



Canadian Grain  
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# Sampling and sample preparation for testing cereals for deoxynivalenol (DON)

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## Acknowledgements

- Characterizing sources of variability in DON testing:
  - J. Chan, D. Gaba, K. Pleskach
    - Grain Research Laboratory, Canadian Grain Commission
  - J. Osborne
    - Department of Statistics, North Carolina State University
  - A.B. Slate and T.B. Whitaker
    - Biological and Agricultural Engineering Department, North Carolina State University
- Effects of sample processing on variability in testing:
  - R. Blagden, J. Chan, T.L. McMillan, K. Pleskach, M.S. Izydorczyk
    - Grain Research Laboratory, Canadian Grain Commission



## Challenges in measuring bulk material



Ollier *et al.* 2018  
[doi.org/10.1111/pbr.12667](https://doi.org/10.1111/pbr.12667)



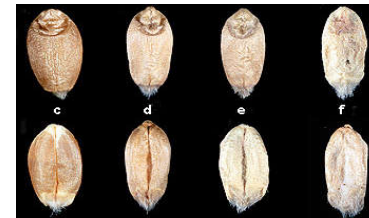
0.22 mg/kg





## Challenges measuring bulk wheat for DON

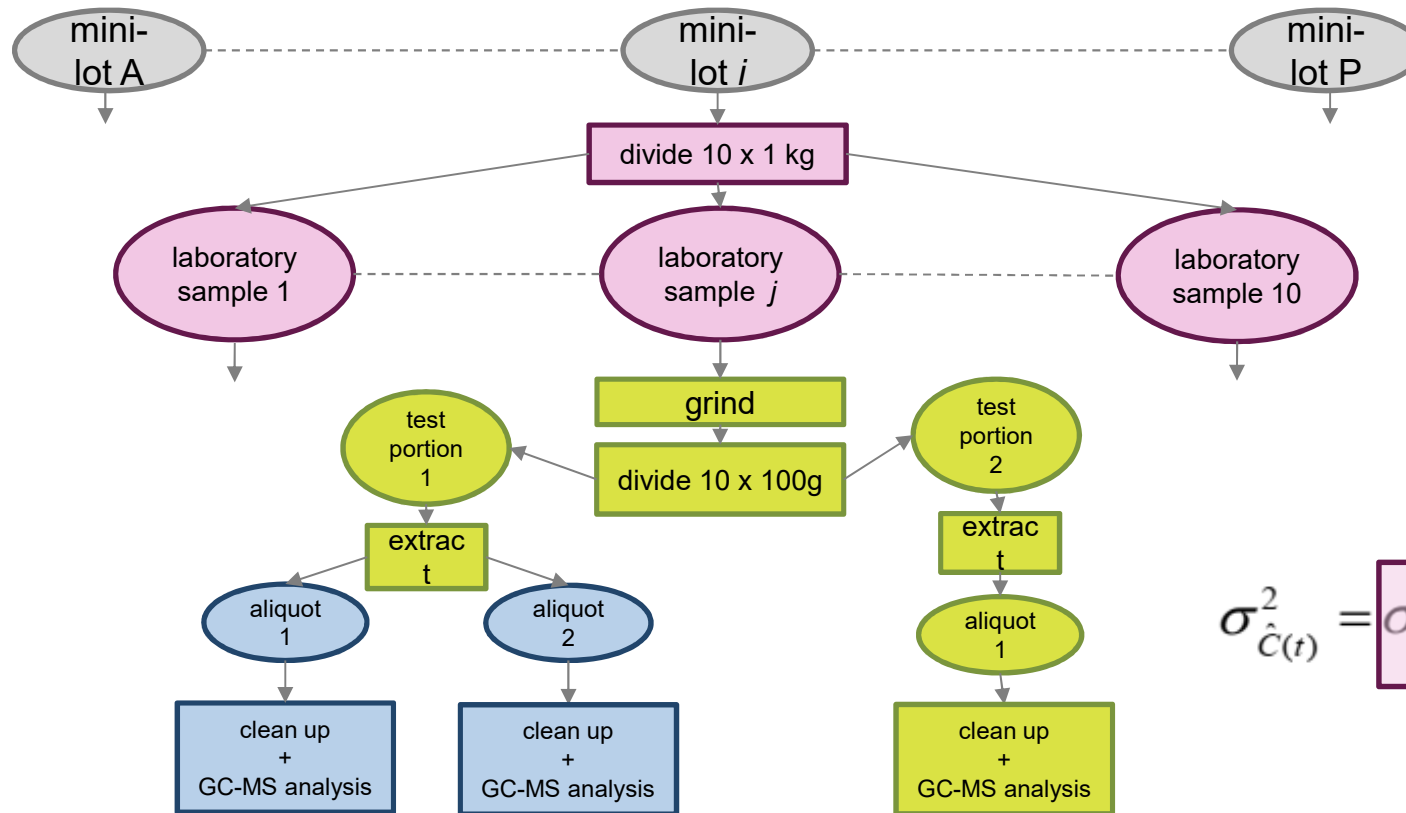
- A number of studies have demonstrated variability of DON in bulk wheat:
  - DON in truckloads and storage: 3-125% CV
  - (Hart and Schabenberger, 1998; Biselli et al., 2008; Mallmann et al., 2018)
- As well as variability within a wheat field:
  - <0.02 – 1.0 mg/kg (Oerke et al., 2010)
  - <0.001 – 0.88 mg/kg (Xu et al., 2008)
- And amongst kernels:
  - <LOD – 600 mg/kg (Sinha and Savard, 1997)







