International Journal of Chemical Studies

P-ISSN: 2349–8528
E-ISSN: 2321–4902
IJCS 2017; 5(5): 475-479
© 2017 IJCS
Received: 08-07-2017
Accepted: 09-08-2017

Candra Irawan
Politeknik AKA Bogor, Indonesia

Swatika Juhana
Politeknik AKA Bogor, Indonesia

Hanafi
Politeknik AKA Bogor, Indonesia

Henny Rochaeni
Politeknik AKA Bogor, Indonesia

Miskah Yumna Fajri
Politeknik AKA Bogor, Indonesia

Rika Perdana Putri
PT. Nilam Widuri, Bogor-Indonesia

Correspondence
Swatika Juhana
Politeknik AKA Bogor, Indonesia

Synthesis and spectral characterization Schiff base cyclantine with GC-MS

Candra Irawan, Swatika Juhana, Hanafi, Henny Rochaeni, Miskah Yumna Fajri and Rika Perdana Putri

Abstract
It has been synthesized Schiff base cyclantine by condensation between cylamenaldehyde dan methylanthranilate. The process of condensation is at temperature 89,5 ℃; 99 ℃; dan 109,5 ℃ with stirrer. The result shows that Schiff base cyclantine spreads colour in bright yellow to dark yellow with floral aroma. The result of the average value of the refractive index of variation of heating temperature is respectively 1,575; 1,538; dan 1,508. The average value of the density of the variation of heating temperature is respectively 1,071 g/mL; 1,065 g/mL; dan 1,063 g/mL. The average value of specific gravity of the variation of heating temperature is respectively 1,074; 1,068; dan 1,066. The results with GC-MS shows Schiff *base cyclantine* has formed m/z= 323,2 that refers to cyclantine compound ($C_{21}H_{25}NO_2$).

Keywords: Schiff base, cyclantine, GC-MS, condensation, cylamenaldehyde, methylanthranilate

Introduction
One of the qualities of a fragrance depends on the quality of the dissolving medium of fragrance. Fragrance quality has a durable aroma resistance. The fragrance dissolving medium having antimicrobial properties can improve the aroma resistance. Schiff base derivates showed a variety of biological and pharmacological activities as antimicrobial, antidepreseeant, anti-HIV, cytotoxicity, analgesic, antileshmanial, anticonvulsant, insecticides, fungicides, anticancer, tuberculostatic, and anti-inflammatory [1-5]. Therefore it is necessary to make Schiff base, one of which makes Schiff base cyclantine. Schiff bases known as imines are compounds containing azomethine group (-CH=N-) and represented by the general formula R3R2C=NR1. Many 2-hidroxy or 2-thiol amines are known to react with aldehydes or ketones to give heterocyclic instead of Schiff base. They are the condensed product of aldehydes or ketones with carbonil compounds and were first reported by Hugo Schiff in 1964 [6]. Originally, the classical synthetic route for synthesis of Schiff bases was reported by Schiff which involves condensation of primary amines with carbonyl compounds [7] under azeotropic distillation with the simultaneous removal of water. According to the Arctander (1969) [8] of making Schiff base cyclantine was made with condensation between cyclamenaldehyde and methylanthranilate. The condensation process takes place
under various conditions, for example by direct heating at a temperature of 90ºC with stirring. Modification of a simple condensation process by varying the heating temperature range needs to be done to become the reference temperature range that can be used for synthesis of sciff base cyclantine in industrial scale. So this research is done which aim to make Schiff base cyclantine with simple condensation at temperatures 89,5 ℃; 99 ℃; dan 109,5 ℃ with stirrer.

