ASM Sc. J., 16, 2021

https://doi.org/10.32802/asmscj.2021.809

Study of Histamine Detection using Liquid Chromatography and Gas Chromatography

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Histamine is a heterocyclic amine shaped by decarboxylation of the histidine. It is a compound that lack chromophore and involatile. However, the detection of histamine is imperative due to the characteristic of histamine has given several disadvantages in food industry. This paper describes methods for histamine detection by employing high performance liquid chromatography and gas chromatography. The derivatization techniques required for both methods in order to increase the sensitivity of chromatography analysis. Two derivatizing agents were applied in this study such as 9-flourenilmethyl chloroformate (FMOC – Cl) for HPLC analysis whereas for GC analysis a *N,O*-bis (trimethylsilyl)acetamide (BSA) was used. Method validation was in accordance to Commission Decision 657/2002/CE. The validation of specificity, linearity, precision, accuracy, detection limit and quantitation limit results indicate that the methods were acceptable. The linear range for both methods were at 0.16 – 5.00 $\mu g \cdot mL^{-1}$. The determination of histamine using GC showed the superiority of this instrument compared to HPLC. Method applicability was also checked on real sample namely mackerel in order to acquire a satisfactory recovery for both methods.

Keywords: histamine; HPLC; GC; fluorescence; FID; derivatizing reagent

I. INTRODUCTION

Histamine is a compound that containing nitrogenous and having low molecular weight. It is also considered as one of the biogenic amines which are very toxic and easily found in food protein according to the European Food Safety Authority (EFSA) (Parchami *et. al.*, 2017; Liu *et al.*, 2020). The presence of histamine in foods formed through histidine decarboxylation with the presence of a specific bacteria or shaped by amination and transamination of ketones and aldehydes (Gama & Rocha, 2020).

Histamine detection is very needed due to several reasons such as to modify the current methods so the best method acquired and to analyse histamine content of foods protein from different countries and dealing with it (Bogdanovic *et al.*, 2020). Food and Drug Administration (FDA) has reported that histamine can be safely consumed below 50 ppm, it has also related to Scombroid poisoning owing to fish consumption that related to *Scombroidae* family such as sardine, mackerel, tuna and mahi – mahi (Qiao *et al.*, 2020).

Several approaches such as chromatography and electrochemical techniques have been applied and studied to detect histamine in foods. Liquid and gas chromatography are the techniques that have been applied in decades. These methods are well developed, sensitive and very selective (Plakidi *et. al.*, 2020; Zhang *et. al.*, 2020; Bogdanovic *et. al.*, 2020; Kamankesh *et. al.*, 2019; Wojnowski *et. al.*, 2019; Jia *et al.*, 2020). However, histamine has a significant shortage when analysed using chromatography approaches such as involatile and the absence of a specific chromophore. These conditions setting off histamine need to be derivatized before analysed using liquid or gas chromatography. Furthermore, the derivatizing agents applied will also influence the application of chromatography (Munir & Badri, 2020), 9 – fluorenylmethyl chloroformate chloride (FMOC–Cl), 6–aminoquinolyl-*N*-hydroxysuccinimidy (AQC), O – phthaldialdehyde (OPA), dansyl-, dabsyl-, and benzoyl chloride are derivatizing reagents generally used to derivatize histamine before analyse using HPLC (Angulo *et. al.*, 2020; Bogdanovic *et. al.*, 2020; Lkhagva *et. al.*, 2020; Plakidi *et al.*,

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2020), whereas for GC analysis difference reagents used to derivatize histamine such as silylation, alkylation and acylation groups (Munir & Badri, 2020; Wojnowski *et. al.*, 2019; Papageorgiou *et al.*, 2018) as shown in Figure 1.

In this study, histamine was derivatized using FMOC–Cl before analyzed using HPLC that equipped with fluorescence detector whereas for GC analysis, histamine was derivatized using one of the silylation compounds namely *N,O* – bis (trimethylsilyl) acetamide (BSA) that equipped with flame ionization detection (FID) and verify the structure of histamine – derivatizing agent using mass spectrometry (MS). The recovery study of both methods were also studied by using fish mackerel.

