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Design and use of quality control samples in a collaborative study of trace metals in daily diets

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Planung und Verwendung von Qualitätskontrollproben bei einem Ringversuch für Spurenmetalle in der täglichen Nahrung

Summary. A series of six simulated diets consisting of mixtures of a limited number of foodstuffs have been produced, primarily to be used as external quality control samples in the ongoing WHO/UNEP HEAL-project. Lead and cadmium in the simulated diets have been determined by several reference laboratories. Zinc, copper, iron, manganese, chromium, nickel, calcium, aluminium, cobalt and arsenic have so far been analysed by at least two different laboratories. Examples of reference values and of the practical use of the diets in analytical quality assurance are given.

Background

One part of the Human Exposure Assessment Location, "HEAL", project [2] is concerned with the analysis of total diets for lead and cadmium. The subjects (in Sweden 10–15 women) collect duplicate diets during one week, divided into seven 24-h sampling periods.

During the initial discussions of the diet part of the HEAL project, which currently involves five countries, one of the questions raised was how the quality of the lead and cadmium analyses carried out in different countries could be ensured so that the comparison of the dietary intake of these metals in different countries would be valid. The analysis of both external (EQC) and internal (IQC) quality control samples was considered vital (EQC = concentrations of metals unknown to the laboratories; IQC = concentrations of metals known to the laboratories).

The available Certified Reference Materials (CRM), from e.g. the U.S. National Bureau of Standards, were considered to be either in an inappropriate matrix, or contained lead and cadmium at unsuitable levels. In addition, the use of only one or two different reference materials would probably not reveal any systematic analytical errors.

To overcome these problems we decided to produce a series of composite reference diets with metal levels covering the anticipated range. These quality control diets were to be used in sets of three or four at a time. The analytical performance evaluation was based on the regression line of reported (Y) versus reference values (X). We adopted the evaluation criteria used for lead and cadmium in blood in a previous UNEP/WHO programme [1]. The acceptance/rejection is based on the Maximum Allowable Deviation (MAD) of the empiric regression line from the ideal line $Y = X$.

The MAD criteria for both metals, with all results in mg/kg dry weight, were tentatively set as $Y = X \pm (0.1X + 25)$, where 0.1 is a relative error (of 10%) and 25 is two times the estimated error of the method. For further details regarding the statistical background to the MAD-lines see ref. [1]. In addition to the analysis of lead and cadmium, a number of other metals have been determined in the diets.

Results from training

When used for training the laboratories participating in the HEAL project, both random and systematic errors were detected. Generally, performance was improved between EQC Rounds 1 and 2. The results from two laboratories are shown as examples in Fig. 1.

Composition of diets

Ingredients

It was decided to use ingredients with a natural content of the metals, instead of spiking the diets to different levels. The following ingredients were therefore selected:

Table 1. The results of Pb and Cd analyses from a number of reference laboratories

Reference laboratory	Analytical method	Levels of metals in µg/kg	
		Pb	Cd
National Food Administration, Sweden	AAS	89	212
Swedish Environmental Institute, Sweden	AAS	135	270
National Institute of Environmental Medicine, Sweden	AAS	96	217
Technical Research Centre, Finland	AAS	110	190
Kernforschungsanlage Jülich, FRG	DPASV	102	200
Ministry of Agriculture, Fisheries and Food, UK	ICP-MS	170	197
	Mean =	117	214
	Standard deviation =	30	29

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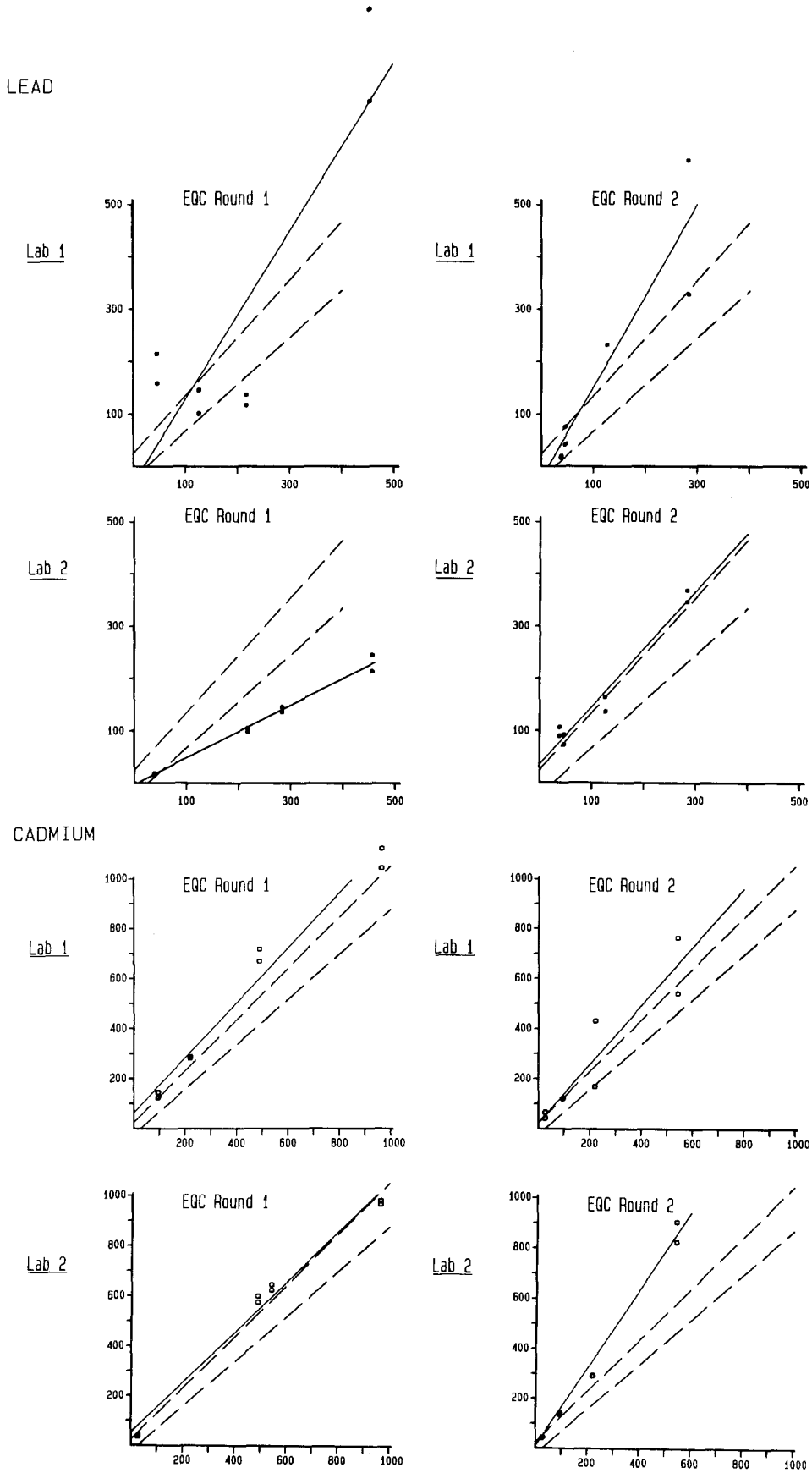

