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## Full Length Article

## 4-Methoxy-*ortho*-phthalaldehyde: a promising derivatizing agent for the fluorimetric evaluation of histamine in seafood

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## ABSTRACT

Histamine, the molecule associated with food poisoning, is nowadays considered as a reliable marker of seafood freshness and therefore its rapid and easy quantification appears critical. Existing method like HPLC or direct fluorimetric analysis employing *ortho*-phthalaldehyde (OPA) can be time-consuming and tedious. To overcome this problem, we propose in this paper to evaluate the application potential of 4-methoxy-*ortho*-phthalaldehyde (MO-OPA) for the rapid and easy evaluation of histamine in seafood samples. For this purpose, the histamine complexation conditions were studied by molecular fluorescence spectroscopy using MO-OPA as derivatization agent and then compared with the reference compounds: OPA. MO-OPA proved to be superior to OPA for histamine detection in terms of linearity range ( $1.10^{-6}$ – $1.10^{-4}$  mol.L<sup>-1</sup>,  $R^2 > 0.99$ ,  $n = 10$ ) and detection limit ( $9.5.10^{-7}$  mol.L<sup>-1</sup>). The optimal conditions were next applied to the analysis of histamine in a mixture of biogenic amines standards and finally on real samples of yellowfin tuna of known histamine concentration.

## Introduction

Food safety and quality are increasingly at the center of preoccupation for consumers as well as health agencies. Consequently, a permanent evaluation of the freshness and toxicity index of foods is required and involves in particular the characterization of spoilage markers such as biogenic amines (BAs). In food, these non-volatile nitrogenous organic bases result from the decarboxylation of their amino acids precursors generated by pathogenic microorganisms as well as by maturation and fermentation processes. [1,2] While BAs are essential components in the process aiming to regulate physiological functions in the human body, they can also pose a serious risk to the consumers health due to their relatively low toxic threshold. [3–5] Detection and quantification studies of BAs are extensively illustrated in the literature, particularly those addressing histamine (HA). Indeed, the presence of HA in seafood is regulated by health authorities like the European Commission [6] or the U.S. Food and Drug Administration (FDA) [7] since it is responsible for histamine intoxication. Currently, other BAs such as putrescine and cadaverine are also studied considering that they can potentiate the toxic effect of HA. [8] As a result of its toxicity and regulation, HA analysis techniques are widely documented, including colorimetric, [9–11] enzymatic [12,13], electrochemical [14–16] and chromatographic methods [17–25]. Fluorescence spectroscopy

analysis is also a commonly employed technique [26–28] especially the one developed by the AOAC which is currently approved by the FDA [7,29,30]. Since HA does not contain any fluorophore moiety, a derivatization procedure is crucial in order to allow an analysis by molecular fluorescence spectroscopy. Several derivatizing agents have been employed and the most commonly used ones are dansyl chloride, [31,32] fluorescamine, [33,34] 2,3-naphthalenedicarboxaldehyde [35,36] and *ortho*-phthalaldehyde (OPA). [29,37–39] Employed since 1959, [40] the OPA takes the lead over other reagents since it enables the detection of BA at low concentrations and without HPLC separation. The selectivity over other aminated compounds is achieved through the specific excitation and emission wavelengths of each BA/OPA complexes. [41] Nevertheless, OPA derivatization leads to poorly stable compounds. The addition of thiol reagents such as 2-mercaptoethanol, [42] 3-mercaptopropionic acid [42,43] or N-acetylcysteine [42,43] was proposed in order to create less unstable OPA/thiol/HA complexes, to extend the pH range required for the formation of compounds as well as to facilitate its applicability. With a similar aim, we propose here to study the behavior of HA fluorescent analysis of a new derivatization agent possessing a methoxy group directly linked to the aromatic core of the OPA: 4-methoxy-*ortho*-phthalaldehyde (MO-OPA) (Fig. 1). As far as literature is concerned, derivatives based on an OPA core have scarcely been used as derivatization agents and were never applied to BAs

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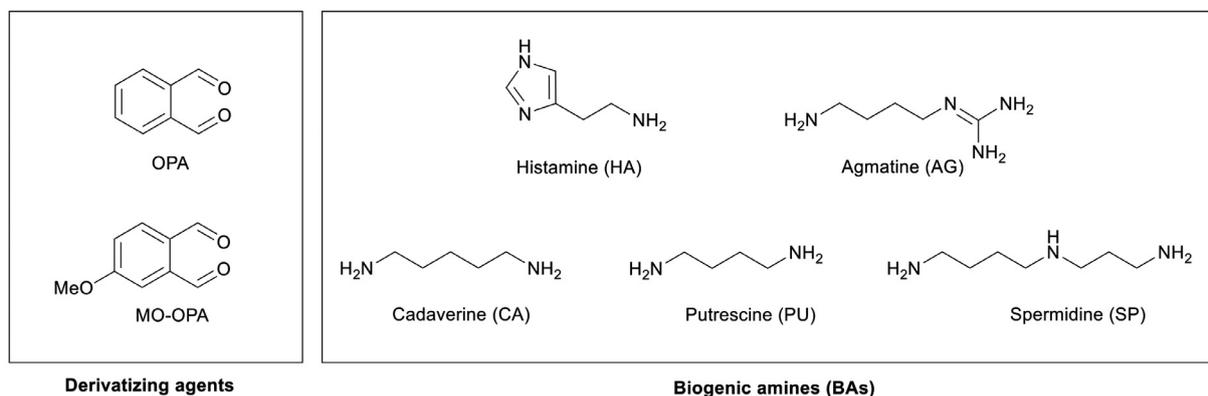


Fig. 1. The chemical structures of *ortho*-phthalaldehyde and 4-methoxy-*ortho*-phthalaldehyde (left) and biogenic amines (right) used in this study.

