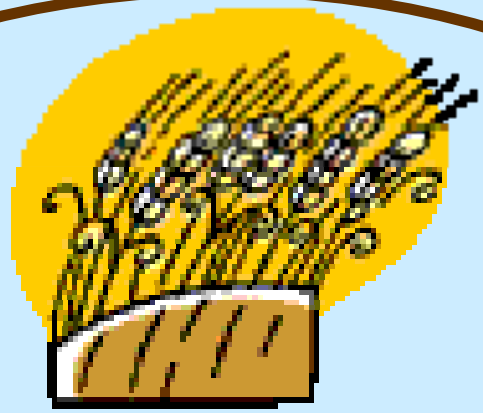

Repeatability reproducibility with a standard method ISO 5725-2

Tuesday 30th August 2005

7th ISTA seminar on statistics

Hohenheim Germany

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ISO standards

- ISO/IEC 17025:1999 General requirements for the competence of **testing** and calibration **laboratories**
- ISO 5725-1:1994 Accuracy (trueness and precision) of measurement methods and results -- Part 1: **General principles and definitions**
- ISO 5725-2:1994 Accuracy (trueness and precision) of measurement methods and results -- Part 2: **Basic method for the determination of repeatability and reproducibility of a standard measurement method**
- ISO 5725-3:1994 Accuracy (trueness and precision) of measurement methods and results -- Part 3: **Intermediate measures of the precision of a standard measurement method**
- ISO 5725-4:1994 Accuracy (trueness and precision) of measurement methods and results -- Part 4: **Basic methods for the determination of the trueness of a standard measurement method**
- ISO 5725-5:1998 Accuracy (trueness and precision) of measurement methods and results -- Part 5: **Alternative methods for the determination of the precision of a standard measurement method**
- ISO 5725-6:1994 **Practical examples**

repeatability and reproducibility of a standard measurement method

ISO 5725-2 *when test result is a numerical value (obtained or computed), where participating laboratories used a given standard method, to test a set of samples, with replicates*

repeatability is a measure for the within-laboratory consistency. It is a variance (variability of measurements)

between-laboratory variance is a measure for variation between the participating laboratories.

reproducibility is the sum of repeatability and between-laboratory variance.

value = for a lab, a method, a sample;
each sample analysed at least twice in a lab for a method

ISO 5725-2 when test result = numerical value obtained or computed on a continuous scale

The repeatability is a measure for the within-laboratory consistency.

Repeatability variance: $$S^2_{rj} = \frac{\sum_{l=1}^{Pj}(n_{lj} - 1)S^2_{lj}}{\sum_{l=1}^{Pj}(n_{lj} - 1)}$$

The between-laboratory variance is a measure for variation between the participating laboratories.

Between-laboratory variance: $$S^2_{Lj} = \frac{S^2_{dj} - S^2_{rj}}{\bar{n}_j}$$

The reproducibility is the sum of repeatability and between-laboratory variance.

Reproducibility variance: $S^2_{Rj} = S^2_{rj} + S^2_{Lj}$

NB :value = for a lab, a method, a sample; each sample analysed at least twice in a lab for a method

where $$S^2_{dj} = \frac{1}{p_j - 1} \sum_{l=1}^{Pj} n_{lj}(\bar{z}_{lj} - \bar{z}_j)^2$$

and $$n_j = \frac{1}{p_j - 1}\left[\sum_{l=1}^{Pj} n_{lj} - \frac{\sum_{l=1}^{Pj} n^2_{lj}}{\sum_{l=1}^{Pj} n_{lj}}\right]$$

