



OFFICE NATIONAL  
INTERPROFESSIONNEL  
DES GRANDES CULTURES



ARVALIS  
Institut du végétal



# Sampling program 2005-2007 Caroline DESGRANGES (ONIGC)



*Fifth Fusarium-toxin forum – 11 janvier 2008*



OFFICE NATIONAL  
INTERPROFESSIONNEL  
DES GRANDES CULTURES



ARVALIS  
Institut du végétal



Study co-ordinated & undertaken by :

IRTAC, ONIGC and ARVALIS-Institut du végétal

Financed by :

ONIGC, ANMAC, ARVALIS-Institut du végétal,  
Coop de France, FNA, SYNACOMEX, SNIA,  
USIPA, GNIS

*Fifth Fusarium-toxin forum – 11 janvier 2008*



OFFICE NATIONAL  
INTERPROFESSIONNEL  
DES GRANDES CULTURES

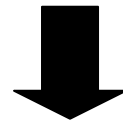


ARVALIS  
Institut du végétal



## Problem posed

Regulation 401-2006 : 100 increments / 500 tonnes



Evaluate the accuracy using less than

100 increments / 500 t.

☛ Norm EN ISO 24333

*Fifth Fusarium-toxin forum – 11 janvier 2008*

## Available Data

		Regulation EC Sept 07 -Maximum levels in foods- (ppb)	Mean values of studied silos (ppb)
<b>DON</b>	<i>Wheat</i>	1 250	477 to 1 988
	<i>Maize</i>	1 750	2 633
<b>Fumonisines B1+B2</b>	<i>Maize</i>	4 000	534 to 7 132
<b>Zearalenone</b>	<i>Maize</i>	350	139 to 683

