Determination of Water Content in Soluble Coffee by Karl Fischer Method

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Presentation Outline

- Why do we need a precise method to determine the water content in soluble coffee ?
- What are the possible methods ?
- What do the measure ?
- How do they perform ?

Why do we need a precise method ?

- Legal Norms (Import/Export)
  - More products are exported into other countries.
  - Different legislation applies different methods.
- Process Control
  - Product consistency (composition, physical)
  - To guarantee stability during product shelf-live.
- To study the behavior of soluble coffee during processing and storage.

Method used

- P_2O_5 at 85°C for 2 hours
- P_2O_5 at 48°C for 2 weeks
- Karl Fischer
- Standard oven, 95°C, 2 hours, low RH
- Standard oven under tropical conditions (high RH), 95°C, 2 hours
- Vaccum oven, ISO 3726-1983, 70°C, 50 mbar, 16 hours
- Near infrared Spectroscopy as supporting technique

The Karl-Fischer method allows to determine the total water content

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1 $CH_3OH + SO_2 + RN$ Ü Þ $[RNH]SO_3CH_3$

2 $H_2O + I_2 + [RNH]SO_3CH_3 + 2RN$ Ü Þ $[RNH]SO_4CH_3 + 2[RNH]I$

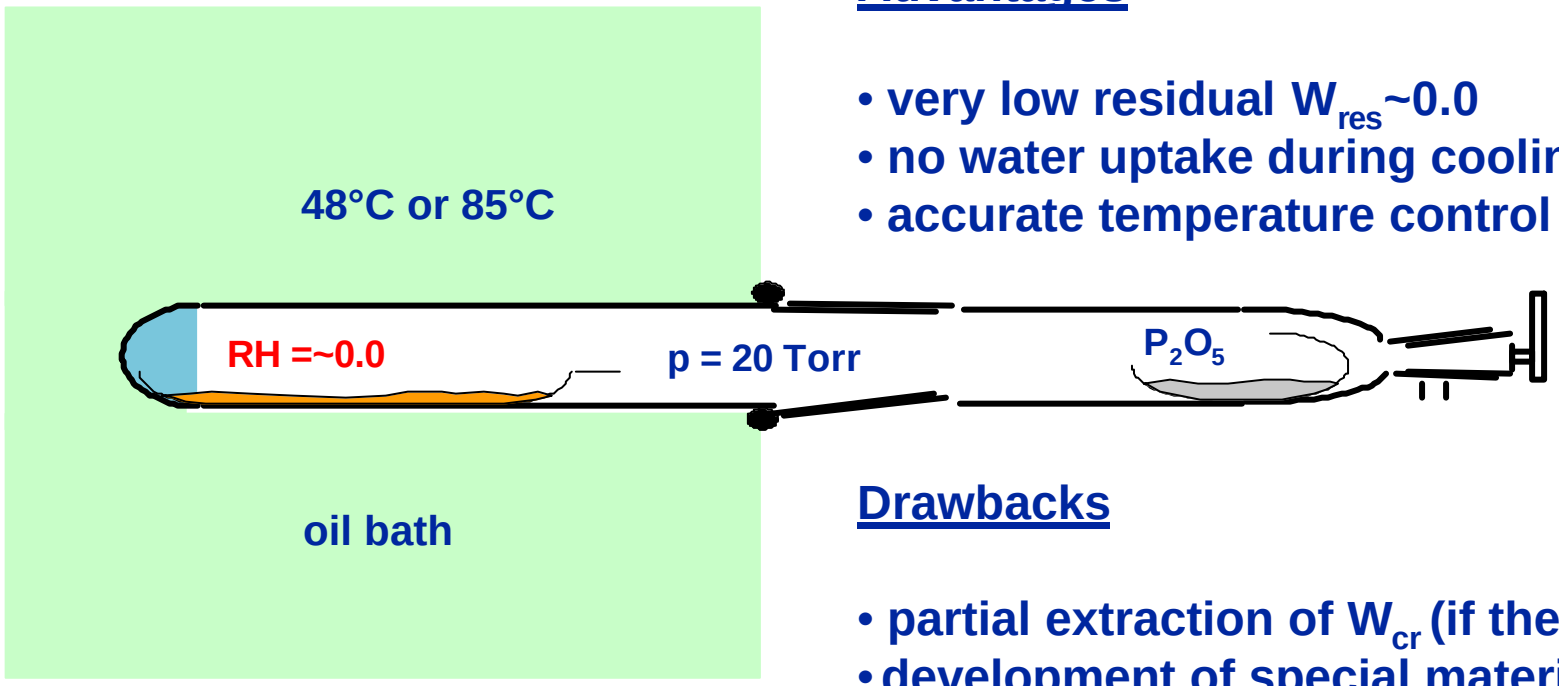
Solvent : **Methanol changed every analysis**

