

# Synthesis 3,4-Dimethoxybenzyl-2,4-Dihydroxyphenyl Ketone from Eugenol

Matsjeh S., Anwar C., Sholikhah E. N., and Alimuddin A. A.

**Abstract**—The main content of clove oil is eugenol, amounting to 80-90%. The aims this study to utilize eugenol from clove leave oil as raw material in the synthesis of 3,4-dimethoxybenzyl-2',4'-dihydroxyphenyl ketone with 3,4-dimethoxybenzylcyanide as intermediate compound. Converting eugenol into 3, 4-dimethoxybenzyl cyanide via several stages of reactions. Methylation of eugenol using dimethylsulphate and NaOH as catalyst was produced methyleugenol as much as 89.78% with a purity of 93.6%. Isomerization of methyleugenol using alkaline potassium tertiary butoxide (t-BuOK) in the medium dimethyl sulfoxide (DMSO) was resulted in 87.24 % yield methylisoeugenol with a purity of 88.89%. Methylisoeugenol was oxydated using potassium dichromate ( $K_2Cr_2O_7$ ) in a phase transfer catalyst system (PTC) by Polysorbate 80 into a 3,4-dimethoxybenzaldehyde as much as 85.36 % with a purity 83.46 %. Reduction of 3,4-dimethoxybenzaldehyde using sodium borohidrat ( $NaBH_4$ ) was produced 3,4-dimethoxybenzylalcohol as much as 98 % with a purity 100 % level. The 3,4-dimethoxybenzylalcohol was halogenated using  $SOCl_2$  was produced 3,4-dimethoxybenzylchloride which reacted further with sodium cyanide to produce 3,4-dimethoxybenzylcyanide as much as 89.5 % with a purity 99.24%. The acylation reaction of acid 3,4-dimethoxyphenylacetic acid with resorcinol to 3,4-dimethoxybenzyl-2,4-dihydroxyphenyl ketone obtained is a white a solid product having a melting point of 171-172°C. Rendamen generated from this experiment by 76%.

**Index Terms**—Eugenol, 3,4-dimethoxybenzylcyanide, 3,4-dimethoxybenzyl-2', 4'dihydroxyphenylketone.

## I. INTRODUCTION

Almost Indonesia's vast territory are very rich with aromatic essential oil plants.that essential oils which are the main content of aromatic compounds. One of them is the clove leaf oil. Clove leaf oil contains 80 % eugenol. One of the very important chemical properties of eugenol is eugenol have allyl group. The allyl group in eugenol, allowing eugenol can be converted into 3,4 - dimetoksibenzil carboxylic acid ( 3,4-dimethoxyphenyl acetic acid) . The compound have the ability to act as a raw material to synthesis of isoflavones. Isoflavone are subclass of flavonoids which are found in fruits, nuts, soybeans, and soybased products [1]. Isoflavones have demonstrated a variety of important biological activities, including

antioxidant [2], antibacterial [3], osteoporosis [4], anticancer effects [1]-[5]. The biological activity of these compounds is associated with beneficial effects on human health has become an interesting topic in the study of isoflavones including synthetic methods.

Many synthetic methods have been developed for the synthesis of isoflavones, among others are compounds chalcone [6], [7], chromon [8] and deoksibenzoin [9], [10]. The two most popular synthesis of isoflavon are the deoxybenzoine. (2-hydroxyphenyl benzyl ketone) and the chalcon routes. The Starting material for synthesis isoflavone via deoxybenzoine route used benzylcarboxylic acid (phenylacetic acid) and substituted phenols. The formation of intermediates deoxybenzoine by Hoeben-Hoesch reaction between phenol and benzyl cyanide [11], [12].

This research relates to synthesis of 3, 4-dimethoxybenzyl -2, 4-dihydroxyphenyl ketone from eugenol through 3, 4-dimethoxybenzylcyanide as an intermediate. Eugenol was chosen as a raw material, related to its structure which have allyl group attached in phenyl. The oxidation of allyl group will obtained substituted phenylacetic acid [12], [13].

