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Sampling of cereals and cereal
products

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Sampling of cereals and cereal products

- Correct sampling is an operation that requires the most careful attention
- Necessity of a **representative sample** from a lot of cereals, pulses or cereal products...
- **Personnel** employed for sampling should have been suitably **trained**
- **Cereal products as flour : more homogeneous material → plans less restricting needed**

Contexte réglementaire

3 directives for samplinf of cereal products (official controls):

- ❖ Directive 2002/27/CE for **aflatoxines**
- ❖ Directive 2002/26/CE for **ochratoxine A**
- ❖ Directive 2005/38/CE for **fusarium toxins** (JO L143/18 of 7 june 2005)

1 regulation draft : all the sampling directives

JO in february ?

**Possibility to have an alternative method on big lots ;
commission is writing a guide on sampling (for
summer 2006)**

Directives Echantillonnage

Modalités d'échantillonnage pour le contrôle des aflatoxines (directive 98/53/CE) de l'ochratoxine A (directive 2002/26/CE) des fusariotoxines (directive 2005/38/CE)

Poids du lot en T	Poids ou nombre de sous lots	Nbre de prélèvements élémentaires	Aflatoxines		ochratoxineA/fusariotoxines	
			Masse prélèvement élémentaire en g	Masse ech global en Kg	Masse prélèvement élémentaire en g	Masse ech global en Kg
>=1500	500T	100	300	30	100	10
>300 et<1500	3 sous lots	100	300	30	100	10
>=50 et<=300	100T	100	300	30	100	10
<50		10-100		1 à 10		1 à 10

Chaque sous lot doit faire l'objet d'un échantillonnage séparé

Poids des sous lots peut dépasser le poids indiqué jusqu'à concurrence de 20%

Sampling Directives : the reality

Exemple of a silo of 10 000T

factors	Régulation	Draft standard	« Experimental »
Number of increments	100/500T	50/500T	10/500T
Total	2000	1000	200
Sampling time	470h - 67 days	185h - 26,5 days	30h
Cost for sampling and reduction of global sample	10300 euros	4000 euros	650 euros

Regulation on sampling for cereal products : the reality

- **example of time needed for sampling of flowing cereals : lot of 3000 t**

Note : real time from tests in silos

Flowing rate	Regulation
500 t / hour	1 increment every 36 seconds during 6 hours
400 t / hour	1 increment every 45 seconds during 7,5 hours
250 t / hour	1 increment every 72 seconds during 12 hours

Standardisation on sampling for cereal products

- **2 international standards** (standard ISO 6644:2002 on flowing cereals ; standard ISO 13690:1999 on static cereals) : only for homogeneously distributed characteristics

No standard for mycotoxins or contaminants heterogeneously distributed

⇒ no harmonized procedures at an international or european level !

Working group on sampling

– 3 working group :

- 2 Groups dealing with standardization

- First, at a french level with the national standard body : AFNOR

- » Working group to prepare a working document on sampling, particularly on contaminants as mycotoxins

- based on ISO standard 13690 and 6644 and regulation on mycotoxins (Fusarium toxins versus OTA)

- applicable in silos...by store keepers (taking into account the possibilities of equipment...) and millers ...: « workable » sampling schemes

– Second, at a european level : CEN (European Committee for standardization CEN 338)

- » sampling : item registered in the work program of CEN 338 « Cereals and Cereal Products »
- » draft have circulated for comments in 2004-11-15 to the european member bodies and ISO members ; dead line : 2005-01-15
- » establishment of the working group experts for the same date : the CEN and ISO members have nominated experts ⇒ WG 5
- » convenors : Mrs VERON-DELOR (IRTAC/ANMAC)

European working group on standardisation

- Creation of the international working group WG5 of CEN TC338 on January 2005, with French convenor
 - 22 experts designated by national body organizations of standardisation :
 - from CEN : France, Germany, United Kingdom, Austria, Spain, Portugal and Netherlands
 - from ISO : Iran, Canada, Thailand, China
 - draft of standard circulated between experts and comments received in 2005
- **a standard in 2007**