Sample Preparation: **fresh in mortar before every analysis**

Ipol : **5 uA**

Titer determination: **2 drops of water**

The P_2O_5 method results in reproducible and accurate determinations under well defined conditions



Advantages

- very low residual W_{res}~0.0
- no water uptake during cooling
- accurate temperature control

Drawbacks

- partial extraction of W_{cr} (if there is)
- development of special material
- problems related to P_2O_5

We use these method as a Nestlé reference method.
Conditions are individually adapted to the product.

The determination by the oven method can be very sensitive to laboratory conditions (RH, Temp.)

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Drying Conditions:	2 gram
Standard oven:	95°C
	2 hours
Lab. Cond:	25 +/- 2°C
	36 +/- 5 RH
Standard oven tropical:	95°C
	2 hours
Lab. Cond:	32 +/- 1°C
	63 +/- 2 RH
Vacuum oven:	70°C
	50 mbar
	16 hours

Advantage

- **no extraction of cryst. water (if there is)**
- **easy to use method**
- **no special material**

Drawbacks

- **residual water depending on laboratory conditions**
- **risk of water uptake during cooling**
- **temperature variations effects**

Repeatability is excellent for all methods

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Table 1: Standard Deviation of Repeatability SD(r) for different methods		
Method	No of samples	SD(r) robust
Karl Fischer 2001	45	0.06
Oven 95°C 2h 2001 40%RH	21	0.04
Oven 95°C 2h 2001 63% RH	21	0.04
Vacuum Oven 70°C 2001.	22	0.03
P2O5 85°C 2h 2001	55	0.03
Karl Fischer 1996 freeze-dried	32	0.04
Oven 95°C 2h 1996 freeze-dried	32	0.03
P2O5 48°C 1 week 1996 freeze-dried	24	0.04
Karl Fischer 1996 agglomerated	32	0.03
Oven 95°C 2h 1996 agglomerated	32	0.03
P2O5 48°C 1 week 1996 agglomerated	24	0.03

Statistical comparison of results of the different methods

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Table 2 : Bias and Standard Deviation of Differences SD(d) for different Methods					
Method 1	**Method 2**	**No. of samples**	**Bias**	**SD(d)**	**Statistically significant**
P_2O_5 2001	KF 2001	37	0.11	0.121	YES
P_2O_5 1996	KF 1996	24 freeze dried	0.11	0.109	YES
P_2O_5 1996	KF 1996	24 agglom.	0.11	0.091	YES
P_2O_5 2001	oven stand	21	-0.07	0.093	YES
P_2O_5 1996	oven 1996	24 freeze dried	- 0.12	0.083	YES
P_2O_5 1996	oven 1996	24 agglom.	-0.10	0.125	YES
oven stand 2001	KF 2001	17	0.13	0.244	NO
oven 1996	KF 1996	24 freeze dried	0.21	0.097	YES
oven 1996	KF 1996	24 agglom.	0.22	0.095	YES
KF 2001	oven vac	17	-0.43	0.140	YES
P_2O_5 2001	oven vac	23	-0.40	0.112	YES
oven stand 2001	oven vac	17	-0.31	0.132	YES
P_2O_5 2001	oven trop	21	-0.26	0.059	YES
oven stand 2001	oven trop	21	-0.17	0.059	YES

Correlation of Different Methods with P_2O_5 (2001 Study)