Fig. 1. Examples of results from the use of the diets as EQC-samples. Maximum Allowable Deviation is indicated by the *broken lines*

Lean beef, skimmed milk powder: Both products are low in lead and cadmium and provide a difficult matrix.

Potatoes, rye and wheat flour: Give a somewhat higher lead and cadmium level.

Swine kidney: Increases the cadmium level.

Reindeer liver: Is naturally high in both lead and cadmium and provides a complex matrix.

White wine: Increases the lead level without colouring the diet.

The diets were produced as follows:

Mix ingredients → Homogenize (meat-mincer) → Freeze-dry → Grind (hammer mill) → Homogenize → Pack in 30 ml HD polyethylene bottles.

Composition

The target was to produce six diets from these ingredients. The composition of the diets was as shown in the scheme below.

Diet	Parts of ingredients			
	1	2	3	4
A	Rye-wheat-flour	Potatoes	Skimmed milk powder	Lean beef
B	Rye-wheat-flour	Swine kidney	Skimmed milk powder	Lean beef
C	Reindeer liver	Skimmed milk powder	Lean beef	Rye-wheat-flour
D	Skimmed milk powder	Reindeer liver	Rye-wheat-flour	Potatoes
E	White wine	Potatoes	Reindeer liver	Rye-wheat-flour
F	Potatoes	White wine	Rye-wheat-flour	Reindeer liver

Each diet resulted in 250–300 bottles containing 9–11 g. All bottles were then randomly numbered to disguise their individual identity.

Metal content of diets

Homogeneity

The homogeneity of the diets was checked by analysing at least five samples of each diet at two sample weights, 2.0 and 0.4 g. The results indicate that the homogeneity is good with the exception of Cr and Ni, which is probably due to contamination from the milling equipment.

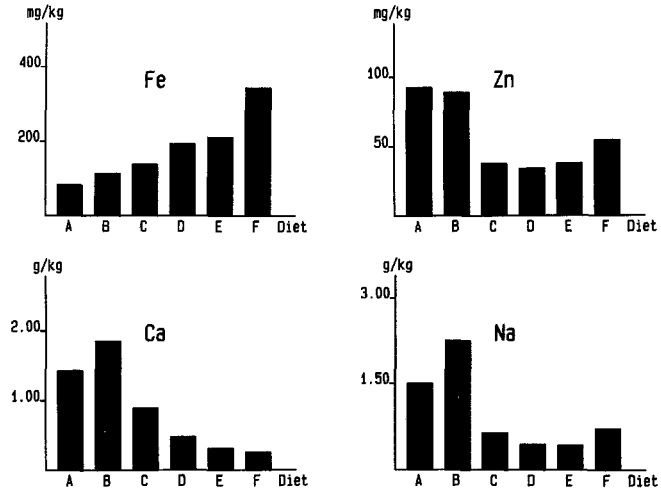

Fig. 2. Levels of Fe, Zn, Ca and Na in the diets, preliminary results

Reference values

In order to establish reference values, the various samples were sent for analysis to a number of well qualified laboratories. Currently 14 metals have been determined by at least two different laboratories:

Pb(6) Cd(5) Cr(4) Ni(4) Ca(3) Fe(5) Zn(4) Cu(4) Mn(3) Co(3) Al(2) K(2) Na(2) As(2).

The reference results received so far for lead and cadmium in *one* of the diets are shown in Table 1.

The preliminary results for Fe, Zn, Ca and Na in *all* the diets are shown in Fig. 2 as examples of the different gradients encountered.

References

1. Vahter M (ed) (1982) Assessment of human exposure to lead and cadmium through biological monitoring, GEMS. Prepared for UNEP/WHO
2. Vahter M, Friberg L, Lind B (1988) Fresenius Z Anal Chem 332:726–731

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