Repeatability of the Analysis of Selenium in Selenium-Enriched Cereal Grain-Based Supplements

C.R. Stockdale, M.L. Jenkin, J.W. Heard and P.T. Doyle

Dept of Primary Industries, Future Farming Systems Research, Kyabram Centre, Kyabram Vic. 3620

Elevated selenium (Se) concentrations in milk from dairy cows can be obtained by supplementing diets with organic Se yeast (Heard *et al*. 2007), and dairy companies are increasing their interest in developing speciality Se products. To meet market specifications for Se in milk products, farmers need to offer their cows feeds with known measured concentrations of Se. Recent research provided an opportunity to test the repeatability of the analysis of Se concentrations in Se-enriched pelleted cereal grain-based concentrates.

During 2004-05, two 6-week experiments were undertaken by Heard *et al*. (2007) where samples of Se-enriched pellets (with Se yeast at nominal amounts from 0-16 mg Se/kg DM) were taken each week to assess Se concentration. In 2006, the original samples were again sub-sampled and analysed for Se concentration, and at the same time, additional duplicate sub-samples were taken from half the original samples. The same analytical technique was used throughout, and all analyses were done at DPI's Environmental Health and Chemistry laboratory at Werribee. Total Se concentrations were determined after digesting sub-samples in a mixture of nitric and perchloric acid by using test tubes fitted with reflux funnels in an electrically heated digestion block. In the end stage of the digestion process, hydrochloric acid was added to the tubes to reduce selenium(V) to selenium(III), with the final matrix being 30% hydrochloric acid. The digestates were diluted (to 20 mL final volume) prior to analysis by inductively coupled plasma mass spectrometry/vapour generation. Duplicate samples were included at 3 to 4/100 samples, and reproducibility was at least 90%. Both standard reference material and 'in-house'-prepared laboratory control samples were used in all analyses. The standard reference materials were NIST standard 1549 (non-fat milk powder) and standard 1577b (bovine liver). Laboratory control samples were ovine kidney, chicken meat and non-fat milk powder. In each run, 2 samples were spiked, with acceptable recoveries being 80-120%, and 3 blanks were also included. Results of the assays were analysed by regression analysis of repeated measures of samples against each other.

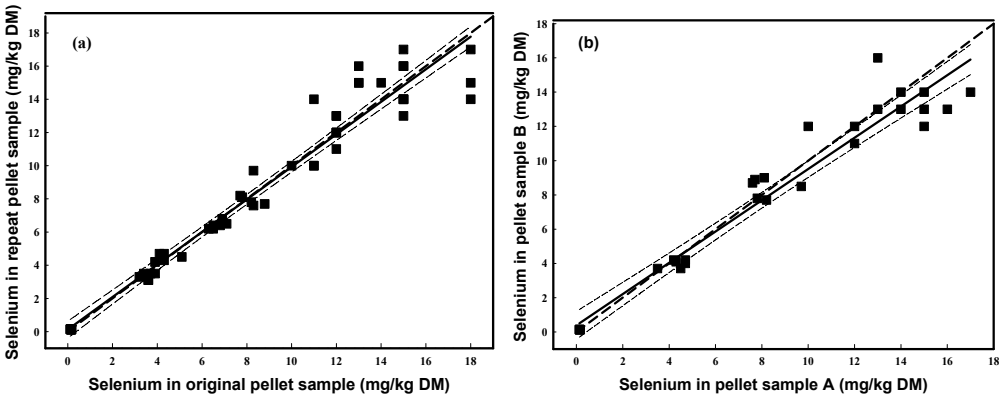

Figure 1. Correlations between a) selenium (Se) concentrations measured in Se-enriched pellets at the time of feeding and those measured in similar samples 1-2 years later (r = 0.98; CV = 14.9%; n = 60), and b) Se concentrations in duplicate samples of Se-enriched pellets (r = 0.97; CV = 16.1%; n = 30). The full lines are the curves of best fit, and the thin broken lines represent the 95% confidence intervals about this curve. The thicker broken line extending to each right-hand top corner is the 1:1 line.

In both assessments, the results across the range of Se concentrations showed that while correlations were good (Figure 1), coefficients of variation were moderately high (15-16%) due to increasing variability as Se concentrations increased. It is suggested that much of this variability was associated with the analytical technique rather than due to mixing or sampling issues since samples were ground and thoroughly mixed before sampling. It is concluded that more sub-samples should be analysed for Se to get a better measure of Se intake as increasing quantities of Se yeast are included in supplements fed to dairy cows. Currently in excess of 50 mg Se/cow have been fed daily in an on-farm pilot to produce Se-enriched milk (PT Doyle, unpublished results).

Heard, J.W., Stockdale, C.R., Walker, G.P., Leddin, C.M., Dunshea, F.R., McIntosh, G.H., Shields, P.M., McKenna, A., Young, G.P. and Doyle, P.T. (2007). *J. Dairy Sci.* **90:** 4117.

Email: richard.stockdale@dpi.vic.gov.au