## II. EXPERIMENTAL SECTION

### A. Materials

Clove leaf oil, eugenol, DMS, NaOH, petroleum ether, dichloromethane,  $K_2Cr_2O_7$ ,  $KMnO_4$ ,  $H_2SO_4$ ,  $NaBH_4$ , DMSO, t-BuOK, polysorbate 80, methanol, sodium cyanide, sodium iodide, ether,  $BF_3 \cdot Et_2O$  chloroform, ,  $SOCl_2$ , acetone, HCl,  $NaHCO_3$ , resorcinol glacial acetic acid, anhydrous  $Na_2SO_4$ , toluene, sodium bisulfit, sodium acetate aluminum  $F_{254}$  TLC plate.

### B. Instrumentation

Set of reflux, magnetic stirrer, electric heating, Buchi rotary evaporators, vacuum pumps, separating funnel, a set of TLC, UV light, stir bar, desiccator, a set of column chromatography and apparatus others of laboratory glassware..FT - IR spectrometer (Shimadzu FTIR - 820 PCs), GC - MS spectrometer (Shimadzu QP 5000) and H - NMR ,  $C^{13}$  NMR (JEOL JNM - MY 60) and GC-MS spectrometer.

### C. Methode

#### 1) Synthesis of methyleugenol

Eugenol (41 g, 0.25 mol) was placed in a 2 L glass beaker, added with stirring 120 mL of NaOH solution (12 g in 120 mL of distilled water, 0.3 mol). After the last 20 minutes of the addition, the mixture was transferred to a separator funnel and allowed to stand at night. Inorganic layer was extracted with 3 × 25 mL petroleum ether. Then transferred into a 1 L three-necked RB flask equipped with reflux condenser,

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dropping funnel and thermometer. 19.2 g (0.15 mol) dimethylsulfat (DMS) to add part-wise for 1.5 hours at room temperature. The mixture was heated under reflux for 3 hours at 110-120 °C, cooled and then 60 mL of distilled water was added. The solution was extracted with 3x 25 mL of petroleum ether and then washed with 10% NaOH.

#### 2) *Synthesis of methylisoeugenol*

To a three-neck flask of 100 ml capacity equipped with a dropper funnel, thermometer, cooler balls, silica gel blue, silica tube and magnetic stirrer, water bath fill in *t*-BuOK 10.1 g (0.09 mol) and 25 mL of DMSO and stirred until dissolved. Once dissolved, enter into the solution 16 g metileugenol (0.09 mol) and followed by reflux with stirring for four hours. Results refluxing mixture was poured into ice-water-petroleum ether (1:1) in a separating funnel and shaken for five minutes. Petroleum ether layer was separated.

#### 3) *Synthesis of 3,4 - dimethoxybenzaldehyde*

To a three-neck flask of 500 ml capacity equipped with a cooling balls, thermometer, stirrer and ice bath enter 5.5 g (0.031 mol) metilisoeugenol, 80 mL of distilled water, 3 mL of glacial acetic acid, 8 g of 50 % H<sub>2</sub>SO<sub>4</sub>, 0.15 polysorbate 80 g catalyst and 150 mL of dichloromethane. The mixture was stirred until homogeneous, then added 25 g (84.9 mmol) K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and the temperature was kept less than 30 °C. After all K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> was added, the mixture refluxed for 30 minutes and then cooled. Added 50 % H<sub>2</sub>SO<sub>4</sub> to pH 1-2. The organic layer is separated from the water layer. Water layer was extracted with 30 mL dichloromethane. The organic layer was collected.