Wheat : Sampling by vacuum spear during storage

Maize : Sampling in flowing grain at exit of silo

Mass of each increment : 750 à 2700g of grain



OFFICE NATIONAL  
INTERPROFESSIONNEL  
DES GRANDES CULTURES



ARVALIS  
Institut du végétal



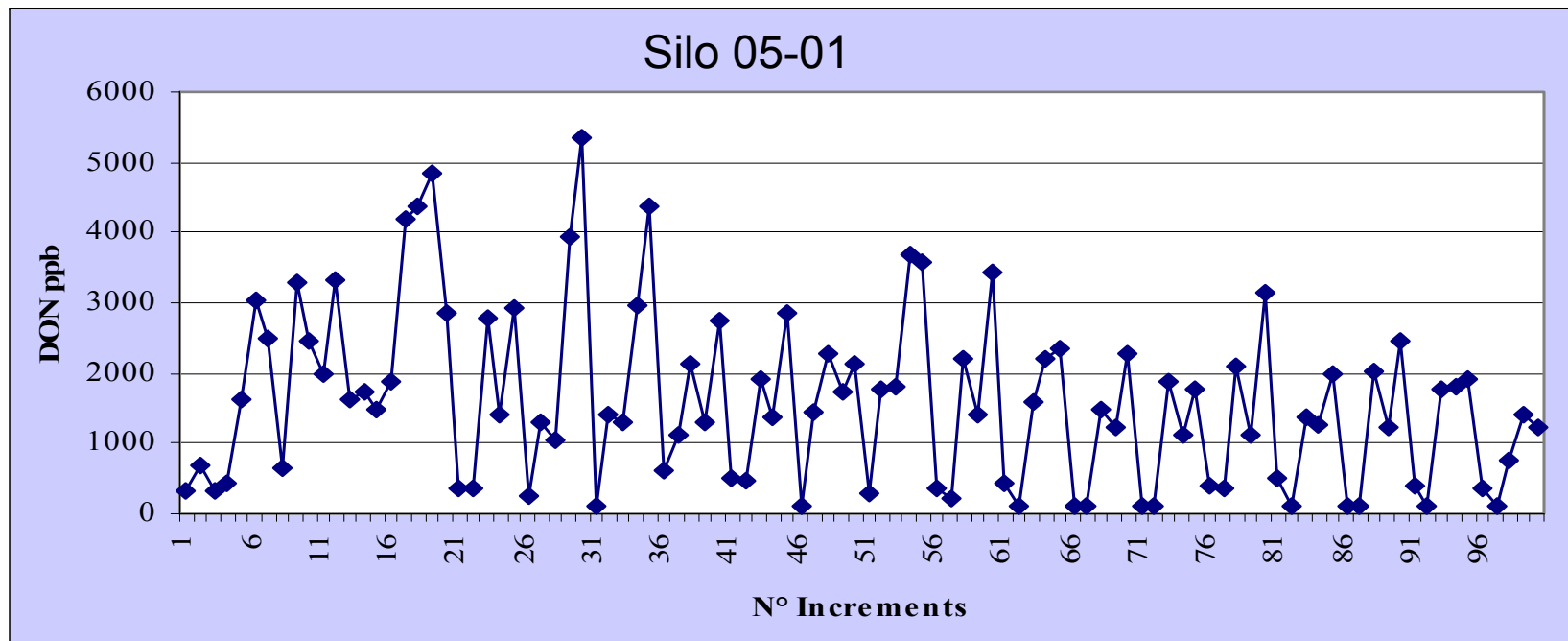
## Available Data

1 582 mycotoxin analysis

- ➔ Quantification of fumonisines B1+B2 by HPLC fluorometry with solid phase extraction clean up - NF EN ISO 13585
- ➔ Quantification of zearalenone by HPLC fluorometry and immunoaffinity column clean up – normative project EN ISO 17372
- ➔ Quantification of deoxynivalenol by HPLC UV and immunoaffinity column clean up – normative project EN ISO DON CEN/TC275/WG5/M383

