A new approach to the method approval system in *Codex Alimentarius*: perspectives of near infrared spectroscopy for official methods

Mária Váradi,[a*] Ilona Boros,[b] Sándor Turza[a] and Judit Budai[c]

[a]*Central Food Research Institute, PO Box 393 H-1537 Budapest, Hungary.*

[b]*Research Institute of Hungarian Sugar Industry, H-1084 Budapest, Tolnai L.-u 25, Hungary.*

[c]*National Institute of Measures, H-1124 Budapest, Németvölgyi út 37-39, Hungary.*

The *Codex Alimentariu*s Commission is the international body responsible for the execution of joint FAO/WHO Food Standard Programme. Created in 1962 by FAO and WHO, the Programme is aimed at protecting the health of consumers and facilitating international trade in foods. Now it has more than 160 member countries and observers from international associations including food industry, trade and consumers as well. The *Codex Alimentariu*s is a collection of international food standards, guidelines and other recommended measures. The *Codex Alimentariu*s includes provisions in respect to the hygienic and nutritional quality of food, including microbiological norms, provisions for food additives, pesticide residues, contaminants, labelling, presentations and methods of analyses and sampling. The work is carried out in eight General Subject Committees (Methods of Analysis and Sampling, Food Labelling, Food Hygiene, etc.) 17 Commodity Committees (Milk and Milk Products, Fish and Fishery Products, Fats and Oils, etc.) and five Regional Coordinating Committees.[1]

The present procedure for the adoption of methods of analysis within the Codex System requires the Codex Committee of Methods of Analysis and Sampling (CCMAS) to consider and endorse methods of analysis proposed by Commodity Committees in the elaboration of their Codex Standards. In addition, CCMAS may propose methods of analysis of general applicability (for example, for trace elements). According to the present situation, the following four types of methods are distinguished in Codex: Defining Methods, Reference Methods, Alternative Approved Methods, and Tentative Methods.[2] Among these methods rapid methods can not usually be found due to the lack of method performance data calculated from the results of inter-laboratory (collaborative) studies, reliability and proper sensitivity.

It was stated recently that there were a number of criticisms to be made of this codex procedure:

- the analyst is denied freedom of choice and thus may be required to use an inappropriate method in some situations
- the procedure inhibits the use of automation,
- it is administratively difficult to change a method found to be unsatisfactory or inferior to another currently available

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This was the reason that in its last session the CCMAS has accepted in principle an alternative approach whereby a defined set of criteria to which methods should comply without specifically endorsing specific methods should be adopted. These are the following:

- accuracy
- applicability (matrix, concentration range and preference given to "general" methods)
- detection limit
- determination limit

precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories), but generated from collaborative trial data rather than measurement uncertainty considerations

- recovery
- selectivity
- sensitivity
- specificity (interference effects etc.)

In this way, a new possibility is being opened to introduce rapid methods such as near infrared (NIR) spectroscopy for Codex purposes.

Table 1. Moisture in dried vegetables. Comparison of methods: calculated from data of *Rader,* (1967) [*Journal of the AOAC* 50(3)].

Product	Method	*m* %	*r*	*R*	s_R*c.s.*	RSD_R*c.s.*	RSD_R*H*	HO_R
Carrot	VO	4.80	0.16	0.22	0.079	1.64	3.16	0.52
	KF	4.83	0.23	0.43	0.154	3.18	3.16	1.01
	NIR	5.06	0.20	0.34	0.121	2.40	3.13	0.77
Squash	VO	6.55	0.21	0.29	0.104	1.58	3.01	0.52
	KF	6.47	0.23	0.40	0.143	2.21	3.02	0.73
	NIR	6.78	0.18	0.50	0.179	2.63	3.00	0.88
Cabbage	VO	7.67	0.19	0.55	0.196	2.56	2.94	0.87
	KF	7.65	0.36	0.79	0.282	3.69	2.94	1.25
	NIR	7.88	0.23	0.87	0.311	3.94	2.93	1.34
Onion	VO	4.37	0.12	0.73	0.261	5.97	3.20	1.86
	KF	4.58	0.28	0.35	0.125	2.73	3.18	0.86
	NIR	4.59	0.13	0.55	0.196	4.28	3.18	1.35