#### 4) *Synthesis of 3,4-dimethoxybenzyl alcohol*

The 3,4-dimethoxybenzaldehyd as much as 0.97 g (6 mmol) and 50 mL of methanol is added to the round bottom flask size 100 mL, then added 0.35 g (9.2 mmol) of sodium borohydrat (NaBH<sub>4</sub>) while stirring. After stirring for one night, the mixture is evaporated to remove methanol. The residue was diluted with 25 mL of distilled water and extracted with 3x25 mL dichloromethane. The organic layer at the bottom was collected.

#### 5) *Synthesis of 3,4-dimethoxybenzylcyanida*

The 3,4-dimethoxybenzyl alcohol 2.0 g (12 mmol) in 15 mL of ether cooled in ice bath to a temperature of 0 °C was added 5 g (18 mmol) in SOCl<sub>2</sub> drops while stirring with a magnetic stirrer. Continue stirring the mixture at room temperature for 2 hours. The reaction was monitored by TLC analysis. After the reaction is complete, water is added to a solution of 50 mL of ice, then transferred into a separator flask and extracted with 3x50 ml ether. Organic layer dried with anhydrous sodium sulfate and evaporated to remove the solvent. Halogenated products was obtained directly towards nitrilisation. To a three-neck round bottom flask equipped with a magnetic stirrer, balls and catcher gas cooling, added 1.5 g of 3,4- dimethoxybenzyl chloride (9.5 mmol), 0.76 g of sodium cyanide, 0.1 g of sodium iodide and 5 mL of dry acetone. The heterogeneous mixture refluxed with vigorous stirring for 20 hours, then cooled and filtered. Filtered residue was washed with acetone. The filtrate obtained was added 5 mL of toluene and extracted with 3x5 ml of warm water.

#### 6) *Synthesis of 3,4-dimethoxyphenylacetic acid*

The 3,4-dimethoxy benzylcyanide 5 grams (30.6 mmol) was reflux with 250 mL 2M HCl for one hours, cooled overnight at 4°C in the refrigerator, the precipitate which occurred filtered, washed, recrystallized with methanol. Identify the structure of the reaction products using FT-IR, 1H NMR and GC-MS.

#### 7) *Synthesis of 3,4-dimethoxybenzyl- 2,4- dihydroxyphenyl ketone*

Resorcinol (0.003 mol, 0.33 g) was added to the acid mixture containing 3.4 - dimethoxyphenylacetic (0.003 mol, 0.5 g) and BF<sub>3</sub>.Et<sub>2</sub>O (4.5 mL). The reaction mixture was refluxed for 1.5 hours and cooled, and added to 60 mL of water; water layer was extracted with ethyl ether (3 x 50 ml). The combined ether layer was washed with saturated sodium acetate solution (30 ml) and saturated NaHCO<sub>3</sub> (15 mL), respectively. Layers were separated, and the ether layer was dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>.

### III. RESULTS AND DISCUSSION

#### A. *Synthesis of Methyleugenol*

Methyleugenol can be prepared by alkylation of eugenol with dimethyl sulphate as alkylation agent. The product was yellow oil to yield 89.78%. The chromatogram of gas chromatography of methyleugenol showed that the purities of product was 93.61%.

#### B. *Synthesis of Methylisoeugenol*

Methylisoeugenol can be prepared by isomerization of Methyleugenol with *t*-BuOK and DMSO, was produced as much as 87.24 % rendement. , form of colorless fragrant liquid. Mass spectra of the results showed ion molecularwer peak at *m* / *z* 178 which is the molecular ion of methylisoeugenol. FT - IR spectra showed absorption characteristics methyleugenol - CH = CH<sub>2</sub> on wave number 995 cm<sup>-1</sup> and 912 cm<sup>-1</sup>, will change form into the cis and trans structures methyleugenol. This change is indicated by the absorption peak at wavenumber 964.4 cm<sup>-1</sup> (cis - form) and 732.9 cm<sup>-1</sup> (trans - form) which replaces the absorption characteristics of the - CH = CH<sub>2</sub>. 1H NMR spectra are presented peak at 5.7 to 5.9 ppm chemical shift signals an H atom of the = CH - CH<sub>3</sub> with the appearance of a doublet peak. Peak at 6.1 ppm is the signal region of the proton - CH = CH with the appearance of a peak multiplets. Peak at 3.8 ppm chemical shift signals an H atom from the methoxy group is strengthened by the results of the integration of the equivalent of six hydrogen atoms. Peak at 1.8 ppm with the integration of the three hydrogen atoms showed a signal of the methyl group. Based on data from FT-IR and 1H - NMR concluded that the compounds produced from the isomerization reaction is methylisoeugenol.

