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Collaborative Tests to Improve Moisture Determination in Food

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Why do we organize internal Collaborative Tests ?

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The Nestlé Proficiency Test is obligatory for laboratories performing tests in the area of

- **Food safety**
- **Nutritional labelling**
- **Regulatory limits**
- **Product release**

For Nestlé Product Compliance and Product Quality is of key importance. Analytical Capabilities are one element to be able to produce superior products.

Scope of the Chemical Test

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- To provide help and advice to the laboratories in order to help them to build up trust and obtain global comparability of their analytical results.
- To provide an external control tool to the participants in order to help them manage and improve the quality of their analytical work.
- To provide a tool for the assessment of the group's laboratories performance in the analytical area.
- To generate Nestlé Reference Materials (NRM) for chemical and contaminants tests or "follow-up samples" straight after the tests for the microbiological tests.

The Nestlé Chemical Proficiency Test

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- 14 products based on standard food products
- 149 parameter
- 422 laboratories including factory laboratories, central laboratories, R&D laboratories and co-manufacturers.
- Only 5% of 5 the laboratories participate with more than 30 parameters. These laboratories are central or R&D laboratories.
- Additional Nestlé proficiency tests are run for contaminants, pesticides and microbiology.
- We also participate to other with outside organisations like FAPAS on determinations like allergens, GMO, fatty acids, antibiotics, etc.

Products used in the Chemical Test

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Symbol	Product name
BFF	Cereals product
BSI	Bouillon
DDP	Dietetic milk powder
FRJ	Fruit juice concentrate
KTA	Soluble coffee
LCS	Sweetened condensed milk
LNS	Unsweetened condensed milk
MCL	Chocolate
MIL	Milo
PET	Dry petfood
PTV	Vegetable paste
TMS	Tomato product
V-MIX	Vitamins premix

The products are especially produced for this test either in a factory or in a product development centre.

We then use rapid methods to check the homogeneity of the products.

Products are either standard products or within the range of those.

The test should be as near as possible to standard laboratory procedures.

The Chemical Test includes the following Analysis

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- Macronutrients analyses
  - **Moisture**
  - **Dry matter**
  - Fat
  - Carbohydrates (Glucose, sucrose, lactose)
  - Protein
- Micronutrients and Additives analyses
  - Vitamins,
  - Minerals
  - Other Nutrients
- Physical Tests
  - Particle size distribution
  - Viscosity
  - pH
  - Acidity
  - **Water activity**

