Analysis of N-nitrosodiprophylamines Carcinogenic Compound in Meat-Processing using Headspace-Single Drop Microextraction-Gas Chromatography-Flame Ionization Detector (HS-SDME-GC-FID)

Teguh Hari Sucipto[1], Ganden Supriyanto[2], and Yanuardi Raharjo[2]

Abstract - Analysis of N-nitrosodiprophylamines carcinogenic compound in processed meat especially hamburger and kebab had occured by HS-SDME-GC-FID technique. The results were obtained determining the optimum pH was 4, the optimum stirring speed was 6 scale, and the temperature of extraction was 30 ºC. It was obtained in this study that the detection limit of 78 ppb, the percent recovery of 101,18%, precision between 0,089% to 0,566%, and the true enrichment factor was 3372,66 times. From the results of the study was concluded that HS-SDME-GC-FID technique can be used to analyze the carcinogenic compound N-nitrosodiprophylamines (NDPA) found in meat-processing (hamburger and kebab) by the concentration of each samples as follows, hamburger I of 0,27 ppm, hamburger II of 0,73 ppm, hamburger III of 1,39 ppm, and kebab I of 3,13 ppm.

Index Terms - HS-SDME-GC-FID technique, N-nitrosodiprophylamines, Meat-processing

INTRODUCTION

The cause of cancer was caused by nitrosamine compound that attacks on certain organs, such as stomach [1]. The results of the various species of animals declared that nitrosamines were carcinogenic. In addition, nitrosamines were also toxic and mutagenic [2]. The level of tolerance N-nitrosamines in the human body ranges from 5 to 10 mg/kg of weight human body [3].

Based on the description above, considered the N-nitrosamines in this case NDPA was carcinogens in the human body and cause cancer, the need for an analytical technique that was simple and has a high sensitivity properties to detect the presence of N-nitrosodiprophylamines (NDPA) in the food. Based on the properties of N-nitrosamines volatile (volatile), the HS-SDME extraction techniques (Headspace-Single Drop microextraction) very efficiently can be used. HS-SDME extraction technique has several advantages, namely avoiding the extraction with organic solvents when the contaminant in samples that may interfere with the analysis. In addition, HS-SDME extraction technique was also simple, easy, and does not require a long time of extraction. The existence of N-nitrosamine compounds can be identified using the instrument GC (Gas Chromatography). Gas chromatography (GC) was an analytical technique that can be used to identify chemical compounds with properties easily evaporated [4] and can detect samples up to μg/L.

[1]Teguh Hari Sucipto, is with Institute of Tropical Disease, Universitas Airlangga, Surabaya, Indonesia. E-mail: teguhharisucipto@gmail.com.

[2]Ganden Supriyanto, and Yanuardi Raharjo are with Departement of Chemistry, Faculty of Science and Technology, Universitas Airlangga, Surabaya, Indonesia. E-mail: ganden88@yahoo.com; raharjo84@gmail.com.

METHODS

In this study used to extract compounds toluene nitrosodipropilamin (NDPA). A total of 10 ml of standard solution (for example, a standard solution of 6 ppm NDPA) was inserted into the bottle containing a magnetic stirring bar. Microsyringe already contain organic solvents (eg, toluene as much as 3 mL) was inserted into the bottle vertically up hanging over the standard solution. Then the microsyringe tip was pressed so that the organic solvent hangs at the end of the needle. Then NDPA standard solution was stirred using a magnetic stirrer. After the extraction process was completed, the organic solvent was pulled back into a microsyringe and injected directly into the GC-FID instruments, and the resulting area for the standard concentration.

VALIDATION OF ANALYTICAL METHODS

The calculation of the limit of detection (LOD) NDPA, earned value detection limit of 78 ppb. This value was the smallest concentration limits can still be responded by Gas Chromatography. While the limit of detection for the calibration curve obtained without the extraction of 0.86 ppm. By comparing the value of the detection limit without extraction of NDPA measurement and limit detection NDPA measurements with HS-SDME extraction showed that the HS-SDME extraction method capable of increasing the sensitivity of GC-FID to provide

a response to NDPA. It can be concluded that with the extraction method using the HS-SDME sensitivity GC-FID being very high [5].

