

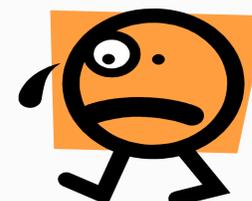


Evaluation of sampling uncertainty of loose material

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Is it possible to take a sample without uncertainty ?

INTRODUCTION

The main approach for sampling uncertainty is presented in the Eurachem Guide (1). Based on this guide the Nordtest TR 604 handbook was elaborated (3).

AIM

The poster shows a simple way of sampling uncertainty evaluation of loose materials, generally based on the Eurachem Guide (1) and Nordtest Handbook (2), using an example of analyses of mineral components in feedingstuffs.

MATERIAL AND METHODS

The essence of the proposal lies in separating two components of results' variability, analytical variability of a method (repeatability) and technical variability corresponding with the heterogeneity of the analyzed material. Variability of results for a mineral component in incremental samples, expressed as variation coefficient CV_m (%), depends on method repeatability CV_r (%) and heterogeneity CV_h (%), in compliance with the Gauss' law of error propagation:

$$CV_m = \sqrt{CV_r^2 + CV_h^2} \quad [1] \quad CV_h = \sqrt{CV_m^2 - CV_r^2} \quad [2]$$

Heterogeneity CV_h (%) calculated from the formula [2] may be applied as the measure of sampling standard uncertainty for a batch of the analyzed material when the sampling method has been adjusted to the requirements or to legal recommendations, e.g. Commission Regulation (EC) No 152/2009 in case of feedingstuffs sampling. Analytical variability CV_r was calculated from the range of duplicate analyses, following Nordtest Handbook (1) – see Table 1.

RESULTS AND DISCUSSION

Taking into account the above assumptions, sampling uncertainties for chlorides, calcium, zinc and copper measurement in compound feed were calculated. Series of ten incremental samples were taken from the analyzed feedingstuffs batch. In each series mass of the incremental sample was 50, 100, 150, 200 or 250 g. It was assessed that the mass of the incremental sample and physicochemical form of mineral component (salt, oxide, degree of fineness) affects sampling uncertainty. In the case of an incremental samples with the mass of 100 g, heterogeneity CV_h (%) as the measure of standard sampling uncertainty for determination the content of chlorides (added as NaCl), calcium ($CaCO_3$), zinc (ZnO) and copper ($CuSO_4 \cdot 5 H_2O$) amounted to 3.25%, 3.16%, 4.45% and 9.54%, respectively. Expanded sampling uncertainty ($U = 2 \cdot CV_h$) calculated for zinc (8.9%) and copper (19.1%) in the tested feed mixture remained within the range of acceptable technical tolerance (up to 20%) for these feed additives at the feed mixture level, recommended in Regulation (EU) No 939/2010.

Table 1. Calculation of repeatability of a method for chloride determination in feedingstuffs (g/kg) from the range acc. to Nordtest (1)

n	X1	X2	Mean value	d	r%
1	2,45	2,46	2,455	-0,01	0,4073
2	2,42	2,43	2,425	-0,01	0,4124
3	2,44	2,4	2,42	0,04	1,6529
4	2,40	2,52	2,46	-0,12	4,878
5	2,48	2,43	2,455	0,05	2,0367
6	2,58	2,57	2,575	0,01	0,3883
7	2,49	2,48	2,485	0,01	0,4024
8	2,51	2,54	2,525	-0,03	1,1881
9	2,41	2,4	2,405	0,01	0,4158
10	2,49	2,43	2,46	0,06	2,439
			SD =	0,0512	
				CV_m = 2,08%	
			X = 2,4665		1,4221
				d2=1,128	
					CV_r = 1,2607

Table 2. Calculation of heterogeneity of minerals in compound feed (incremental sample equals to 100 g, n=10)

Analyzed component	Mean values	CV _m %	CV _r %	CV _h %	U=2 x CV (%)
Ca, g/kg	8,06	3,30	0,54	3,25	6,50
Cl, g/kg	2,46	2,08	1,26	1,66	3,32
Zn, mg/kg	100	4,67	1,40	4,45	8,90*
Cu, mg/kg	16,2	9,75	2,00	9,54	19,1 *

* permitted technical tolerance acc. to Commission Regulation (EU) No 939/2010 amount to 20%

CONCLUSIONS

The poster presentation provides simple method of sampling uncertainty estimation of loose materials based on separating technical variability from analytical variability. The method is especially useful for evaluation of permitted technical tolerances for feed additives according to reg. 939/2010. However, further testing is considered necessary for evaluation an influence of material heterogeneity on sampling uncertainty for different feed products.

References

1. Estimation of measurement uncertainty arising from sampling. Eurachem/Eurolab/Citac/ Nordtest Guide. www.eurachem.org
2. Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories Nordtest TR 537, Version 3, 2008
3. Uncertainty from sampling. A Nordtest handbook for sampling planners on sampling quality assurance and uncertainty estimation www.nordicinnovation.net