analysis. In a recent report, Zhang et al. [44,45] proposed a synthetic route to MO-OPA and 4,5-dimethoxy-*ortho*-phthalaldehyde to achieve a fluorescence probe for ammonium traces detection with the successful development of a hand-held portable fluorometer. They report that the addition of an electron-donating group allows to increase the electron density of the aromatic core leading to an enhancement of the fluorescence intensity. Moreover, it enables faster reaction with analytes at room temperature. [44] In this paper, we take advantage of our precedent expertise on MO-OPA synthesis [46] to evaluate its efficiency on HA fluorescent analysis. To fulfill this goal, the complexation conditions of HA with MO-OPA as well as the molecular fluorescent parameters were investigated and then properties of MO-OPA as derivatization agent were compared with standard OPA. These optimal conditions were next applied to the fluorimetric analysis of HA with MO-OPA in a mixture of BAs standards and finally on real yellowfin tuna (*Thunnus albacares*) samples.

## Experimental section

### Reagents

All the chemicals were purchased from Acros Organic or Sigma-Aldrich and were used as received without further purification unless specified. Biogenic amines were purchased as: Agmatine sulfate salt ( $\geq 97\%$ ), cadaverine dihydrochloride ( $\geq 99\%$ ), histamine dihydrochloride ( $\geq 99\%$ ), putrescine dihydrochloride ( $\geq 99\%$ ), spermidine trihydrochloride ( $\geq 99\%$ ), *ortho*-phthalaldehyde ( $\geq 99\%$ ); recrystallized from *n*-hexane). 4-methoxy-*ortho*-phthalaldehyde (MO-OPA) was synthesized according to the procedure of Moitessier et al. [46]

HPLC grade water was used throughout the study for the preparation of analytical solutions. The pH was adjusted using sodium hydroxide 1M or hydrochloric acid 1% aqueous solutions.

### Tuna samples

Two doped canned samples of yellowfin tuna (*Thunnus albacares*) were provided by the French Agency for Food, Environmental and occupational Health & Safety (ANSES) with known final concentration of HA at  $15.79 \pm 2.53$  mg/kg and  $148.64 \pm 7.99$  mg/kg respectively.

### Apparatus

UV-Vis measurements were performed at 20 °C on a Varian Cary 100 spectrophotometer. A 100-OS 10 mm pathlength optiglass cuvette was used. Fluorescence measurements were performed at 20 °C on a Varian Cary Eclipse spectrofluorimeter. A 100-QS 10 mm pathlength quartz fluorescence cuvette was used.

### Procedures

#### Solution preparation

Stock solutions of HA ( $1.10^{-2}$  mol.L $^{-1}$ ) and derivatization agent (OPA or MO-OPA;  $1.10^{-2}$  mol.L $^{-1}$ ) were prepared by dissolving the compounds in water. Serial dilutions were performed to obtain working standard solutions. All solutions were protected against light and stored at 4 °C prior to use unless specified. The shelf-life of the solutions must not exceed one week. [29] BAs and derivatization agent were mixed during the final dilutions prior to fluorimetric analysis. The solutions were degassed in an ultrasonic bath during their preparation and before the recording of molecular fluorescence spectra.

#### Analytical measurements

For the fluorimetric method, the fluorescence intensity was monitored at the fixed analytical excitation ( $\lambda_{ex}$ ) and emission ( $\lambda_{em}$ ) wavelengths of the complexes. Linear calibration curves were obtained at these  $\lambda_{ex}$  and  $\lambda_{em}$  values by measuring the spectra height signal. All fluorescence measurements were corrected for the solvent signal with the appropriate blank.

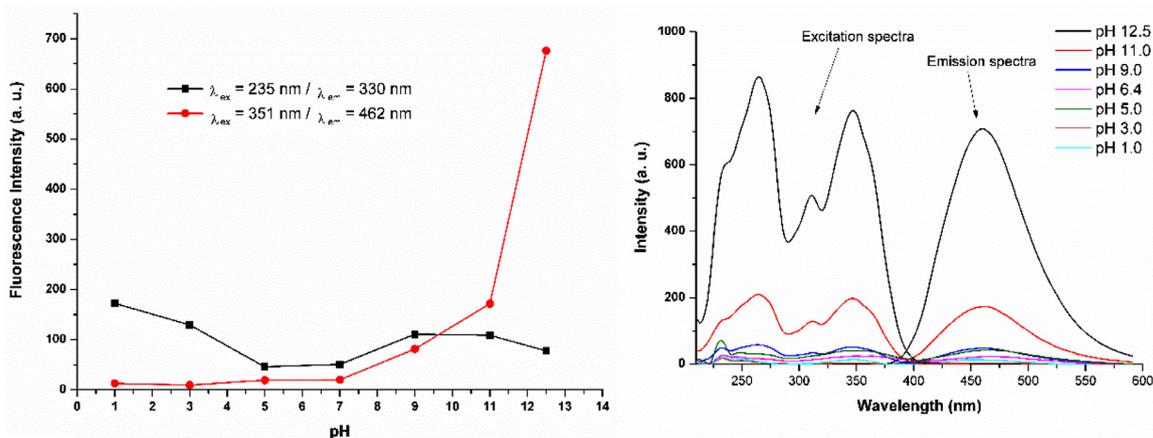
#### Fish samples preparation

Two tuna cans of known level of HA ( $15.79 \pm 2.53$  mg/kg and  $148.64 \pm 7.99$  mg/kg) were processed according to a modified procedure based on Bjornsdottir-Butter et al. [30] work. Briefly, methanol (75% v/v; 50 mL) was added to 10 g of tuna and the mixture was vigorously stirred during 30 min. The sample was then filtered over fritted glass, evaporated and put back in solution in HPLC grade water. The aqueous sample was transferred into a 50 mL volumetric flask and completed with water. 1 mL of the resulting solution was diluted with water into a 10 mL volumetric flask containing 1 mL of MO-OPA aqueous solution ( $1.10^{-3}$  mol.L $^{-1}$ ; finale concentration  $1.10^{-4}$  mol.L $^{-1}$ ). This solution was stored at 4 °C in the dark pending the analysis by molecular fluorescence spectroscopy.

