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Sampling of cereals and cereal products

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Regulation on sampling for cereal products

- **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 !**

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

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 netherland
  - from ISO : Iran, Canada, Thailand, China

• draft of standard circulated between experts and comments received in 2005

→ **a standard in 2007**

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

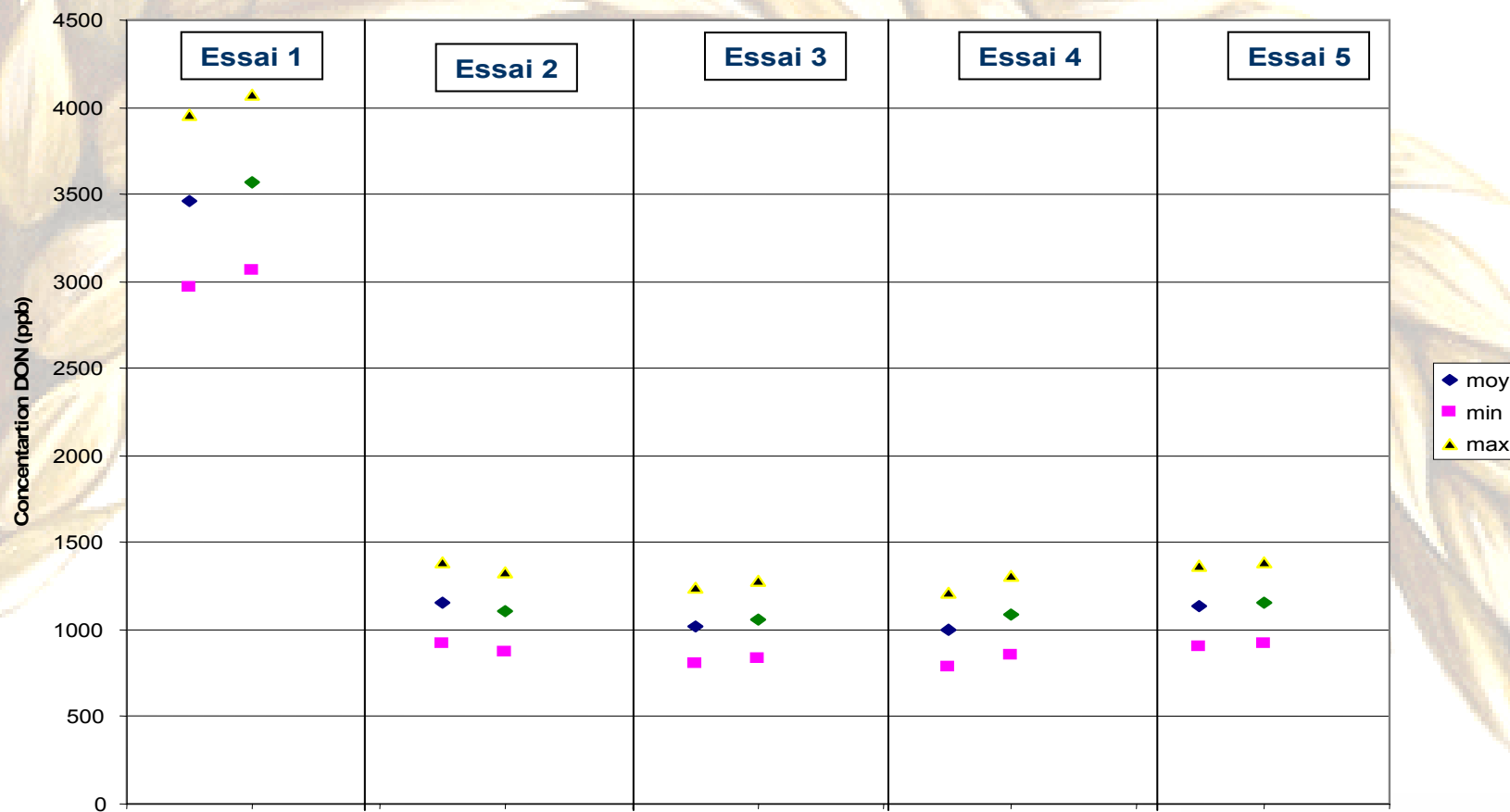2. **heterogeneity of distribution of DON in silos**

    ▪ 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. **Résults of the comparison of sampling plans :**