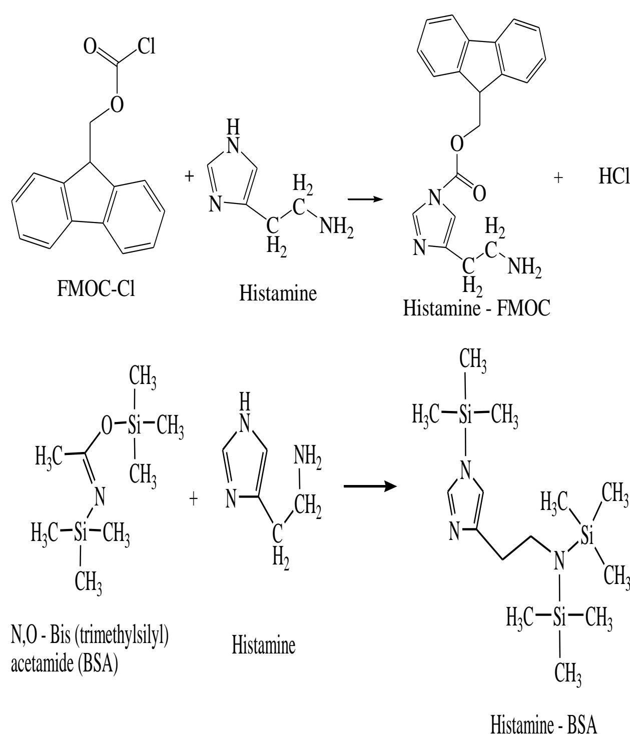


Figure 1. Reaction of FMOC – Cl and *N,O*–bis (trimethylsilyl)acetamide (BSA) as derivatizing reagents with histamine

II. MATERIALS AND METHOD

A. Standard, Chemicals and Reagents

Histamine dihydrochloride (HIS), 9-flourenilmethyl chloroformate (FMOC-Cl), N, O – bis (trimethylsilyl) acetamide (BSA), trichloroacetic acid (TCA), dichloromethane (DCM), HPLC grade acetonitrile and HPLC grade methanol, hydrochloric acid (HCl), potassium borate, glycine, sodium hydroxide, sodium hydrogen carbonate, acetone and glutamic acid were purchased from Sigma-Aldrich Sdn. Bhd. Deionized water was prepared with Premier equipment and used for all solutions. Standard (stock) solution of histamine was prepared at a concentration around 10 mg·L^{-1} in 0.1 M HCl and was further diluted for experiments.

B. Preparation of Histamine Standard Solution

Stock solution (10 mg·L^{-1}) of a solution of histamine was prepared 0.1 M HCl in a volumetric flask (10 mL). The solution was stored in the fridge at 4°C. Standard solutions were prepared by diluting from this stock solution and used to obtain the calibration curves (0.16 – 5.00 µg·mL^{-1}) and for validation purposes.

C. Methods for Derivatized Histamine

1. Histamine derivatized by FMOC - Cl

The derivatization of histamine using FMOC – Cl was performed by the methods of Lkhagva *et al.* (2020) with some modifications. 10 µL of histamine were mixed with a saturated solution of 2 M NaOH (10 µL), $NaHCO_3$ (100 µL) and 1 mL of FMOC – Cl. The mixture was heated on a hot plate (80°C) for 10 min. After the reaction, the excess of FMOC – Cl was removed by using 1 mL of glutamic acid (50 mg of glutamic acid in 1 mL of deionized water). After 30 minutes, the mixture was adjusted to 5 mL with acetonitrile. The solution was filtered using Whatman paper before injected onto HPLC.

2. Histamine derivatized by BSA

The derivatization method was adapted from Jia *et al.* (2020) with several modifications. Precisely 10 µL of histamine was put into the vial and evaporated with nitrogen gas. Afterward, 1 µL of BSA was added into the vial and heated at 80°C for 10 min. Then, cooled for 15 min and the derivatized solution was evaporated using nitrogen gas and the residue was dissolved with 1 mL of dichloromethane. 1 µL of the solution was injected into GC.