Flowing cereals – mechanical or manual means

In accordance with a possible regulation . Draft standard ; « Practical »

	Increment	Minimum number of increments	Sample division	Laboratory sample
Mechanical means	200g	100 increments /500T	Conical divider	10Kg
		100 increments /500T 1 prélèvement par 5T	Mechanical rotary divider	10Kg
		(Predetermined intervals of time ; depends of the flowing) 100 increments /500T		10Kg
Manual means	400g	100 increments /500T 25 increments /500T 10 increments /500T	Conical divider Mechanical rotary divider	10Kg 8 à 9 kg

*: sampling should be adjusted so that the sizes of samples given in the table are obtained

Sampling of static batches (mechanical means recommended)				
	Increment	Minimum number of increments	Sample division	Laboratory sample
Lorry	2000g	<p>100 increments /500T</p> <p>Lot constituted by several lorries : 3 increments by lorry</p> <p>Lot constituted by 1 lorry : 5 increments</p> <p>3 increments</p>	<p>Conical divider</p> <p>Mechanical rotary divider</p>	<p>10 kg</p> <p>6 kg</p>
wagons	2000g	<p>100 increments /500T</p> <p>6 increments by wagon</p> <p>1 increment by wagon ; lot : train of 10 to 12 wagons</p>	<p>Conical divider</p> <p>Mechanical rotary divider</p>	10Kg

*: sampling should be adjusted so that the sizes of samples given in the table are obtained

Sampling of static batches (mechanical means recommended)

	Increment	Minimum number of increments	Sample division	Laboratory sample
Barges	2000g	100 increments /500T 50 increments /500T 10 increments /500T	Conical divider Mechanical rotary divider	10Kg
Ships	2000 g	100 increments /500T 25 increments by lot (1 ship) 10 increments by lot	Mechanical rotary divider	
Silos	2000g	100 increments /500T 50 increments /500T (each 2 meters on 1 height of taking samples is considered as 1 increment ; Repeat) 10 increments / 500 t	Conical divider Mechanical rotary divider	10Kg

Consequences for grain stakeholders and actions decided

- for grain stakeholders, problems arising from :
 - differences between official controls and day to day controls made by cereal storekeepers
 - absence of harmonised procedures of sampling (trade practices)
 - absence of validated alternative protocols, technically and economically acceptable

⇒ **decision of professional organizations of Intercereales to work on alternative sampling plans with an aim of having a validated european standard**

- 1 Group dealing with tests in silos : french cereals Group
 - Tests in real conditions : manual or mechanical sampling, static batches of cereals or flowing cereals and cereal products...in silos, lorries, ship, barges, wagons...
 - Making a comparison between :
 - » a regulation scheme based on OTA or fusariotoxins regulation
 - » the draft standard of the standardization group
 - » a more « practical » plan for daily controls by storekeepers
 - Taking into account of all the existing equipment
 - on cereals and cereals products

Working group on tests in cooperatives : DON

1. comparison of 2 sampling plans :

- regulation ; draft standard
- tests in real conditions :
 - flowing cereals : 5 tests (4 on maize - 1 on wheat) ; 4 in silos ; 1 on train
 - Static cereals : 14 lorries (6 of wheat - 8 of maize) and 3 type of spears ; 2 vertical silos and 1 flat silo