## Results and discussion

### Determination of optimal complexation parameters

An iterative process was used to understand the experimental parameters (pH, stoichiometry, kinetics) that influences the formation of fluorescent complexes between HA and MO-OPA. Based on our experience and taking into account that Oyelakin et al. indicate that the of OPA/PU complexes fluorescence emission is optimal only after 24 h, [47] two conditions were evaluated through this study: the first one at  $t=15$  min at 20 °C and the second one after 24 h at 4 °C in the dark.



**Fig. 2.** Maximum emission intensity of the MO-OPA/HA complexes at 330 nm ( $\lambda_{\text{ex}} = 235$  nm) and at 462 nm ( $\lambda_{\text{ex}} = 351$  nm) as a function of pH (left). Excitation ( $\lambda_{\text{em}} = 462$  nm) and emission ( $\lambda_{\text{ex}} = 351$  nm) spectra of MO-OPA/HA complexes as a function of pH (right). Conditions: [MO-OPA] =  $4.10 \cdot 10^{-5}$  mol.L $^{-1}$ ; [HA] =  $8.10 \cdot 10^{-6}$  mol.L $^{-1}$ ; T = 20°C; t = 15 min

### pH influence

In order to achieve the maximum fluorescence intensity for the MO-OPA/HA complexes, it was essential to determine the optimal excitation wavelength as a function of pH. For OPA/HA complexes, the literature indicates that excitation and emission wavelengths differ according to pH and that a basic pH (greater than 11.5) allows a higher fluorescent emission intensity compared to an acidic medium. [41,48] In this study, the same findings were obtained for OPA complexes (see supplementary data Fig. S1). A UV-spectra of MO-OPA/HA complexes was recorded in water and present two maximal absorption peaks at 235 nm and 351 nm. Excitation of the complexes at 235 nm lead to a moderate fluorescence intensity at 330 nm while excitation at 351 nm lead to a strong fluorescence emission centered around 462 nm in basic medium.

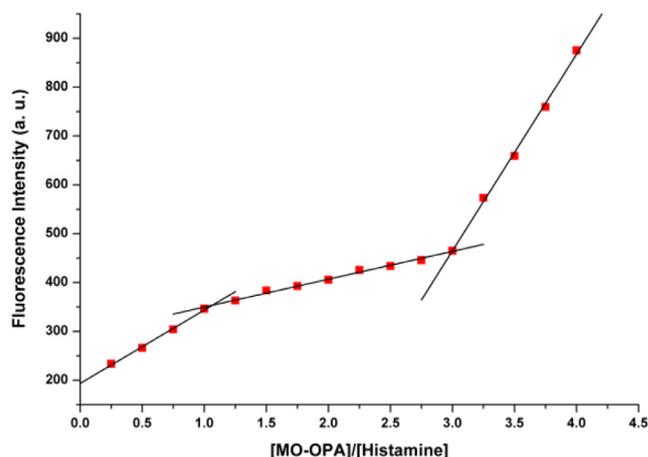
The study of the influence of the pH on the fluorescence intensity was carried out by modulating the pH of a solution containing MO-OPA and HA and recording the maximal intensity for each excitation/emission pair previously determined. Results are presented in Fig. 2 and clearly indicate that MO-OPA operate as his parent molecule OPA by displaying a maximal fluorescence intensity at basic pH. Therefore, a pH of 12.5 was fixed for the remainder of this study.

### Stoichiometry

To get a better insight on the possible stoichiometry of the complexes, the molecular fluorescence intensity of the MO-OPA/HA complexes were recorded at fixed HA concentration by adding incremental amount of MO-OPA. From those experiments, one can observe three linear domains (Fig. 3). The first one was characterized by a MO-OPA/HA ratio (R) less than or equal to 1 which was attributed to the predominant formation of a 1:1 complex (1 molecule of OPA with 1 molecule of HA). The second linear range with R comprised between 1 and 3 was attributed to the main formation of 2:1 complex while the last linear range with R above 3 was attributed to the formation of higher order complexes. It is important to mention here that, at the current state of knowledge, the exact structures of OPA/BAs complexes are still not known with certainty even if mechanisms were proposed in the 1980s for OPA/HA complexes [37,38].

### Kinetics

In our experience, OPA/HA complexes have proven to be unstable and their analysis required a long stabilization period (about 25 h at 4 °C) to reach maximum intensity (see supplementary data Fig. S2) as well as a stable fluorescence emission wavelength (see supplementary data Fig. S3). It therefore seemed interesting to study the kinetics of formation of the MO-OPA/HA complexes and to compare it with the



**Fig. 3.** Maximum fluorescence emission intensity ( $\lambda_{\text{ex}} = 351$  nm;  $\lambda_{\text{em}} = 462$  nm) as a function of MO-OPA/HA ratio. Conditions: [HA] =  $8.10 \cdot 10^{-6}$  mol.L $^{-1}$ ; T = 20°C; t = 15 min; pH = 12.5.

standard derivatization reagent OPA. The maximum fluorescence intensity and associated wavelength were measured when MO-OPA and HA were brought into contact (t = 1 min) and then every 15 min for 50 h (Fig. 4, some points were omitted for clarity). For this purpose, and for the entire duration of the analysis, the samples were either temperature-controlled at 20 °C or stored at 4 °C between each spectrum recording. Remarkably, the maximum emission wavelength did not evolve over time at 4 °C as is the case for the OPA (see supplementary data Fig. 3) and remained stable around 462 nm. Delightfully, fluorescence intensity obtained during 50 h at 4 °C were stable and identical to those obtained after 15 min at 20 °C (Fig. 4) which is not the case for OPA (see supplementary data Fig. S2 and Fig. S3). Fortunately, this stability allows us to avoid the timekeeping of manipulations and in doing so, to analyze larger numbers of samples compared to the 6-10 samples recommended by the AOAC, which constitutes a real improvement over known methods.