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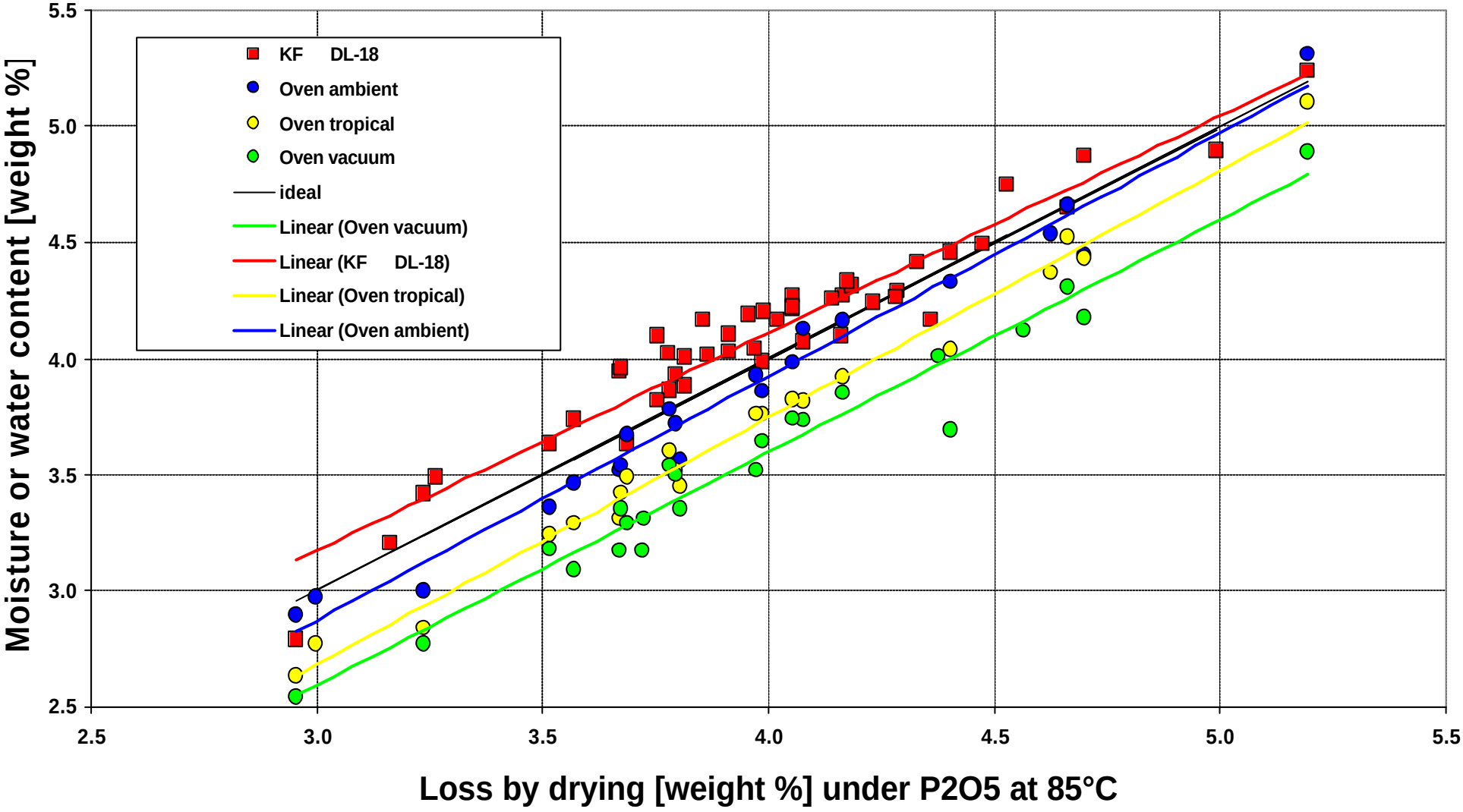

Comparison of Drying Techniques (2001 Study)