– Flowing grains :
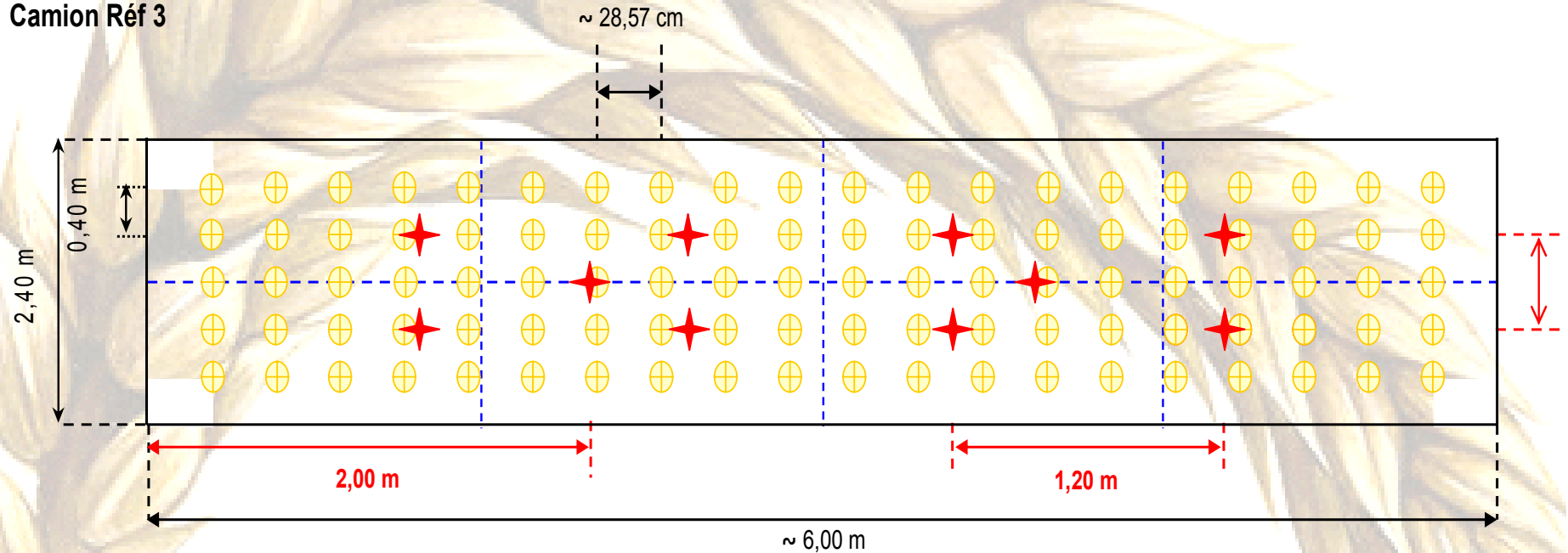
Prélèvements en mouvement