D. Analysis of Derivatized Histamine with Chromatography Approaches

1. HPLC analysis

The HPLC analysis was carried out on a Waters 2475 – C18 column (150 mm × 2.1 mm ID, 3 µm particle size), equipped with fluorescence detector (ex: 267/ em: 314). Chromatographic separation was carried out using an isocratic elution. The mobile phase was acetonitrile: deionized water (63:37, v/v) and the flow rate was kept at 0.34 mL·min^{-1} with injection of 0.5 µL. The mixture was filtered using membrane filter (0.45 nm) and degassed in an ultrasonic bath for 10 min before being analyzed. The target compound was identified according to the retention time of its corresponding standard. Quantification was measured using the calibration curves of the standard that undergone similar derivatization step of sample preparation.

2. GC analysis

Gas chromatography (GC) unit equipped with two different detectors: Flame ionization detector (FID) and mass spectrometer (MS). FID used in order to acquire quantitative analysis whereas MS applied in order to verify the structure of biogenic amines derivatized. This procedure was followed Espalha *et al.* (2019) with some modifications. Both of detectors were optimized under similar temperature in order to analyse the samples. The temperature program was 110°C for 2 minutes and increased to 190°C at the rate of 5°C/min maintained for 3 minutes. Separation was achieved using a HP-5 phenyl methyl siloxane (30m, 0.25mm and 0.25µm) silica capillary column installed in a Hewlett Packard 6890 equipped with FID. Carrier gas was hydrogen. Whereas analyses derivatised biogenic amines using MS was performed on a capillary BPX-5 column (30m, 0.25mm and 0.25µm).

D. Recovery Study for Both Analytical Techniques

Validation of analytical procedure was performed to determine whether the procedure of extraction was eligible or otherwise. Recovery study generally used to validate the analytical procedure, spiking a particular amount of histamine to fish muscle and the recovered its by extracting with a particular solvent (Elik 2021). The extract was also derivatized prior analysed using HPLC and GC. Muscle of fish mackerel (*Scomberomorus guttatus*) used in this study. Approximately 5 g of fish muscle was blended and placed into an Erlenmeyer flask. Afterwards, fish muscle was spiked with 1 mL of 5 µg·mL^{-1} histamine standard and homogenized using centrifuge for 1 min. The sample was then dissolved with 10% TCA and centrifuged for 5 min at 2000 rpm. The supernatant was filtered through a filter paper and rinsed with 10 % TCA. The sample was divided into two categories, one sample derivatized using 5 mM FMOC-Cl and the other was derivatized using BSA. The homogenate was stored at 4°C before further analysis using HPLC and GC.

$$\frac{\text{Peak area of spiked sample}}{\text{Peak area of derivatized histamine}} \times 100 = \text{Recovery (\%)} \qquad (1)$$

E. Validation Study for Both Analytical Techniques

Both methods were validated in terms of: accuracy, precision, linearity, limit of detection (LOD) and limit of quantification (LOQ). LOD was measured using equation $(y + 3s_{y/x})/b$, where y – intercept of the calibration line, $s_{y/x}$ – standard deviation in the y – direction of the calibration line and b – the slope of the calibration line, whereas LOQ was measured using equation $10s_{y/x}/b$ (Jastrzebska *et al.*, 2016). Quantitative analysis was performed using the calibration curve and the accuracy of the method was validated based on recovery test on the standard solution of histamine derivative. Studies of the correlation of determination and linear regression, assessment of repeatability, measurement of mean, standard and relative standard deviation were measured using Microsoft Excel 2020 software.

III. RESULT AND DISCUSSION

A. Method Validation of Derivatized Histamine Analysis using HPLC and GC

The analytical methods were optimized by determining the linear range, accuracy, precision, detection and quantification limit and recovery. Results are presented in Table 1. Linearity of the calibration curves was established by injecting six standard histamine derivatized with various concentrations ranging from 0.16 to 5 µg·mL^{-1}. Satisfactory linearity of both methods was acquired between the peak area

and concentration of the histamine derivatized (Figure 2). The detection limit was validated using formula that have been mentioned above. Both of them were reflected by the limit of detection (LOD) and limit of quantification (LOQ) values (Fu *et. al.*, 2016; Jia *et al.*, 2013).

The repeatability and reproducibility of the method was measured by injecting derivatized histamine (1.25 and 5 μg·mL⁻¹) at six replicates on the same day (intraday) and over seven days (inter-day), respectively. They are presented in Table 1.