### Limits of detection (LOD), quantification (LOQ) and Linearity

To assess the applicability of MO-OPA for HA sensing, limits of detection and quantification as well as linearity range were evaluated. The LOD was assessed by evaluating the fluorescence response of a range of five solutions with concentrations from  $1.00 \cdot 10^{-7}$  mol.L $^{-1}$  to  $1.00 \cdot 10^{-6}$  mol.L $^{-1}$ . It was found that at  $9.5 \cdot 10^{-7}$  mol.L $^{-1}$ , the spectrum

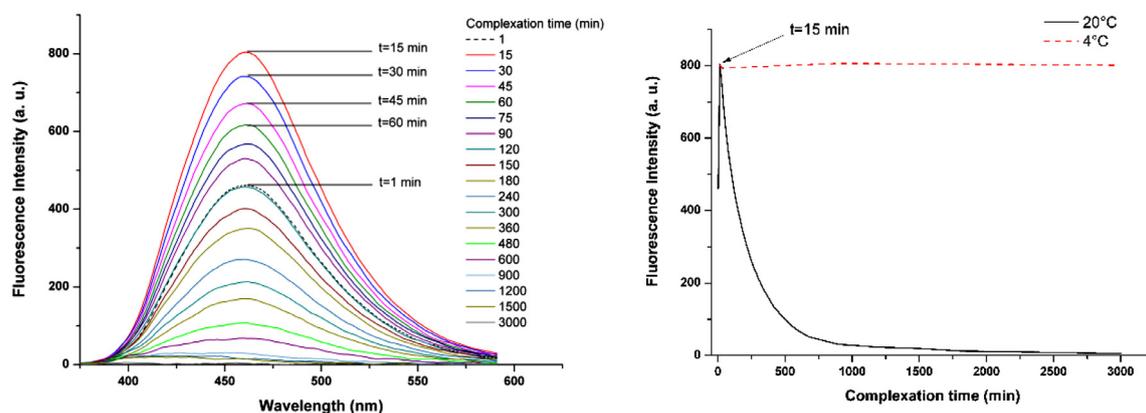


Fig. 4. Fluorescence emission spectra ( $\lambda_{\text{ex}} = 351\text{nm}$ ) of MO-OPA/HA complexes as a function of time at 20°C (left). Evolution of the maximum emission intensity of MO-OPA/HA complexes as a function of time at 4°C and 20°C (right). Conditions:  $[\text{MO-OPA}] = [\text{HA}] = 8.10^{-6} \text{ mol.L}^{-1}$ ;  $\text{pH} = 12.5$ .

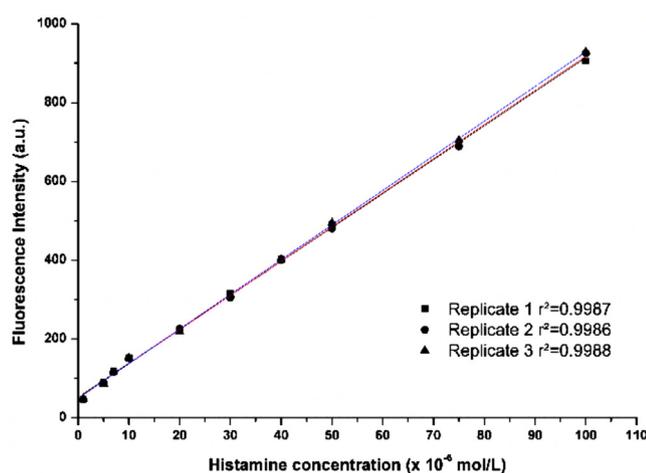


Fig. 5. Fluorescence emission intensity ( $\lambda_{\text{ex}} = 351 \text{ nm}$ ) of MO-OPA/HA complexes as a function of histamine concentration. Conditions:  $[\text{MO-OPA}] = 5 [\text{HA}]$ ;  $T = 20^\circ\text{C}$ ;  $t = 15 \text{ min}$ ;  $\text{pH} = 12.5$

presents some irregularities incompatible with quantification but was significantly above baseline. Agreeably, the molecular fluorescence intensity revealed to be linear between  $1.10^{-6}$  and  $1.10^{-4} \text{ mol.L}^{-1}$  (Fig. 5) with coefficients of determination greater than 0.99 ( $n=10$ ). Pleasingly, this range is significantly improved by about one order of magnitude compared to that of the OPA which, in our hands, has been found to be between  $5.10^{-6}$  and  $5.10^{-5} \text{ mol.L}^{-1}$  (see supplementary data Fig. S4).