#### C. *Synthesis of 3,4-Dimethoxybenzaldehyde*

Methylisoeugenol was converted to 3,4-Dimethoxybenzaldehyde by oxidation use K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> with a phase transfer catalyst polysorbate 80 (KTF). KTF needed to accelerate reactions that take place in a two- phase system. Product is a white solid withrendamen 85.36 %. The data obtained from the GC - MS showed a molecular ion peak at

m/z 166 with a purity of 83.79 %. This the same with molecular weight of the compound 3,4-dimethoxybenzaldehyde. FT-IR spectra of the product was showed typical absorption peak at wave number  $1681\text{ cm}^{-1}$  in the form of sharp bands indicates that the carbonyl group absorption bands in the region supported by 2841 and  $2729\text{ cm}^{-1}$  for the CH bond of the aldehyde.  $^1\text{H-NMR}$  spectrum showed the suitability of the oxidation products of chemical shifts. A peak at a chemical shift ( $\delta$ ) of 9.8 ppm is a characteristic peak for the chemical shift of the aldehyde hydrogen atom. Peaks at chemical shifts ( $\delta$ ) 6.9 to 7.3 hydrogen atoms of aromatic rings, this was confirmed by the results of the integration which is equivalent to three hydrogen atoms. Peak at 3.9 ppm chemical shift signals an H atom from the methoxy group is strengthened by the results of the integration of the equivalent of six hydrogen atoms. Based on data from FT-IR and  $^1\text{H-NMR}$  concluded that the compounds resulting from the oxidation reaction is 3,4-Dimethoxybenzaldehyde .

#### D. Synthesis of 3,4-Dimethoxybenzyl Alcohol

Reduction of 3,4-dimethoxybenzaldehyde compounds using  $\text{NaBH}_4$  to produce compounds such as 3,4-dimethoxybenzyl alcohol is white liquid with a yield of 98%. Mass spectra of compounds results showed reduction in the molecular ion m/z 168 which is the molecular ion of 3,4-dimethoxybenzyl alcohol. Typical absorption peaks in FT-IR spectra showed that the reduction of the carbonyl group of 3,4-dimethoxybenzaldehyde have produced a sharp absorption band is the loss of the carbonyl group at wave numbers  $1712.79\text{ cm}^{-1}$ . The absorption peaks are replaced by a broad absorption band in the wavenumber region  $3387\text{ cm}^{-1}$  which shows the absorption of the hydroxyl group.

$^1\text{H-NMR}$  analysis showed that peak at the chemical shift ( $\delta$ ) of 6.6 ppm is characteristic peaks of aromatic ring hydrogen atom. Peak at a chemical shift ( $\delta$ ) 3.6 ppm is the signal of the methoxy H atoms were reinforced by the results of integration is equivalent to six hydrogen atoms. Peaks at chemical shifts ( $\delta$ ) 4.4 ppm with the integration of two hydrogen atoms showed proton signals of methylene group. Based on the data from FT-IR and  $^1\text{H-NMR}$  concluded that the process of reduction of 3,4-dimethoxybenzaldehyde has produced compounds 3,4- dimethoxybenzyl alcohol .