*Fifth Fusarium-toxin forum – 11 janvier 2008*

## Distribution within silos -DON-



*Fifth Fusarium-toxin forum – 11 janvier 2008*



OFFICE NATIONAL  
INTERPROFESSIONNEL  
DES GRANDES CULTURES

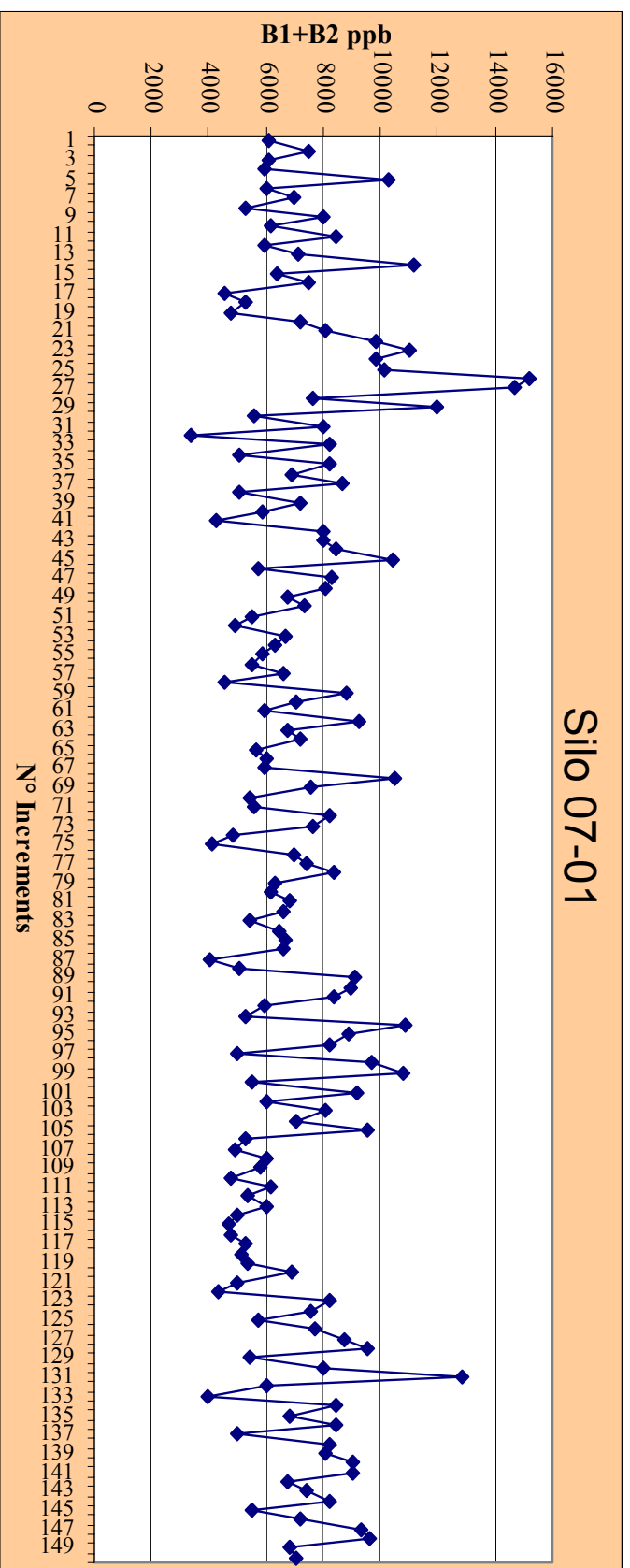


ARVALIS  
Institut du végétal



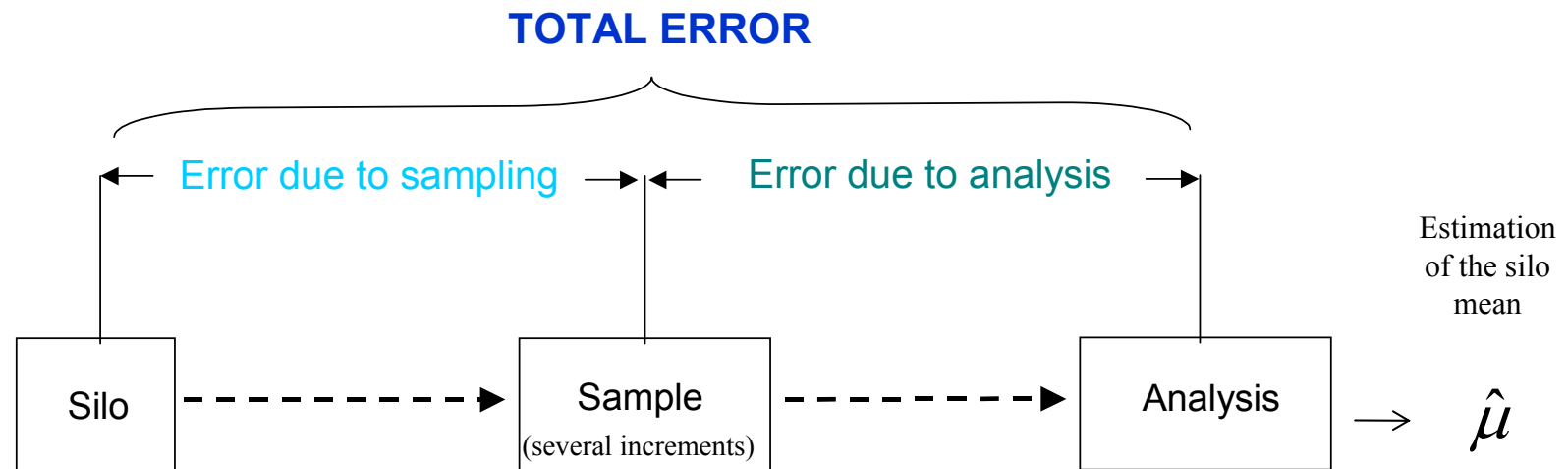
# Distribution within silos -Fumonisines-