l = laboratory j = method

n_{lj} = Number of test results obtained by laboratory l for method j

P_j = Number of laboratories with non-missing results for method j

S_{lj} = Standard deviation for method j and laboratory l

$\bar{Z}_j$ = overall mean for method j

Z_{lj} = mean for method j and laboratory l

Type of data needed

lab	sample	rep	value
lab01	sple1	rep1	4.44
lab01	sple1	rep2	4.39
lab01	sple2	rep1	9.34
lab01	sple2	rep2	9.34
lab01	sple3	rep1	17.4
lab01	sple3	rep2	16.9
lab01	sple4	rep1	19.23
lab01	sple4	rep2	19.23
lab01	sple5	rep1	24.28
lab01	sple5	rep2	24
lab02	sple1	rep1	4.03
lab02	sple1	rep2	4.23
lab02	sple2	rep1	8.42
lab02	sple2	rep2	8.33
lab02	sple3	rep1	14.42
lab02	sple3	rep2	14.5
lab02	sple4	rep1	16.06
lab02	sple4	rep2	16.22
lab02	sple5	rep1	20.4
lab02	sple5	rep2	19.91
…	…	…	…

3 to 30 laboratories

2 to XX samples

2 to 10 replicates

quantitative values

Visible on this screen:

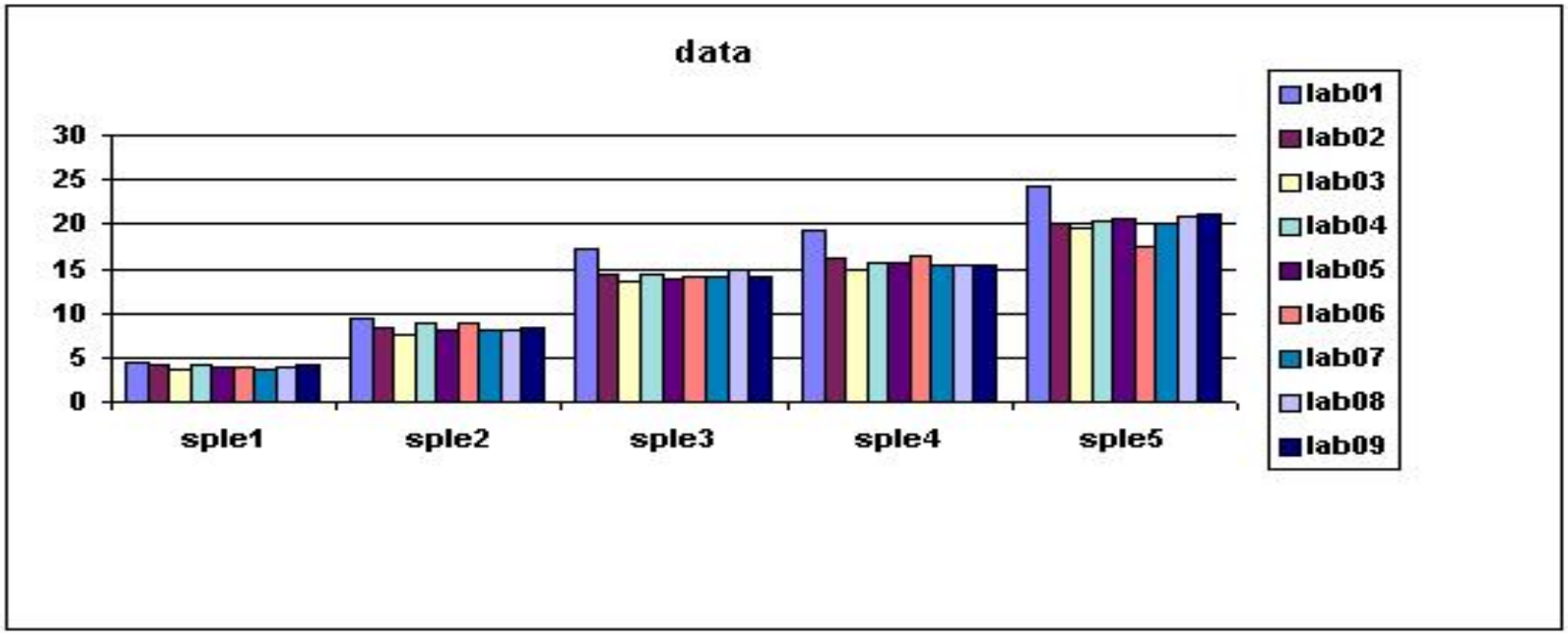
2 labs

5 samples

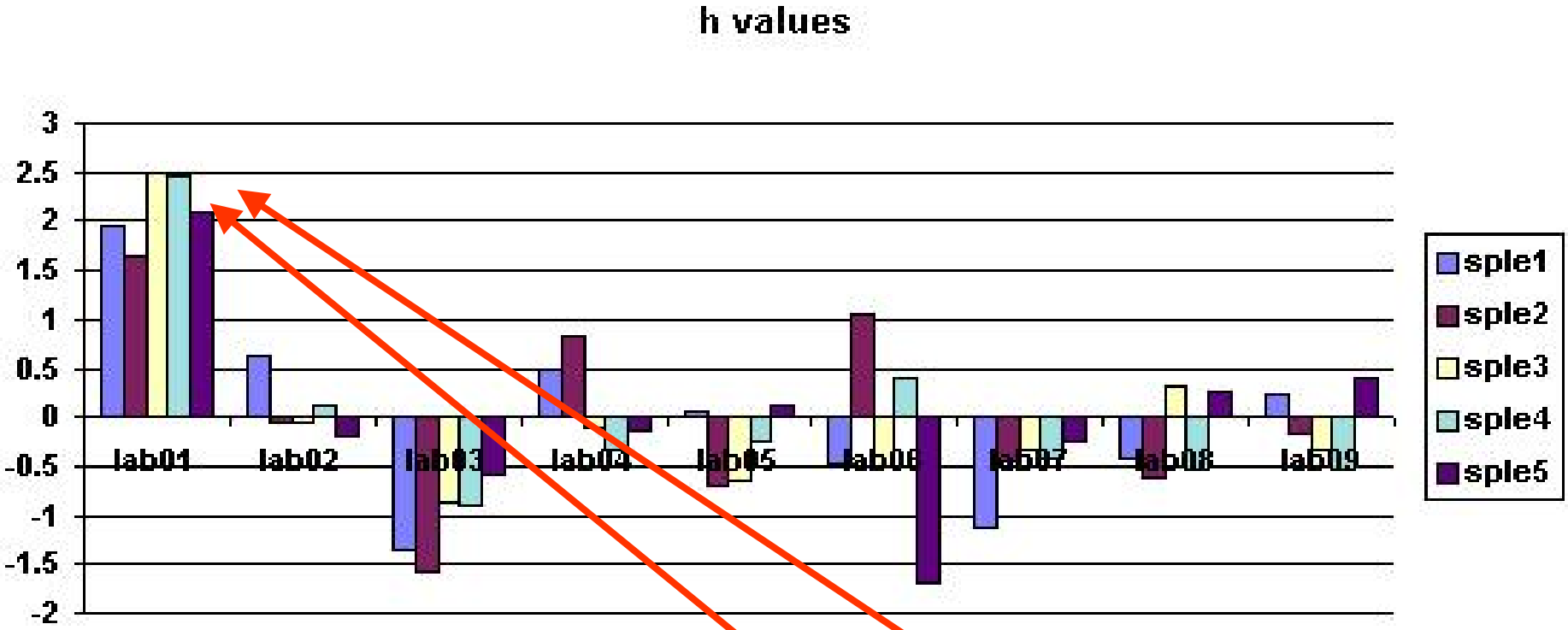
2 reps

Free Tool Includes

- raw data, to be kept and if possible to be reproduced in extenso in report
- means and variances by laboratory and by laboratory X sample
- repeatability and reproducibility values
- h and k values and the test of their significance