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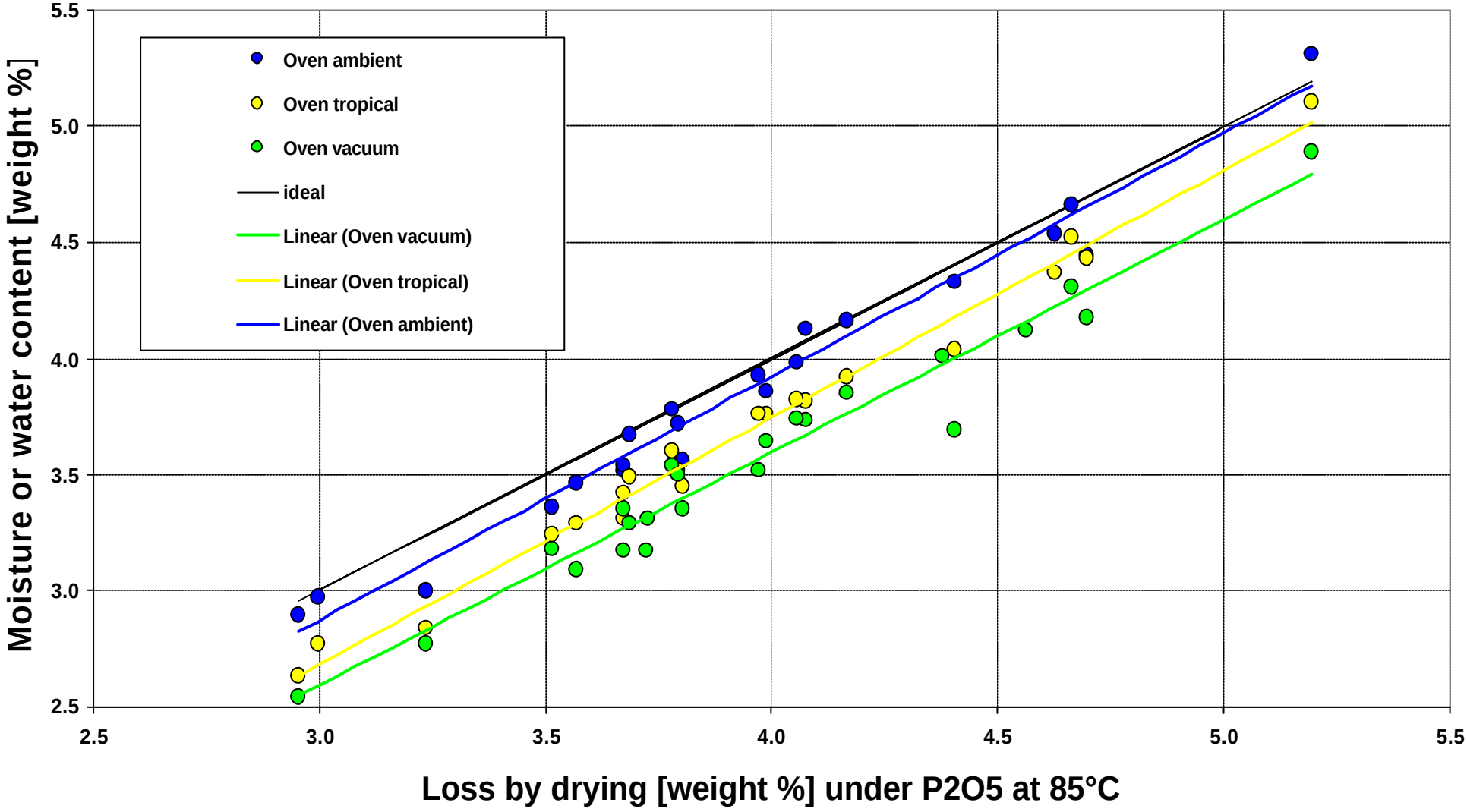

Karl Fischer and Oven Method versus P_2O_5 ,Freeze dried Coffee (1996 Study)