Experimental
Material and measurements
The chemicals used are cyclamendehyde solution, methylanthranilate solution, aquadest, 95% ethanol, and 99% ethanol. The equipment used in this research is GC-MS *Agilent 5975C inert MSD*, refractometer *Atago R-5000α*, hotplate stirrer *IKA C-MAG HS 7*, analytical balance *Denver Instrument TP-214* and analytical balance *ML-204*, cup glass 50 mL, alcohol thermometer, pycnometer 10 mL, vial, magnetic stirrer,

Synthesis Schiff Base Cyclantine

Solution cyclamenaldehyde is weighed as much as 9,5 grams to 50 mL cup glass, then added 8 grams of solution methylanthranilate, then heated and stirred over the hotplate sirer for 2 hours at temperature 89,5 ℃; 99 ℃; dan 109,5 ℃. Solution is saved at room temperature. The test observation was performed in the condition of silent 7 days after making the Schiff base cyclantine.

Analysis by GC-MS

This product is weighed 0,1 grams to vial and diluted to 1 gram in 99% ethanol then is measured with GC-MS. Confirmation of the molecular weight of cyclantine in the product is performed by fragmentation pattern making from the top of the mass spectrum of the product. The measurement is done on GC-MS instrument condition can be seen in Table 1.

Table 1: GC-MS Method for Schiff base cyclantine

Condition	Information
Column	Capiler column (Semi Polar) : HP–5 5% *Phenyl Methyl Siloxan* 325 °C
	Long = 30 m; Internal diameter = 320 μm; Film coating thickness = 0,25 μm
Carrier gas pressure	7,0531 psi
Injector temperature	100°C
Volume of Injection	0,2 μl
Injection Technique	Split
Split Ratio	80 : 1
Program temperature (Column) : 300°C to 5,399E-315 minutes	
Initial Temperature	100°C, held for 5 minutes
Temperature Rise Rate	15°C/min
The Final Temperature	250°C, held for 5 minutes
Interval	20 minutes

Measurement of Refractive Index on Products Using Refractometer

Measured product used refractometer *Atago RX-5000α*. Temperature of refractometer regulated 20 ºC. The solution of product is dripped over the prism surface until evenly distributed and then closed again. The result of the measurement is analyzed by the relationship of value refractive index the product making on temperature variation 89,5 ℃; 99 ℃; dan 109,5 ℃.

Measurement Density and Spesifisic Gravity

Solution from products is measured with pycnometer (10 mL) with room temperature (25 ºC). Pycnometer is cleared with 95% ethanol and dried. Empty pycnometer is weighed and then filled with aquadest until full and weighed. The determination of the solution pycnometer is the same as that.

Result and Discussion

The result of Schiff base cyclantine synthesis produces a color density sequence as in Fiqure 1.

Fiqure 1 shows that the Schiff base cyclantine synthesis solutions bright yellow to dark yellow. The color of Sciff base cyclantine shows the higher the heating temperature during synthesis the more concentrated the resulting Schiff base cyclantine color. The addition of excess methyl anthranilate also can cause the reaction results to dilute and minimize darkening of the color.

(a) (b) (c)

Fig 1: Results of Synthesis Schiff base cyclantine at Temperature (a). 89,5 ºC (b). 99 ºC (c).109,5 ºC

Chemical Structure and Composition

Based on the analysis with GC-MS on the resulting product, then obtained data analysis of GC-MS of target compound (Schiff base cyclantine) can be seen in Table 2.

From the result GC-MS analysis to product synthesis Schiff base cyclantine with different temperature synthesis 89,5 ℃; 99 ℃; dan 109,5 ℃ shows five peaks to cyclantine compounds.

Table 2: Data analysis of GC-MS of target compound (Schiff base cyclantine)

Repeat	Peak *Cyclantine*	Product synthesis at 89,5 ℃		Product synthesis at 99 ℃		Product synthesis at 109,5 ℃	
		Time of Retention	Similarity (%)	Time of Retention	Similarity (%)	Time of Retention	Similarity (%)
1	6	15,958	99	15,958	99	15,950	99
	7	16,491		16,491		16,476	96
	8	16,736		16,729		16,721	99
	9	16,808		16,808		16,801	
	10	17,521		17,507		17,500	
2	6	15,958		15,958		15,958	
	7	16,491		16,491		16,484	95
	8	16,736		16,729		16,729	99
	9	16,808		16,808		16,808	
	10	17,521		17,507		17,507	
3	6	15,958		15,951		15,958	
	7	16,491		16,484		16,491	
	8	16,736		16,736		16,729	
	9	16,808		16,808		16,808	
	10	17,514		17,507		17,514	