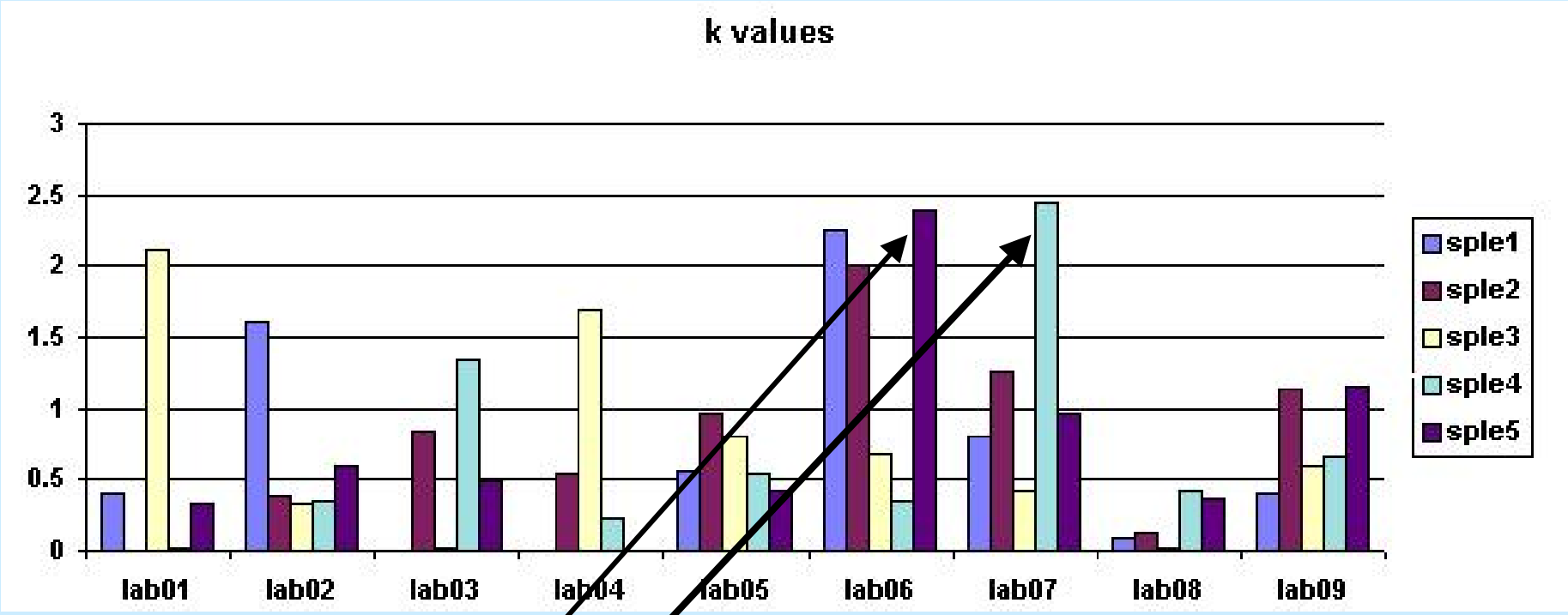


h values shows if a lab has a tendency to under or over estimate



lab	sple1	sple2	sple3	sple4	sple5
lab01	5%		5% 1%	5% 1%	5%
lab02					
lab03					
lab04					
lab05					
lab06					
lab07					
lab08					
lab09					