#### E. Synthesis of 3,4-Dimethoxybenzylcyanide

Halogenated compounds 3,4-dimethoxybenzyl alcohol using  $\text{SOCl}_2$  generates 3,4-dimethoxybenzyl chloride compounds. Chloride compound is reacted with KCN obtained directly without further purification because the resulting halogen compounds are not stable. The yield of the product obtained by 89.5%. Product nitrilisation further analyzed by a FT-IR,  $^1\text{H-NMR}$  and GC-MS spectrophotometer. Gas chromatography analysis of the compound nitrilisasi results showed that the purity of the product was 99.24 %. Mass spectra of compounds results showed molecular ion at m/z 177 which is the molecular ion of 3,4 - dimethoxybenzylcyanide .

The compound 3,4-dimethoxybenzylalcohol have a wide absorption peak of hydroxyl groups on the local wave number  $3387\text{ cm}^{-1}$  which will be lost after the substitution reaction with halogen followed by nitrilisation to produce compounds 3,4-dimethoxybenzylcyanide . The compound

3,4-dimethoxybenzylcyanide have FT-IR spectra are typical of the sharp uptake of cyanide groups ( $-\text{CN}$ ) at wave number  $2245\text{ cm}^{-1}$ .

A proton in the chemical shifts ( $\delta$ ) 6.9 ppm is the characteristic for the aromatic ring hydrogen atom. Proton chemical shift at 3.9 ppm is the absorption of the methoxy H atoms are strengthened by the integration of the results of the equivalent of six hydrogen atoms. Proton chemical shift at 3.7 ppm with an integration of the two hydrogen atoms of the methylene group signal. Based on data from FT-IR and  $^1\text{H-NMR}$  spectroscopy concluded that the compound is produced 3,4-dimethoxybenzylcyanide

#### F. Synthesis of 3,4-Dimethoxyphenyl Acetic Acid

The 3,4-dimethoxyphenyl acetic acid starting material for synthesis keton was derived from 3,4-dimethoxybenzylcyanide. Infrared spectra of this product shows the presence of broad O-H hydroxyl stretch absorption at  $3448.72\text{ cm}^{-1}$  which is supported with the presence of sharp peak C=O stretch at  $1712.79\text{ cm}^{-1}$ . which showed the absorption of carbonyl groups. Absorption band in the region  $3000\text{--}3100\text{ cm}^{-1}$  indicate the presence of a bond = CH (sp<sup>2</sup>-s) that is reinforced by the absorption band at  $1643.35\text{ cm}^{-1}$  which is the characteristic absorption of the bond between sp<sup>2</sup> carbon clusters C = C ( $1600\text{--}1700\text{ cm}^{-1}$ ). Absorption band at wave number  $1512.19\text{ cm}^{-1}$  indicates that the double bond derived from an aromatic core ( $1450\text{--}1600\text{ cm}^{-1}$ ). Uptake in the area of  $2839.22\text{--}2939, 52\text{ cm}^{-1}$  indicate the presence of vibration Csp<sup>3</sup>-H bonds are backed by the absorption at  $1420\text{ cm}^{-1}$  is the absorption of the methylene group ( $-\text{CH}_2-$ ) and absorption at daerah  $1396, 46\text{ cm}^{-1}$  indicates the presence of a methyl group ( $-\text{CH}_3$ ). The presence of absorption in the region  $1000\text{--}1300\text{ cm}^{-1}$  indicate the presence of ether groups (COC) to reinforce the notion that the methyl group is a methoxy group. From the analysis of the IR spectrum characteristic absorption peaks indicate that the product compound is 3,4-dimethoxybenzyl carboxylate acid.

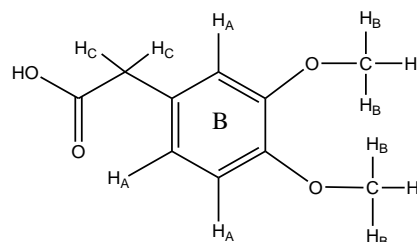


Fig. 1. The structure of 3, 4-dimethoxybenzyl carboxylic acid.

