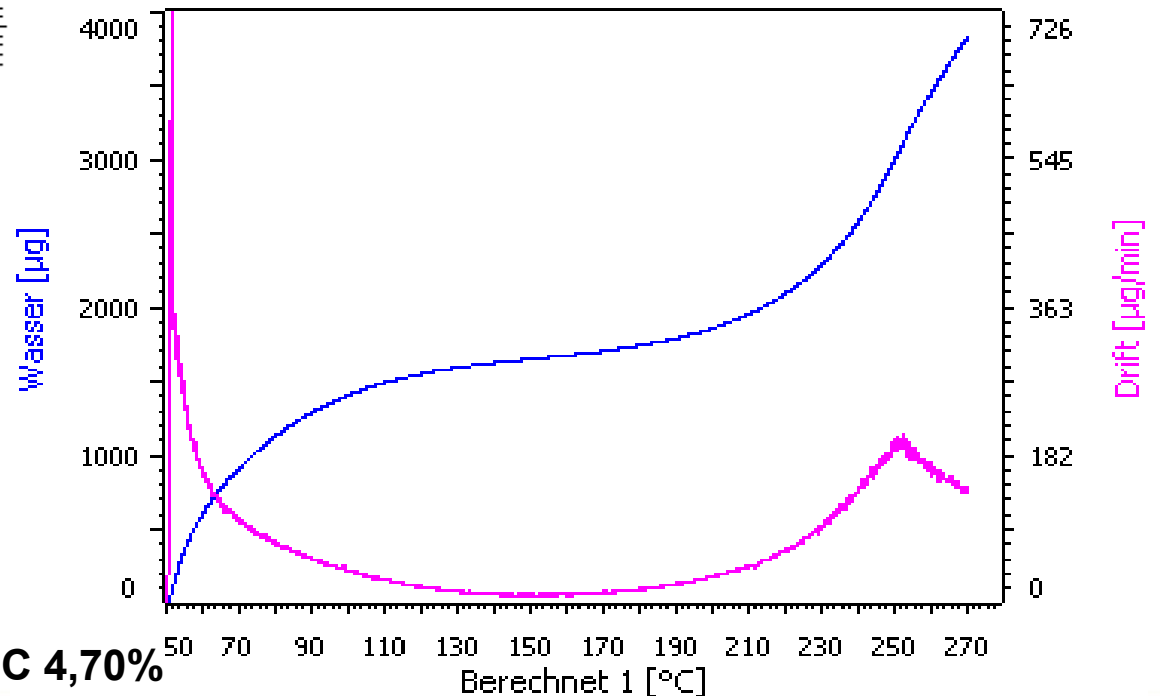
- | | | |
|----|--|------------------------|
| 1. | 50°C in 40 mL Methanol | 4,14% H ₂ O |
| 2. | 50°C in 20 mL Methanol + 20ml Formamide 1:1 | 4,46% H ₂ O |
| 3. | 50°C in 20mL Methanol + 20mL Formamide 1:1 + 7g salicylic acid | 4,46% H ₂ O |
| 4. | In boiling Methanol | 4,61% H ₂ O |
| 5. | 150°C in a KF oven | 4,70% H ₂ O |



Coffee Industrial Grinded Curves

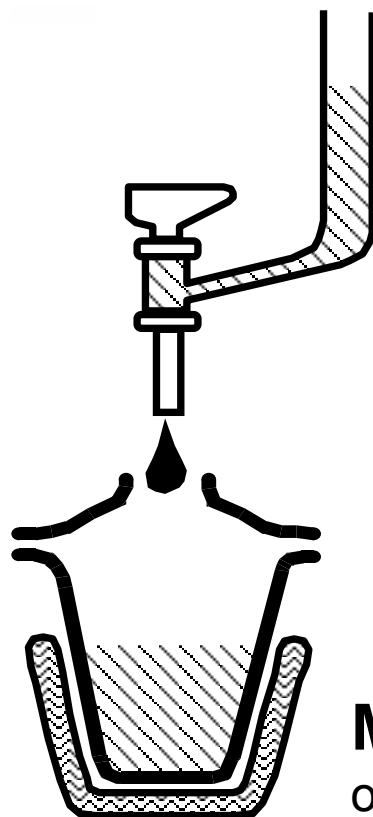


Temperature ramp 50°C – 250°C



KF Oven 150°C 4,70%

The titration at 40°C or 50°C
accelerates the extraction of the water or the
dissolution of the sample



Titration agent
containing all KF
reactants necessary
plus buffer dissolved in
e.g. DEGEE