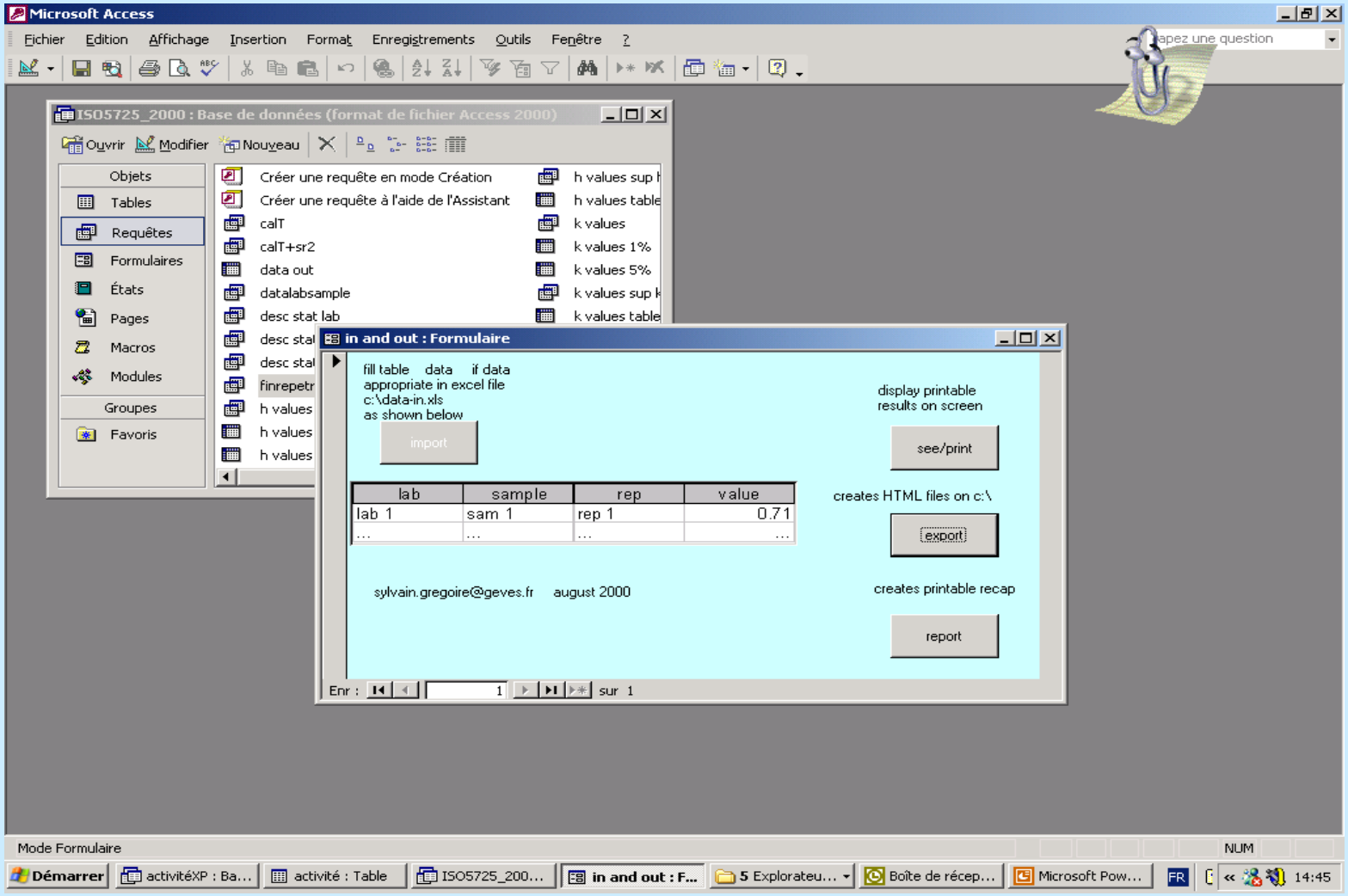
k values indicates the relative variability of the results



lab	sample	k values	k 1%	k 5%	k crit 1%	k crit 5%
lab01	sple3	2.11		5%	2.29	1.9
lab06	sple1	2.26		5%	2.29	1.9
lab06	sple2	2.01		5%	2.29	1.9
lab06	sple5	2.39	1%	5%	2.29	1.9
lab07	sple4	2.45	1%	5%	2.29	1.9

!

Guided tour


Microsoft Access
Fichier Edition Affichage Insertion Format Enregistrements Outils Fenêtre ?
ISO5725_2000 : Base de données (format de fichier Access 2000)
Ouvrir Modifier Nouveau
Objets
Tables
Requêtes
Formulaires
États
Pages
Macros
Modules
Groupes
Favoris
Créer une requête en mode Création
Créer une requête à l'aide de l'Assistant
calT
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desc stat lab
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h values
k values
k values 1%
k values 5%
in and out : Formulaire
fill table data if data appropriate in excel file c:\data-in.xls as shown below
import
display printable results on screen
see/print
lab sample rep value
lab 1 sam 1 rep 1 0.71
creates HTML files on c:\
export
sylvain.gregoire@geves.fr august 2000
creates printable recap
report
Enr : 1 sur 1
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in and out : F...
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Boîte de récep...
Microsoft Pow...
FR
14:45