2. heterogeneity of distribution of DON in silos

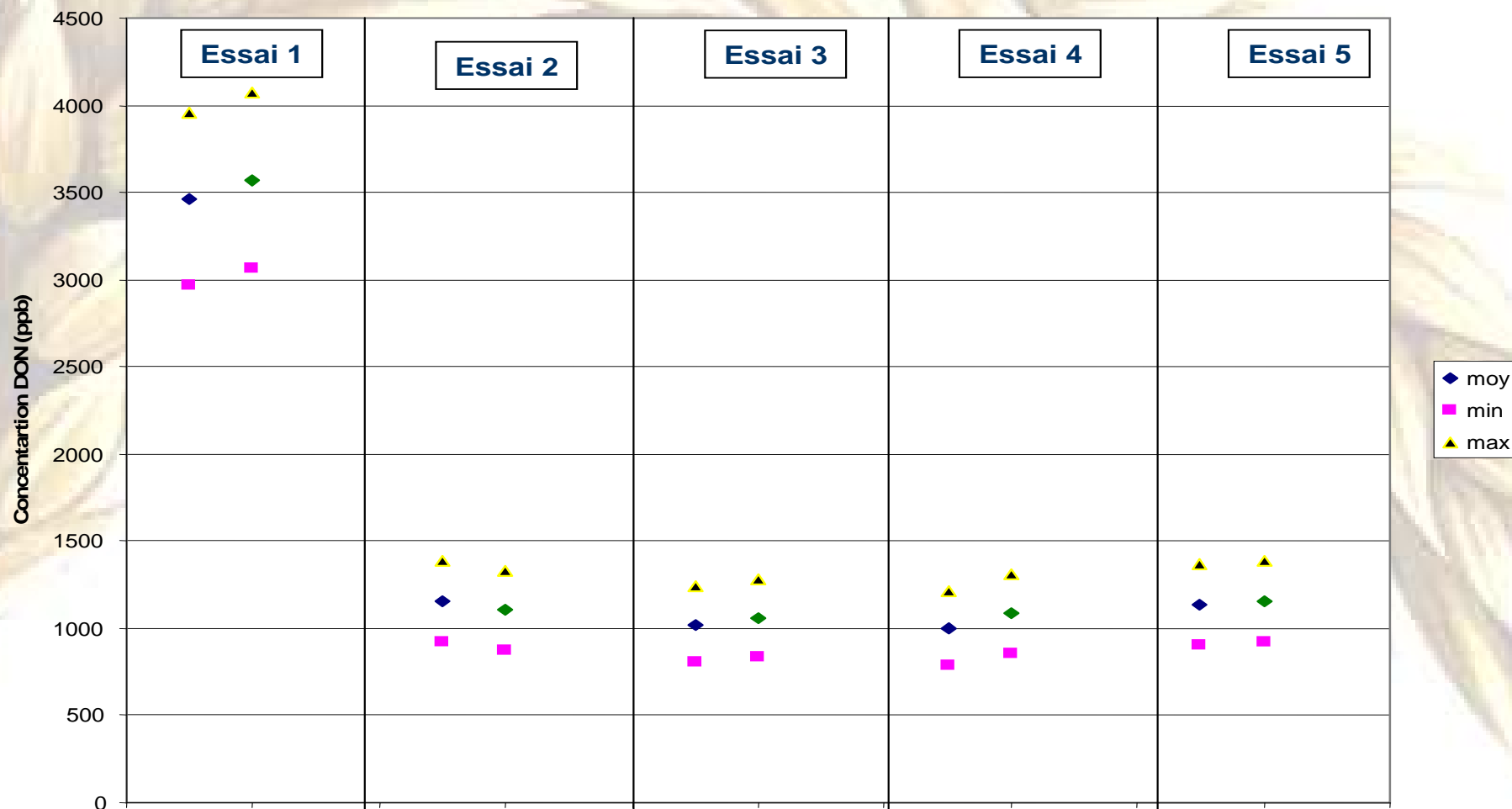
- 1 vertical silos and 2 flat silos ; on wheat ; 100 increments by silos (silos of about 500 t) ; each increment analysed

Results of the study on DON

1. Résultats of the comparison of sampling plans :

- Flowing grains : 100 versus 25 (regulation / standard)