Table 2 shows that analysis of GC-MS on the resulting product having % similarity of target compound is cyclantine above 90% from base data of library (library search report Libr (TP).L). Cyclantine is one of Schiff bases. Based on the separation of the compound with GC-MS from the product obtained by the cyclantine appears at the peaks 6, 7, 8, 9, 10. Product of analysis with GC-MS from cyclantine with highest percent of highest area shows at Figure 2.

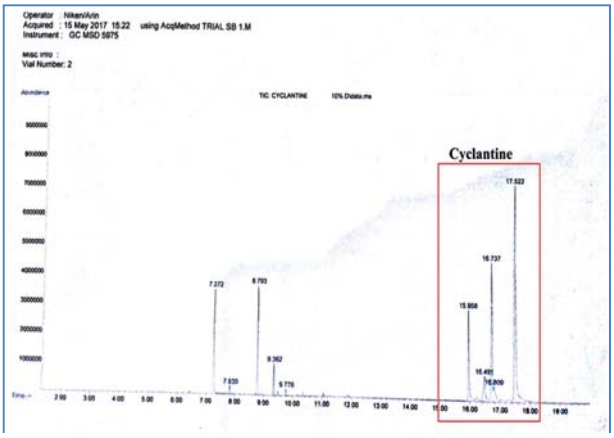


Fig 2: (a) Product of analysis GC-MS of cyclantine at peaks 6, 7, 8, 9, 10

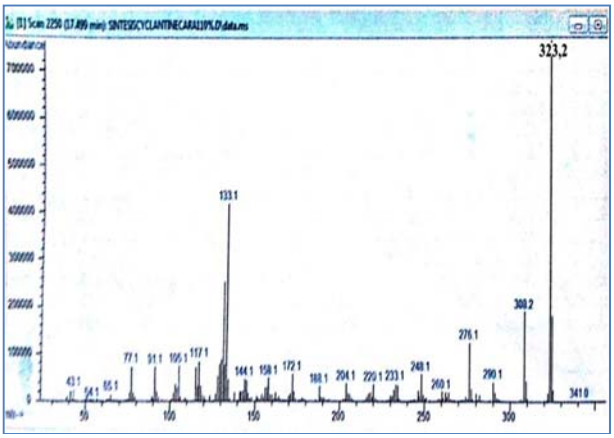
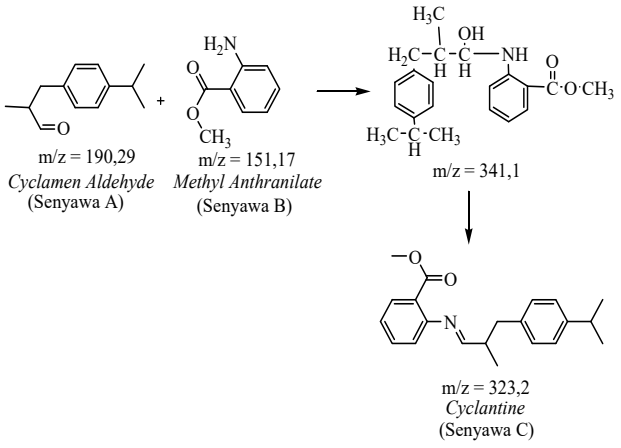