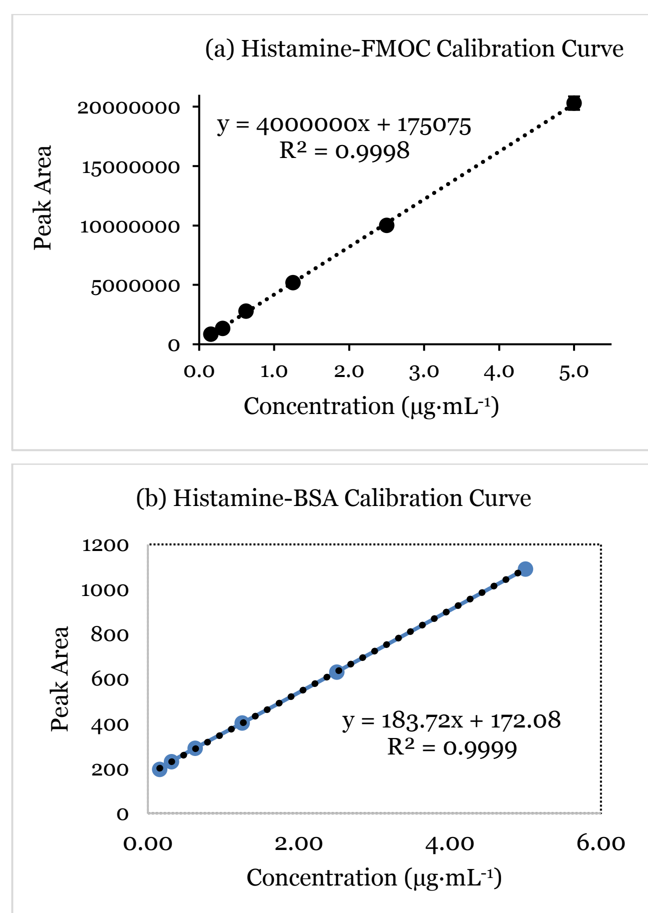


Figure 2. (a) Calibration curve of histamine – FMOC and (b) Calibration curve of histamine – BSA, peak area vs. concentration

Several HPLC methods have been applied for histamine determination, nevertheless the application of C18 reversed – phase columns and gradient acetonitrile – water mobile phase with satisfactory detection limit obtained (Li *et. al.*, 2021; de Lira *et al.*, 2019). A fish mackerel was selected to conduct the precision and accuracy assay. The intraday precision was measured from the result of six replicates analytes prepared at 1.25 and 5 μg·mL⁻¹ (medium and high concentration was chosen) of derivatized histamine to fish sample in a single day whereas for the inter-day precision was determined from the analytes for seven consecutive days. The mixture was derivatized prior to HPLC and GC analysis. Satisfactory recovery for histamine was also acquired for both methods and shown in Table 1. It can be concluded that tricholoroacetic acid (TCA) can be applied to extract histamine from fish mackerel.

Table 1. Linear regression calibration parameters of histamine determination by HPLC and GC after derivatization with chosen derivatizing agent

Analyte	Parameters validated	Technique applied	
		HPLC analysis	**GC analysis**
Histamine	Linear range (μg·mL⁻¹)	0.16, 0.31, 0.63, 1.25, 2.50 and 5.00	
	Calibration curve	y = 4E+06x + 175075	y = 183.72x + 172.08
	R^2	0.9998	0.9999
	LOD (μg·mL⁻¹)	0.10	0.06
	LOQ (μg·mL⁻¹)	0.30	0.18
	Intraday (%RSD)	1.73	0.23
	Interday (%RSD)	7.38	0.78
	Recovery Study (%)	103	105

The typical chromatogram of derivatized histamine standard analysed by HPLC equipped with fluorescence detector presented in Figure 3. FMOC – Cl that used to derivatize histamine cannot be verify with GC – MS due to the characteristic of derivatizing agent is not appropriate with MS detector, furthermore, only silylation, alkylation and acylation group can be used and analysed by MS detector. However, several studies have been carried out in order to verify the derivatized histamine by FMOC – Cl. The use of glycine as an internal standard also increased the selectivity of detection. Several studies by researchers have proven that in order to determine histamine using HPLC, the application of derivatizing agent is compulsory (Sentellas *et al.*, 2016). According to Figure 3, there were three components appeared at retention time of 2.080, 3.510 and 3.839 minutes, respectively.