Prélèvements en mouvement

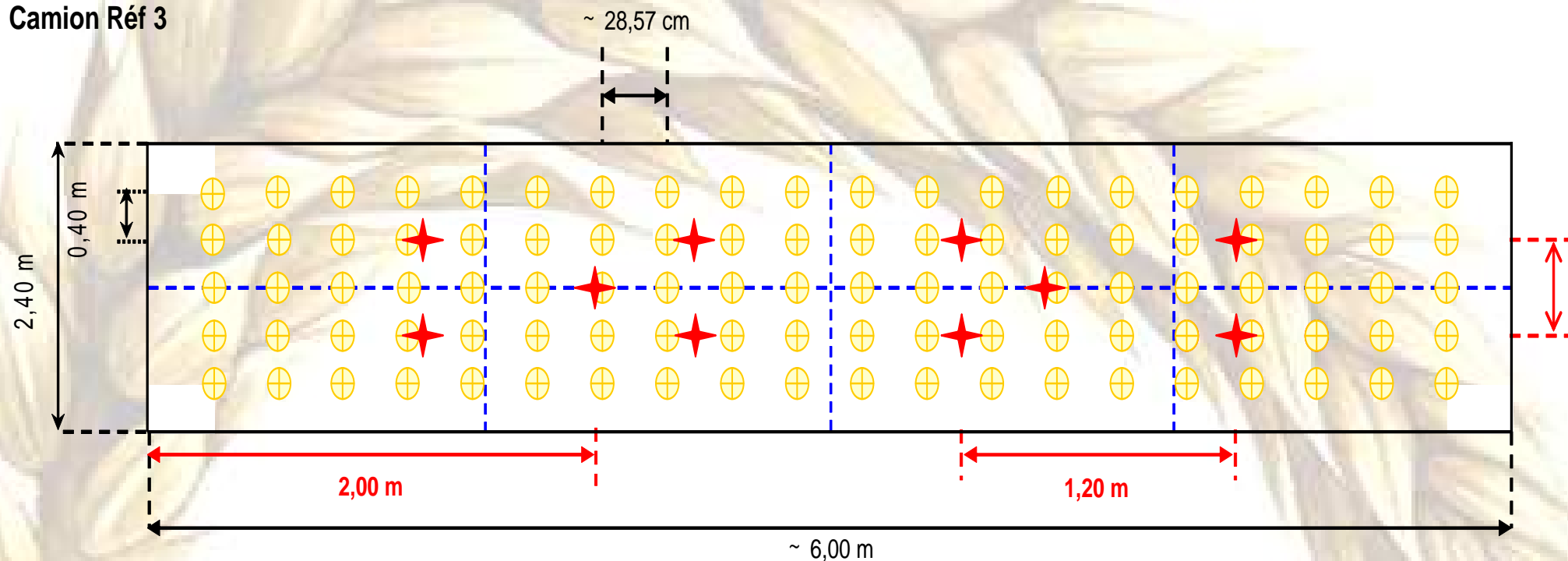


Results of the study on DON

1. Résultats of the comparison of sampling plans :

– Static grains in lorries :