#### Evaluation of other BAs interferences

HA is not the only BA produced during fish alteration and therefore evaluation of interferences from other BAs must be conducted. To this end, 4 common BAs were selected to challenge the method. Cadaverine (CA) and putrescine (PU) were selected since they are listed as potentiators of HA effects. [8] Agmatine (AG) and HA were already known to interfere during analyses with OPA [49] and spermidine (SP) was examined to assess the impact of a secondary aliphatic amine. The robustness of HA quantification was evaluated in harsh condition i.e. with BAs amounts similar or even higher to the HA amount and without purification on column to avoid extra handling. [30]

Interferences of BAs on the molecular fluorescence intensity of MO-OPA/HA complexes was firstly evaluated individually (data not shown) then collectively with the following conditions:  $\text{pH} = 12.5$ , complexation time = 15 min at 20 °C,  $\lambda_{\text{ex}} = 351 \text{ nm}$  and  $\lambda_{\text{em}} = 462 \text{ nm}$ . Advantageously CA, PU and SP does not induce any significant change on the molecular fluorescence intensity, alone or collectively, even at concentration two

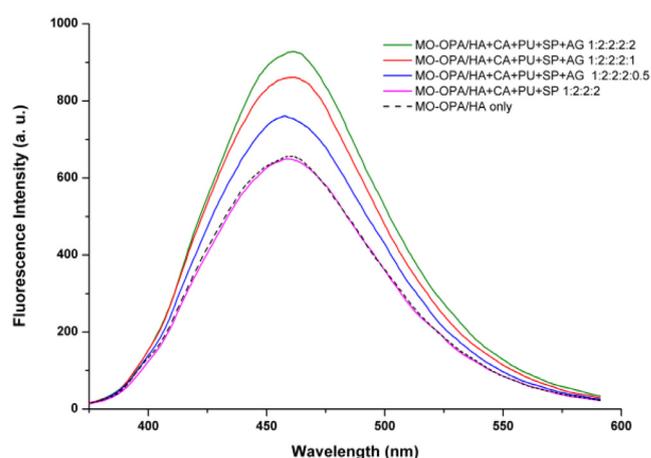


Fig. 6. Evaluation of interfering biogenic amines (CA, PU, SP and AG) on the fluorescence emission intensity of MO-OPA/HA complexes ( $\lambda_{\text{ex}} = 351\text{nm}$ ). Conditions:  $[\text{MO-OPA}] = 10^{-4} \text{ mol.L}^{-1}$ ;  $[\text{HA}] = 8.10^{-6} \text{ mol.L}^{-1}$ ;  $T = 20^\circ\text{C}$ ;  $t = 15 \text{ min}$ ;  $\text{pH} = 12.5$ .

times greater than HA (Fig. 6). It was attributed to an absence of fluorescence emission of MO-OPA/BAs complexes at an excitation wavelength of 351 nm. However, as for analysis with OPA, the presence of AG induces a noteworthy fluorescence enhancement up to 33% for HA and AG in equimolar proportions since MO-OPA/AG complexes proved to be slightly fluorescent at this excitation wavelength. This enhancement rises to 39 % when OPA is used in place of MO-OPA.

#### Histamine analysis in fish sample

To estimate the potential of MO-OPA for the analysis of real samples, HA analysis was performed in duplicate on two doped canned samples of yellowfin tuna (*Thunnus albacares*) provided by the French Agency for Food, Environmental and occupational Health & Safety (ANSES) with known final concentration of HA at  $15.79 \pm 2.53 \text{ mg/kg}$  and  $148.64 \pm 7.99 \text{ mg/kg}$  respectively. After opening the sealed can, the tuna meat is extracted with methanol according to a modified protocol described by Bjornsdottir-Butter *et al.* [30] then solutions were analyzed by molecular fluorescence spectroscopy in two replicate according to previously optimized parameters with both, MO-OPA (Fig. 7) and OPA (see supplementary data Fig. S5) as derivatizing agent. For real samples, fluorescence intensities were plotted against known concentration of HA.

In both cases fluorometric analysis lead to an overestimation of HA amount but, interestingly enough, this overestimation is less marked

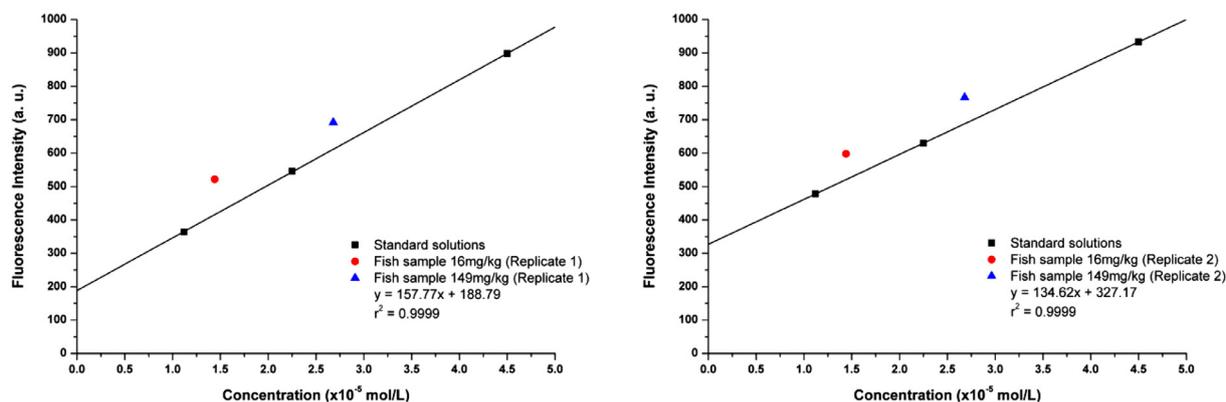


Fig. 7. Fluorescence analysis of real tuna samples in the presence of MO-OPA. Replicate 1 (left) and replicate 2 (right).

**Table 1**

Comparative table of mass fractions obtained from the maximum molecular fluorescence intensities of the OPA/HA and MO-OPA/HA complexes.

Derivatizing agent	Sample 1 <sup>a</sup>		Sample 2 <sup>b</sup>	
	Replicate 1	Replicate 2	Replicate 1	Replicate 2
MO-OPA	21.1 mg/kg	20.1 mg/kg	159.5 mg/kg	163.4 mg/kg
OPA	22.0 mg/kg	21.4 mg/kg	166.2 mg/kg	170.4 mg/kg

<sup>a</sup> sample doped at  $15.79 \pm 2.53$  mg/kg.