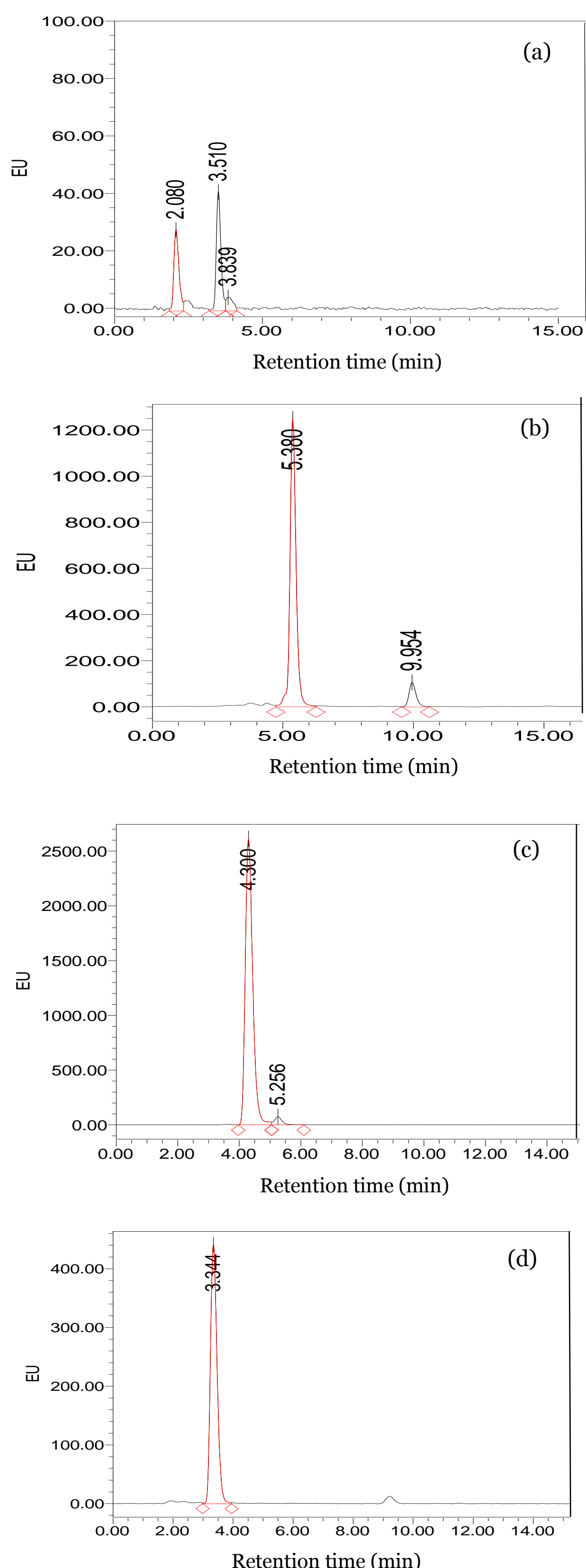


Figure 3. (a) The chromatogram of histamine standard derivatized by FMOC-Cl (ex: 267/ em: 314), (b) The chromatogram of FMOC-Cl, (c) the chromatogram of FMOC-Cl and glycine and (d) the chromatogram of FMOC-Cl and TCA

The peak for derivatized histamine standard appeared at retention time of 2.080 min. The peak for FMOC-Cl was detected at 5.380 min, glycine appeared at 4.310 min, and TCA appeared at 3.344 min. All chromatogram peaks are presented in Figure 3.

HPLC analysis equipped with fluorescence detector allows to detect histamine with satisfactory LOD with linearity between 0.25 – 10 μg·mL^{-1} (Sentellas *et. al.*, 2016; Comas – Baste *et. al.*, 2017; Wang *et al.*, 2018). Generally, a derivatization step with a chromophore or fluorophore is required to increase the sensitivity and selectivity during analysis. The application of derivatization needs sample preparation in order to avoid derivatization agents react with undesired matrices. As a result, the step is very tedious and time – consuming. Furthermore, HPLC is a quantitative technique that suitable for histamine analysis when HPLC is coupled with a variety of detectors such as UV and fluorescence detectors (Gagic *et al.*, 2018). Several researchers have been attempted to avoiding derivatization step for histamine analysis, nevertheless, owing to its high polarity of histamine that causing weak interaction with C18 of HPLC, the attemptation is still a great challenge for researchers. Table 2 presents various studies using HPLC and GC for histamine detection.