Rules for Sample Handling

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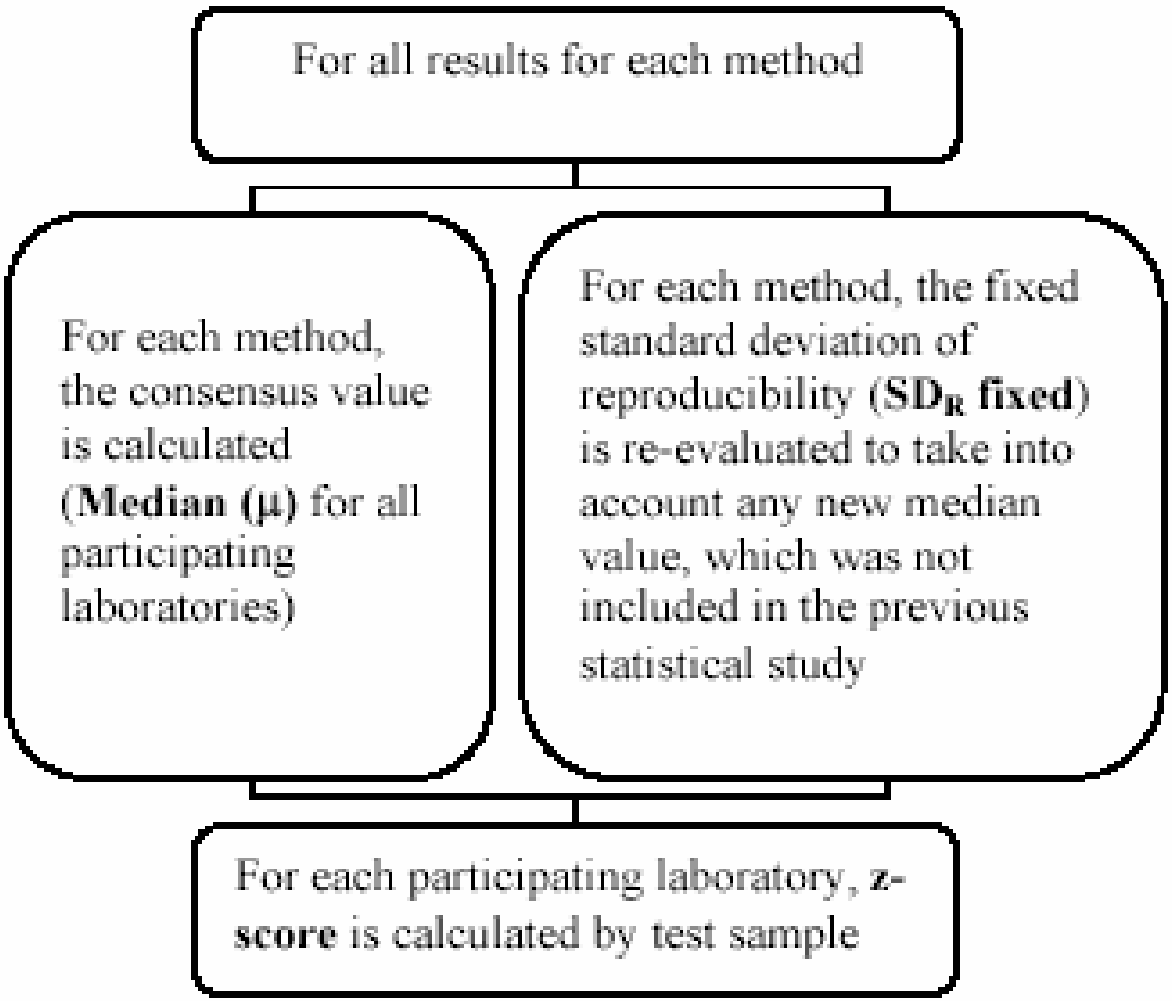
- Treat the samples as routine samples and fit them in the daily series.
- Determinations must be carried out by the analyst doing them routinely.
- The two samples must be analysed on two different test portions. Each determination must be analysed in duplicate on the same day.
- Before taking the test portions, ensure adequate preparation of the sample by following the LI "preparation of the sample" for the product involved.

The Results are treated statistically



For each analytical result a z-Score is calculated

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The z-score value is the ratio between the deviation of each individual result from the consensus value (μ), and the fixed standard deviation of the reproducibility, **SD(R) fixed**:

$$z - score = \frac{(x - \mu)}{SD(R)\ fixed}$$

where:

x	Result of participating laboratory
μ	Consensus value (Median)
SD(R) fixed	The fixed SDR value

Each analysis can be now judged compared to the established consensus value.
Based on all analytical results the laboratory receives a judgement.

The Focus of this Presentation is the Moisture Content

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- Water in food has significant influence on the product quality related to physical, chemical or microbiological stability.
- Consistent laboratory performance on the determination of moisture content in food increases accuracy and precision of the production monitoring and in consequence product consistency.
- Therefore the moisture and dry matter content is included for all test samples.
- Additionally a sample for the three principle ranges of water activity are included.

The following Methodologies are covered

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- We cover the Karl Fischer method, the standard oven under various conditions, the vacuum oven and the determination of the water activity.
  - Most methods are adaptations of international standards.
  - We translate these into Nestlé laboratory instructions.
  - These instructions have often improvements compared to the standards and are validated via internal collaborative studies.
  - All laboratories have to monitor the performance of the methods and the collaborative tests are part of this effort.
  - A large number of laboratories is accreditated including the organisation of the collaborative tests.
  - The results related to this tests are part of the determination of the measurement uncertainty.