<sup>b</sup> sample doped at  $148.64 \pm 7.99$  mg/kg.

when using MO-OPA as evidenced by results summarized in Table 1. MO-OPA proved to be more accurate than its parent OPA and therefore could be a great tool in the future to estimate the degree of freshness of fishery products.

## Conclusion

To conclude, we have successfully studied the application potential of MO-OPA for the fluorometric analysis of HA in seafood. Performance of MO-OPA was compared to the reference compounds OPA and it was demonstrated that MO-OPA possesses several advantages. Indeed, it has an extended linear range of work of about one order of magnitude compared to the standard OPA. In addition to that, limits of detection and quantification of HA were found to be about 5 times lower than with OPA. The time required to reach the maximum molecular fluorescence intensity has proven to be very fast (15 min) and stable for more than two days for solution kept at 4°C. This outstanding rapidity and stability allow for the analysis of samples without timekeeping operations. Those points constitute a major improvement over the existing derivatization method. Those advantages coupled with the low interferences of other BAs on HA analysis by MO-OPA have permitted the analysis of HA with a slightly lower overestimation than with the use of OPA. MO-OPA therefore appears to be a first-choice candidate for rapid and easy molecular fluorescence evaluation of Ha in fishery products.

## Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.talo.2020.100014.

## References

- [1] A. Halász, Á. Baráth, L. Simon-Sarkadi, W. Holzapfel, Biogenic amines and their production by microorganisms in food, *Trends Food Sci. Technol.* 5 (1994) 42–49 [https://doi.org/10.1016/0924-2244\(94\)90070-1](https://doi.org/10.1016/0924-2244(94)90070-1).
- [2] D. Doeun, M. Davaatseren, M.-S. Chung, Biogenic amines in foods, *Food Sci. Biotechnol.* 26 (2017) 1463–1474 <https://doi.org/10.1007/s10068-017-0239-3>.
- [3] A.R. Shalaby, Significance of biogenic amines to food safety and human health, *Food Res. Int.* 29 (1996) 675–690 [https://doi.org/10.1016/S0963-9969\(96\)00066-X](https://doi.org/10.1016/S0963-9969(96)00066-X).
- [4] M. Papageorgiou, D. Lambropoulou, C. Morrison, E. Kłodzińska, J. Namieśnik, J. Plotka-Wasyłka, Literature update of analytical methods for biogenic amines determination in food and beverages, *TrAC Trends Anal. Chem.* 98 (2018) 128–142 <https://doi.org/10.1016/j.trac.2017.11.001>.
- [5] C. Ruiz-Capillas, A.M. Herrero, Impact of biogenic amines on food quality and safety, *Foods* 8 (2019) 62 <https://doi.org/10.3390/foods8020062>.
- [6] European Commission, Commission Regulation (EC) No 2073/2005 of 15 November 2005 on microbiological criteria for foodstuffs, *Official J. Eur. Union* (2005).
- [7] FDA, Fish and Fishery Products Hazards and Controls Guidance, Fourth Edition, 2019. <https://www.fda.gov/food/seafood-guidance-documents-regulatory-information/fish-and-fishery-products-hazards-and-controls-guidance>.
- [8] L. Prester, Biogenic amines in fish, fish products and shellfish: a review, *Food Additiv. Contaminants* 28 (2011) 1547–1560 <https://doi.org/10.1080/19440049.2011.600728>.
- [9] A. Ghosh, S. Talukdar, K. Ghosh, T. Das, D. Das, Colorimetric and fluorescence recognition of tryptophan and histidine using phthalaldehyde based probe: experimental, computational, cell imaging and fish tissue analysis, *RSC Adv.* 4 (2014) 55286–55289 <https://doi.org/10.1039/C4RA10158H>.
- [10] T. Dole, S. Koltun, S.M. Baker, R.M. Goodrich-Schneider, M.R. Marshall, P.J. Sarnoski, Colorimetric evaluation of mahi-mahi and tuna for biogenic amines, *J. Aquat. Food Prod. Technol.* 26 (2017) 781–789 <https://doi.org/10.1080/10498850.2017.1297879>.
- [11] L. González-Ceballos, B. Melero, M. Trigo-López, S. Vallejos, A. Muñoz, F.C. García, M.A. Fernandez-Muiño, M.T. Sancho, J.M. García, Functional aromatic polyamides for the preparation of coated fibres as smart labels for the visual detection of biogenic amine vapours and fish spoilage, *Sens. Actuators B* 304 (2020) 127249 <https://doi.org/10.1016/j.snb.2019.127249>.
- [12] A. Arulkumar, G. Karthik, S. Paramasivam, M.A. Rabie, Histamine levels in Indian fish via enzymatic, TLC and HPLC methods during storage, *Food Measure* 11 (2017) 281–289 <https://doi.org/10.1007/s11694-016-9395-z>.
- [13] S. Gone, N. Kosa, J. Krebs, J. Hungerford, M. Trucksess, C. DeWitt, Validation Study of MaxSignal® Histamine Enzymatic Assay for the Detection of Histamine in Fish/Seafood, *J. AOAC Int.* 101 (2018) 783–792 <https://doi.org/10.5740/jaoacint.17-0289>.
- [14] A. Veseli, M. Vasjari, T. Arbneshi, A. Hajrizi, L. Švorc, A. Samphao, K. Kalcher, Electrochemical determination of histamine in fish sauce using heterogeneous carbon electrodes modified with rhenium(IV) oxide, *Sens. Actuators B* 228 (2016) 774–781 <https://doi.org/10.1016/j.snb.2016.01.085>.