The results of  $^1\text{H-NMR}$  analysis showed the presence of peaks in the proton chemical shifts ( $\delta$ ) 7.15 ppm is the chemical shift of the characteristic peaks for the aromatic ring hydrogen atom. Aromatic protons in the region  $\delta$  6.0 to 8.0 is indicated by the appearance of a singlet, this was confirmed by the results of the integration is equivalent to three (H<sub>A</sub>) hydrogen atom. The peak at 4.2 ppm chemical shift signals an H atom of the methoxy group (H<sub>B</sub>) which is reinforced by the results of the integration of the equivalent of six hydrogen atoms. Absorption peaks at 3.8 ppm chemical shift with integration showed two hydrogen atoms is a signal of a methylene group (H<sub>C</sub>). There are three types of proton magnetic resonance. Chemical shifts ( $\delta$ ) at 7.15 ppm is stated three aromatic protons at the same chemical shift. Range

characteristics of aromatic hydrogen appears in the range from 6.5 to 8.5 ppm. Chemical shifts ( $\delta$ ) of protons of a methoxy group at 4.25 ppm indicated, the chemical shift at 11.0 is the proton of the hydroxy group and a carboxyl group at 3.87 ppm chemical shift correlated with the protons of the methylene. Based on spectral data on mass spectrometry, the molecular mass is 196. This corresponds to a molecular mass of 3,4-dimethoxyphenylacetic acid.

### G. Synthesis of

#### 3,4-dimethoxybenzyl-2,4-dihydroxyphenyl ketone

The 3, 4-dimethoxybenzyl-2,4-dihydroxyphenyl ketone (deoxybenzoin) obtained by acylation Friedel-Crafts reaction of 3,4-dimethoxybenzyl carboxylic acid (3,4-dimethoxy-phenylacetic acid) and resorcinol using boron trifluoride etherate ( $\text{BF}_3 \cdot \text{Et}_2\text{O}$ ) as catalyst and solvent. Acylation reaction for 1.5 hours at 60-80°C, obtained a solid product having a melting point of 171-172°C. Rendamen generated from this experiment is 76%. The spectrometry analysis with IR, GC-MS and  $^1\text{H}$  NMR spectrophotometer of this product was identical with chemdraw estimation of the chemical shift of 3,4-dimethoxybenzyl-2,4-dihydroxyphenyl ketone.

The formation of an acyl cation ( $\text{RC}=\text{O}$ ) from the reaction between 3,4-dimethoxybenzyl carboxylic acid and  $\text{BF}_3 \cdot \text{Et}_2\text{O}$ . The Acyl cation formed then reacts with resorcinol produce 3,4-dimethoxybenzyl-2,4-dihydroxyphenyl ketone as shown in under reaction

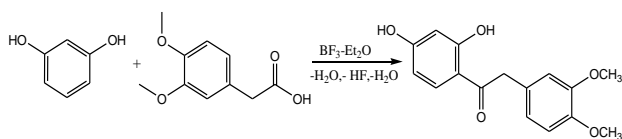


Fig. 2. The reaction synthesis of 3,4 dimethoxybenzyl-2,4-dihydroxyphenyl ketone from 3,4 - dimethoxyphenyl acetic acid and resorcinol.