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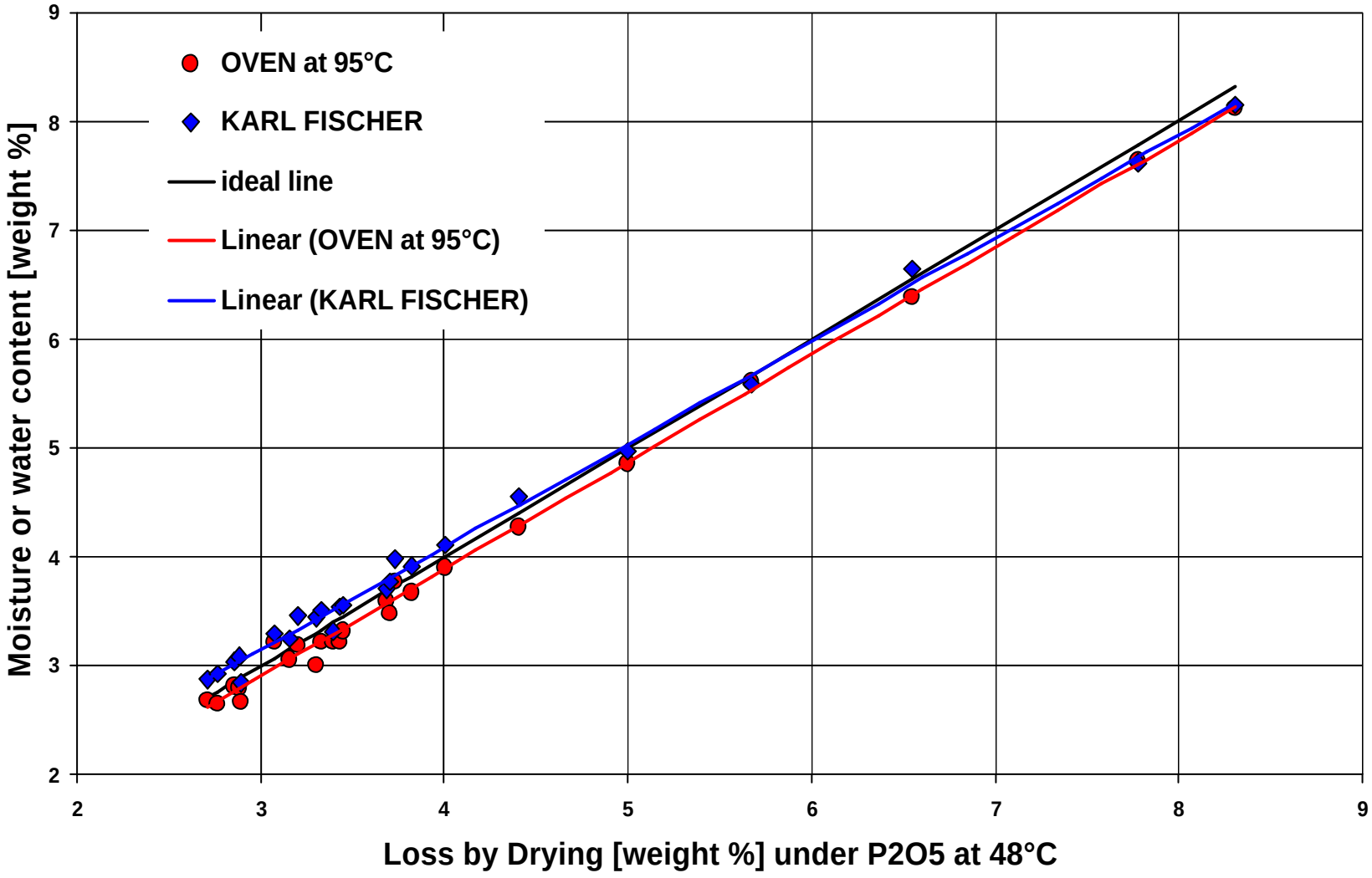

Karl Fischer and Oven Method versus P_2O_5 ,Agglomerated Coffee (1996 Study)