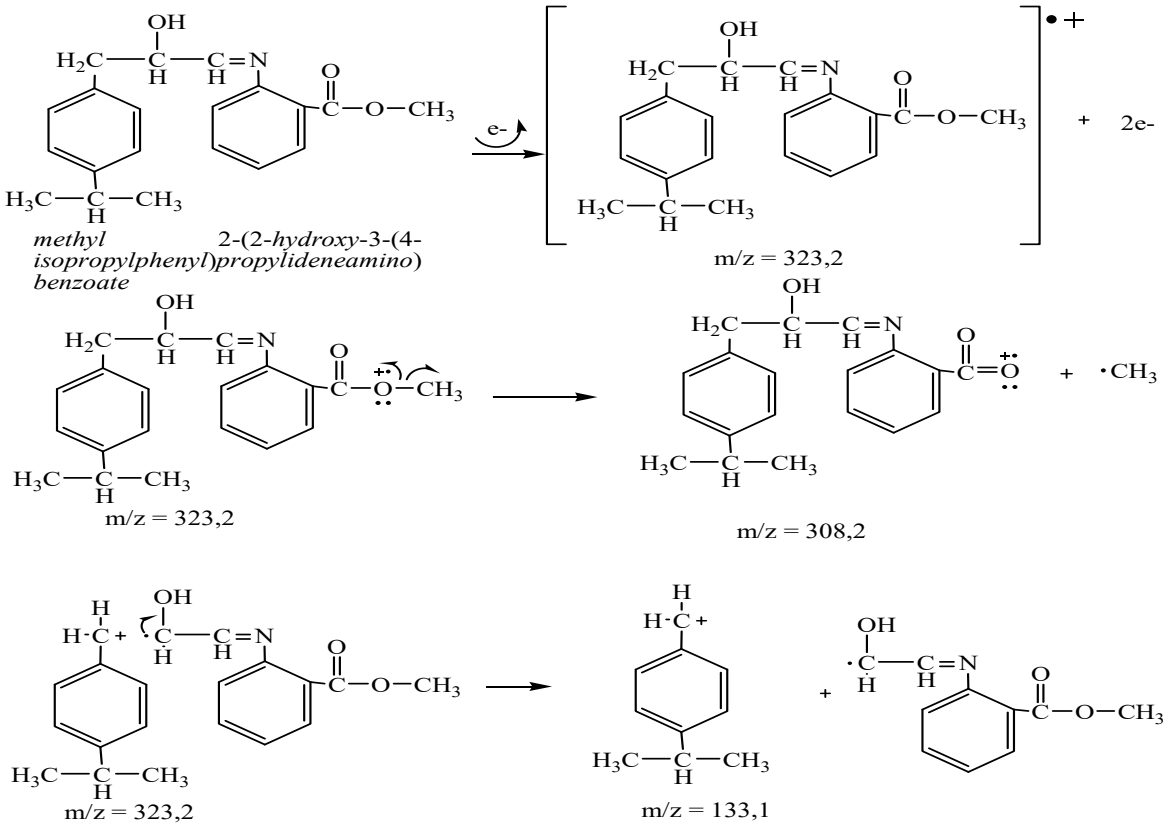
Fig 2: (b) Mass spectra data cyclantine compounds synthesis

Based on Figure 2, peaks 6, 7, 8, 9, 10 show cyclantine with retention of time like at Table 2, formula of compound $C_{21}H_{25}NO_2$, molecular weight 323, 44, and % similarity above 90%. Cyclantine compounds can make base peaks of five peaks, allegedly because boiling point cyclantine very high (low volatility) with large molecular weight can lead to less effective GC separation. The boiling point cyclantine is at 424 ºC while injector temperature is set at 100 ºC and column temperature program is at 300 ºC. So not all the gas phases of cyclantine are evenly formed in the inlet and are perfectly segregated in the GC column.