- [15] Y.-T. Lin, C.-H. Chen, M.S. Lin, Enzyme-free amperometric method for rapid determination of histamine by using surface oxide regeneration behavior of copper electrode, *Sens. Actuators B* 255 (2018) 2838–2843 <https://doi.org/10.1016/j.snb.2017.09.101>.
- [16] K. Zeng, H. Tachikawa, Z. Zhu, V.L. Davidson, Amperometric detection of histamine with a methylamine dehydrogenase polypyrrole-based sensor, *Anal. Chem.* 72 (2000) 2211–2215 <https://doi.org/10.1021/ac9911138>.
- [17] P. Malle, M. Vallé, S. Bouquelet, Assay of biogenic amines involved in fish decomposition, *J. AOAC Int.* 79 (1996) 43–49.
- [18] G. Nouadje, N. Siméon, F. Dedieu, M. Nertz, Ph. Puig, F. Couderc, Determination of twenty eight biogenic amines and amino acids during wine aging by micellar electrokinetic chromatography and laser-induced fluorescence detection, *J. Chromatogr. A* 765 (1997) 337–343 [https://doi.org/10.1016/S0021-9673\(96\)00925-9](https://doi.org/10.1016/S0021-9673(96)00925-9).
- [19] Á.K.A. Salazar, J.J.L. Castro, Á.K.A. Salazar, J.J.L. Castro, Central composite design to optimize the derivatization procedure for analysis of biogenic amines by HPLC-UV, *J. Braz. Chem. Soc.* 28 (2017) 575–581 <https://doi.org/10.5935/0103-5053.20160200>.
- [20] A. Ščavničar, I. Rogelj, D. Kočar, S. Kse, M. Pompe, Determination of biogenic amines in cheese by ion chromatography with tandem mass spectrometry detection, *J. AOAC Int.* 101 (2018) 1542–1547 <https://doi.org/10.5740/jaoacint.16-0006>.
- [21] L. Mattsson, J. Xu, C. Preininger, B. Tse Sum Bui, K. Haupt, Competitive fluorescent pseudo-immunoassay exploiting molecularly imprinted polymers for the detection of biogenic amines in fish matrix, *Talanta* 181 (2018) 190–196 <https://doi.org/10.1016/j.talanta.2018.01.010>.
- [22] A. Kaufmann, K. Maden, Easy and fast method for the determination of biogenic amines in fish and fish products with liquid chromatography coupled to orbitrap tandem mass spectrometry, (2018). <https://doi.org/info:doi/10.5740/jaoacint.17-0407>.
- [23] H.K. Mayer, G. Fiechter, UHPLC analysis of biogenic amines in different cheese varieties, *Food Control* 93 (2018) 9–16 <https://doi.org/10.1016/j.foodcont.2018.05.040>.
- [24] B.M. Fonseca, A.C. Cristóvão, G. Alves, An easy-to-use liquid chromatography method with fluorescence detection for the simultaneous determination of five neuroactive amino acids in different regions of rat brain, *J. Pharmacol. Toxicol. Methods* 91 (2018) 72–79 <https://doi.org/10.1016/j.vascn.2018.02.002>.
- [25] G. Duflos, C. Dervin, P. Malle, S. Bouquelet, Relevance of matrix effect in determination of biogenic amines in plaice (*Pleuronectes platessa*) and whiting (*Merlangius merlangus*), *J. AOAC Int.* 82 (1999) 1097–1101.
- [26] D. Seto, N. Soh, K. Nakano, T. Imato, Selective fluorescence detection of histamine based on ligand exchange mechanism and its application to biomonitoring, *Anal. Biochem.* 404 (2010) 135–139 <https://doi.org/10.1016/j.ab.2010.04.038>.
- [27] T. Nakano, K. Todoroki, Y. Ishii, C. Miyauchi, A. Palee, J.Z. Min, K. Inoue, K. Suzuki, T. Toyooka, An easy-to-use excimer fluorescence derivatization reagent, 2-chloro-4-methoxy-6-(4-(pyren-4-yl)butoxy)-1,3,5-triazine, for use in the highly sensitive and selective liquid chromatography analysis of histamine in Japanese soy sauces, *Anal. Chim. Acta* 880 (2015) 145–151 <https://doi.org/10.1016/j.aca.2015.04.059>.
- [28] A. Han, L. Xiong, S. Hao, Y. Yang, X. Li, G. Fang, J. Liu, Y. Pei, S. Wang, Highly bright self-assembled copper nanoclusters: a novel photoluminescent probe for sensitive detection of histamine, *Anal. Chem.* 90 (2018) 9060–9067 <https://doi.org/10.1021/acs.analchem.8b01384>.
- [29] Association of official analytical chemists, histamine in seafood - fluorometric method, AOAC official method 977.13. (1977) 1–3.
- [30] K. Bjornsdottir-Butler, F.A. Bencsath, R.A. Benner, Modification and single-laboratory validation of AOAC official method 977.13 for histamine in seafood to improve sample throughput, *J. AOAC Int.* 98 (2015) 622–627 <https://doi.org/10.5740/jaoacint.14-194>.
- [31] R. Jeya Shakila, T.S. Vasundhara, K.V. Kumudavally, A comparison of the TLC-densitometry and HPLC method for the determination of biogenic amines in fish and fishery products, *Food Chem.* 75 (2001) 255–259 [https://doi.org/10.1016/S0308-8146\(01\)00173-X](https://doi.org/10.1016/S0308-8146(01)00173-X).
- [32] J.J. Learey, S. Crawford-Clark, B.J. Bowen, C.J. Barrow, J.L. Adcock, Detection of biogenic amines in pet food ingredients by RP-HPLC with automated dansyl chloride derivatization, *J. Sep. Sci.* 41 (2018) 4430–4436 <https://doi.org/10.1002/jssc.201800455>.