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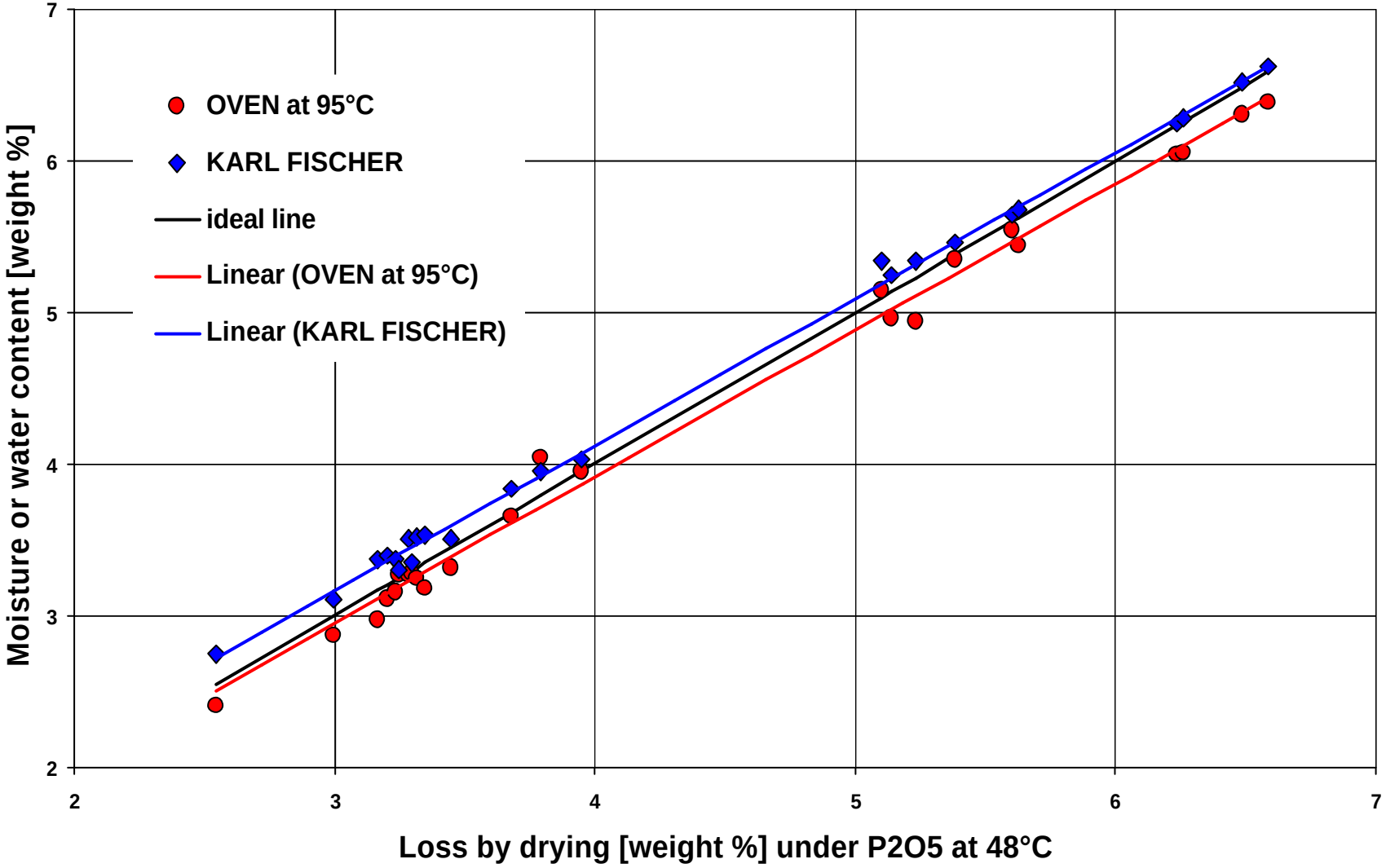
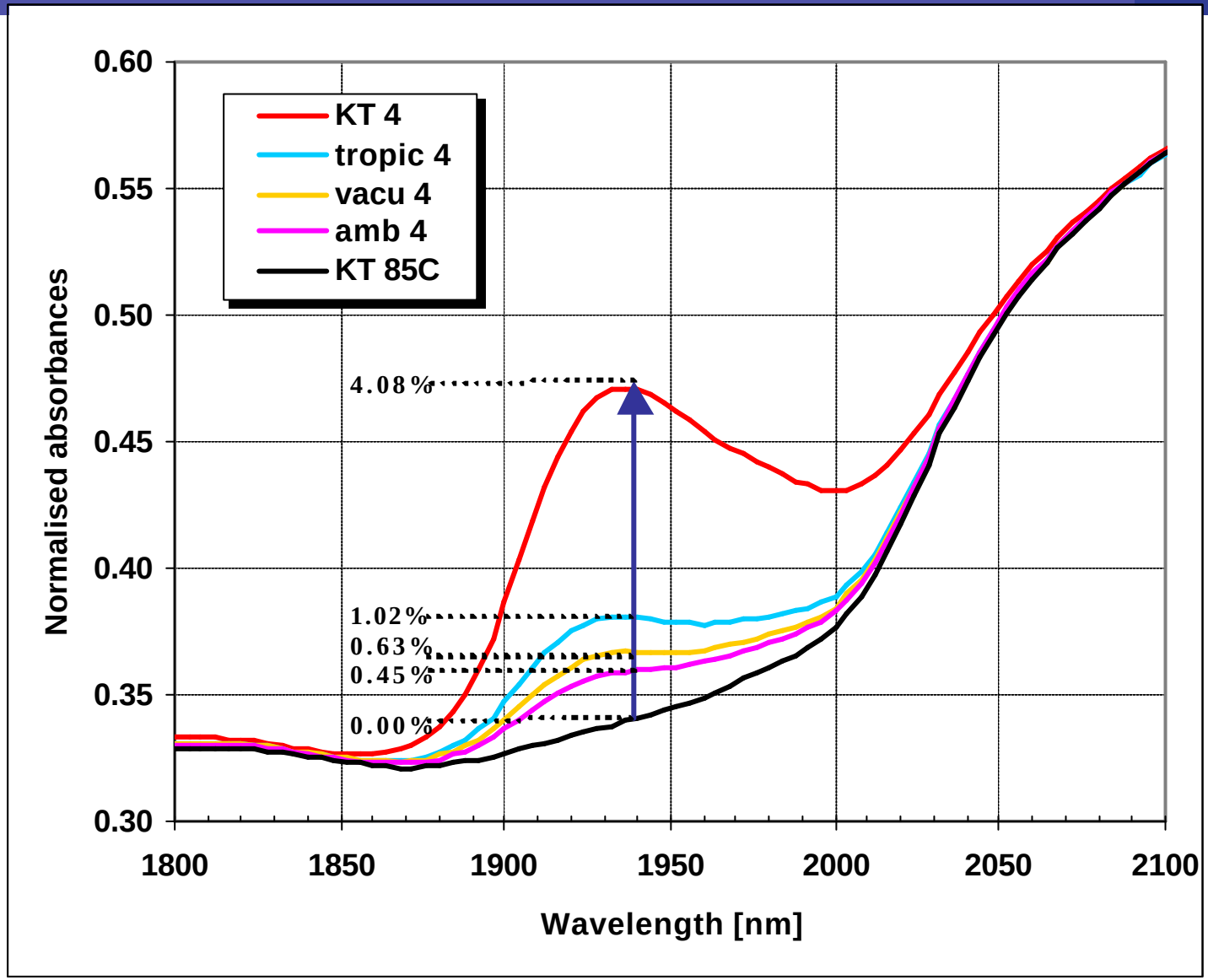