Silo 07-01



*Fifth Fusarium-toxin forum – 11 janvier 2008*

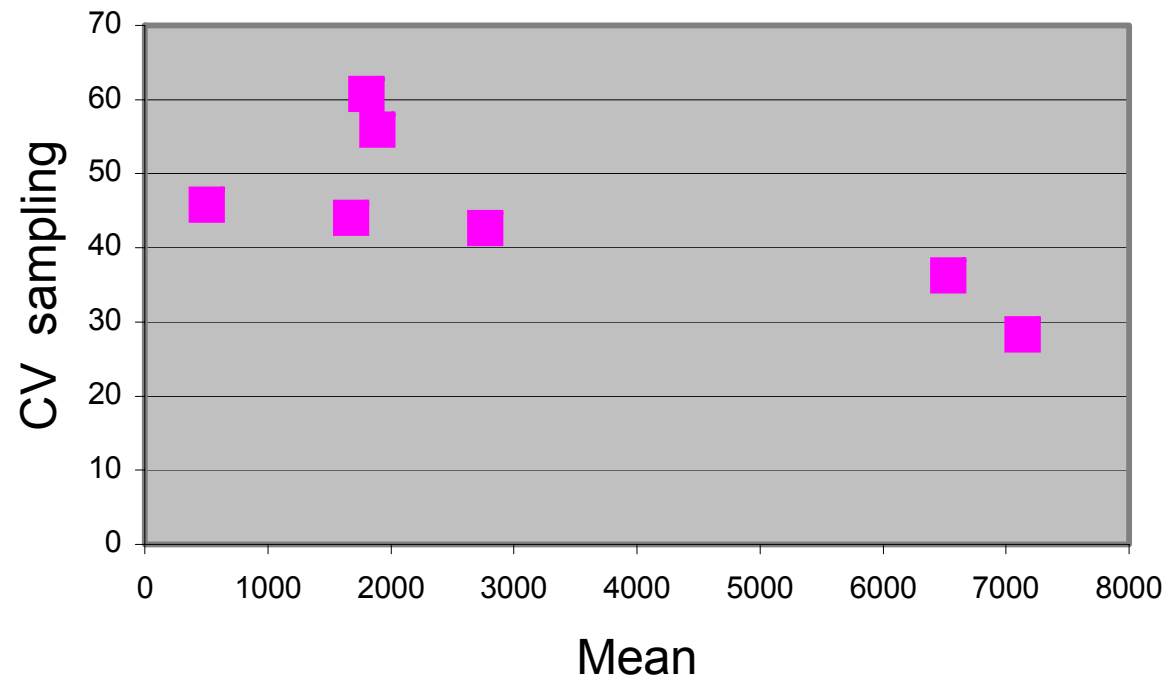
# Statistical Analysis



$$\boxed{\text{Total Error}} = \boxed{\text{Error due to sampling}} + \boxed{\text{Error due to analysis}}$$