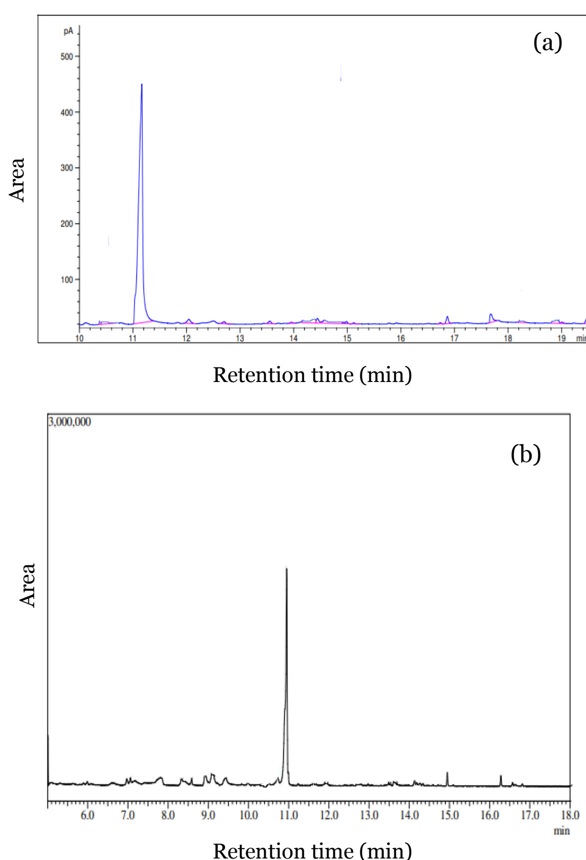
Table 2. Various studies for histamine detection using liquid and gas chromatography

Method used	LOD (μg·mL^{-1})	Ref.
HPLC (FLD & UV)	1.6 & 0.17	Francisco *et al.*, 2019
HPLC (FLD & UV)	0.1 & 0.08	Plakidi *et al.*, 2020
HPLC – FLD	0.24	He *et al.*, 2020
HPLC (QO – MS)	0.07	Myslek & Leszczynska 2018
HPLC – FLD	0.10	This study
GC – (QO – MS)	0.002	Jia *et al.*, 2020
GC – MS	0.06	Kamankesh *et al.*, 2019
GC – MS	0.003	Alizadeh *et al.*, 2017
GC – (FID & MS)	0.06	This study

HPLC: High performance liquid chromatography, GC: Gas chromatogaphy, FLD: Fluorescence detector, UV: Ultraviolet, MS: Mass spectrometry, QO: Quadrupole orbitrap, LOD: Limit of detection

The next method used in this study in order to compare to HPLC was analysis of derivatized histamine by GC – FID and GC – MS. Different reagent used to derivatized histamine before analysed using GC, namely BSA. Using this reagent, it

is possible to detect analyte using MS detector in order to verify the structure of derivatized histamine. Figure 4 shows the chromatogram of derivatized histamine detected by GC – FID and GC – MS. The specific peak was presence in the GC chromatogram at 11.2 min. Owing to FID as a detection that lacks of capability to confirm peak identity. Furthermore, GC – MS analysis is really required to identify and confirm the structure of derivatized histamine. Mass spectrometer is an excellent qualitative technique that providing confirmatory analysis guidelines to investigate the presence or absence of analyte(s) in a sample. Analysis of derivatized histamine standard by MS detector is also imperative to ensure that the derivatization actually reacts to histamine or otherwise (Gagic *et al.*, 2019). According to this study, the application of GC can decrease the detection limit of histamine. Several studies reported that histamine detection using GC was applied to detect histamine in cheese sample. Nevertheless, the derivatization step is imperative and required in order to increase the selectivity of GC (Mohammadi *et. al.*, 2017; Ramezani *et al.*, 2015).