## Experimental design

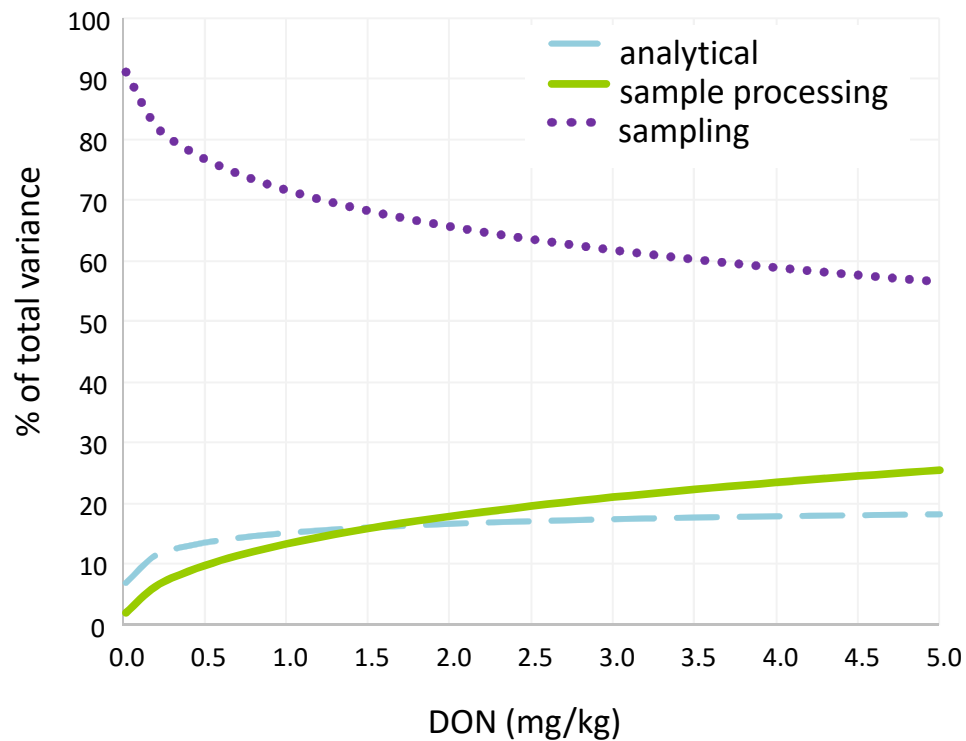


$$\sigma_{\hat{C}(t)}^2 = \sigma_{\hat{C}(s)}^2 + \sigma_{\hat{C}(sp)}^2 + \sigma_{\hat{C}(a)}^2$$