Methanol

Methanol + Formamide
or other solubilizer

40 °C - 50°C

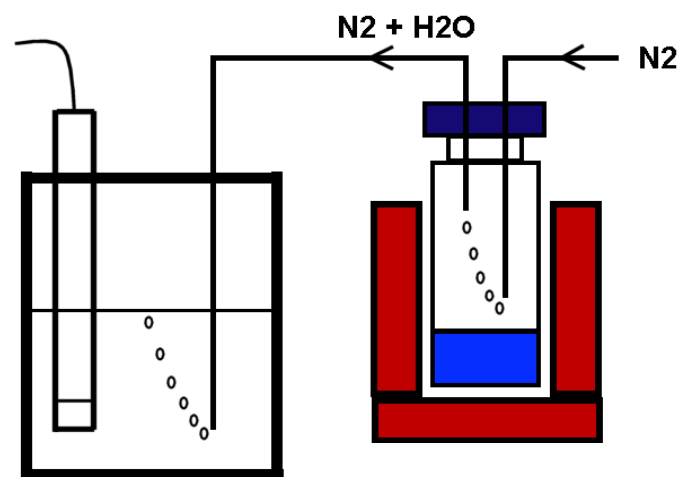
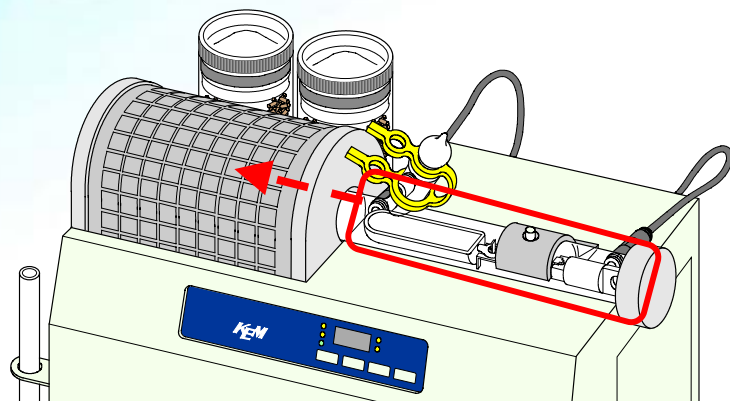
Titration in boiling Methanol



Change of
titration
parameters not
necessary

2. How to solve solubility problems by an indirect method

Sample evaporation in a KF oven



Results in an Overview



- **LOD**

2h 150°C	5,69%, 5,62%, 5,65%	Mean= 5,65%
24h 150°C	7,33%, 7,19%, 7,26%	Mean= 7,26%

Comment: Water, CO₂ and gases do volatile

- **KF titration**

150°C KF oven

4,70%

50°C in Methanol

4,14%

50°C + Formamid

4,46%

66°C in Methanol

4,61%

Related products

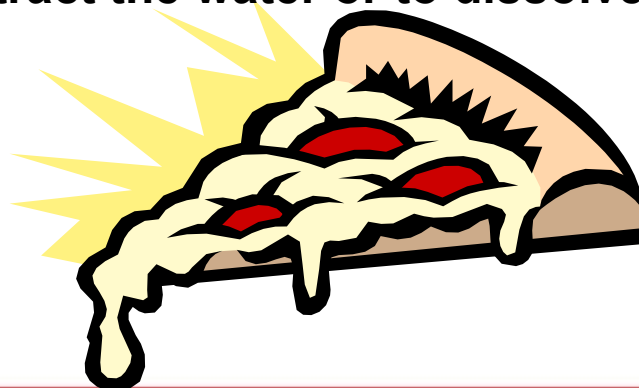


Coffee Cream

Can be titrated easily by volumetric titration, the limitation in accuracy is given by the handling of the small sample size for the high water content.

Cake and Cookies

Require like all natural products and sweets the titration at 50°C and the addition of formamide in order to extract the water or to dissolve the sugar.



Determination of water in various types of coffee and related products

Comprehensive: Grinded Coffee Beans

LOD Results can just be compared using same parameters strictly.

Direct KF titration

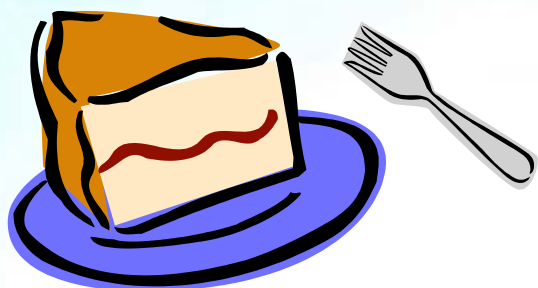
Results in boiling methanol seem to be most reliable.

KF oven

Results found using the KF oven and in boiling methanol are comparable

Comprehensive: Coffe Extracts

Important to lower the pH by addition of salicylic acid in order to avoid a secondary reaction.



Thank you!

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