Exercise 1

- Open charbonISO 5725_2000.mdb Access database
- Obtain a report and look at it
- Look at table data
  - How many labs did the test?
  - How many samples were used?
  - What is approx. the repeatability?
  - Look at graphs mean by lab, data and h values
  - Look at graph k values
  - Look at h and k critical values
- Discussion

More methods, or factors

- You may wish to use the program in cases where you have different methods, or made a factor vary in a method

=>you can simply split in different analysis per level of factor (factor=a, factor=b,…)

- You may want to design an experiment with many factors and for each factor have different modalities.

=>In that case use ANOVA or GLM or … You are too far from the ISO standard definition

*For each combination of XXX*YYY I have repeats*

Lab	Lot/Sample	Repeat	Value
Lab	Method	Repeat	Value
Method	Lot	Repeat	Value
XXX	YYY	Repeat	Value

10 labs 4 lots 3 methods -> 1 report per method (or per lot)

8 labs, 3 methods, 3 levels of presence -> 1 report per method (or level)

3 methods 4 levels of presence 10 labs -> “labs as repeats”

Etc…

Remember repeatability, reproducibility is computed by “YYYY” (lot/sample)

h and k values

- Critical h values at 1% and 5%

$$h_{ij} = (\bar{y}_{ij} - \bar{\bar{y}}_j) / \sqrt{1/(p_j - 1)\sum_{i=1}^{p_j} (\bar{y}_{ij} - \bar{\bar{y}}_j)^2}$$

High h value =over estimate / general mean

Low h value =under estimate / general mean

- Critical k values at 1% and 5%

High k value, greater variability in repeats

Low k values are not spotted on the critical values

$$k_{ij} = s_{ij}\sqrt{p_j} / \sqrt{\sum s_{ij}^2}$$

If Nb Labs* Nb samples = 100 -> 1 and 5 are expected at 1% and 5%

With 10 labs 3 samples = 30 ->0 or 1 at 1% 1 or 2 at 5% are expected

- Over-estimate compared to mean of all values; it does not tell who is right who is wrong
- No true value is given in the computations, no reference laboratory is given in the computations
- These are alerts to take into account by the experts

Exercise 2 two differences to discover

- Open charbon2ISO5725_2000.mdb Access database
- Obtain a report and look at it
- Change the heading of the report, with your name
- Look at table data
- Use of data set charbon2
  - How many labs did the test? (see names)
  - How many samples were used? (see names)
  - What is approx. the repeatability?
  - Look at graphs mean by lab, data and h values
  - Look at graph k values
  - Look at h and k critical values
- discussion

Exercise 2 two differences to discover

- Open harbon2ISO5725_XX.mdb Access database
- Obtain a report and look at it
- Change the heading of the report, with your name
- Look at table data
- Use of data set charbon2
  - How many labs did the test? (see names)
  - How many samples were used? (see names)
  - What is approx. the repeatability?
  - Look at graphs mean by lab, data and h values
  - Look at graph k values
  - Look at h and k critical values
- discussion