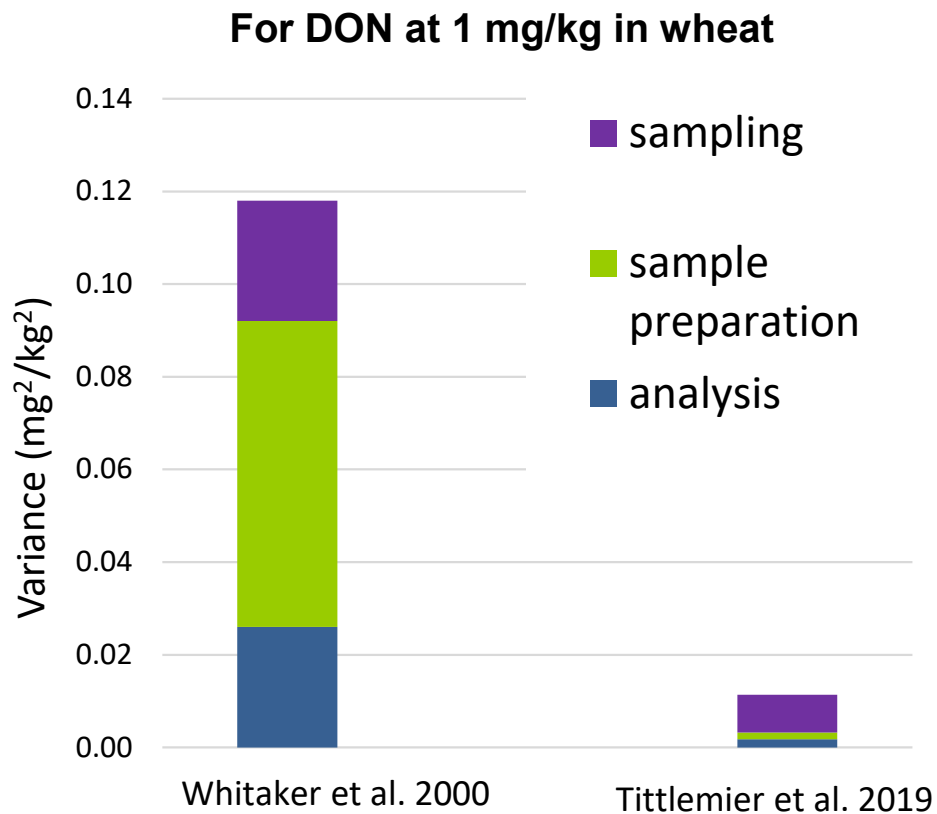
## Contributions to total variance

$$S_t^2 = 0.0081DON^{1.6372} + 0.0015DON^{2.1874} + 0.0017DON^{1.8991}$$





## Main contributors to DON test results variance

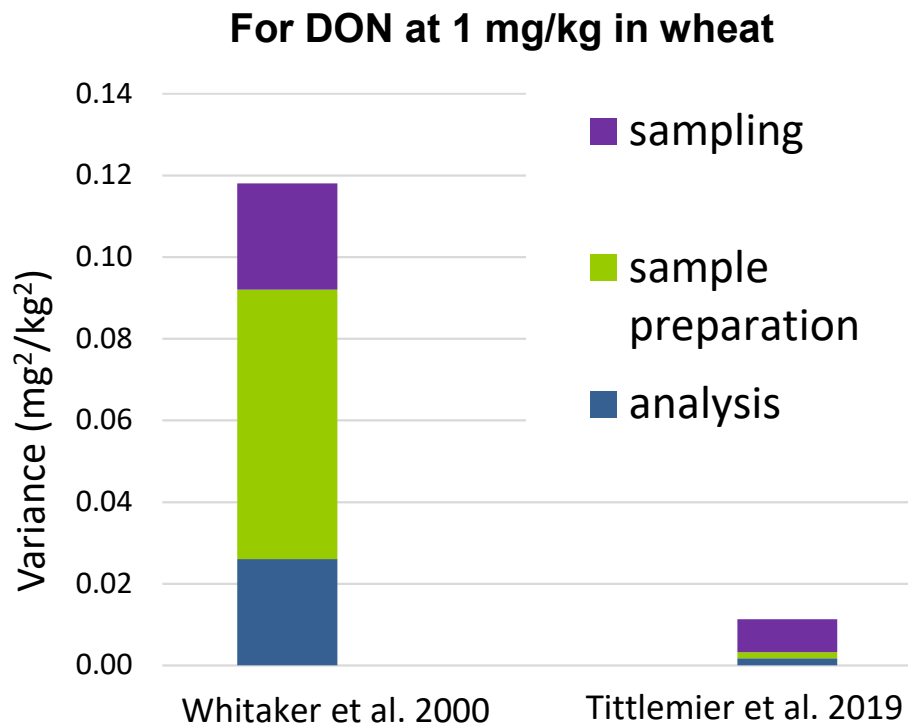


- 2019 vs 2000
  - total variance ~10x less
  - Initial sampling step contributed the most to total variance