## Statistical Analysis



The coefficient of variation (CV) is independant from the mean

*Fifth Fusarium-toxin forum – 11 janvier 2008*

# Statistical analysis

Sampling error



CV = 45%

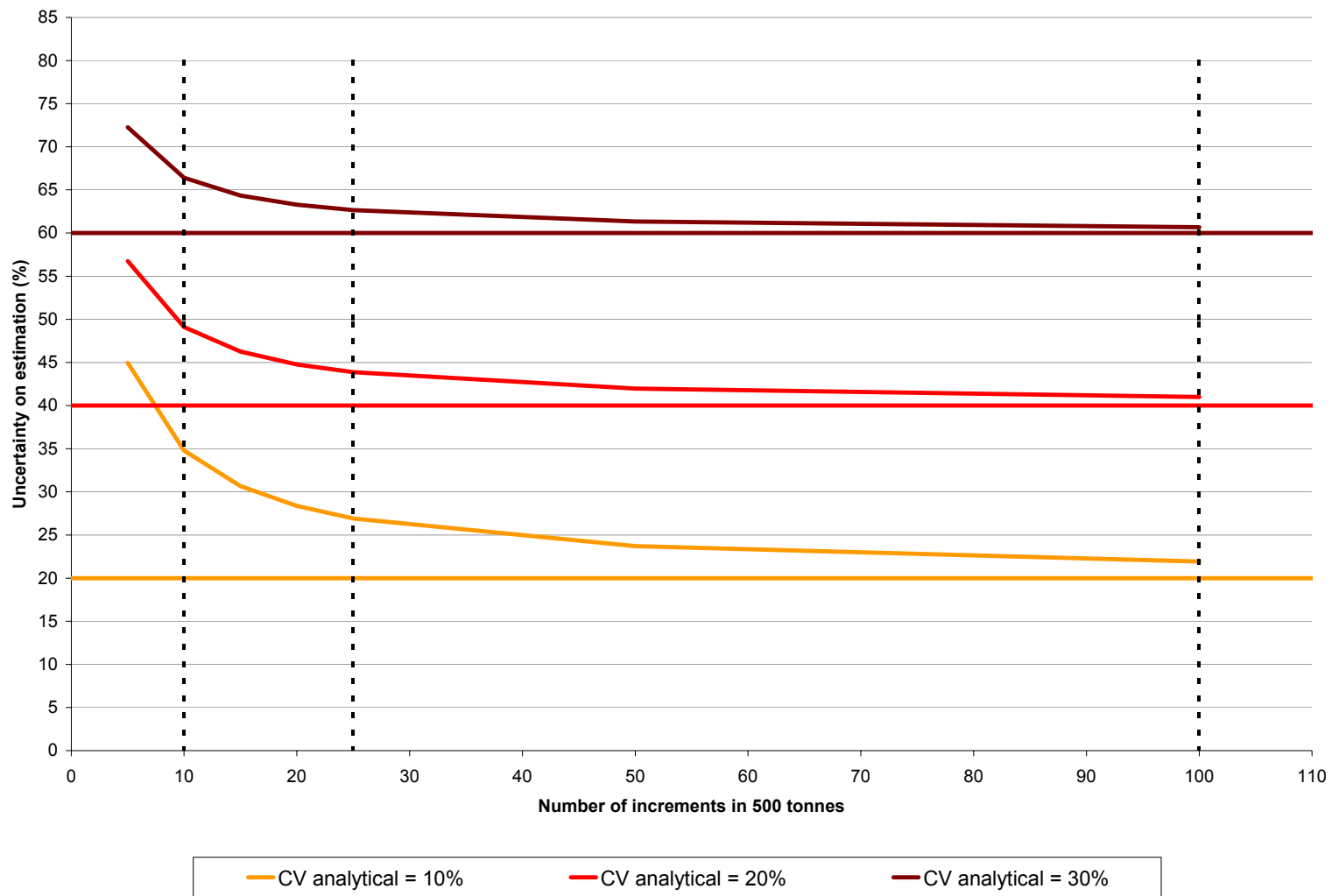
Analytical error



3 working hypothesis

CV = 10%, 20% et 30%

## Conclusions



*Fifth Fusarium-toxin forum – 11 janvier 2008*

## Conclusions

- Overall accuracy of the result cannot be lower than analytical accuracy given by  $CV_{\text{analytical}}$
- From a given number of increment samples the gain in precision becomes insignificant :

=> 100 increments +  $CV_{\text{analytical}}$  of 20% : uncertainty at +/- 41%

=> 25 increments +  $CV_{\text{analytical}}$  of 20% : uncertainty at +/- 44%

=> 10 increments +  $CV_{\text{analytical}}$  of 20% : uncertainty at +/- 50%



- 3 points

- 6 points



OFFICE NATIONAL  
INTERPROFESSIONNEL  
DES GRANDES CULTURES



ARVALIS  
Institut du végétal



## Conclusions

- Total Error = Sampling Error + Analytical Error
- This model is validated by an empirical approach  
(simulation of the composition of 10 000 samples using 5, 10, 15... increments)
- This model is valid for :
  - DON, Fumonisines, Zearalenone
  - Wheat and Maize
  - Large panel of contaminations

*Fifth Fusarium-toxin forum – 11 janvier 2008*

## Application : Decision aiding table

Relative analytical uncertainty (%) Number of increments	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40	42	44	46	48	50	52	54	56	58	60
5	41	42	43	43	44	45	46	47	48	49	50	51	53	54	55	57	58	60	61	63	64	66	67	69	71	72
10	30	31	32	33	34	35	36	37	39	40	41	43	44	46	47	49	51	52	54	56	58	59	61	63	65	66
15	25	26	27	28	29	31	32	33	35	36	38	40	41	43	45	46	48	50	52	53	55	57	59	61	62	64
20	22	23	25	26	27	28	30	31	33	34	36	38	40	41	43	45	47	48	50	52	54	56	58	60	61	63
25	21	22	23	24	25	27	28	30	32	33	35	37	38	40	42	44	46	48	49	51	53	55	57	59	61	63
50	16	17	19	20	22	24	25	27	29	31	33	34	36	38	40	42	44	46	48	50	52	54	55	57	59	61
100	13	15	17	18	20	22	24	26	28	29	31	33	35	37	39	41	43	45	47	49	51	53	55	57	59	61
Infinity	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40	42	44	46	48	50	52	54	56	58	60

## International normalization

- 2 norms in preparation :
  - European & international norm : EN ISO 24333  
*reviewing between march to october 2008 ; publication in 2009*
  - Technical report (supporting norm EN ISO 24333), gathering French, UK and German sampling studies : project EN ISO 29263  
*reviewing between may to july 2008 ; publication end of 2008*



OFFICE NATIONAL  
INTERPROFESSIONNEL  
DES GRANDES CULTURES



ARVALIS  
Institut du végétal



# Sampling program 2005-2007



*Fifth Fusarium-toxin forum – 11 janvier 2008*