Mass spectra of compounds at Figure 1b shows peaks m/z = 341,1 and m/z = 323,2. Peak at m/z = 341,1 is allegedly compounds of methyl 2-(1-hydroxy-3-(4-isopropylphenyl)-2-methylpropylamino) benzoate. The compounds are suspected to be result of cyclamenaldehyde and metilanthranilate passing through the stages of the aldol condensation reaction. The base peak with the greatest abundance at m/z = 323,2 is cyclantine. Following Schiff base cyclantine reaction:



From the analysis, a base peak fragmentation of the cyclantine compound having the formula of IUPAC methyl 2-(2-hydroxy-3-(4-isopropylphenyl)propylideneamino)benzoate is presented as follows:



Based on fragmentation pattern, the peaks with larges abundance have m/z = 323, 2, m/z = 308,2 dan m/z = 133,1. At the stage os Schiff base synthesis which has various the temperature 89,5 ℃; 99 ℃; dan 109,5 ℃ has shown in mass spectrophotometer that the cyclantine is identified in the resulting product.

Analysis of Refractive Index *Schiff Base Cyclantine*

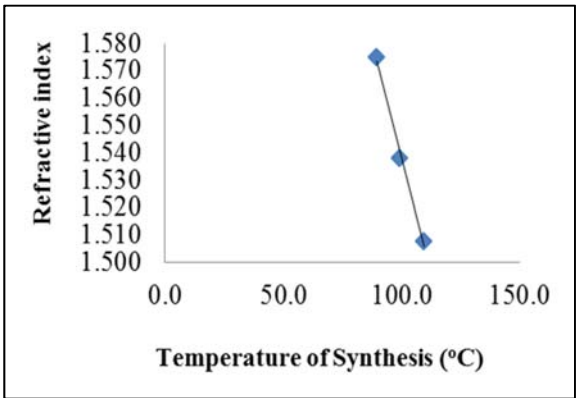


Fig 3: Refractive index value of Schiff base cyclantine average at temperature 89,5 ℃; 99 ℃; and 109,5 ℃

Based on Figure 3, is known that the higher the heating temperature the refractive index value will decrease. The Schiff base cyclantine that is too dilute may cause the refraction of light away from the normal line.

Analysis Density and Spesific Gravity of Schiff base cyclantine

Testing of density of product maked at temperature 89,5℃; 99℃; dan 109,5℃ produces an average density value of each 1,071 g/mL; 1,065 g/mL; dan 1,063 g/mL.

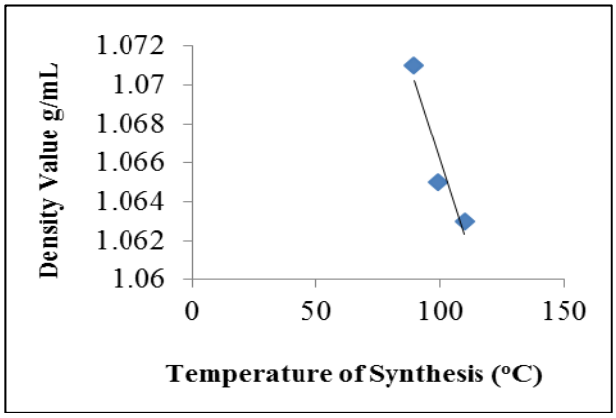


Fig 4: Density value of Schiff base cyclantine shows temperature averages at 89,5℃; 99℃; and 109,5℃

Based on Figure 4, it is known that the higher the synthesis temperature the density value will decrease. This is because the mixture (large mass) is too dilute due to higher heating temperatures.

Testing of specific gravity of product made at temperature 89,5 ℃; 99 ℃; dan 109,5℃ produces an average specific gravity value of each1,074; 1,068; dan 1,066.

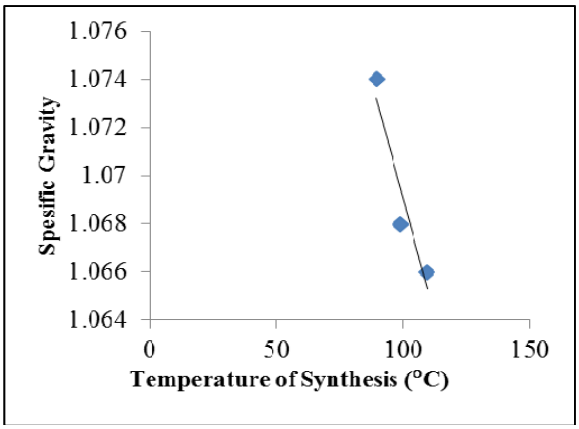


Fig 5: Spesific Gravity Value of Schiff base cyclantine average at temperature 89,5 ℃; 99 ℃; and 109,5℃