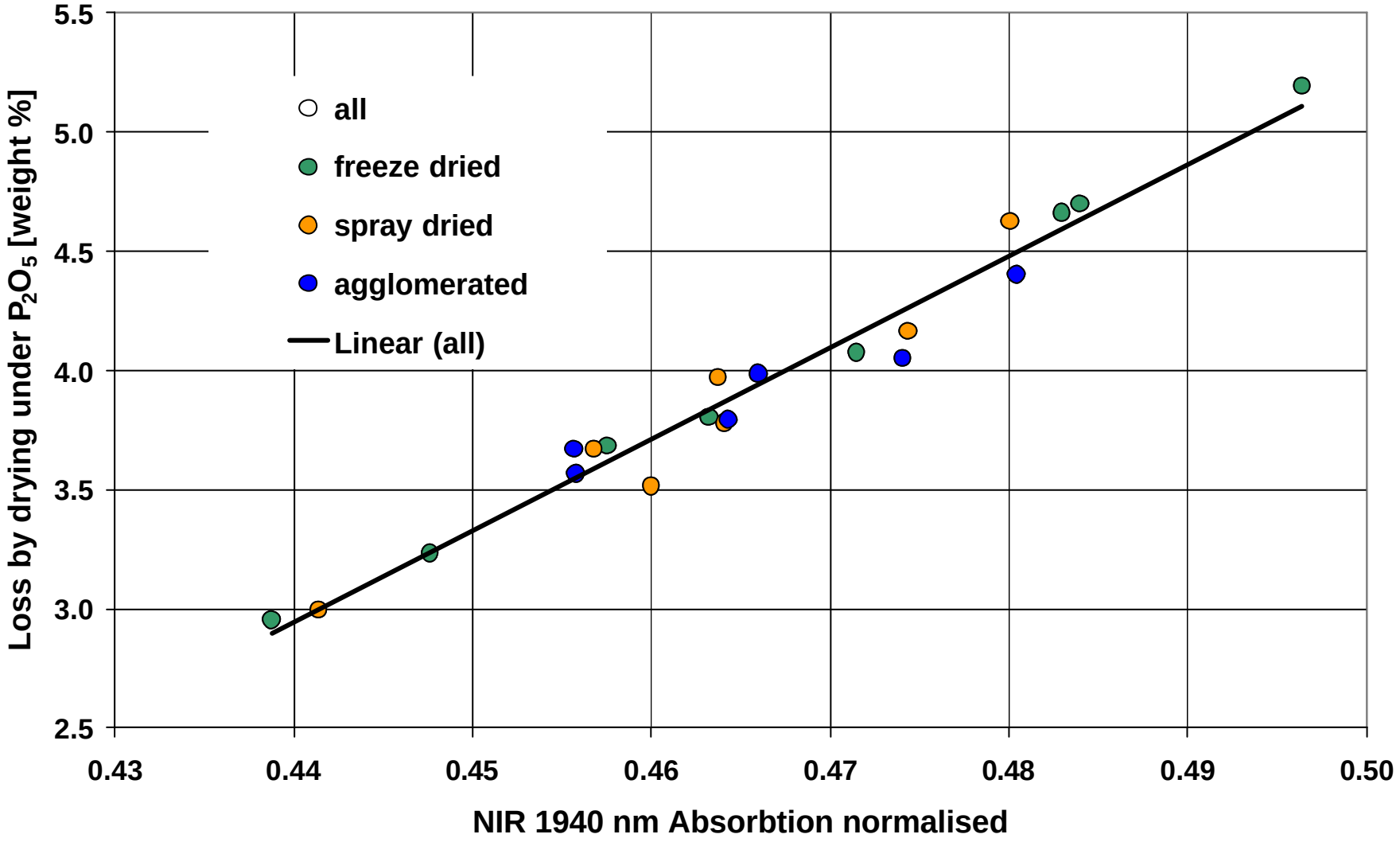
Normalized NIR Absorbance Spectra for soluble Coffee dried by various Methods