Results of the study on DON

1. **Résults of the comparison of sampling plans :**

– Static grains in lorries :
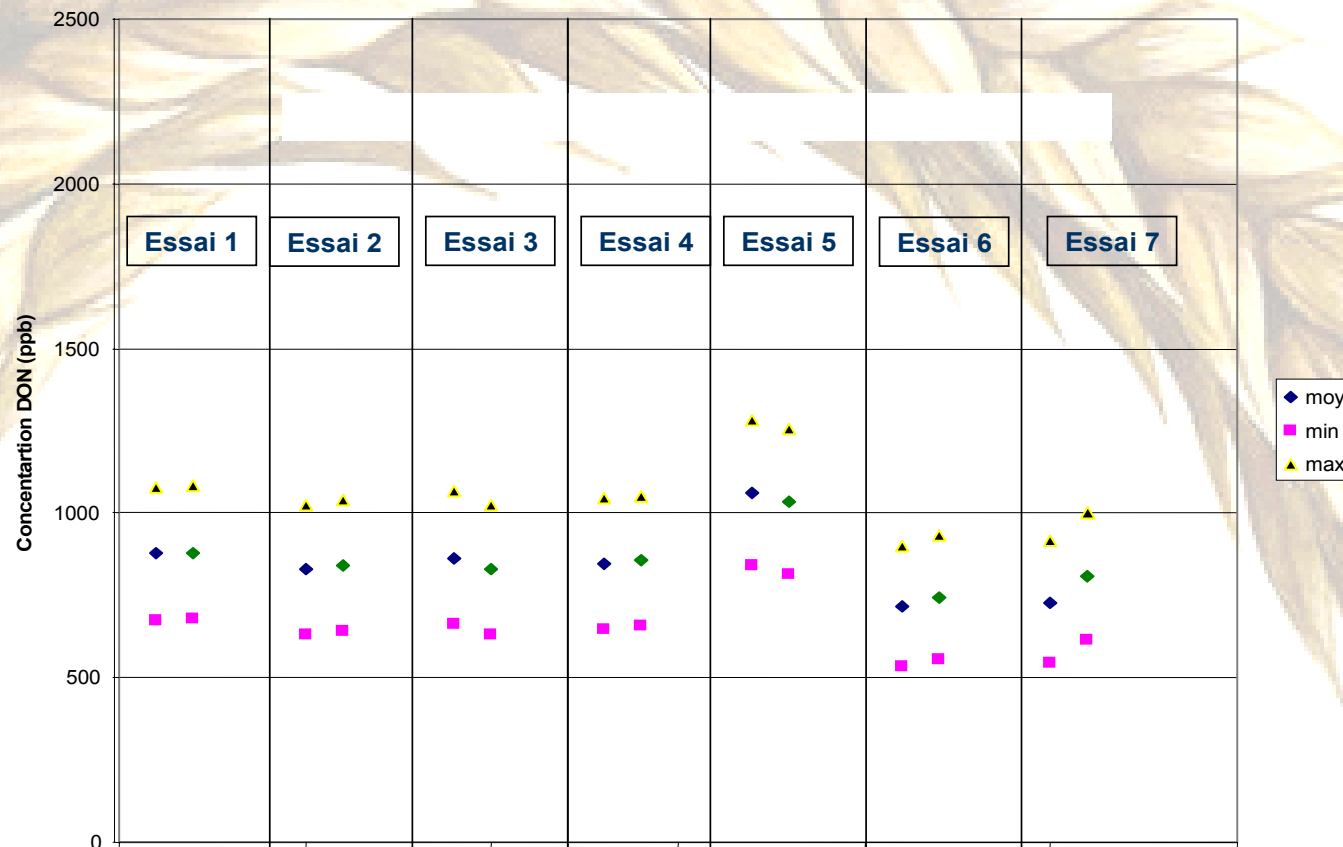
Camion Réf 3

⊕ : 100 points de prélèvement

✦ : 10 points de prélèvement

Results of the study on DON

1. **Résults of the comparison of sampling plans :**

– Example of sampling of static grains in lorries :