Based on Figure 5, it is known that the higher the heating temperature the specific gravity value will decrease. This is because the Schiff base cyclantine in the product which has undergone a higher warming will cause the composition of the lighter mass material or mixture is too dilute.

Analysis of Aroma Description Schiff Base Cyclantine

Based on testing result by the panelist, it is known that same aroma between Schiff base cyclantine at temperatures 89,5ºC, 99ºC, 109,5ºC lies in the aromas of attribute Floral, Sweet, White Flower, Lily of the Valley, Orange Blossoms, Green – Floral, while there is an with additional flavor attribute at 89,5ºC which has Herbaceous aroma and at temperature 99ºC has a Neroli and tuberose aromas. So the description of the aroma of panelist results in the floral smell family.

Conclusion

Schiff base cyclantine can be produced from condentation reaction between cylamenaldehyde dan methylanthranilate at temperatures 89,5℃; 99℃; dan 109,5℃. Schiff base cyclantine is coloring light yellow to dark yellow with aroma description floral. Result of testing product is synthesized at temperatures 89,5℃; 99℃; dan 109,5℃. The result of the average value of the refractive index of variation of heating temperature is respectively 1,575; 1,538; dan 1,508. The average value of the density of the variation of heating temperature is respectively 1,071 g/mL; 1,065 g/mL; dan 1,063 g/mL. The average value of specific gravity of the variation of heating temperature is respectively 1,074; 1,068; dan 1,066. The results with GC-MS shows Schiff base cyclantine has formed m/z= 323,2 that refers to cyclantine compound ($C_{21}H_{25}NO_2$).

Acknowledgement

The authors acknowledge the kindness and co-operation of the informal and local administrators in the study area, and supported by the Politeknik AKA Bogor, Indonesia.

Reference

1. Bhanupriya B, Devender P, Nadeem S, Shamsher MA, Waquar A. Search for Biologically Active Isatins: A Short Review. Journal of Pharmaceutical Science and Drug Research (IJPSDR). 2010; 2:229-235.
2. Manju P, Neeraj KS, Priyanka KK, Jha. Synthetic and Biological Multiplicity of Isatin. Journal of Advance Scientific Research. 2011; 2:35-44.
3. Aditya J, Patidar A, Manocha N, Gupta D. Synthesis, Characterization and Antimicrobial Activity of Novel Schiff Base of Isatin Derivatives. International Journal of Pharmaceutical Science and Drug Research (IJPSDR). 2012; 4:260-266.
4. Manjusha V, Surendra NP, Krishna NS, James PS. Anticonvulsant Activity of Schiff Bases of Isatin Derivates. Acta Pharmaceutica. 2011; 54:49-56.
5. Joaquim FMS, Simon JG, Angelo CP. The Chemistry of Isatins: a Review From 1975 to 1999. Journal of Brazilian Chemical Society. 2011; 12(3): 273-324.
6. Ibrahim MN, Hamad KJ, Al-Joroshi SH. Synthesis and Characterization of Some Schiff Bases. Asian Journal of Chemistry. 2006; 18(3):2404-2406.
7. Saravanan PS, Ramamoorthy S, Veerendiran M, Kaliyan S, Dakhshnamoorthy K, Kaliyaperumal R *et al*. Synthesis, Spectral Correlations and Biological Evaluation of Some Aryl (E)-N-Benzylidene-3-nitrobenzenamines. International Journal of Scientific Research in Knowledge. 2013; 1(11):472-483.
8. Arctander S. Perfume and Flavor Chemicals. Montclair N.J.USA. 1969.