



## Aurantiol Schiff base as A Raw Material in Fragrance Industry Synthesized by Simple Condensation Method and Its Characterization Using GC-MS

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<http://dx.doi.org/10.13005/ojc/360332>

(Received: May 06, 2020; Accepted: June 07, 2020)

### ABSTRACT

Fragrance seeds are produced from a combination of various kinds of raw materials, both natural and synthetic. One of the synthetic raw materials added in making fragrances is Schiff base. In this study, aurantiol Schiff base was made from raw material of methyl anthranilate and hydroxy citronellal by simple condensation at 110°C for 30 minutes. The color of the product obtained is observed and compared to the standard color, which is dark yellow. Furthermore, characterization was carried out using gas chromatography and mass spectrophotometry. The chromatogram results showed that aurantiol with the molecular formula  $C_{18}H_{27}NO_3$  had similarities above 90%.

**Keywords:** Fragrance, Aurantiol, Schiff base, Condensation method, GC-MS.

### INTRODUCTION

Schiff base (azomethine/anils/imines) which has azomethine (-CH = N-) group belongs to a group of compounds that have various biological activities such as anticancer, antimicrobial, antiviral, antitumor, antineoplastic, antitubercular, anti-HIV, anthelmintic, anticonvulsant, antimalarial, antiplatelet, antibiotic, diuretic, anti-inflammatory, and analgesic.<sup>1,2,3</sup> In addition, Schiff base is also often used in spices and fragrances because of its characteristic odor.<sup>4</sup> Some Schiff bases are reported to give the smell of orange flowers.<sup>5,6</sup>

Schiff base is a compound obtained from a condensation reaction between aldehydes and primary amines and produces water as a by-product.<sup>7,8</sup> The amines which are commonly used in the fragrance production are methyl anthranilate and ethyl anthranilate, while aldehydes commonly used in fragrances are alkyl aromatic or aldehyde, terpene aldehyde, such as hydroxycitronellal.<sup>9</sup> There are many types of Schiff base, such as lilyantine, jasmea, verdantiol,<sup>10</sup> aurantiol,<sup>11</sup> lyrame,<sup>12</sup> cyclantine,<sup>13</sup> and citral-methylantranilate.<sup>14</sup> The most famous type of Schiff base and widely used in various kinds of flower fragrance seeds are verdantiol, aurantiol, and lyrame.<sup>15</sup>



Aurantiol has the IUPAC name: methyl-N-3,7-dimethyl-7-hydroxyoctyliden anthranilate, and several trade names such as aurantiol, auriol, auranol, auralva, antralal, aurangeol, aurantine, and bigariol. Aurantiol has the molecular formula  $C_{18}H_{27}NO_3$ , with a molecular weight of 305.43 g/mol.<sup>16</sup> In the previous study, aurantiol was synthesized with simple condensation at a temperature of about  $90 \pm 5^\circ\text{C}$  with a time variation for 15, 30, 60, 120, 180, and 240 minute. The results showed that the optimal synthesis time about 30 to 60 min can be used as a reference for aurantiol synthesis in an industrial scale.<sup>11</sup> In this experiment, synthesis of aurantiol was carried out with simple condensation by increasing the temperature at  $110^\circ\text{C}$  for 30 minute. It is expected that by increasing the temperature can optimize the synthesis process.

## EXPERIMENTAL

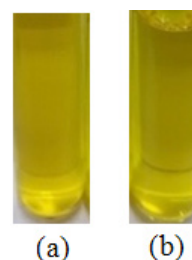
Aurantiol Schiff base was prepared by mixing methyl anthranilate solution and hydroxycitronellal solution. Then, the mixture was heated and stirred on a hotplate magnetic stirrer with a temperature of  $110^\circ\text{C}$  for 30 minute. The color of aurantiol Schiff base was compared to the standard colors. In order to confirm the obtained aurantiol schiff base, Agilent 5975C Gas Chromatography–Mass Spectrometer was applied. The aurantiol schiff base could be detected from the mass spectra. Finally, fragmentation patterns were made to confirm the obtained compounds. The analysis on condition of GC-MS can be seen at Table 1 below.<sup>12</sup>

**Table 1: GC-MS Analysis Condition**

Information	
Column	Capillary Column : HP-5 (5% Phenyl Methyl Siloxane $325^\circ\text{C}$ )
Carrier Gas	Helium
Carrier Gas Pressure	7.0531 psi
Injection Technique	Split
Injector Temperature	$100^\circ\text{C}$
Injection Volume	$0.2 \mu\text{L}$
Split Ratio	80 : 1
Temperature Program (Column)	
Initial Temperature	$100^\circ\text{C}$ hold for 5 min
Temperature rate	$15^\circ\text{C}$ per min
Final Temperature	$250^\circ\text{C}$ hold for 5 min
Interval	25 min

## RESULT AND DISCUSSION

The obtained aurantiol schiff base compared to standard product of PT Nilam Widuri is shown in Fig. 1. The colour of aurantiol was dark yellow and the standard had a thicker color. The aurantiol Schiff base is formed from hydroxycitronellal which has a carbonyl group. It reacts with the amine group in methyl anthranilate through the cross-aldol condensation reaction stage.