Infrared spectra of this product showed that this product was 3,4-dimethoxybenzyl-2,4-dihydroxyphenyl ketone compound. This proved by the disappearance of broad O-H hydroxyl stretch absorption at  $3448.72 \text{ cm}^{-1}$  indicating acylation process between 3,4-dimethoxyphenylacetic acid and resorcinol have been done. The product seen several changes including the loss of carboxylate bands at wavenumber region ( $\nu$ )  $3400 \text{ cm}^{-1}$  which shows that the starting material 3,4-dimethoxybenzyl carboxylic acid has undergo substituted electrophilic reaction with resorcinol to 3,4 dimethoxybenzyl-2,4-dihydroxyphenyl ketone. This was confirmed by the appearance of a sharp absorption band at wave number  $1712.79 \text{ cm}^{-1}$  which shows the absorption of carbonyl groups Absorption band at wave number  $1512.19 \text{ cm}^{-1}$  indicates that the double bond derived from an aromatic core ( $1450\text{-}1600 \text{ cm}^{-1}$ ). Uptake in the area of  $2839.22 \text{ cm}^{-1}\text{-}2939.52 \text{ cm}^{-1}$  indicated the presence of vibration Csp<sup>3</sup>-H bonds are backed by the absorption at  $1420 \text{ cm}^{-1}$  is the absorption of the methylene group (-CH<sub>2</sub>-) and absorption at daerah  $1396, 46 \text{ cm}^{-1}$  indicates the presence of a methyl group (-CH<sub>3</sub>). The presence of absorption in the region  $1000\text{-}1300 \text{ cm}^{-1}$  indicate the presence of ether groups (COC) to a methoxy group.

There are five types of magnetic environment of the

protons. Chemical shift ( $\delta$ ) appear at two magnetic field correspond to the six aromatic protons in ring A and B. Chemical shift ( $\delta$ ) at 6.9 ppm indicated three proton of ring A, and 6.45 ppm indicated two proton of ring B. One aromatic proton from ring A at 7.8 ppm. Chemical shift ( $\delta$ ) of methoxy group indicated by 3.85 ppm, than chemical shift at 4.2 ppm correlated to proton of methylene.  $^1\text{H}$ -NMR spectrum of 3,4 dimethoxybenzyl-2,4-dihydroxyphenyl ketone showed significant difference with the spectrum of the 3,4-dimethoxybenzyl carboxylic proton. Peaks at chemical shifts ( $\delta$ ) 11 ppm which indicates the hydroxyl group of the carboxyl protons at 3,4-carboxylic dimethoxybenzyl lost after the product is formed.

$^{13}\text{C}$ -NMR spectroscopic analysis gives an overview of the molecular carbon skeleton synthesis product compounds that will strengthen the results of the analysis of the IR spectrum and  $^1\text{H}$ -NMR.  $^{13}\text{C}$ -NMR spectra of these appear in the absorption signal of the carbonyl carbon, aryl and methoxy. Carbon with oxygen as carbonyl signal appeared at 202.4 ppm. Carbon with oxygen as alcohol aromatic signals appeared at 102.48 ppm and 108.25 ppm. Carbon for ether methoxy-OCH<sub>3</sub> appeared at 55.45 ppm and 55.48 ppm. Methylene carbon appeared at  $\delta = 43.67$  ppm.

Based on the spectra data of mass spectrometer, the molecular mass of this product is 288. This is likely to be the molecular mass of 3,4-dimethoxybenzyl-2,4-dihydroxyphenyl ketone.

## IV. CONCLUSION

Preparation of 3, 4-dimethoxybenzyl cyanide basic ingredients of eugenol can be done through the stages of eugenol methylation reaction, isomerization, oxydation, reduction, halogenation and nitrilisation. Methylation process of eugenol produced methyleugenol from as much as 89.78%. Isomerization of methyleugenol yield methylisoeugenol 87.24 %. 3, 4-dimethoxybenzaldehyde obtained from the oxidation process of methylisoeugenol as much as 85.36 %. Reduction of 3, 4-dimethoxybenzaldehyde using  $\text{NaBH}_4$  produces 3, 4-dimethoxybenzyl alcohol as much as 98%. Halogenated 3, 4-dimethoxybenzyl alcohol produces 3,4-dimethoxybenzylchloride which reacted further to 3,4-dimethoxybenzylcyanide without prior separation as much as 89.5 % with a purity of 99.24%. Acylation of 3,4-dimethoxyphenylacetic acid was resulted 3,4-dimethoxybenzyl-2,4-dihydroxyphenyl ketone 76% at melting point 171-172 °C.

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