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Validation of standard method EN ISO 19343 for the detection and quantification of histamine in fish and fishery products using high-performance liquid chromatography

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ABSTRACT

In contaminated fish, bacterial decarboxylases produce histamine from histidine, thereby causing scombroid fish poisoning. European Regulation (EC) No 2073/2005 on microbiological criteria for foodstuffs requires using a fully validated, standardized reference HPLC method for detecting and quantifying histamine. After optimizing this reference method for the quantification of histamine in fish muscle, we organized an inter-laboratory study in 2013 across nine laboratories from seven European countries using defined criteria of method performance. The optimized, validated method was standardized (Standard EN ISO19343) as part of Mandate M381 from the European Commission to the European Committee for Standardization (CEN), signed in December 2010. The standard method was validated for three types of foodstuffs (fish with enzymatic maturation, fish without enzymatic maturation and fish sauce).

1. Introduction

Histamine and biogenic amines occur in different types of food, including fish, meat, chocolate, coffee fruit, vegetables and beverages (Papageorgiou *et al.*, 2017). Histamine derives from the decarboxylation of histidine and can be produced in significant quantities by microbiological histidine decarboxylase in some fish species that naturally contain histidine, mainly in *Scombridae*, *Engraulidae* and *Clupeidae* (Guillier *et al.*, 2011). The poisoning associated with histamine in fish is called scombroid fish poisoning. Depending on the type of product and storage conditions, a wide variety of bacteria can produce histidine decarboxylase, such as *Enterobacteriaceae*, *Clostridium*, *Lactobacillus*, *Vibrio*, *Pseudomonas* and *Photobacterium*.

Various methods have been investigated to characterize and quantify histamine in seafood: rapid methods with commercial enzyme-linked immunoassay (ELISA) kits (Köse *et al.*, 2011) and chromatographic methods. In addition to gas chromatography (Huang *et al.*, 2016), the methods described in the literature to quantify histamine accurately in fish are based on liquid chromatography, with some variants: pre-column derivatization (Duflos *et al.*, 1999; Zotou and Notou, 2013), post-column derivatization (Veciana-Nogues *et al.*, 1994), ionic chromatography (Antolini *et al.*, 1999) and ultra-high pressure liquid chromatography (Mayer *et al.*, 2010). Furthermore, different types of sample preparation (Saaid *et al.*, 2009; Wu *et al.*, 2015) or detection methods (UV light, fluorimetry, tandem mass spectrometry) can be used (AOAC, 1990; Nalazek-Rudnicka and Wasik, 2017).

European Regulation (EC) No 2073/2005 (Anonymous, 2005a) amended by Regulation (EC) No 1441/2007 on microbiological criteria for foodstuffs set specific criteria for histamine in fish. The sampling plan requires nine samples from a batch (n), the mean histamine concentration of these nine samples must be less than 100 $mg{\cdot}kg^{-1}$ (m), two samples (c) are allowed to have a concentration between 100 and 200 $mg{\cdot}kg^{-1}$ but none can exceed 200 $mg{\cdot}kg^{-1}$ (M). The values of m and M are multiplied by a factor of 2 for fishery products that have undergone an enzyme maturation treatment in brine. European Regulation (EC) No 1019/2013 (Anonymous, 2013a) having amended Regulation 2073/2005 gives the possibility of taking only a single sample from a retail point, with a maximum allowable histamine concentration (M) of less than 200 $mg{\cdot}kg^{-1}$. In this amendment, fish sauces produced by fermentation of fishery products were added (n = 1, m = M = 400 $mg{\cdot}kg^{-1}$). The reference method associated with these criteria is an HPLC quantification method (Duflos *et al.*, 1999).

The first clinical signs of histamine poisoning are similar to those of

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an allergic reaction: facial flushing, rash, facial oedema, burning in the throat and a taste of pepper in the mouth, itching and tingling of the skin (Hungerford, 2010).

Working Group 6 'Microbiology of the food chain' of Technical Committee 275 'Food analysis-Horizontal methods' of the European Committee for Standardization (CEN/TC275/WG6), in charge of standardization in the field of the microbiology of the food chain at the European level, has received a mandate from the European Commission (EC) (Mandate M381 between the EC and CEN, signed in December 2010) to validate inter-laboratory studies and standardize a set of 15 reference methods in food chain microbiology. These methods are cited or are expected to be cited as reference methods in European Regulation (EC) No 2073/2005 (Anonymous, 2005a) on microbiological criteria for foodstuffs.