**Prélèvements sur camions - statique**

Results of the study on DON

1. **Résults 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

2. heterogeneity of distribution of DON in silos
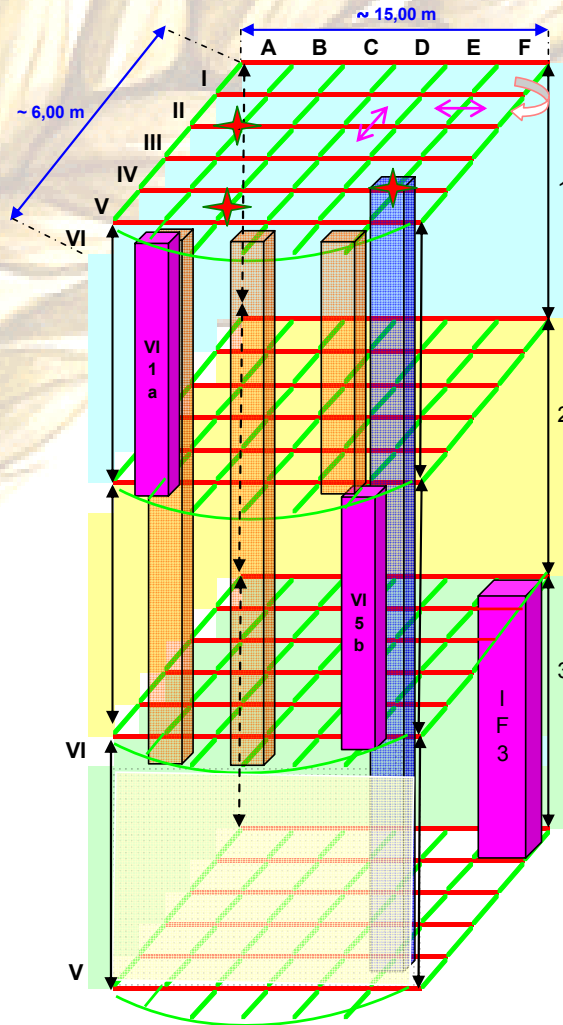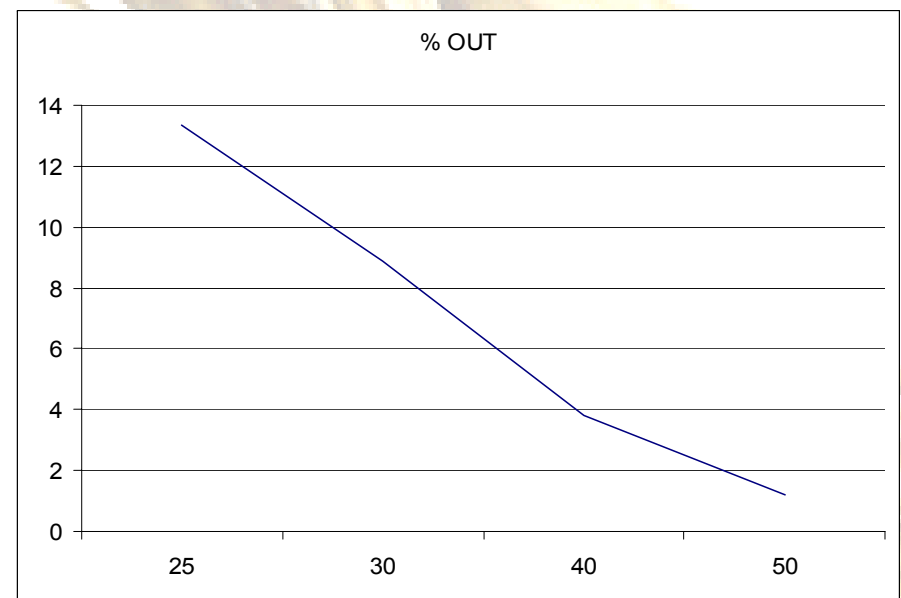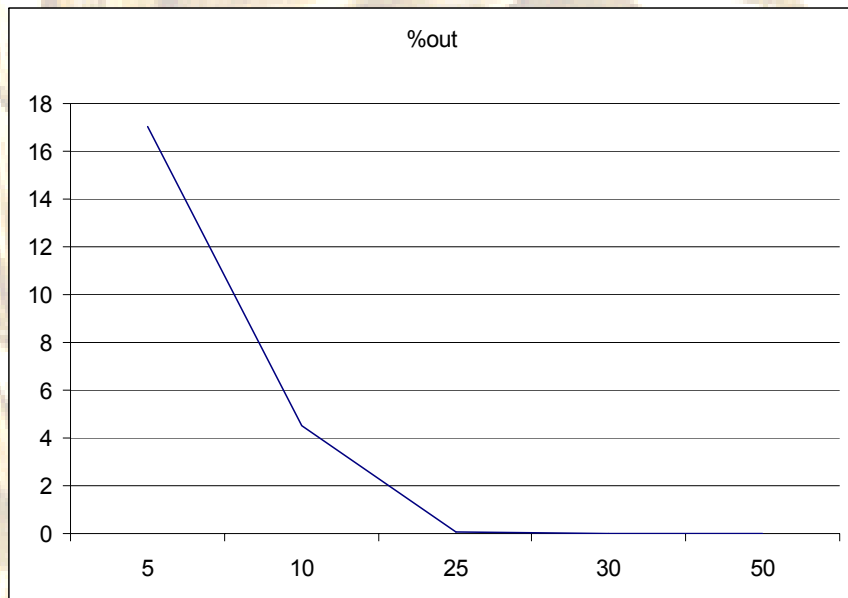

-100 samples analysed

- depth : 9 m

Results of the study on DON

– Statistical results :

- Modelisation of the number of samples (increments) and relation with consumer risk in 2 silos

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 theorical 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 A

- • 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 <u>validated european standard</u> for different mycotoxins**

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