## How can DON test results variance be reduced?



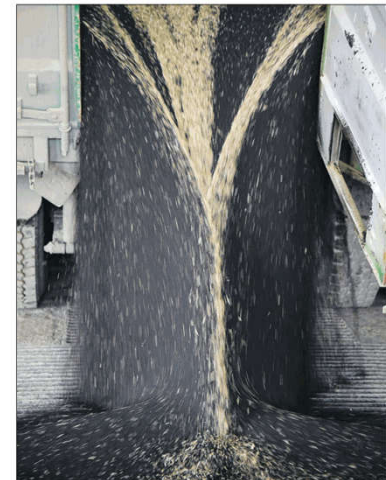
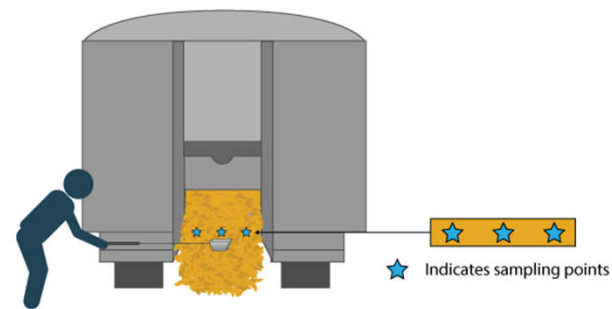
	2000 study	2019 study
Lab sample	0.454 kg	1 kg
Test portion	25 g	100 g
Grinder	Burr mill	Rotor beater
Test method	ELISA	GC-MS

- increase sample size
- decrease particle sizes
- increase precision of test method



## Maximize sample representativeness

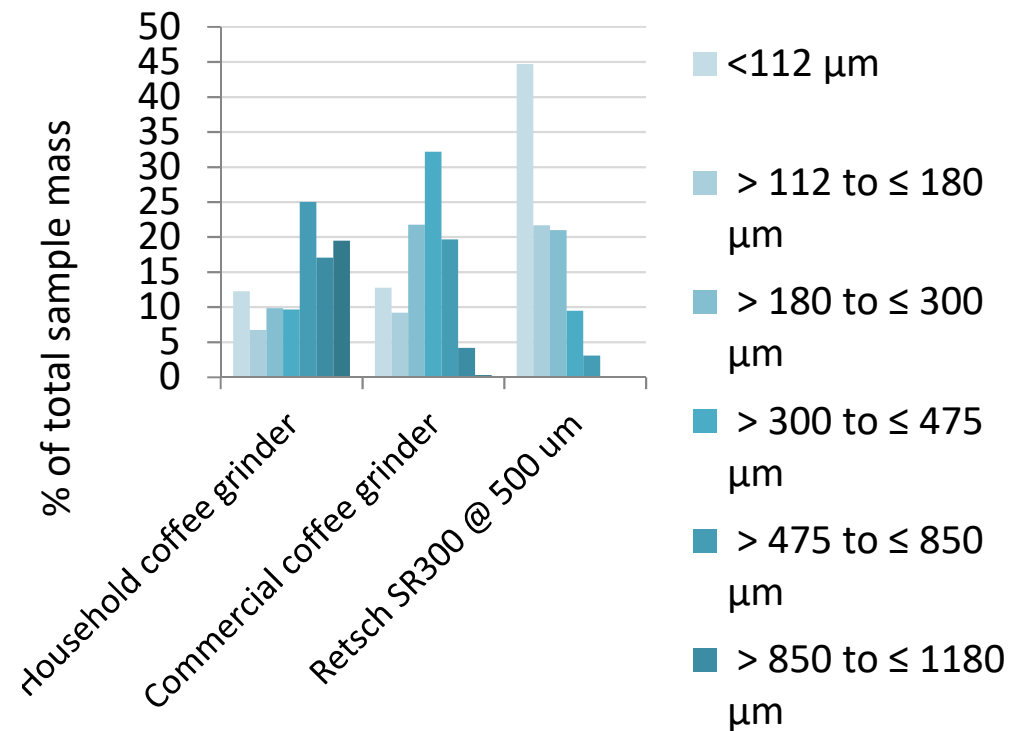
- Take increments to prepare composite samples
- Use proper tools and procedures when sampling and dividing
- Optimize sample size



Canadian Grain Commission - Guide to Taking a Representative Sample



## Grind well to reduce distributional heterogeneity





## Maintain sample representativeness

- Use proper tools and procedures when mixing and dividing whole grain
- Shaking or hand mixing is not a good procedure!



