RETROSPECTIVE ON PROFICIENCY TESTS FOR NUTRIENT COMPONENTS IN MEAT PRODUCTS

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Abstract – For some nutrients (protein, total fat, carbohydrate, sugars, salt), and some cooked meat products (corned-beef, liver pate, ham pate), standard deviation for proficiency assessment were estimate from a retrospective of 1995–2014 proficiency tests. The results of proficiency tests are also compared to DGSANCO's tolerances for nutrition labeling.

Key Words – nutrition declaration, standard deviation for proficiency assessment, tolerances.

I. INTRODUCTION

In order to help the laboratories, Ifip (and before 2006 the CTSCCV – Centre Technique de la Salaison, de la Charcuterie et des Conserves de Viandes) provides proficiency tests (PTs) for chemical determinations in meat products. Ifip is the manager of these comparisons, from the choice of sample to the statistical treatment of the datas. A total of 75 to 92 have participated during the last 20 years, mostly came from France, the other from Europe. They received two or three canned samples tested for their homogeneity and stability, twice a year. The statistical analysis was performed, according to ISO 13528 [1].
One goal of this paper was the estimation of standard deviation for proficiency assessment.

The mandatory nutrition declaration is include in regulation (EU) n°1169/2011 (or INCO regulation) [2] and will be applied in Europe until December 14th 2016 on the majority of pre-packed meat products. The European Commissions Directorate General for Health and Consumer Protection (DGSANCO) provided a guidance document [3] which gave tolerances for nutrient values declared on a label. The other goal was to see the application of these tolerances for meat products.

II. MATERIALS AND METHODS

Ifip used to employ canned corned-beef from 1995 to 2007, liver pate from 1995 to 2014 and ham pate from 2007 to 2014. Corned-beef was homogenized in our laboratory, liver pate and ham pate were made by an industrial producer. For most laboratories, these types of matrix were regular and anyway, they didn't require a cautious manipulation. According to ISO 17043 [4], homogeneity and stability of the samples were kept to assess laboratories performance, they were estimated as described in ISO 13528 [1].
Each laboratory can perform all or only a part of the measures, two results of each component are required. Laboratory could use the method of its choice, it gives the reference for standard methods or the principle for other methods.

All the results were quantitative, if some answers were qualitative they had been removed (sugars and carbohydrate). For each round the statistical analysis was performed, for the present paper, according to ISO 13528 [1]. In this procedure, robust statistics were used to minimize the influence of the outliers instead of other methods for their detection and rejection; thus, the impact of extreme values on the average and the standard deviation was down weighted. But results which presented a manifest error like scale error, are rejected first.

Calculations were done with 9.4 SAS software version (SAS Institute, USA).

III. RESULTS AND DISCUSSION

The number of laboratories depends on nutrient component, and on the year of the PT (cf. table 1). Participants were all laboratories that routinely perform analyses with meat products. They are commercial, or public or of food industries laboratories.

Table 1 number of laboratories

Nutrient component	Number of laboratories Minimum	Maximum
Protein	67	92
Total fat	37	57
Total carbohydrate	28	39
Sugars	40	76
“Salt” = 2.5x sodium	22	40

Most employed methods were:
- Protein: Kjeldahl method (NFV 04-407 or ISO 937 standards),
- Total fat: extraction after boiling of a test portion with hydrochloric acid to free the occluded and bound lipid fractions (NFV 04-402, ISO 1443 standards). These two standard have not been revised since 1968 and 1973 respectively, laboratories used some adaptations.
- Carbohydrate: the Bertrand’s method [5] or by difference (100-(ashes + fat + protein + humidity)).
- Sugars: Bertrand’s method [5], it’s used more for technological survey than nutrient component, it’s not a standard, but it was often used for meat products.
- Sodium: incineration and determination by atomic absorption spectrometry [6] or ionic chromatography. The first method has not been revised since 1977, the second one is not yet a standard in Europe in the scope of meat products. Sodium is expressed in salt (equivalent content calculated using the formula: salt = sodium × 2.5).

The standard deviation for proficiency assessment is not dependent of the time or the level for the studied components and matrices. Illustrations are shown for fat in liver pate and ham pate (cf. figures 1 and 2).