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

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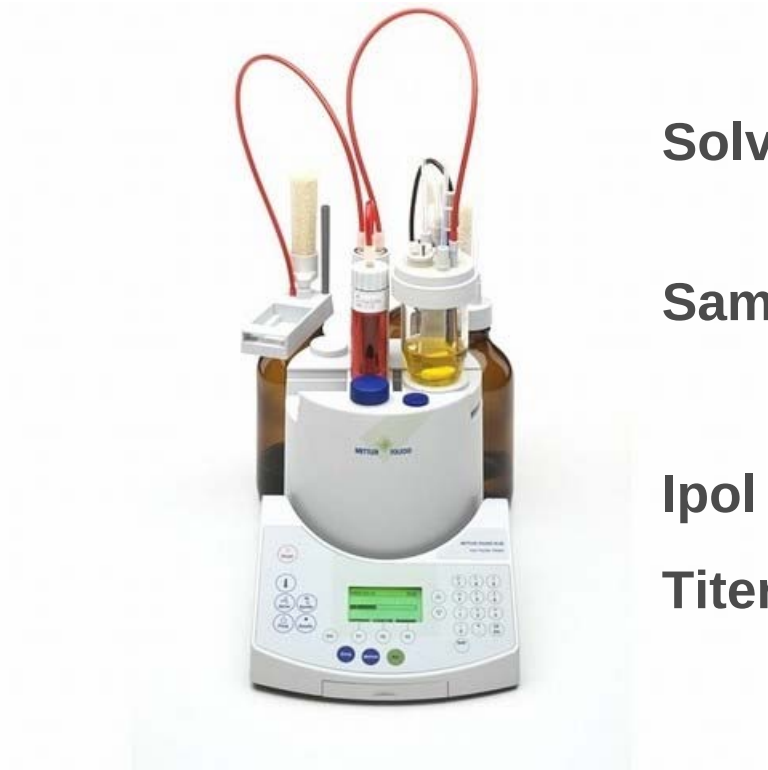
1 $CH_3OH + SO_2 + RN \Leftrightarrow [RNH]SO_3CH_3$

2 $H_2O + I_2 + [RNH]SO_3CH_3 + 2RN \Leftrightarrow [RNH]SO_4CH_3 + 2[RNH]I$



Solvent : Methanol changed every 3-4 analysis

Sample Preparation: None

500 mg of sample DDP

Ipol : 5 uA

Titer determination: 2 drops of water

The oven method can be very sensitive to laboratory conditions

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2 g sample DDP, 102°C, 2h

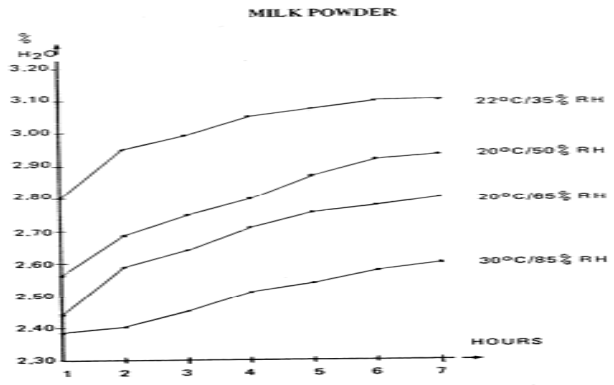
Nestle Laboratories are controlled for temperature and relative humidity.