The aim of this article is to present the optimization and validation of the method currently cited in Regulation (EC) No 2073/2005 for histamine quantification. This validated method is now specified in the recently published Standard EN ISO 19343 (2017), prepared by the Technical Advisory Group for histamine (TAG 11) of CEN/TC 275/WG 6.

2. Materials and methods

2.1. Design of the trial

Nine laboratories from seven European countries participated in the inter-laboratory study organized in 2013 in accordance with ISO 5725-2 (1994). Canned fish used for this inter-laboratory was prepared by the ANSES Laboratory for Food Safety, Boulogne-sur-mer, France. Three fish species (tuna, mackerel and herring), were used for the inter-laboratory ring trial. For each matrix, three levels of contamination were prepared in duplicate. Thus, in total, 18 samples were sent to each laboratory. Homogeneity and stability of samples were evaluated according to an internal procedure based on ISO 13528 (2005) and the IUPAC Procedure (Anonymous, 2006). The test materials were shipped to the participants by post at ambient temperature. Samples were randomly coded to identify test materials and to avoid collusion between participants. Instructions and deadlines for analysing test materials were defined in an accompanying letter.

2.2. Preparation of test materials

A total of 7.8 kg of fish flesh was required to produce all the 60 g samples of canned fish for this study, i.e. 2.6 kg of flesh of each species (tuna, mackerel and herring). Before preparing test material, each matrix was analysed by the organizing laboratory (the ANSES laboratory) using the HPLC method to ensure that samples were free of histamine. For spiking, a histamine solution (1333 mL) was prepared in the laboratory from dihydrochloride histamine (Sigma Aldrich, Saint-Quentin-Fallavier, France) and bidistilled water were added to 4 kg of flesh (2.6 kg necessary for the ring trial). The formula used to determine the mass of histamine necessary to prepare this solution is the following:

$$(final\ concentration / histamine\ purity) \times 100 \times (weight_{sample} + weight_{water}).$$

The three contamination levels, 25, 100 and 220 $mg \cdot kg^{-1}$, were prepared respectively as follows: 220.97 g, 883.87 g and 1944.52 g of histamine in 1333 mL of double-distilled water. These test materials were distributed in 60 g cans, which were then crimped and sterilized according to the following process: 121 °C during 7–8 min and 116 °C/60 min.

2.3. HPLC method

2.3.1. Sample preparation

Samples (fish flesh) were homogenized by grinding in a mixer. A portion of 5 g (± 0.1 g) of the fish homogenate was transferred to a centrifuge tube.

2.3.2. Extraction

For extraction, 10 mL of perchloric acid and 100 µL of 1,7-diaminoheptane (6.4 $mg \cdot mL^{-1}$) were added to 5 g of fish in the centrifuge tube and mixed with an Ultra-Turrax homogenizer. After complete homogenization, the tube was centrifuged at 8000 × *g* for 5 min at 4 °C.

2.3.3. Derivatization

For derivatization, 100 µL of supernatant was transferred in a tube, 300 µL of sodium carbonate solution and 400 µL of dansyl chloride solution were added. The tube was vortexed and incubated for 5 min in the dark at 60 °C. The tube was cooled under cold tap water and 100 µL of L-proline solution was added. The tube was vortexed and placed in the dark for 15 min.

2.3.4. Purification

For purification, 500 µL of toluene were added to the tube and vortexed. Histamine is contained in the organic phase composed of toluene. This phase can easily be recovered by freezing the aqueous phase (< −18 °C for 30 min minimum) or by transferring as much as possible of the upper organic phase into a new tube. The organic phase containing histamine dansyl derivates was evaporated under nitrogen flow for 5 min. The dry residue was re-suspended in 200 µL of a solution containing 60% acetonitrile and 40% water (v/v), vortexed and then filtered at 0.2 µm.

2.3.5. Standard samples

To avoid matrix effects and bias, the calibration curve was performed on the same matrix (histamine-free) as the analysed sample. Standard samples were prepared by adding a histamine solution composed of 1.1168 g of histamine dihydrochloride in 100 mL of bidistilled water (Sigma Aldrich, Saint-Quentin-Fallavier France). This addition of histamine was performed after the grinding step in a homogenized sample of fish (tuna, mackerel or herring according to the analysed matrix to avoid matrix effect) (Anonymous, 2013b). The calibration curve was carried out at five different concentration levels (0, 25, 50, 100 and 250 $mg \cdot kg^{-1}$ of histamine).