m: mean value
r: repeatability
R: reproducibility
s_R *c.s.*: reproducibility variance from collaborative study
RSD_R *c.s.:* reproducibility relative standard deviation from collaborative study
RSD_R*H:* reproducibility relative standard deviation predicted from Horwitz equation
HO_R: Horwitz ratio = RSD_R *c.s.* / RSD_R*H*
VO: vacuum oven method
KF: Karl Fischer method
Horwitz equation: $RSD\ H = 2^{(1-0.5*\log C)}$

It means that NIRS should be evaluated according to the criteria system. In some fields there are promising results. For example, in the case of moisture, crude protein analysis for feed and forages NIR method is accepted by AOAC-International. To select the methods of analysis Codex prefers the official methods of analysis elaborated by international organizations occupying themselves with a food or group of foods. Furthermore the method selected should be chosen on the bases of practicability and preference should be given to methods, which have applicability for routine use. In addition, these AOAC methods have been collaboratively tested, and their method performance parameters are published.[3,4]

As Codex deals only with food, we looked for examples of food testing methods to show what method performance parameters can be calculated from inter-laboratory studies.

Our first example is from an early NIR publication.[5] We used the data of Rader on moisture in dried vegetables measured with NIR and two classical methods (vacuum oven and Karl Fischer methods). Mean, repeatability and reproducibility values were calculated according to ISO 5725-2, and Horrat (Horwitz ratio) values for all sample types and methods.[6] These results are summarized in Table 1.

As it can be seen from the results, Horrat values for NIR method are less than 2 that is satisfactory for in all sample types and all the three methods. In case of two sample types Horrat value for the NIR method is even lower than Horrat value for one of the classical methods. It means that in these cases the differences between the results of different laboratories were smaller with NIR method than with the classical method.

In our second example, which is shown in Table 2, three parameters of wheat (protein, moisture and wet gluten) were measured in 11–14 laboratories with NIR and classical laboratory methods. The data were originated from the National Institute of Measures, Hungary. This Institute has been organizing proficiency testing for wheat testing laboratories in Hungary for several years. For demonstra-

Table 2. Wheat components. Comparison of methods: caclulated from data of the National Institute of Measures.

Component	Method	*m* %	*r*	*R*	s_R *c.s.*	SD_R *c.*	RSD_RH	HO_R
Protein	Lab	10.56	0.18	0.39	0.139	1.32	2.81	0.47
	NIR	10.72	0.39	1.47	0.525	4.90	2.80	1.75
Moisture	Lab	12.35	0.08	0.72	0.257	2.08	2.74	0.76
	NIR	12.10	0.20	1.13	0.404	3.34	2.75	1.21
Wet gluten	Lab	24.28	0.55	1.20	0.429	1.77	2.47	0.71
	NIR	23.97	0.35	2.52	0.900	3.75	2.48	1.51

m: mean value
r: repeatability
R: reproducibility
s_R *c.s.*: reproducibility variance from collaborative study
RSD_R *c.s.:* reproducibility relative standard deviation from collaborative study
RSD_RH*:* reproducibility relative standard deviation predicted from Horwitz equation
HO_R: Horwitz ratio = RSD_R *c.s.* / RSD_RH
Horwitz equation: $RSD\ H = 2^{(1-0.5*\log C)}$

tion purposes a part of the data measured in a proficiency testing this year were processed as the results of an inter-laboratory method performance study.

The results show that Horrat values are also under two, that is satisfactory for NIR methods for all the three components, but the classical laboratory methods gave always lower between laboratory differences. The cause of the poorer performance of the NIR methods can be that the types of NIR instruments used and the calibrations were different in the participating laboratories.

In conclusion it can be stated that the method performance data calculated from the results of inter-laboratory studies proved to be satisfactory. We are planning to widen the inter-laboratory study in Hungary and in collaboration with other countries. By this way we can help that the NIR technique be accepted as an official method in the Codex system. The new method approval system in the Codex also supports the application of NIR in the field of food regulation and harmonization.

References

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