Figure 1. Fat standard deviation for proficiency assessment in liver pate and ham pate for different levels.

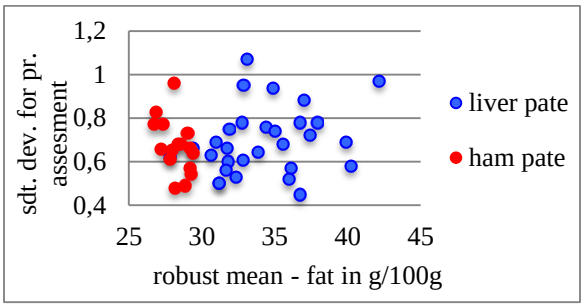
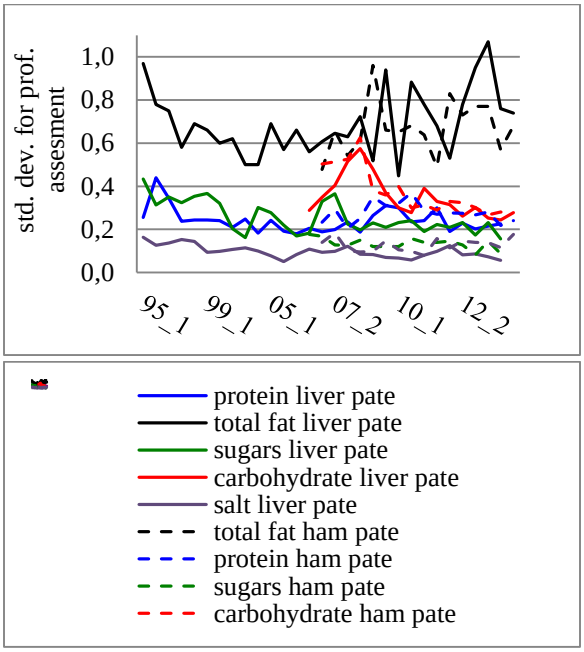


Figure 2. Liver pate and ham pate standards deviation for proficiency assessment, 1995-2014



The mean of standard deviations for proficiency assessment could be estimated with results from the studied period (cf. table 2).

Table 2 Standard deviations for proficiency assessment

Nutrient component	Liver pate 95-2014*	Ham pate 2007-2014	Corned-beef 95-2007
Protein	0.24	0.28	0.42
Total fat	0.69	0.67	0.39
Carbohydrate	0.35	0.37	/
Sugars	0.26	0.13	0.14
Salt = 2.5xsodium	0.10	0.13	0.12

*carbohydrate 2007-2014

From the nutrition declaration of INCO regulation [2] results of protein, fat, salt, carbohydrate and sugars are reported in figures 3 to 7, for liver pate between 1995 and 2014. Robust means and robust standard deviations [1] were represented, they were chosen as the labeling nutritional values. Then tolerances were calculated from the DGSANCO's guidelines [3] (cf. table 3) and included in the figures.

Table 3 tolerances including measurement uncertainty (adapted from [3])

Nutrient component	Tolerances (includes uncertainty of measurement)
Protein, Carbohydrate, Sugars	<10 g per 100 g: ±2 g 10-40 g per 100 g: ±20%
Fat	10-40 g per 100 g: ±20% >40 g per 100 g: ±8 g
Salt (= 2,5x sodium)	<1.25 g per 100 g: ±0.375 g ≥1.25 g per 100 g: ±20%