Advantage

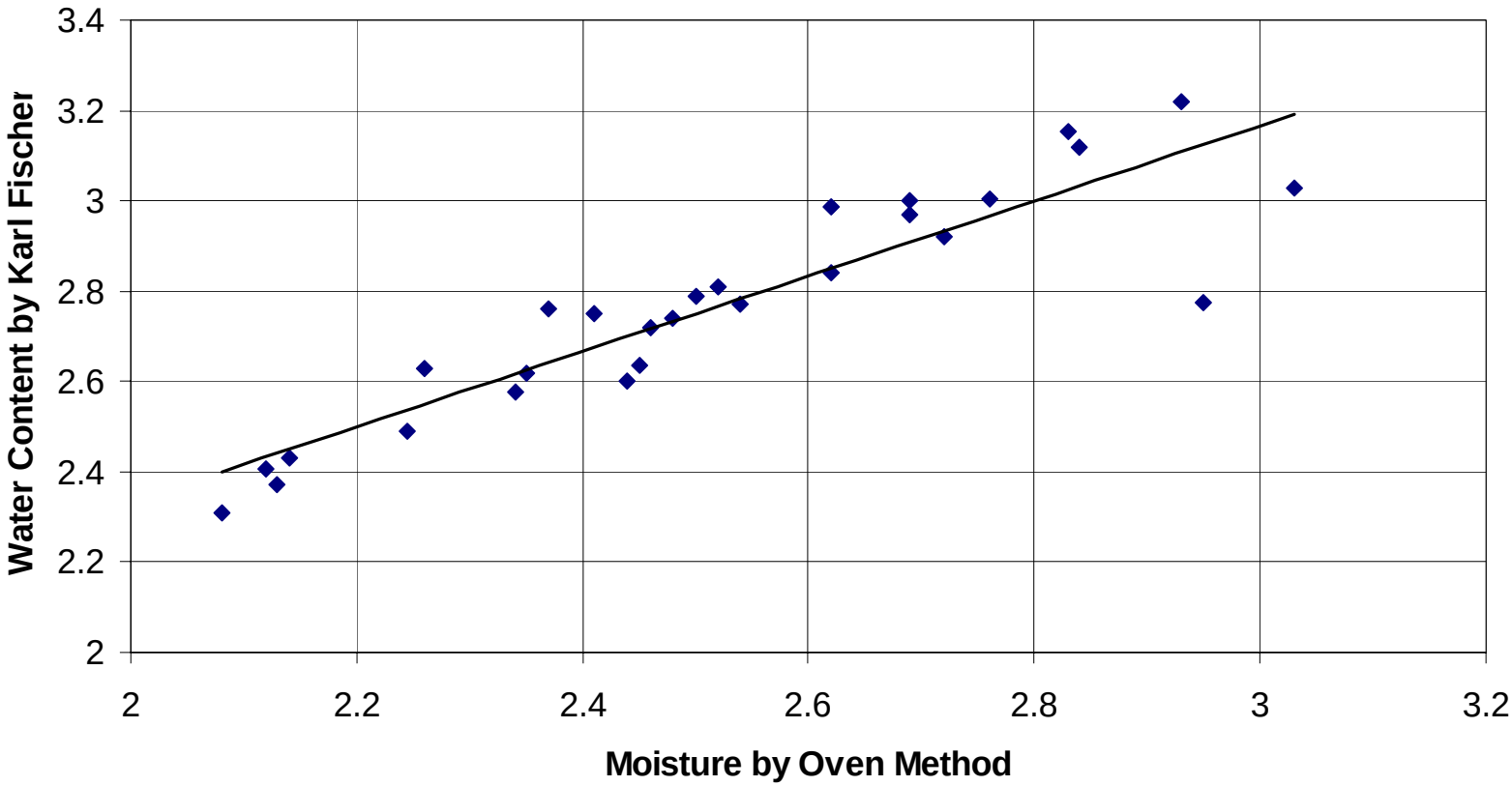
- no extraction of cryst. water (if there is)
- easy to use method
- no special material