**Fig. 1. Color comparison of (a) the aurantiol standard (b) and the obtained aurantiol**

The confirmation of aurantiol Schiff base was carried out by GC-MS. The results are presented in Table 2.

**Table 2: The GC result of aurantiol Schiff base**

Retention Time (Min)	Compounds	Competition (%)	Molecular Formula
8.680	Hydroxycitronellal	20.68	$C_{10}H_{20}O_2$
9.465	Methyl Anthranilate	32.82	$C_8H_9NO_2$
16.923	Aurantiol	30.85	$C_{18}H_{27}NO_3$
16.981	Aurantiol	15.65	$C_{18}H_{27}NO_3$

Table 2 shows aurantiol with the molecular formula  $C_{18}H_{27}NO_3$  (molecular weight = 305.42 g/mol) and similarities above 90%. The aurantiol compound was identified at last, because the large molecular weight of aurantiol can cause the compound to be strongly bound to the stationary phase. Aurantiol boiling point is high (low volatility) of  $241^\circ\text{C}$ , while the injector temperature is set at  $100^\circ\text{C}$  and the maximum temperature of program column is  $325^\circ\text{C}$  can cause the gas phase of aurantiol not to form homogeneously since it is in the inlet, so aurantiol is identified more than one peak with different time retention. From this result, it was shown that the product contained not only aurantiol schiff base, but it also contained hydroxycitronellal and methyl anthranilate. The mass spectrum of the aurantiol compound in the product can be seen in Figure 2.

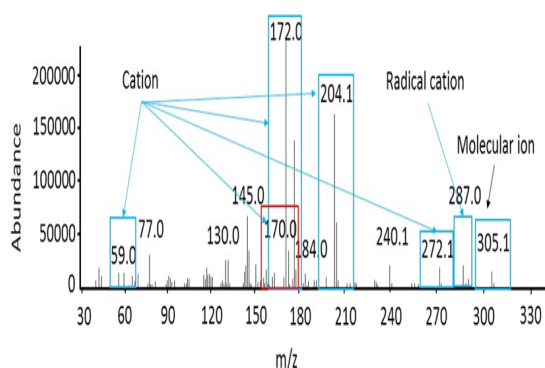


Fig. 2. Mass spectra of aurantiol schiff base

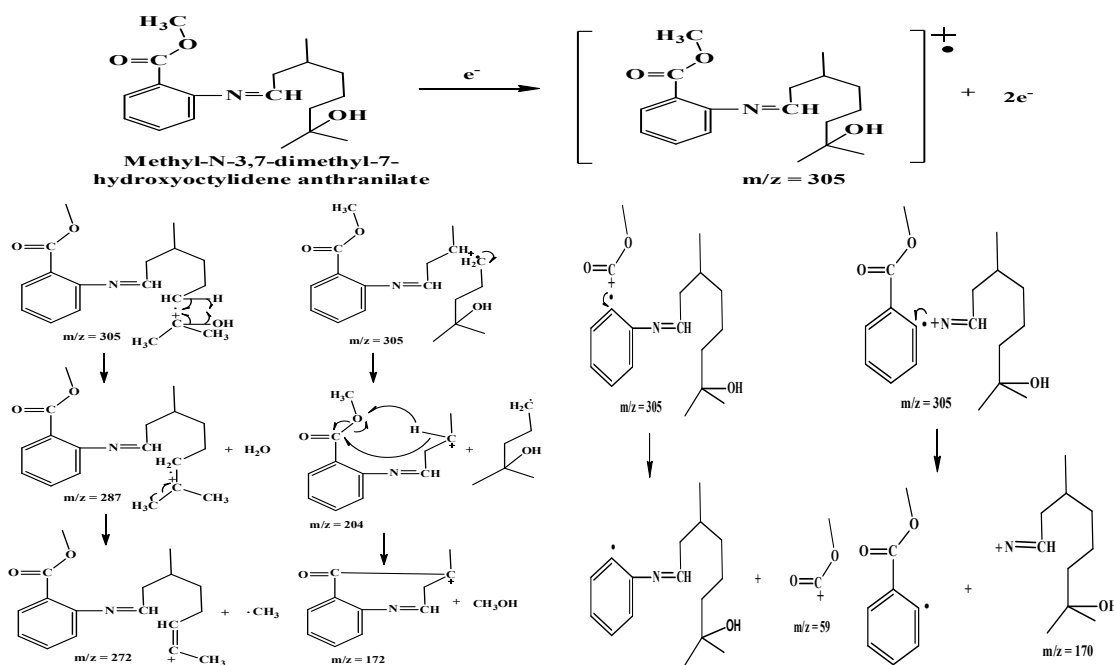


Fig. 3. Fragmentation of aurantiol Schiff base

## CONCLUSION

The aurantiol schiff base was successfully synthesized by a simple condensation at 110°C for 30 minutes. From the chromatogram results, 2 peaks were identified as aurantiol at retention times of 16.923 and 16.981 minutes. Based on mass spectrum, the molecular ion of aurantiol Schiff base was detected at m/z value of 305.1.

## ACKNOWLEDGEMENT

We would like to thank Politeknik AKA Bogor to fully support our research and PT Nilam Widuri for helping us during the experiments.

## Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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