Properly mixed

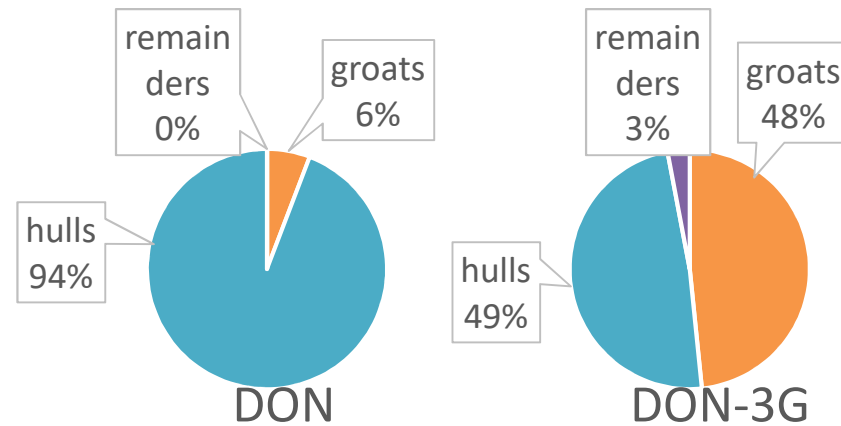


Shaken



## Maintain sample representativeness

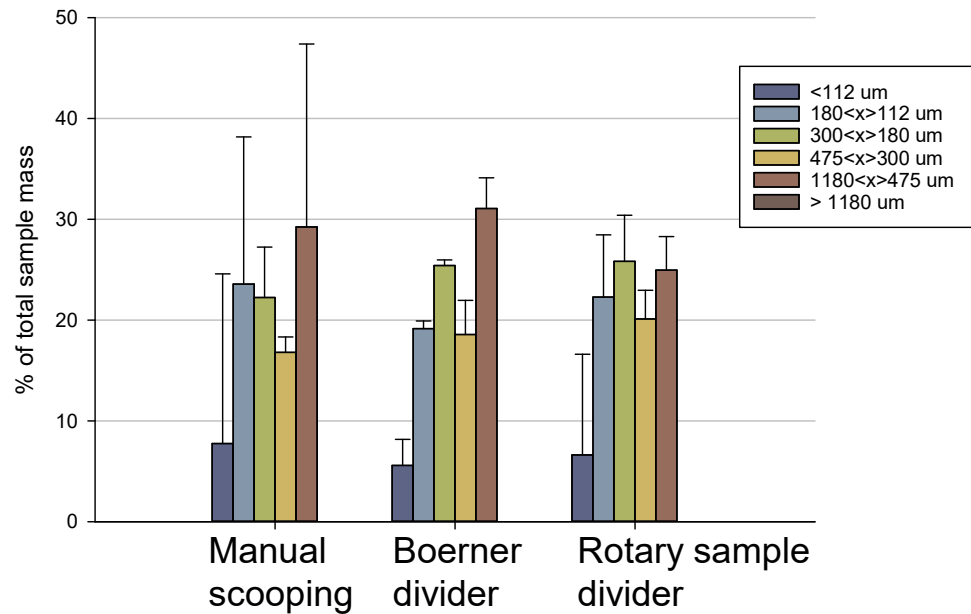
- Use proper tools and procedures when mixing and dividing ground grain
- Shaking or hand mixing is not a good procedure!





## Maintain sample representativeness

- Manual scooping produces more variable sub-sample composition





## Reducing variability in DON test results

- Maximize sample representativeness by:
  - taking increments from the larger bulk to form a composite
  - grind samples before sub-dividing
  - use proper tools and procedures for mixing, grinding, and dividing
  - optimize sample size (laboratory sample and test portion)
- Must balance cost (time, equipment, etc.) with the benefit of a reduction in DON variance



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