2.3.6. HPLC settings

Histamine and internal standards were separated on a Kromasil C18 reverse phase column (250 mm × 4.6 mm × 5 µm) from AIT (Houilles, France) with a water/acetonitrile gradient and a flow of 1 $mL \cdot min^{-1}$. After 30 min of separation, the chromatogram showed the peaks of histamine and the internal standard (1,7-diaminoheptane). Quantification of histamine was performed by calculating each response factor against 1,7-diaminoheptane and using the calibration curve.

2.4. Homogeneity and stability of the test materials

The homogeneity and stability of the test materials were tested according to the usual internal procedure of our laboratory, based on ISO 13528 (2005) and the IUPAC protocol. Ten samples for homogeneity and three for stability were randomly collected and analysed in duplicate. These analyses were carried out for each matrix (tuna, herring and mackerel) and for each concentration level (25, 100 and 220 $mg \cdot kg^{-1}$), 90 cans in total for homogeneity and 27 for stability (analyses performed within the defined deadline).

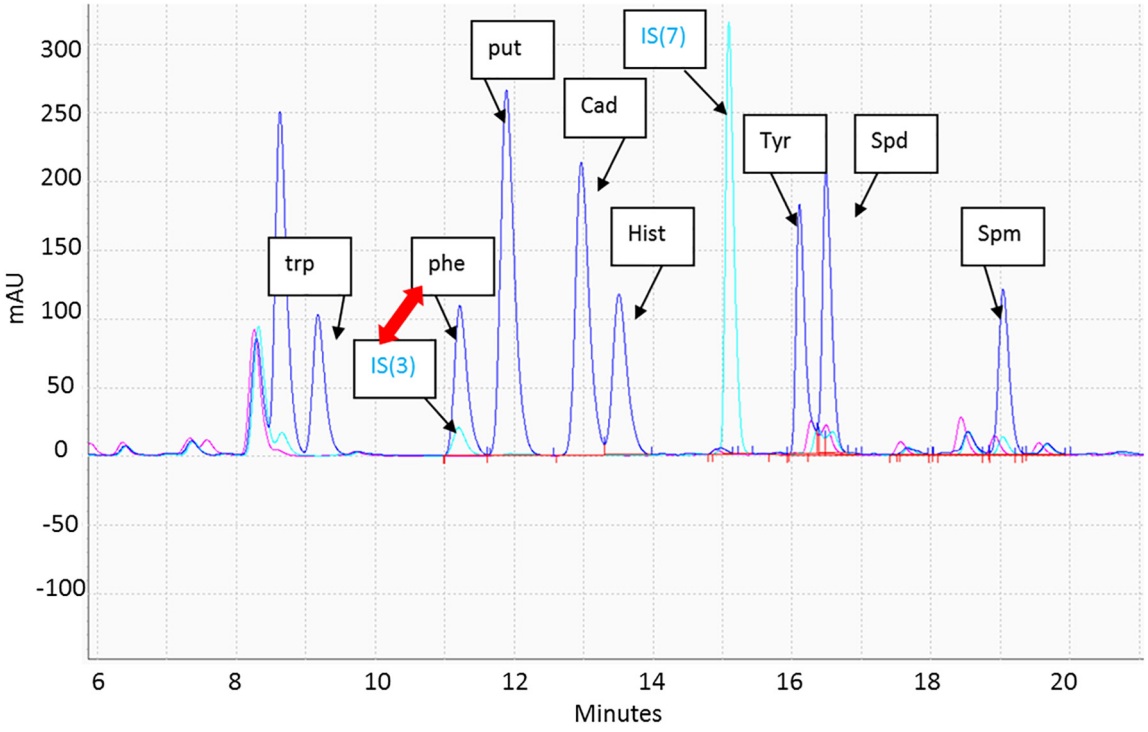

Fig. 1. Chromatograms of tuna samples with biogenic amines (trp = tryptophan; phe = phenylethylamine; put = putrescine; cad = cadaverine; hist = histamine; tyr = tyramine; spd = spermidine; spm = spermine) and internal standards (IS3 = 1,3-diaminopropane; IS7 = 1,7-diaminoheptane).