Figure 3. Liver pate evaluation of protein, 1995-2014

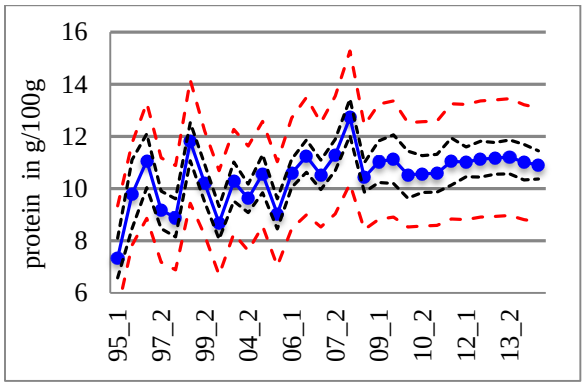


Figure 4. Liver pate evaluation of fat, 1995-2014

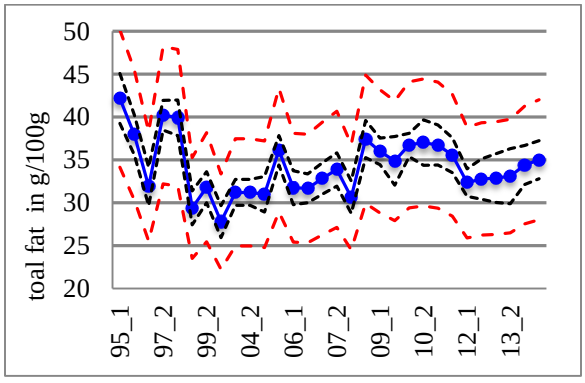


Figure 5. Liver pate evaluation of total carbohydrate, 2007-2014

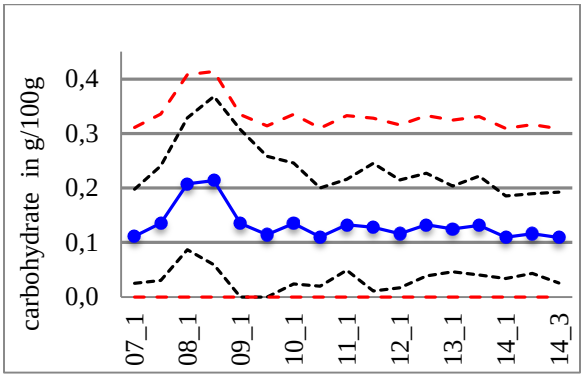


Figure 6. Liver pate evaluation of sugars, 1995-2014

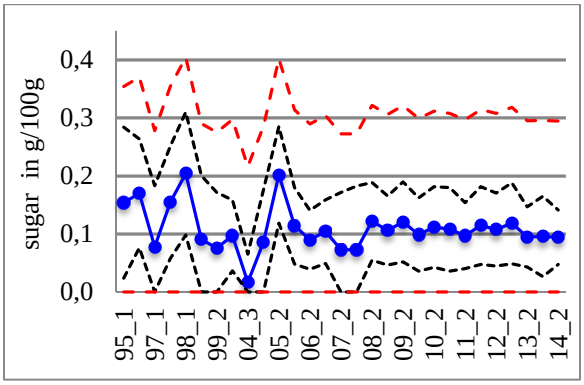
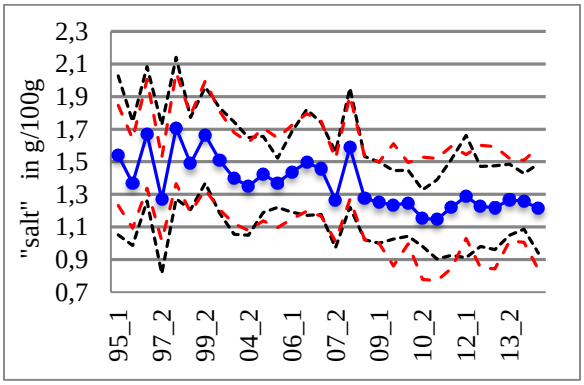


Figure 7. Liver pate evaluation of salt equivalent content calculated using the formula: salt = sodium × 2.5, 1995-2014



The legends for figure 3 to 7:
robust mean
robust mean -3x(robust standard deviation) and robust mean + 3x(robust standard deviation)

- - - lower and upper tolerance for nutrition declaration from DGSANCO's guidelines [3]

In this study, as we worked in each round on single lot, tested for its homogeneity, no variation of production were included to determine the nutrient values. However for protein, fat, carbohydrate and sugars the tolerances would be respected. It would be more difficult for salt calculated from sodium, maybe in relation with the uncertainty of this method, uncertainty of 10% was usual, and the lack of standard method for meat products with added salt.

IV. CONCLUSION

Standard deviations for proficiency assessment had been establish from the results of the PT's 1995-2014, they are now determined for next rounds.
Works are needed to improve measurement of sodium in meat products.

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