Drawbacks

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

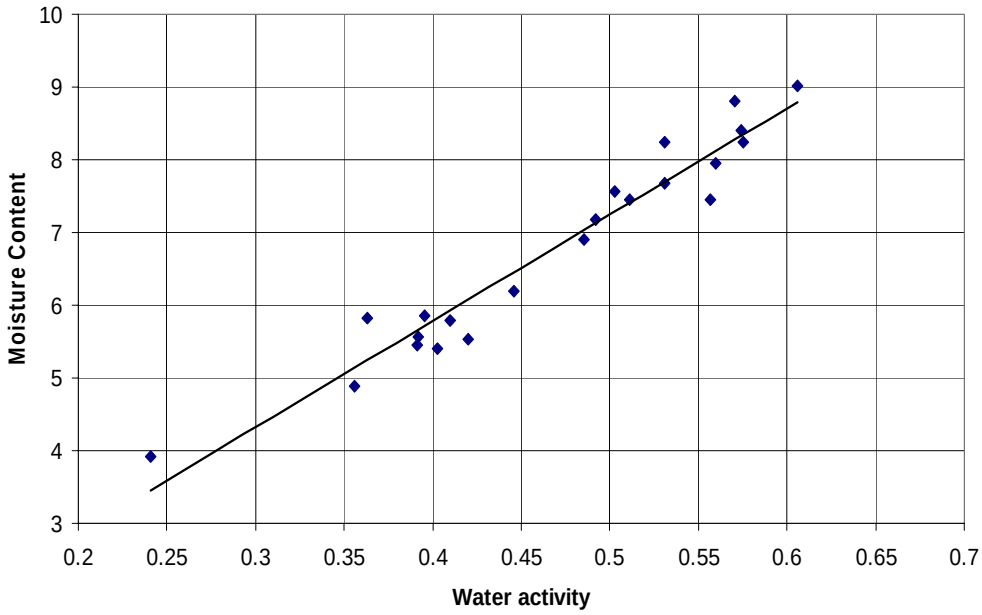


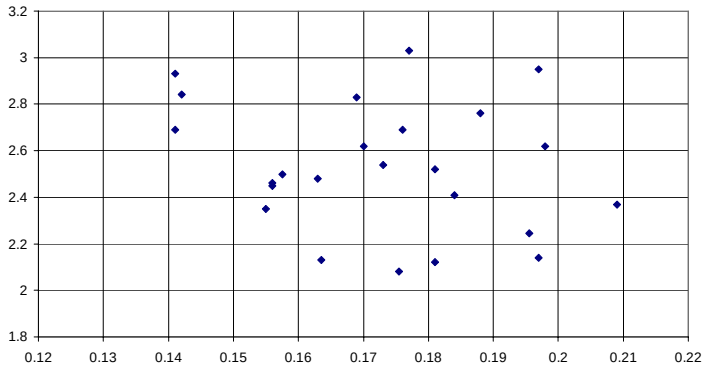
Both Methods correlate for DDP



The Karl Fischer method results in higher results (bias approx. 0.25)
2 outliers are observed (difference can be explained)

Relationship Moisture Content and Water Activity





PET

DDP

For a well defined product we would expect a correlation between the moisture content and the water activity.

For the PET product a quite good correlation is identified, whereas for the DDP we confirm the large variability of the composition and the physico-chemical status of the product.