2.5. Statistical analysis of the data

Data from the participating laboratories were analysed according to the ISO 5725-2 (1994) standard. No results were excluded due to participant deviations from the stipulated instructions.

Performance of the method was assessed in accordance with ISO 5725-2 (1994) using Excel spreadsheets developed in-house according to this standard and also using the software *e-noval V3.0b PROD* by *Arlenda* © (Mont-St-Guibert, Belgium).

Performance assessment made it possible to calculate data on method precision: the mean value ($mg{\cdot}kg^{-1}$), the standard deviation of repeatability s_r ($mg{\cdot}kg^{-1}$), the limit of repeatability r ($mg{\cdot}kg^{-1}$ and %), the standard deviation of reproducibility s_R ($mg{\cdot}kg^{-1}$), the limit of reproducibility R ($mg{\cdot}kg^{-1}$ and %) and the HorRat ratio. The HorRat ratio is determined as follows: R%/PR% where R is the reproducibility from the ring trial and PR is the predicted reproducibility obtained from the Horwitz equation. The Horwitz equation used is $PR\% = 2C^{-0.1505}$, where C is the theoretical concentration. If the HorRat ratio is close to 1, the method precision (in terms of reproducibility) is close to the predicted value.

The repeatability limit r is defined as the absolute difference between two independent single test results obtained using the same method on identical test material in the same laboratory by the same operator using the same apparatus within the shortest feasible time interval. The r value should not be exceeded in more than 5% of the cases. If the difference between replicate results within a laboratory exceeds r, the results should be considered suspect. The reproducibility limit R is defined as the absolute difference between two single test results of the two test results on the normal scale obtained using the same method on identical test material in different laboratories with different operators using different apparatuses. The R value should not be exceeded in more than 5% of cases. If the difference between results in different laboratories exceeds R, the results should be considered suspect.

The coefficient of variation of repeatability CVr (%) is defined as the ratio between the standard deviation of repeatability and the average value of repeatability. The coefficient of variation of reproducibility CV_R (%) is defined as the ratio between the standard deviation of reproducibility and the average value of reproducibility.

3. Results and discussion

3.1. HPLC method optimization

The TAG 11 task force of CEN/TC 275/WG 6 agreed to adapt the method referenced in European Regulation (EC) No 2073/2005 (Duflos *et al.*, 1999). First, the internal standard was changed. The initial method called for 1,3-diaminopropane hydrochloride at a very low concentration with a peak size too small regarding the size of the histamine peak at the regulatory limit of $100\ mg{\cdot}kg^{-1}$. We also demonstrated possible coelution with phenylethylamine, not present in fish but in another matrices (Sengupta and Mohanakumar, 2010; Toro-Funes *et al.*, 2015). Thus, 1,7-diaminoheptane was tested and gave satisfactory results at a sufficiently low concentration ($6.4\ mg{\cdot}ml^{-1}$) without coelution (Fig. 1). The task force thus decided in 2013 to use 1,7-diaminoheptane as the internal standard. The second optimization involved the purification step by using 200 µL of a solution of 60% acetonitrile and 40% water (v/v) to resuspend the dry residue.

3.2. Stability and homogeneity of the test materials

Homogeneity was satisfactory for the herring matrix at all three concentration levels and for mackerel at the lowest two levels. For the other test material (tuna), we used the inter-sample standard deviation for the stability test and participant results.

The analysis period between sample dispatch and stability test was 14 days. All test materials were deemed stable.

3.3. General results of the inter-laboratory trial

A total of nine laboratories from seven European countries participated in the inter-laboratory study. All participants used the same

Table 1
Performance data for tuna matrices in the ring trial.

Theoretical level	Tuna		
	25 mg/kg	100 mg/kg	220 mg/kg
Number of data	36	36	36
Mean value (mg/kg)	24,01	93,10	200,30
Recovery (%)	96,03	93,10	91,05
Repeatability standard deviation s_r (mg/kg)	4,37	5,69	6,42
Coefficient of variation of repeatability CV_r (%)	18,2	6,11	3,20
Repeatability limit ($r = 2{,}8 \times s_r$) (mg/kg)	12,24	15,93	17,98
Repeatability limit ($r = 2{,}8 \times CV_r$) (%)	50,96	17,11	8,97
Reproducibility S_R (mg/kg)	7,74	12,15	19,37
Coefficient of variation of reproducibility CV_R (%)	32,23	13,05	9,67
Reproducibility limit ($R = 2{,}8 \times s_R$) (mg/kg)	21,67	34,02	54,24
Reproducibility limit ($R = 2{,}8 \times CV_R$) (%)	90,26	36,54	27,07
HorRat value according to Horwitz	3,27	1,63	1,36