Camion Réf 3



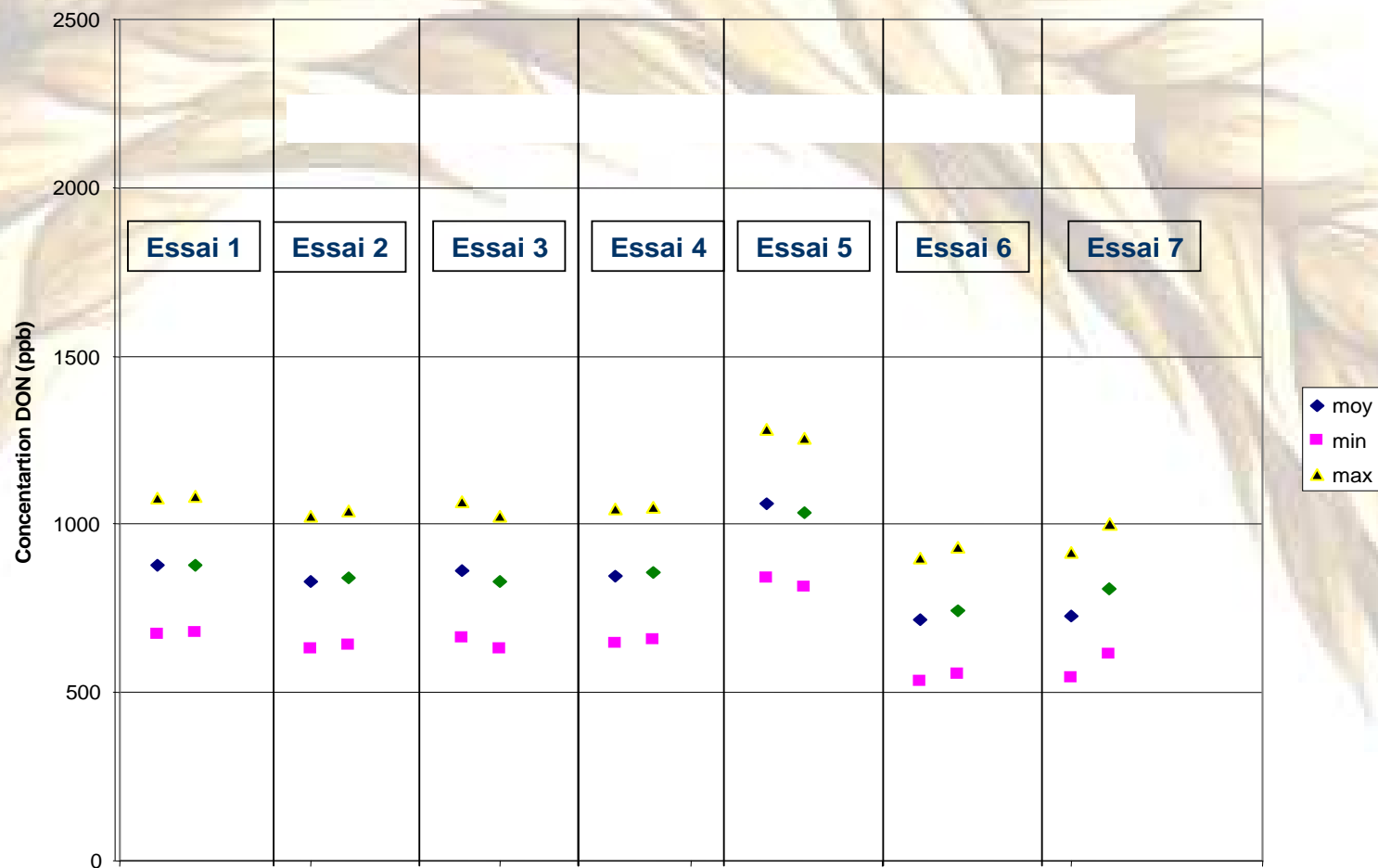
⊕ : 100 points de prélèvement

★ : 10 points de prélèvement

Results of the study on DON

1. **Résultats of the comparison of sampling plans :**
 - Example of sampling of static grains in lorries : 100 versus 5 (regulation / standard)