Moisture desorption isotherm obtained for Skim milk powder

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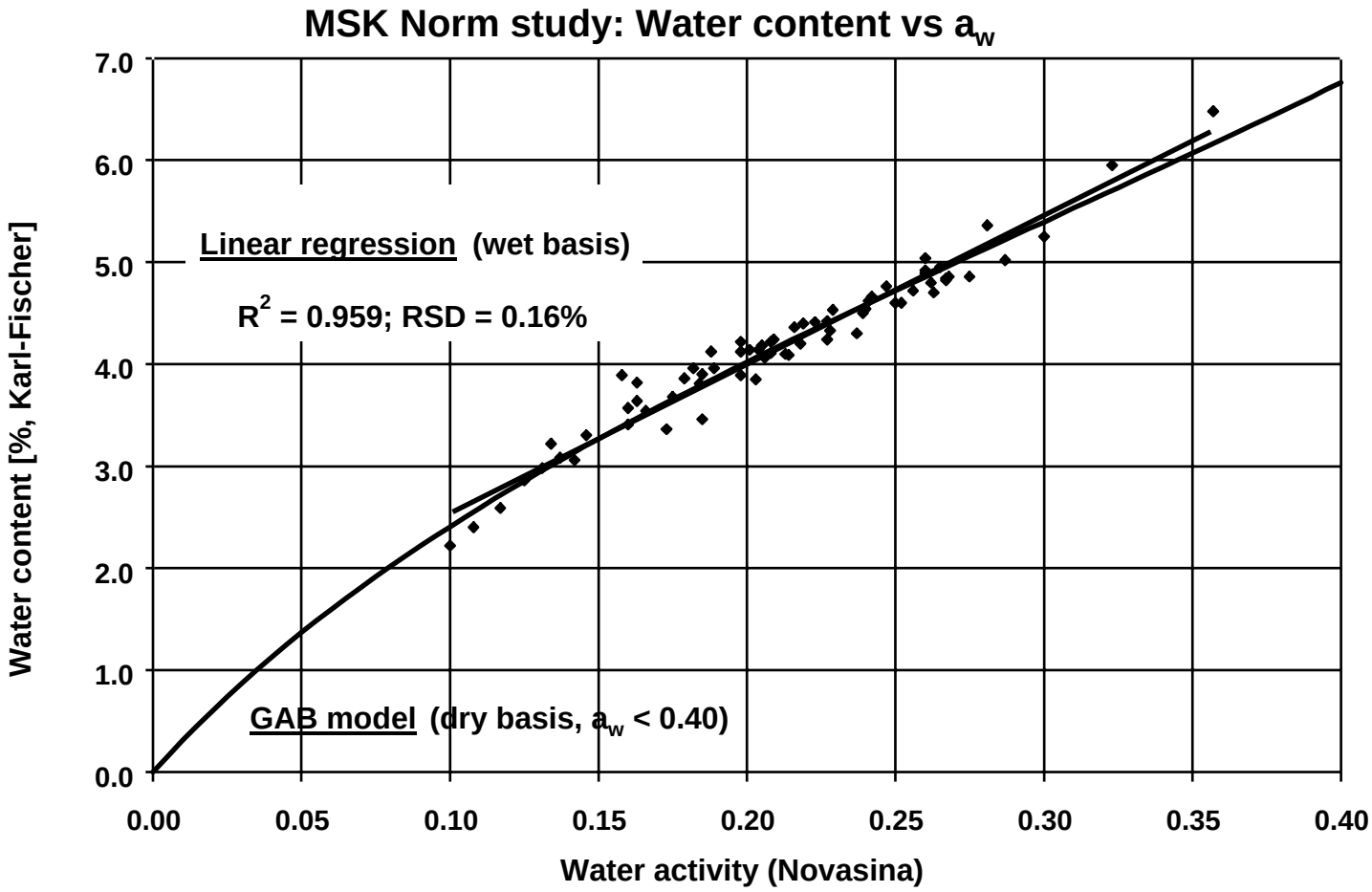
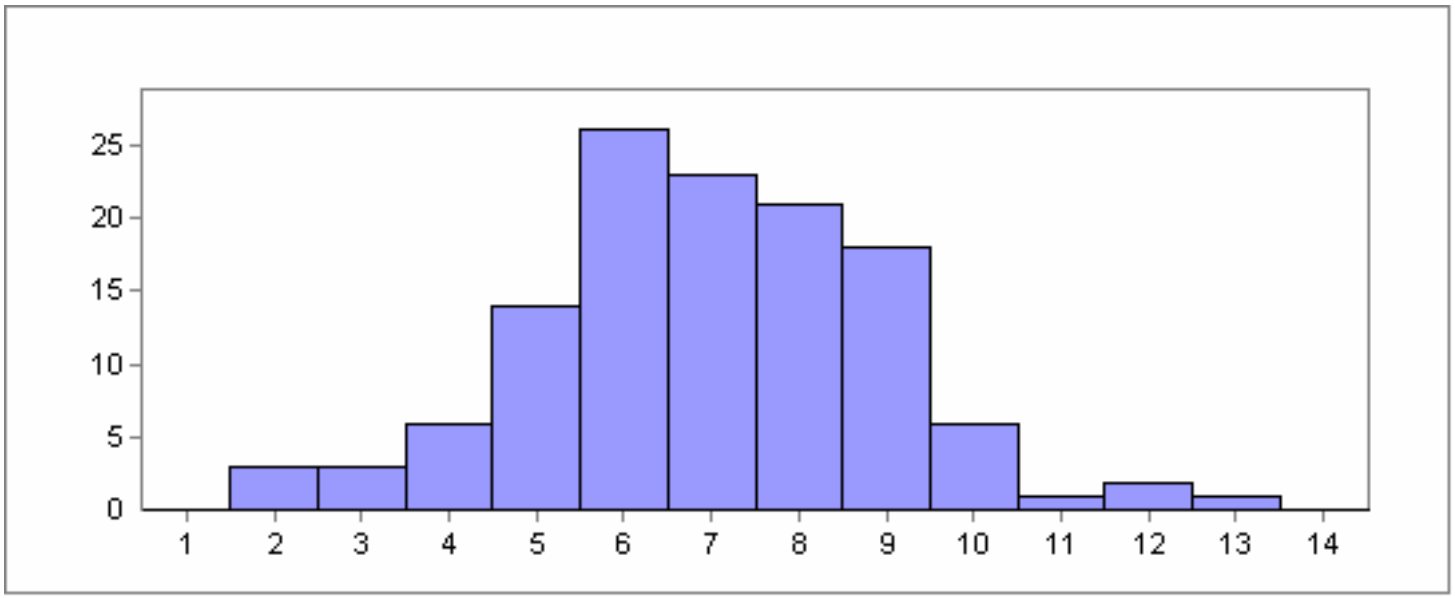
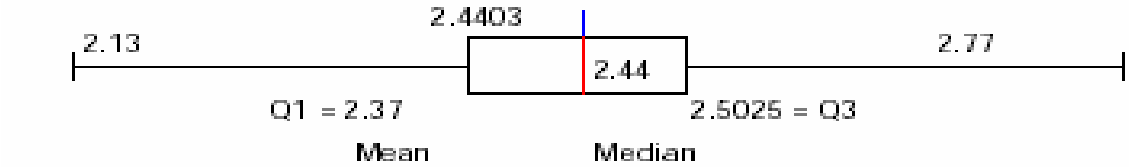


Typical DDP sample





We are checking the normal distribution in order to be sure about lot homogeneity and correct application of the method.
The large majority of tests gives this picture.