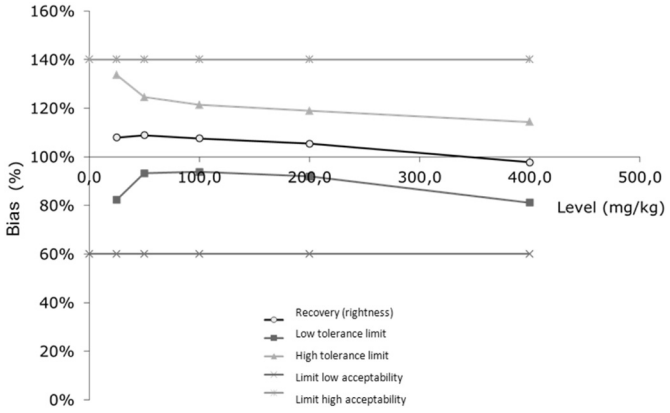

Fig. 2. Accuracy profile defined on tuna.

method with the same reagents (histamine and 1,7-diaminoheptane supplied by us). No data were excluded from statistical analysis.

Regarding tuna, the percentage of recovery for all three tested concentration levels was around 95%. The values for s_r were 4.37 for level 1, 5.69 for level 2 and 6.42 for level 3. In addition, CV_r was 3.20%, CV_R 9.67%.and HorRat value was 1.36 for 220 mg·kg^{-1} (Table 1). These data are in accordance with the accuracy profile (Fig. 2).

For herring, the s_r values were 6.71 for level 1, 1.38 for level 2 and 5.58 for the level 3. Moreover, CV_r was 6.22, CV_R 9.74% and HorRat value was 1.37 for 220 mg·kg^{-1} (Table 2).

For mackerel, the s_r values were 2.69 for level 1, 2.66 for level 2 and 8.79 for the level 3. The CV_r was 5.99%, CV_R 14.82% and HorRat value was 2.09 for 220 mg·kg^{-1} (Table 3).

Table 2
Performance data for herring matrices in the ring trial.

Theoretical level	Herring		
	25 mg/kg	100 mg/kg	220 mg/kg
Number of data	36	36	36
Mean value (mg/kg)	11,07	26,87	89,65
Recovery (%)	44,29	26,87	40,75
Repeatability standard deviation s_r (mg/kg)	6,71	1,38	5,58
Coefficient of variation of repeatability CV_r (%)	61,61	5,14	6,22
Repeatability limit ($r = 2{,}8 \times s_r$) (mg/kg)	18,79	3,86	15,62
Repeatability limit ($r = 2{,}8 \times CV_r$) (%)	169,72	14,38	17,43
Reproducibility S_R (mg/kg)	7,04	3,92	8,73
Coefficient of variation of reproducibility CV_R (%)	63,59	14,59	9,74
Reproducibility limit ($R = 2{,}8 \times s_R$) (mg/kg)	19,71	10,98	24,44
Reproducibility limit ($R = 2{,}8 \times CV_R$) (%)	178,07	40,85	27,27
HorRat value according to Horwitz	6,45	1,82	1,37

Table 3
Performance data for mackerel matrices in the ring trial.

Theoretical level	Mackerel		
	25 mg/kg	100 mg/kg	220 mg/kg
Number of data	36	36	36
Mean value (mg/kg)	18,35	70,11	146,80
Recovery (%)	73,39	70,11	66,73
Repeatability standard deviation s_r (mg/kg)	2,69	2,66	8,79
Coefficient of variation of repeatability CV_r (%)	14,66	3,79	5,99
Repeatability limit ($r = 2{,}8 \times s_r$) (mg/kg)	7,53	7,45	24,61
Repeatability limit ($r = 2{,}8 \times CV_r$) (%)	41,05	10,62	16,77
Reproducibility S_R (mg/kg)	3,87	8,99	21,75
Coefficient of variation of reproducibility CV_R (%)	21,09	12,82	14,82
Reproducibility limit ($R = 2{,}8 \times s_R$) (mg/kg)	10,84	25,17	60,90
Reproducibility limit ($R = 2{,}8 \times CV_R$) (%)	59,05	35,90	41,48
HorRat value according to Horwitz	2,14	1,60	2,09

