Preparation of an infant formula proficiency testing material and assessment of its homogeneity and stability

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Introduction

The European Union Reference Laboratory for Chemical Elements in Food of Animal Origin (EURL-CEFAO) is a PT Provider accredited according to ISO/IEC 17043:2010 and the organization of exercises, devoted to outstanding laboratories of the EU Member States (National Reference Laboratories), is one of its most important and appreciated activities. A lot of exercises have been proposed taking into account matrices, analytes, Maximum Levels (MLs) ruled in Commission Regulation (EC) 1881/2006 and discussions about new or modified MLs. Following this approach an exercise was organized in 2012 on the determination of cadmium (Cd) and lead (Pb) in powdered infant formula based on animal proteins (17th PT).

Production of PT Infant Formula Material

The starting material was a commercial product whose negligible content of Cd and Pb accounted for the necessity of a suitable spiking. The standard solution for spiking was prepared taking into account the lyophilization yield and the planned concentration in the final material. A preliminary test batch had been produced in order to check the preparation procedure that was then used for PT samples production.

Stability and Homogeneity assessment

Homogeneity (Table 1) and stability (Table 2) were evaluated using a standard deviation for proficiency assessment based on an EURL algorithm (σ_{pEURL}) and more restrictive than the Horwitz equation. The procedure of evaluation was based on ISO 13528:2005 and the International Harmonized Protocol.

SELECTION OF STARTING MATERIAL
- Analysis for basal content of Cd and Pb
- Choice of the powder needing the minimum quantity of water for dissolution (shortening of the lyophilisation process)

PREPARATION OF TEST BATCH
- Dissolution of the selected powder with standard solution of Cd and Pb (step of spiking)
- Homogeneisation and lyophilisation of the liquid sample
- Mixing and quartering of the freeze-dried material

PRODUCTION OF PT MATERIAL
- Dissolution and spiking of around 3.0 kg of the selected IF
- Homogeneisation and lyophilisation
- Mixing and quartering of freeze-dried material
- Bottling and labelling of the final material (production of 181 bottles)

Table 1. Parameters from homogeneity check of PT material: grand mean and σ_{pEURL} are in µg/kg

	Cd	Pb
Grand Mean	7.3	30.6
σ_{pEURL}	1.15	5.65
S^2_{an}	0.0096	1.1
S^2_{sam}	0.0036	0.0
S_{an}/σ_{pEURL}	0.085	0.186
c	0.22	6.1
$S^2_{sam} < c$	YES	YES

Note: S^2_{an}= analytical variance; S^2_{sam}= sampling variance that is assumed equal to zero if the estimate from the equation is negative; c= critical value for the test

Table 2. Stability assessment on the test batch (analytes concentration about the same as in PT material**)**

	$\|y_{t0}-y_{t1}\|$ (µg/kg)	$\|y_{t0}-y_{t2}\|$ (µg/kg)	$0{,}3*\sigma_{pEURL}$ (µg/kg)	Criterion: $\|y_{t0}-y_{tn}\| \leq 0{,}3*\sigma_p$
Cd	0.3	0.4	0.4	**MET**
Pb	0.8	0.1	1.4	**MET**

Note: y_{t0}= average concentration at time of preparation; y_{t1}= average concentration after 3 months and a half; y_{t2}= average concentration after 15 months

Conclusion

The material prepared for the 17th EURL-CEFAO PT was found homogeneous and stable over a period of time much longer than the duration of the exercise. These results are outstanding taking into account the low concentration values proposed and the restrictive standard deviation for proficiency assessment used to perform the tests. Furthermore, the PT was particularly useful considering that Commission Regulation (EC) 1881/2006 has been recently amended (CR (EU) 488/2014) with the introduction of the new ML for cadmium in infant formulae (based on cow's milk) that is consistent with the concentration value proposed in the 17th PT.

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