Special Website gives feedback to the Laboratories

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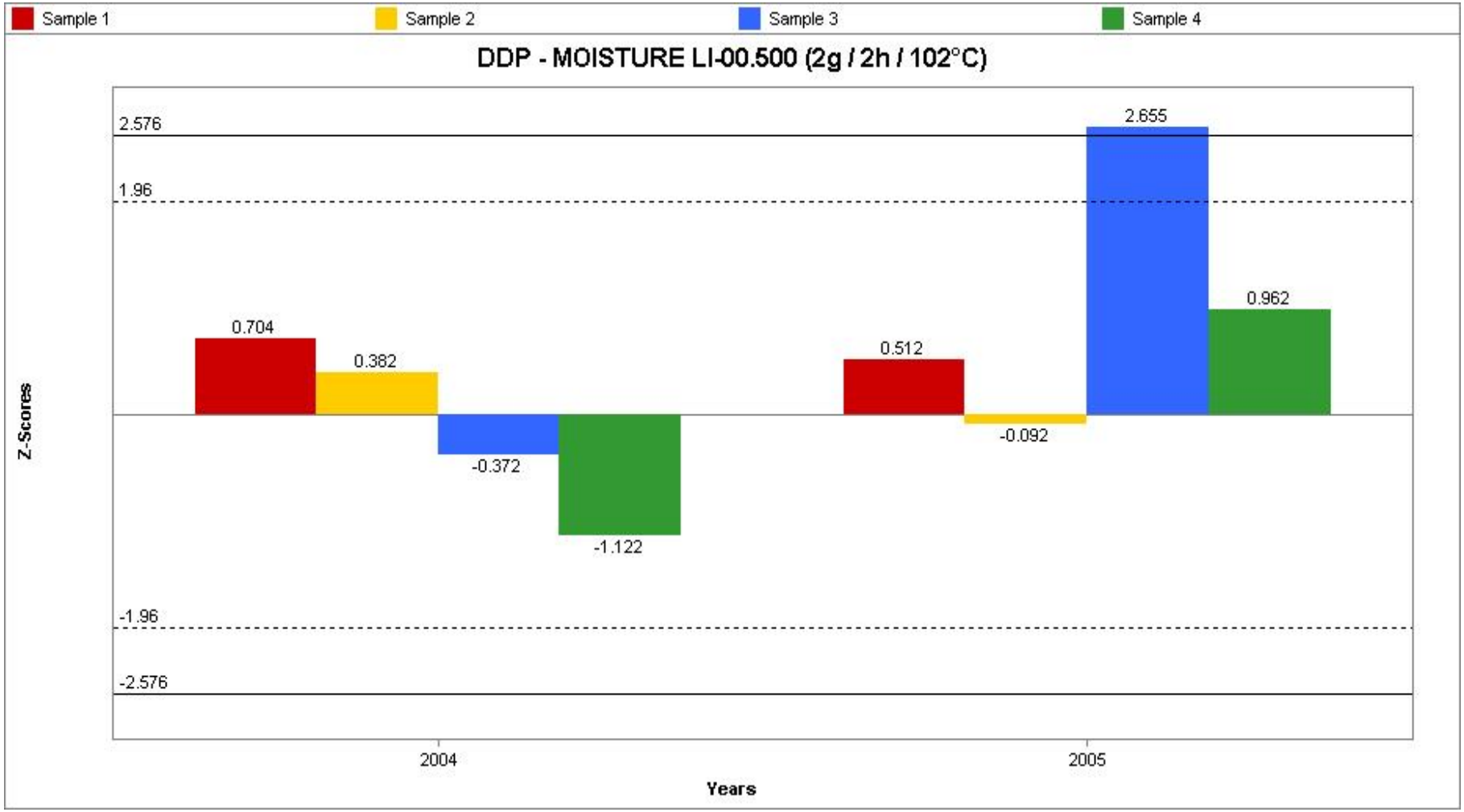

