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Evaluation of uncertainty of feed additives measurement in feedingstuffs and premixtures using data from proficiency testing

Waldemar Korol, Jolanta Rubaj, Grażyna Bielecka, Sławomir Walczyński
National Research Institute of Animal Production, National Laboratory for Feedingstuffs, Lublin, Poland; email – korol@clpp.lublin.pl

INTRODUCTION

Several proficiency testings (PTs) were performed in the years 2005-2010 by national reference laboratories (NRLs) for feed additives from Hungary and Poland. According to the Regulation (EC) No 882/2004, NRLs shall organise PTs between the official national laboratories and ensure an appropriate follow-up of such PTs.

AIM

The poster shows an interlaboratory experimental approach based on data from PTs applied in order to estimate the uncertainty of vitamin A and E, copper, lysine, methionine and threonine measurement in feedingstuffs and premixtures.

MATERIAL AND METHODS

Results obtained by the National Laboratory for Feedingstuffs in these PTs were applied to bias (*b*) calculation as the measure of accuracy. Instead of bias the experimental approach to estimating standard uncertainty (*u*) took into account within-laboratory reproducibility (s_w) as the criterion of measurement precision. Following formulas presented below and published in Eurolab Technical Report (1) and Nordtest Handbook (2) were taken into account.

$$u = \sqrt{s_w^{\,2} + b^2} \qquad [1] \qquad b = \sqrt{\Delta^2 + u_{PT}^{\,2} + \frac{s_{PT}^{\,2}}{n}} \qquad [2]$$

$$\Delta = \sqrt{\frac{\sum (bias_i)^2}{n}} \qquad [3] \qquad u_f = \sqrt{(CL/2)^2 + (0.1C)^2} \qquad [4]$$

Within-laboratory reproducibilities (s_w) were calculated on the basis of control cards (copper), from the range between two replications (amino acids) or were obtained from validation studies (vitamin A and E). Obtained results were summarised in the Table 1 and compared to calculated from Horwitz's and Wood's equations [4], (3). In the Wood's equation u_f means the maximal standard uncertainty, CL means LOQ and C means concentration of determined substances.

RESULTS AND DISCUSSION

Bias obtained from PTs was succesfully used for uncertainty evaluation (Table 1). In some cases precision and bias should be improved, e.g. for vitamin A in feedingstuffs and premixtures (Table 2). Expanded uncertainty for the measurement of amino acids added to feeds in the synthetic form of lysine (8%) and methionine (13%) was consistent with the uncertainty of determining these amino acids used by the laboratories authorized to perform analyses within official control of feedinstuffs in Germany (lysine 10%, methionine 15%). Expanded uncertainties of copper measurement in feedingstuffs (11.6%) and premixtures (10.6%) were consistent with those calculated from the Horwitz's formula for feedingstuffs (Cu 20 mg/kg) and premixtures (Cu 1500 mg/kg), 20.3% and 10.6%, respectively Table 1.

Table 1. Results of uncertainty evaluation for some feed additives in compound feeds and premixtures

Additive	Kind of feed	s_w (%)	bias (%)	u (%)	U = 2 ·u (%)
Vitamin A	Feedingstuff	4.0	12.4	13.1	26.2
	Premixture	2.0	7.2	7.5	15.0
Vitamin E	Feedingstuff	2.0	9.0	9.1	18.2
	Premixture	1.0	6.1	6.4	12.8
Copper	Feedingstuff	3.5	4.6	5.8	11.6
	Premixture	2.7	4.6	5.3	10.6
Lysine	Feedingstuff	3.1	3.6	4.7	9.2
Methionine	Feedingstuff	5.3	5.4	7.6	15.2
Threonine	Feedingstuff	2.7	5.8	6.4	12.8

Table 2. Comparation of obtained uncertainties with calculated from Horwitz's equation and Wood's criteria

Additive	Kind of feed	Mean value	Obtained U = 2 ·u (%)	From Horwitz (%) *	From Wood (%) [4]
Vitamin A	Feedingstuffs	23300 IU/kg	26.2	23.8	20.4
	Premixtures	2533000 IU/kg	15.0	11.8	20.0
Vitamin E	Feedingstuffs	94 mg/kg	18.2	16.1	20.6
	Premixtures	7.0 g/kg	12.8	8.2	20.0
Copper	Feedingstuffs	20 mg/kg	11.6	20.3	23.6
	Premixtures	1.51 g/kg	10.6	10.6	20.0
Lysine	Feedingstuffs	11.0 g/kg	9.2	7.9	20.0
Methionine	Feedingstuffs	4.5 g/kg	15.2	9.0	20.0
Threonine	Feedingstuffs	6.1 g/kg	12.8	8.6	20.0

* Expanded uncertainty for the Horrat value H=1 calculated from the Horwitz' formula $RSD_R = 2 \cdot C^{-0,15}$; U (%) = 2 · RSD_R (accepted Horrat value 0.5 < H < 2)

CONCLUSIONS

Obtained expanded uncertainties may be applied to test result interpretation and in assessment of compliance with rules concerning feed additives, especially permissible tolerances recommended in the Commission Regulation (EU) No 939/2010.

References

1. Eurolab Technical Report 1/2007. Measurement uncertainty revisited: Alternative approaches to uncertainty evaluation, Eurolab, Paris, 2007
2. Handbook for Calculation of Mesurement Uncertainty in Environmental Laboratories Nordtest TR 537, Version 3, 2008
3. Wood R. Method performance and the criteria approach. Problems with prescribing methods of analysis. 12th Meeting CEN/TC 327, 14-15 May 2009, Brussels, Belgium.