Prélèvements sur camions - statique



Réglementaire – normatif

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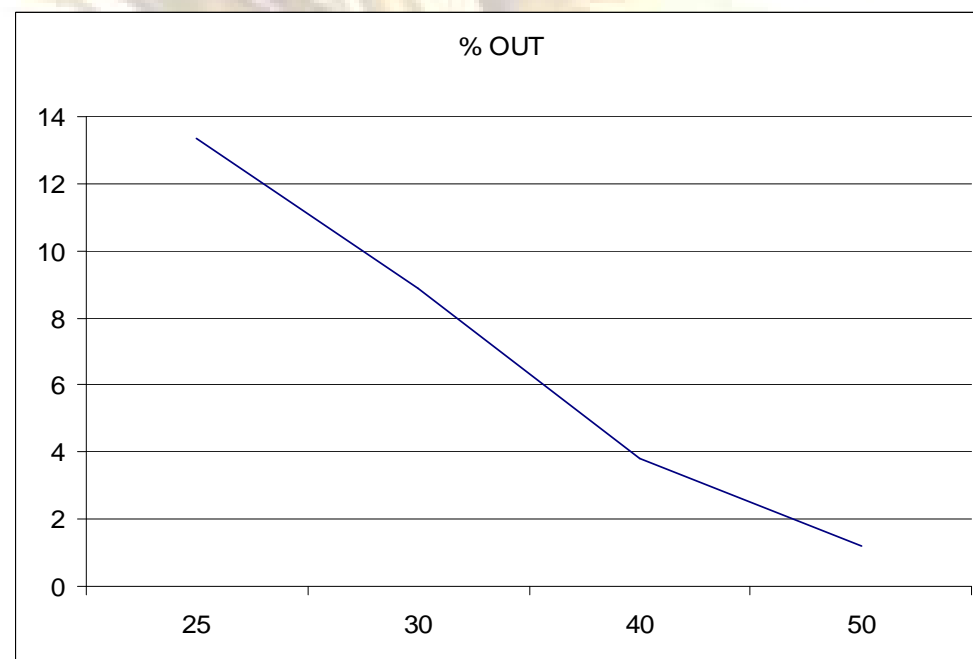
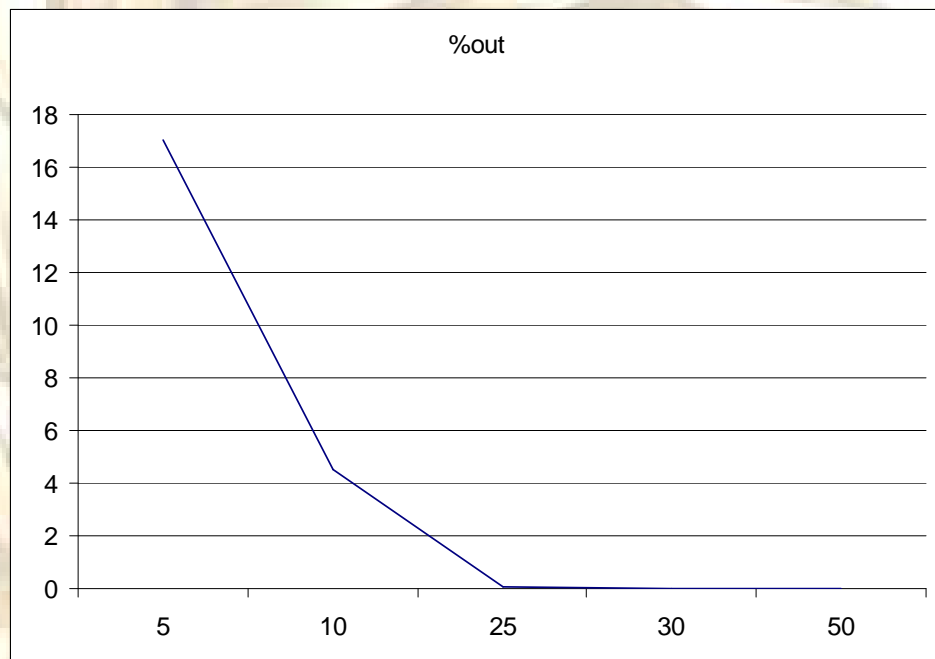
Results of the study on DON

1. Résultats of the comparison of sampling plans :

- The mean value of DON observed in the different lots sampled are comprised between 477 and 3461 ppb : the values frame well the lawful thresholds
- Statistical results :
 - No statistical differences between regulation and standard protocol in the case of flowing cereals or static cereals
 - ⇒ For the same lot, the 2 sampling plans give the same evaluation of the mean DON level
 - No statistical differences between sampling by manual spear or automatic spear but necessity to confirm