Lab 1 lot 1 rep 2 is exotic

Lab5 has less variability

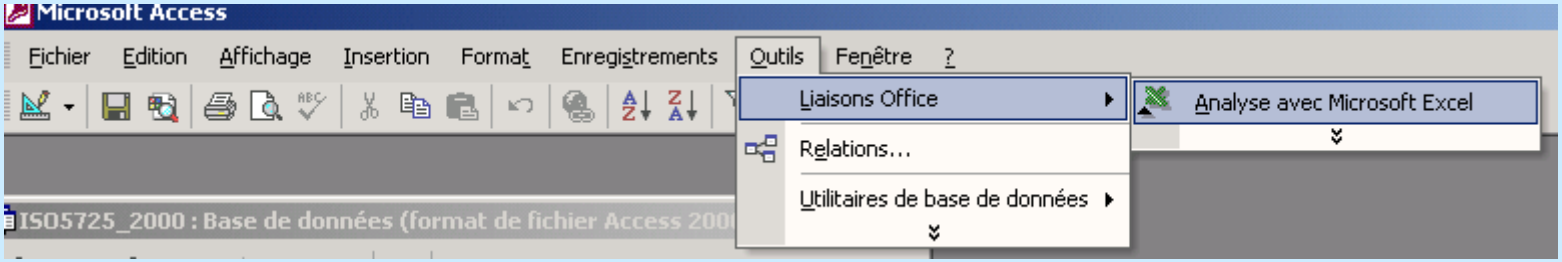
Lab names and sample names changed

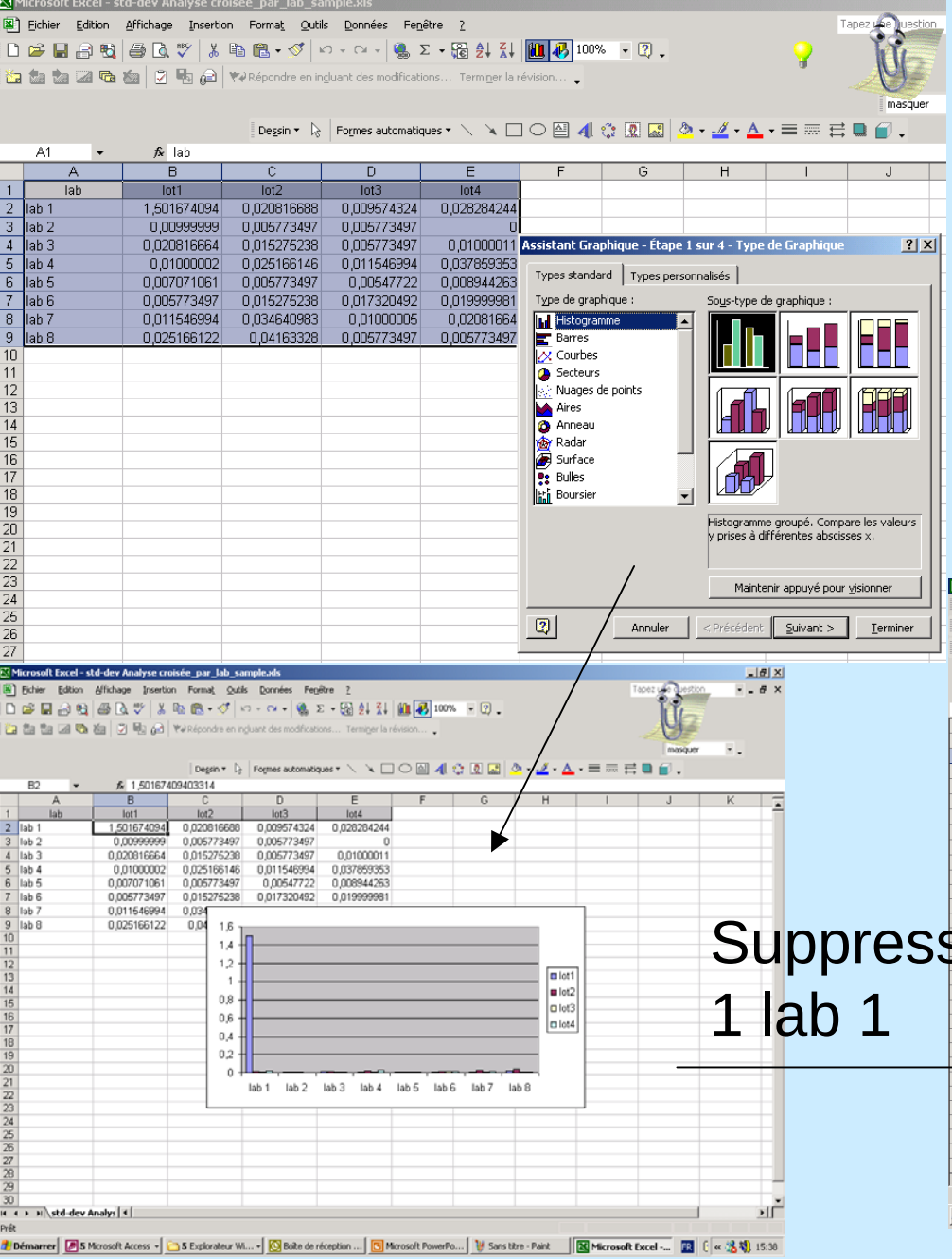
From office Access to Excel

- Button std dev

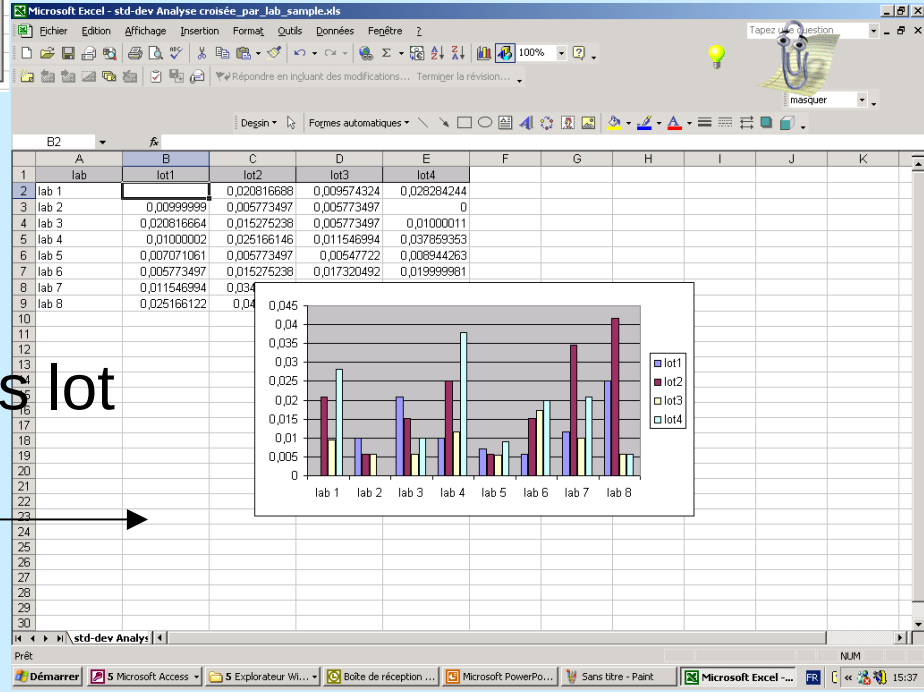
std-dev Analyse croisée_par_lab_sample : Requête Analyse croisée

lab	lot1	lot2	lot3	lot4
lab 1	1,5016740940	0,0208166879	0,0095743242	0,0282842443
lab 2	0,0099999905	0,0057734972	0,0057734972	0
lab 3	0,0208166640	0,0152752377	0,0057734972	0,0100001097
lab 4	0,0100000203	0,0251661460	0,0115469944	0,0378593529
lab 5	0,0070710611	0,0057734972	0,0054772204	0,0089442634
lab 6	0,0057734972	0,0152752377	0,0173204916	0,0199999809
lab 7	0,0115469944	0,0346409831	0,0100000501	0,0208166401
lab 8	0,0251661224	0,0416332803	0,0057734972	0,0057734972





Suppress lot 1 lab 1



Troubleshooting

- Out of capacity <= no variability for 1 (any) sample
- Reproducibility<repeatability <= report with =
- Non normality
  - use and notify in your report
  - transform data, advantage tests are ok if good transformation, disadvantage you loose technical unit

Complete implementation of ISO 5725-2 need more than free tool

- Data check
- Grubbs to detect outliers
- Linearity of repeatability along measures (do it with Excel)
- etc…

SAS, Statgraphics, and other packages can help, specific ISO software are often expensive