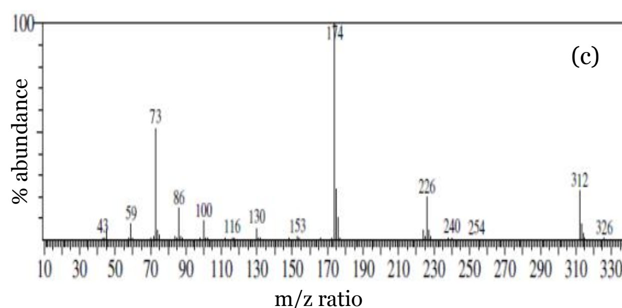


Figure 4. (a) The chromatogram of histamine standard derivatized by BSA analysed by GC – FID, (b) The chromatogram of derivatized histamine verified by GC – MS and (c) Mass spectrum of derivatized histamine standard

The molecular and fragment ions derivatized histamine standard is shown in Figure 5, while electron impact (EI) of mass spectrum for derivatized histamine standard presented in Figure 4. Mass spectrum of histamine derivatized by BSA has a molecular ion with m/z = 326 which can be seen in mass spectrum (Figure 4). The loss of CH_3 (methyl) from a molecular ion (m/z=326) resulted a fragment ion with m/z = 312. The loss of C_2H_6Si (dimethylsilylane) from a fragment ion with m/z = 312 resulted a base peak with m/z = 174. Osorio *et al.* (2011) reported in their study that the molecular ion should not be the most abundant ion or even for some cases they cannot appear in the mass spectrum. Other fragment ions with structural information are shown in Figure 5.

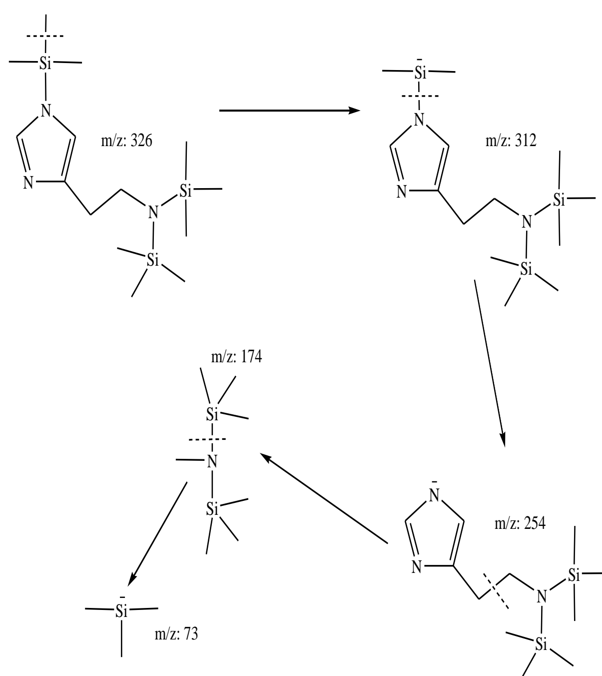


Figure 5. Molecular and fragments ions in mass spectrum of derivatized histamine and their suggested structures

IV. CONCLUSION

This study reported the differences technique of histamine detection using HPLC and GC. Prior analysed using these instruments, histamine needs to be derivatized in order to increase their sensitivity and selectivity of the instruments during analysis. Validation study has been applied for both methods and satisfactory results for both methods have been acquired. Derivatization reagents and methods can also be used for histamine analysis. Nevertheless, GC has shown its superiority compare to HPLC such as the detection limit is low and the structure of derivatized histamine can be verified using MS detector. Histamine can also be derivatized using FMOC – Cl and BSA, both of them showed the by – products obtained after derivatized and can be seen by several peaks appeared beside derivatized histamine. Histamine derivatized by FMOC – Cl also cannot be analysed using MS detector owing to histamine can only be detected after derivatized by specific reagents such as silylation, alkylation and acylation groups.

V. ACKNOWLEDGEMENT

The authors would like to extend their gratitude to Universiti Kebangsaan Malaysia and Malaysia Ministry of Education for providing the necessary funding for this research through the Research Incentive Grant (GGP-2019-021).

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