Graphs allow to identify systematic errors

Some typical results taken 2005

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Product	Determination	No. Lab.	Ref Value	SD(R)
BSI-1	Oven (2 - 3g / 4h / 102°C)	56	5.43	0.10
BSI-2	Oven (2 - 3g / 4h / 102°C)	56	2.13	0.07
BSI-3	Oven (2 - 3g / 4h / 102°C)	56	4.28	0.13
BSI-4	Oven (2 - 3g / 4h / 102°C)	56	2.66	0.07
BSI-1	Oven (2 - 3g / vac 4h / 70°C)	13	5.11	0.23
BSI-2	Oven (2 - 3g / vac 4h / 70°C)	13	1.92	0.13
BSI-3	Oven (2 - 3g / vac 4h / 70°C)	13	3.91	0.18
BSI-4	Oven (2 - 3g / vac 4h / 70°C)	13	2.34	0.06
DDP-1	Oven (2g / 2h / 102°C)	124	2.44	0.12
DDP-2	Oven (2g / 2h / 102°C)	124	2.72	0.12
DDP-3	Oven (2g / 2h / 102°C)	121	2.26	0.10
DDP-4	Oven (2g / 2h / 102°C)	121	2.34	0.09
DDP-1	Karl Fischer	35	2.60	0.11
DDP-2	Karl Fischer	35	2.92	0.10
DDP-3	Karl Fischer	34	2.63	0.08
DDP-4	Karl Fischer	34	2.58	0.09
MIL-1	Oven (2g / 3h / 102°C)	48	2.78	0.12
MIL-2	Oven (2g / 3h / 102°C)	48	2.30	0.16
MIL-3	Oven (2g / 3h / 102°C)	47	2.53	0.10
MIL-4	Oven (2g / 3h / 102°C)	47	2.76	0.10
KTA-1	Oven (2g /2h /95°C)	59	3.31	0.10
KTA-2	Oven (2g /2h /95°C)	59	3.18	0.11
KTA-3	Oven (2g /2h /95°C)	59	0.93	0.10
KTA-4	Oven (2g /2h /95°C)	59	3.25	0.13

Vacuum oven shows larger variability (pressure control)

Sugar content influences the performance of the oven method.
In some samples the physical state influence the results.

Karl Fischer performs slightly better than oven as it has been shown in a number of publications.

Water activity measurements 2005

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Product	No. Lab.	Ref Value	SD(R)
DDP-1	22	0.164	0.017
DDP-2	22	0.198	0.018
DDP-3	21	0.141	0.011
DDP-4	21	0.156	0.014
LCS-1	18	0.854	0.005
LCS-3	18	0.846	0.002
LCS-4	18	0.846	0.004
PET-1	17	0.241	0.010
PET-2	17	0.363	0.014
PET-3	17	0.560	0.011
PET-4	17	0.574	0.010

With higher value the laboratory performance improves.

This should be due to better sample homogeneity and easier sample handling. Especially very dry products tend to pick up water very rapidly.

Microbial safety is more relevant in higher range. Lower range relevant for physico-chemical stability.

7 year average results

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Sample	No	Avg Value	avg SD(R)
DDP oven	28	2.52	0.10
DDP KF	28	2.77	0.10
BFF oven	28	2.05	0.08
BSI oven	28	3.40	0.13
BSI vac oven	28	2.82	0.20
DWP oven	12	1.45	0.12
KTA oven	27	3.19	0.11
MCL oven	28	0.90	0.09
MCL KF	28	0.88	0.08
MIL oven	12	1.83	0.11
PBA oven	20	1.05	0.06
PET oven	26	6.77	0.14
PET vac oven	4	7.08	0.22
PTV DM	27	39.54	0.23
TMS DM	28	17.33	0.15
LCS DM	27	73.48	0.15
LNS DM	26	24.27	0.08
DDP aw	24	0.173	0.017
PET aw	24	0.468	0.014
LCS aw	27	0.845	0.006

7 year results confirm the earlier findings.

Overall results are better than typical results from other collaborative studies. This might be partially due to some specifics of our test (2 samples + 2 reference samples per half year).

Karl Fischer tends to perform slightly better than the oven method.

Matrix influences can be clearly seen.

Conclusions 1

- Global collaborative tests increase the performance of the laboratories due to
  - Monitoring of the laboratory performance
  - Availability of real life samples for method monitoring
  - Continuous improvement of methods
  - Corrective actions in case of deviation
  - Training of the staff
- The results illustrate the high level of reliability Nestlé achieves in it's laboratories.
- The large number of laboratories performing on good and excellent level allow the other laboratories to improve.

Conclusions 2

- Collaborative tests are only one element in the effort to achieve analytical excellence. Other elements are:
  - Good Laboratory Practise (installation, instrumentation)
  - Validated Methods and Standards
  - People Management (selection, training)
  - Central support (instructions, assistance, audits)
  - Management Commitment
- The global harmonisation of especially methods, standards and regulation has a very positive effect on product consistency and finally on consumer satisfaction.
- Consumer satisfaction is the basis of a successful business.