For the herring and mackerel matrices, we observed a low percentage of recovery: respectively around 36% and 70%. This recovery rate for herring corresponds to 44, 27 and 40% recovery for 25, 100 and 220 mg·kg^{-1} histamine. For mackerel, the percentages of recovery were 73, 70, and 66% for 25, 100 and 220 mg·kg^{-1}. These values were relatively constant for each species regardless of the histamine concentration. These results clearly indicate a matrix effect on histamine quantification due to use of calibration curve involved in tuna. Matrix effects are well-known in foodstuff characterization and have been described in a previous study on histamine in fish (Stroka *et al.*, 2014). We confirmed these matrix effects in an additional study that showed better recovery rates (78 to 97%) for herring in different sample preparation conditions (raw, cooked, canned) with calibration curve performed with herring matrix (Table 4).

We therefore recommend using the same matrix (without histamine) for the calibration curve and the sample to ovoid the influence of the matrix for the recovery.

The recovery rates for tuna were slightly better than those reported for other quantification methods, i.e. rapid ELISA it (77 to 110%) (Hungerford and Wu, 2012) or other HPLC methods (80 to 83%) (Muscarella *et al.*, 2013). Nordic committee on food analysis described a method (196–2013) close of this method with similar results for collaborative study. This study showed coefficient of variation of repeatability CVr (%) of 2.3% and coefficient of variation of reproducibility CV_R (%) of 6.1 for a recalculated mean of histamine (171.7 mg/kg). In our study, coefficient of variation of repeatability CVr (%) of 3.2% and coefficient of variation of reproducibility CV_R (%) of 9.67 for a no recalculated mean of histamine (220 mg/kg). Therefore, AOAC Official Method 977.13 present a recovery of 97 to 117% for 50,15 mg/kg and a coefficient of variation of repeatability CVr (%) of 4.15% and coefficient of variation of reproducibility CV_R (%) of 8.87 for a recalculated mean of histamine (158.4 mg/kg).

Table 4
Recovery of histamine from various herring preparations.

Herring		
	Histamine (mg·kg^{-1})	Recovery (%)
+ hist 250 mg·kg^{-1} + UT 2 min	243	97.2
+ hist 250 mg·kg^{-1} + UT 12 min	234	93.6
+ hist 250 mg·kg^{-1} + UT 12 min + Ster	197	78.8
+ hist 250 mg·kg^{-1} + Ster	196	78.4
Precooked herring + hist 250 mg/kg + Ster	202	80.8

Hist: histamine; UT: mixing by Ultra Turrax; Ster: sterilization.

Table 5
Performance data for the three tested matrices at 400 mg·kg^{-1} histamine.

	Tuna	Anchovy[a]	Fish sauce
Sample 1	396.32	414.28	423.19
Sample 2	387.52	365.17	397.62
Sample 3	401.88	421.81	402.08
Sample 4	381.78	378.73	398.44
Sample 5	422.09	434.21	394.99
Sample 6	401.70	425.71	403.28
Mean value (mg/kg)	398.55	406.65	403.27
Recovery (%)	99.64	101.66	100.82
SD	14.02	27.97	16.92

[a] Which have undergone enzyme maturation treatment in brine.

3.4. Implementation on different matrices

This method has been implemented on three matrices (tuna, anchovy and fish sauce), corresponding to the different types of products covered by Regulation No 2073/2005 with a specific standard range. The contamination levels chosen correspond to the upper regulatory limit (400 mg·kg^{-1}) (Table 5). The recovery rate for the three matrices was around 100%. The characterization of performance was defined by an intra-laboratory study on tuna (Fig. 2).

4. Conclusions

The new EN ISO 19343 (2017) standard was validated by an inter-laboratory study and can be considered as a relevant method for the detection and quantification of histamine in fish and fishery products, according to the criteria set in European Regulation (EC) No 2073/2005. This method was adapted slightly from the method referenced in the regulation and can be easily implemented in a laboratory. Due to observed matrix effects, the Technical Advisory Group for histamine (TAG11) of CEN/TC275/WG6 recommend applying a specific standard curve to the matrix analysed.

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