Accuracy in niacin quantification in food matrices, by an RP-HPLC method

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Aim

This work intended to evaluate accuracy of niacin quantification in food matrices, by a RP-HPLC method, in order to validate it for four different food groups: legumes, grains, dairy products and fish.

Accuracy evaluation

Precision was evaluated by testing five matrices in triplicate in four different days. Repeatability and intermediate precision standard deviations (S_r and S_{Pi}) were calculated, for each matrix, trough ANOVA, $p < 0,05$. Repeatability and intermediate precision limits, *r* and *Pi* were also calculated.

Trueness was assessed from the z-scores obtained in four *Breakfast Cereals* FAPAS samples and one NIST *Adult/Infant formula* reference material, tested in duplicate.

Method

Samples – 1 to 10 g
Extraction – 0.1 M HCl, 121°C, 30 min
Stationary phase – Phenomenex Fortis C18 5µ (150 x 4.6 mm).
Mobile phase – 0.07 mol/l phosphate buffer, 0.075 mol/l hydrogen peroxide and $5x10^{-6}$ mol/l copper sulphate, 1 mL/min, isocratic
Injection volume – Standards (50 µl), samples (12.5 to 100 µl)
Derivatization – Post-column, with UV irradiation
Detection – Fluorescence; excitation λ=322 nm; emission λ=380 nm
Quantification – External standard method; five points
Equipment – Waters 2695 HPLC separation module and 2475 Multi-wavelength fluorescent detector

Results

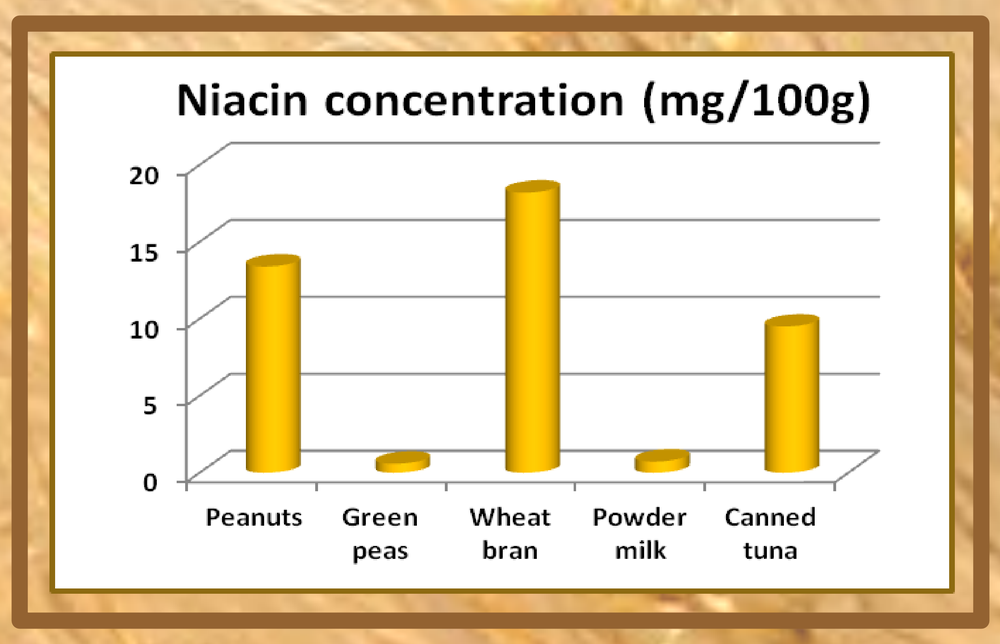

Fig. 1 – Average niacin concentration

Table 1 - Precision data (mg/100 g)

	Repeatability			Intermediate Precision	
	N = 4 (days) x 3 (replicate)				
	S_r	*r*	$\bar{x}$	S_{Pi}	*Pi*
Peanuts	0.52	1.46	13.4	1.25	3.4
Green peas	0.02	0.66	0.59	0.05	1.66
Wheat bran	0.23	0.05	18.2	0.78	0.14
Powder milk	0.02	0.64	0.73	0.6	2.17
Canned tuna	0.24	0.05	9.5	0.6	0.16

$\bar{x}$ - Average niacin concentration, *Sr* - Repeatability standard deviation, *r* - Repeatability limit, S_{pi} - Intermediate precision standard deviation, *Pi* - Intermediate precision limit.

Fig. 2 – Trueness study results

Discussion

The repeatability data obtained in this study, for niacin in different food matrices were in accordance with the ones presented in the method standard EN 15652:2009.

Trueness evaluation using breakfast cereals and formulas revealed a good method/laboratory performance. Accuracy evaluation, using representative matrices and covering usual niacin contents, showed the method suitability to its purpose and to be submitted to accreditation by an external entity.

References

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