Calculation% recovery between 99.87% and 105.65%, of the value can be inferred that the extraction method in determining the HS-SDME NDPA has good accuracy, or it can be said that this extraction method is actually closer proximity NDPA concentration. No recovery value that indicates 105.65% due to other compounds that give the same signal at the retention time NDPA.

Method can be said to have the accuracy or precision was good if the value of the coefficient of variation (CV <3%) [6]. It can be concluded that the accuracy or precision produced by GC-FID used for the analysis of NDPA compounds in the sample, as evidenced by the resulting coefficient of variation of 0.089% to 0.566%.

Theoretical enrichmen factor (EF_{th}) was amounted to 3333.33 times. According to the theoretical concentration that occurs in the extraction process using the HS-SDME NDPA at 3333.33 times. While the actual or true concentration enrichment factor (EF_{tr}) amounted to 3372.52 times. So it can be concluded that the concentration process that occurs in the extraction using the HS-SDME good, because the results have EF_{th} not so much difference with EF_{tr}.

CALIBRATION CURVE OF NDPA

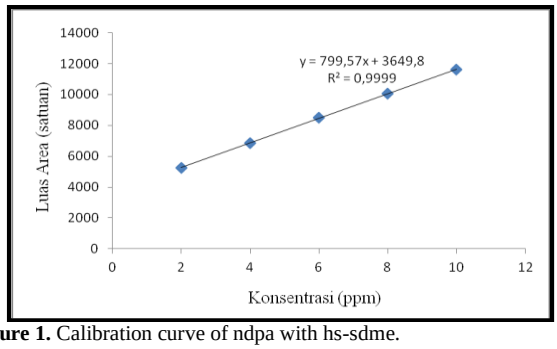

Figure 1. Calibration curve of ndpa with hs-sdme.

Calibration curve of NDPA with HS-SDME the analytic parameters optimum conditions. Calibration curve of NDPA with the extraction of HS-SDME was obtained by linear regression y = 799.57x + 3649.8 with correlation coefficients (R2) of 0.9999. It shows there was a correlation between the concentrations of NDPA an area of the chromatogram.

SAMPLES ANALYSIS

TABLE 1. DATA OF NDPA CONCENTRATION IN THE SAMPLES.

	Concentration (ppm)
Samples	A
Hamburger	0,27
Kebab	3,13

CONCLUSION

Methods of HS-SDME-GC-FID can be used to analyze compounds N-nitrosodiprophylamines (NDPA) was contained in processed meats (hamburger and kebab). This method has a detection limit of the HS-SDME-GC-FID method was 78 ppb, the percent recovery of 101.18%, the precision between 0.089% to 0.566%, the theoretical enrichment factor of 3333.33 times, and a true enrichment factor 3372.66 times.

REFERENCES

[1] Domanska-Blicharz, K., Rachubik J., Kowalski, B., 2005, Occurrence of Volatile N-Nitrosamines in Polish Tinned Foods, *Bull Vet Inst Pulawy*, 49, 319-322.

[2] Andrade, R., Reyes, F.G.R., Rath S., 2005, A Method For The Determination of Volatile N-Nitrosamine in Food by HS-SPME-GC-TEA, *J. Food Chem.*, 91: 173-179.

[3] Filho, P.J.S., Rios, A., Valcárcel, M., Zanin, K.D., Caramão, E.B., 2003, Development of a New Method for The Determination of Nitrosamines by Miceller Electrokinetic Capillary Chromatography, *Water Research*, 37: 3837-3842.

[4] Riccio, D., Wood, D.C., Miller, J.M., 2008, Using Single Drop Microextraction for Headspace Analysis with Gas Chromatography, *J. of Chem. Education*, 85(7), 965-968.

[5] Sleiman, M., Maddalena, R.L., Gundel, L.A., dan Destaillats, H., 2009, Rapid and Sensitive Gas Chromatography-Ion-Trap Tandem Mass Spectrometry Method for The Determination of Tobacco-Specific N-Nitrosamines in Secondhand Smoke, *J. of Chrom. A*, 1216, 7899-7905.

[6] Miller, J.C., dan Miller, J.N., 1988, *Statistics for Analytical Chemistry Second Edition*, Elllis Horwood Limited, England.