- [33] R. Adamou, A. Coly, S.E. Douabalé, M.L.O.C.O. Saleck, M.D. Gaye-Seye, A. Tine, Fluorimetric determination of histamine in fish using micellar media and fluorescamine as labelling reagent, *J. Fluoresc.* 15 (2005) 679 <https://doi.org/10.1007/s10895-005-2975-7>.
- [34] Y. Murakami, N. Nakai, N. Ando, A. Okayama, [Determination of histamine by a rapid fluorescamine-UPLC method], *Shokuhin Eiseigaku Zasshi* 59 (2018) 121–125 <https://doi.org/10.3358/shokueishi.59.121>.
- [35] J.-H. Kim, I.S. Shin, Y.K. Lee, H.J. Oh, S.J. Ban, Improved HPLC method using 2,3-naphthalenedicarboxaldehyde as fluorescent labeling agent for quantification of histamine in human immunoglobulin preparations, *Osong Public Health Res. Perspect.* 2 (2011) 127–134 <https://doi.org/10.1016/j.phrp.2011.07.003>.
- [36] G. Rammouz, M. Lacroix, J.C. Garrigues, V. Poinot, F. Couderc, The use of naphthalene-2,3-dicarboxaldehyde for the analysis of primary amines using high-performance liquid chromatography and capillary electrophoresis, *Biomed. Chromatogr.* 21 (2007) 1223–1239 <https://doi.org/10.1002/bmc.893>.
- [37] A.L. Rönnerberg, C. Hansson, T. Drakenberg, R. Håkanson, Reaction of histamine with o-phthalaldehyde: Isolation and analysis of the fluorophore, *Anal. Biochem.* 139 (1984) 329–337 [https://doi.org/10.1016/0003-2697\(84\)90013-7](https://doi.org/10.1016/0003-2697(84)90013-7).
- [38] T. Yoshimura, T. Kaneuchi, T. Miura, M. Kimura, Kinetic analysis of the fluorescence reaction of histamine with orthophthalaldehyde, *Anal. Biochem.* 164 (1987) 132–137 [https://doi.org/10.1016/0003-2697\(87\)90376-9](https://doi.org/10.1016/0003-2697(87)90376-9).
- [39] M.K. Sasaki, F.R.P. Rocha, A.D. Batista, D.L. Rocha, Flow-based food analysis: an overview of recent contributions, *Anal. Methods* 9 (2017) 6313–6334 <https://doi.org/10.1039/C7AY02350B>.
- [40] P.A. Shore, A. Burkhalter, V.H. Cohn, A Method for the Fluorometric Assay of Histamine in Tissues, *J. Pharmacol. Exp. Ther.* 127 (1959) 182–186.
- [41] S.E. Douabalé, M. Dione, A. Coly, A. Tine, Contributions to the determination of histamine rate by measuring out the histamine–orthophthalaldehyde complex in the absorption and fluorescence, *Talanta* 60 (2003) 581–590 [https://doi.org/10.1016/S0039-9140\(03\)00184-X](https://doi.org/10.1016/S0039-9140(03)00184-X).
- [42] D.A. Larionova, S.N. Shtykov, N.V. Beloglazova, E.N. Koroleva, Effect of nucleophilic agents and organized media on the fluorimetric determination of histamine with o-phthalaldehyde, *J. Anal. Chem.* 63 (2008) 1044–1050 <https://doi.org/10.1134/S1061934808110051>.
- [43] Y. Mengerink, D. Kutlán, F. Tóth, A. Csámpai, I. Molnár-Perl, Advances in the evaluation of the stability and characteristics of the amino acid and amine derivatives obtained with the o-phthalaldehyde/3-mercaptopropionic acid and o-phthalaldehyde/N-acetyl-L-cysteine reagents: High-performance liquid chromatography–mass spectrometry study, *J. Chromatogr. A* 949 (2002) 99–124 [https://doi.org/10.1016/S0021-9673\(01\)01282-1](https://doi.org/10.1016/S0021-9673(01)01282-1).
- [44] Y. Liang, Y. Pan, Q. Guo, H. Hu, C. Wu, Q. Zhang, A novel analytical method for trace ammonium in freshwater and seawater using 4-methoxyphthalaldehyde as fluorescent reagent, *J. Anal. Methods Chem.* (2015) <https://doi.org/10.1155/2015/387207>.
- [45] M. Zhang, T. Zhang, Y. Liang, Y. Pan, Toward sensitive determination of ammonium in field: A novel fluorescent probe, 4,5-dimethoxyphthalaldehyde along with a hand-held portable laser diode fluorometer, *Sens. Actuators B* 276 (2018) 356–361 <https://doi.org/10.1016/j.snb.2018.08.115>.
- [46] C. Moitessier, A. Rifai, P.-E. Danjou, I. Mallard, F. Cazier-Dennin, Efficient synthesis of 4-substituted-ortho-phthalaldehyde analogues: toward the emergence of new building blocks, *Beilstein J. Org. Chem.* 15 (2019) 721–726 <https://doi.org/10.3762/bjoc.15.67>.
- [47] O. Oyelakin, M. Traoré, E.H.B. Mbaye, A. Konté, L. Cissé, A.N. Faye, O. Faye, M. Mbaye, K. Kital, G. Seye, A. Coly, A. Tine, F. Delattre, Spectrofluorometric determination of putrescine using orthophthalaldehyde as a fluorophore: a novel method, *J. AOAC Int.* 99 (2016) 170–173 <https://doi.org/10.5740/jaoacint.15-0212>.
- [48] A. Tine, S.E. Douabalé, New method for determining histamine rate in halieutic products, in: C.D. Geddes (Ed.), *Reviews in Fluorescence* 2008, Springer, New York, NY, 2010, pp. 195–218, [https://doi.org/10.1007/978-1-4419-1260-2\\_8](https://doi.org/10.1007/978-1-4419-1260-2_8).
- [49] P. Nedeljkovic, M. Turel, A. Lobnik, Fluorescence-based determination of agmatine in dietary supplements, *Anal. Lett.* 48 (2015) 1619–1628 <https://doi.org/10.1080/00032719.2014.991962>.