Some Interpretation

- The differences in water content observed by NIR are larger than the bias between the methods.
  - At 1940 nm principally only absorption of water molecules is observed.
  - Therefore decomposition or evaporation of other volatiles must make up the difference.
  - The presence of water at higher temperatures seem to trigger decomposition reactions.
- Nevertheless, calibrations based on the performed samples were excellent (see next slide, correlation P_2O_5 versus normalized NIR absorption)

P_2O_5 versus NIR Absorption at 1940 nm



Comparison Nestle method and ISO proposal ISO TC 34/SC 15

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The addition of formamide to the solvent and salicylique acid does not significantly change the performance on a standard soluble coffee.

	Nestle method	ISO proposal
1	2.56	2.55
2	2.55	2.55
3	2.56	2.56
4	2.60	2.52
5	2.60	2.60
6	2.56	2.52
Average	2.57	2.55
STdev	0.023	0.032

Conclusions

- Excellent repeatability for all methods.
- No significant difference between different soluble coffee types.
- ISO method (vacuum oven) gives lowest result.
- P_2O_5 measures total water content (see NIR).
- Karl Fischer gives similar results to P_2O_5.
- The oven under standard conditions gives almost the same results (degradation compensates for residual water)
- The oven under tropical conditions shows importance of laboratory conditions.

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