Results of the study on DON

- Statistical results :
 - Modelisation of the number of samples (increments) and relation with evaluation of correct mean value in 2 silos (standard versus regulation)



CONCLUSIONS

- The regulatory sampling plans and alternative protocol give the same evaluation for the DON average value of a lot (no statistical difference) for flowing grains or static batch

→ the regulation protocol led to unjustified costs as well for official controls as for intervention

Example of a silo of 500 t :

Type of sampling plan	Number of incremental samples	Time needed for sampling (h) – Aggregate sample weight (kg)	Time needed for homogénéisation and division of the aggregate sample (h)	Time needed for the first grinding of the laboratory sample, the cleaning and decontamination of the grinder (h)	Preparation of laboratory sample (h)	Total time needed for the preparation of laboratory sample before analysis (h) - Total evaluated cost (sample and analysis)
« Régulation »	100	18h – 190 kg	5h30	0h40	1h10min (for 10 kg)	25h20 - 662 euros
« Draft Standard »	50	7h – 105 kg	2h10	0h40	1h10min	11h - 347 euros

CONCLUSIONS

- **Heterogeneity of distribution in a silo**
 - **Sometimes, high variability in a silo**
 - **high variability between silos**
 - **on 3 silos, simulation of the relation between confidence interval on mean value and theoretical number of increments show that the standard deviation due to sampling is less or equal than the analytical standard deviation for 10 to 50 incremental samples**
- **Clearly need of more data on heterogeneity**

FUTURE WORK

- At this point, it's necessary to complete the work on :
 - comparison of protocols of sampling for fumonisines and zearalenone on maize
 - comparison of protocols of sampling for ochratoxine /
 - evaluation of the relation between number of Increments and size of laboratory sample on the mean level of mycotoxins of a lot

FUTURE WORK

- At this point, it's necessary to complete the work on :
 - complement on study of heterogeneity of lots to establish clear relations between number of increments and confidence interval : in order to be able to choose the more appropriate sampling plan (balance between consumer and producer risk)
- ⇒ **Necessity to have refunds from the commission with the aim of having validated european standard for different mycotoxins**

Possibilités et Echéancier correspondant

- **Normalisation : protocole « normatif »**
 - **Réunion d'examen des résultats et modifications du texte par le groupe en avril et juin 2006**
 - **Norme EN/ISO en 2007**
- **Normalisation : protocole de « routine »**
 - **Norme expérimentale F (délai : 3 à 4 mois) ou norme de « routine » F (délai : 7 à 8 mois)**
 - **Technical Specification ou Technical Report EU/ISO (délai : 18 à 24 mois)**

Sampling of cereals and cereal products

- After this study, we can summarize some important factors :
 - most suitable equipment should be chosen taking into account the lot to be sampled
 - Take into account that some grains can be subject to breakage when sampled by vacuum spears
 - As often as possible, favoured automatic samplers versus manual sampling equipment
 - best results will be obtain on flowing cereals when possible

Sampling of cereals and cereal products

- The methods (automatic or manual) of taking samples from flowing batches shall be adapted to the speed at which the products are flowing
- for grain, whichever method of sampling is used, the increments should be taken at regular intervals over the entire width and depth
- for automatic sampling the equipment shall be adjustable so that the size of the increments or the frequency of sampling can be varied over a wide range.

Sampling of cereals and cereal products

- for bulk samples, take into account that the lightest impurities are often at the surface and the heaviest often at the centre
- for bulk static product, manual or automatic equipment can be used up to 2.5 m of depth
- the equipment available allows sampling up to a depth of 9 m maximum : technical necessity to use vacuum spears between 2.5 and 9 m
- The number of increments shall be as high as possible