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# Iron (III) chloride, Aluminium chloride and Zinc chloride as Catalysts in the Synthesis of Tetrahydropentagamavunon-0

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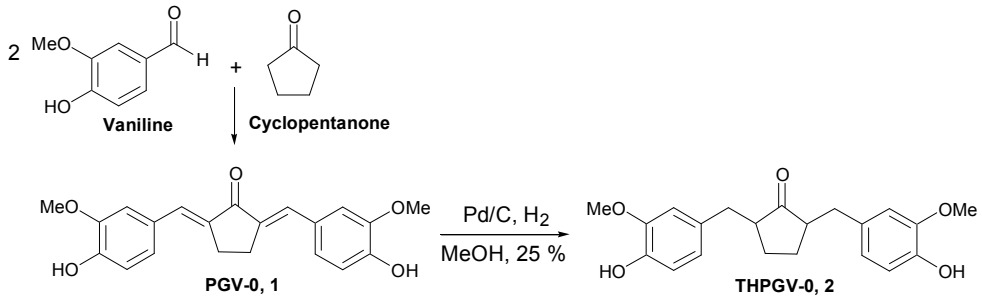
**Abstract**

Tetrahydropentagamavunon-0 (THPGV-0), an analog of tetrahydrocurcumin, has chemical name as 2,5-bis(4'-hydroxy-3'-methoxybenzyl)-cyclopentanone. It can be synthesised from pentagamavunon-0 (PGV-0, 2,5-bis(4'-hydroxy-3'-methoxybenzylidene)-cyclopentanone). The hydrogenation reaction was applied in this synthesis and it was catalysed by palladium on carbon which yielded THPGV-0 in moderate yield. In seeking for a cheap catalyst, the different catalysts was applied in this hydrogenation reaction. The reaction condition was changed by using different catalysts other than palladium on carbon. Among Palladium/carbon (Pd/C) 10 %, Iron (III) chloride ($FeCl_3$), Aluminium chloride ($AlCl_3$) dan Zinc chloride ($ZnCl_2$) only reaction catalysed by Pd/C gave THPGV-0. While others gave products like the side products when Pd/C used. Structure elucidation was carried out by using spectroscopic method. The obtained products from the reaction by using $FeCl_3$, and $AlCl_3$ as catalysts are 1,3-bis(4'-hydroxy-3'-methoxybenzyl)cyclopentane and 2,5-bis(4'-hydroxy-3'-methoxybenzyl) cyclopentanol and by using $ZnCl_2$ as catalyst is 1,3-bis(4'-hydroxy-3'-methoxybenzyl)cyclopentane.

**Keywords:** Iron (III) chloride, Aluminium chloride, zinc chloride, tetrahydropentagamavunon-0

## 1. Introduction

Curcumin as one of the very popular isolated natural product has been investigated as antibacterial (Naz *et al.*, 2010), antioxidant (Jayaprakash *et al.*, 2002), antiinflammatory (Kohli *et al*, 2005), anticancer (Wilken *et al.*, 2011), and antidiabetic (Konatham *et al*., 2010). Tetrahydrocurcumin (THC) which known as one of curcumin metabolites also has been reported to have a good biological activities for example as antibacterial (Singh and Jain, 2012). While Pentagamvunon-0 (PGV-0, 2,5-bis(4'-hydroxy- 3'-methoxybenzylidene)-cyclopentanone), a curcumin analog, has been succesfully synthesised from vaniline and cyclopentanone (Scheme 1) and patented by Faculty of Pharmacy, Gadjah Mada University, Indonesia. Its activity as antibacterial and anti fungi also have been evaluated (Sardjiman, 2000). And tetrahydropentagamavunon-0 (THPGV-0) itself is an analog of THC also has been synthesised by Ritmaleni and Simbara (2010). The synthesis of THPGV-0 was done by using the hydrogenation reaction on PGV-0 at room temperature and catalysed by Palladium/carbon (Pd/C).




Scheme 1. Synthesis of THPGV-0

Catalysts that have been used in the synthesis of THC are Pd/C (Mori et al., 2006) and $AlCl_3$ (Koltunov et al., 2004). Although, hydrogenation by using Pd/C as catalyst is the most popular one but because of its high price, the exploration of catalysts used for this type of reaction is possible. In industrial perspective, using iron as catalyst in

hydrogenation proses is interesting one as we know iron is widely spread in the earth that make the price is very cheap. Rangheard (2010) has used iron in the heterogeneous hydrogenation of olefins and alkynes. Hydrogeantion of α,β-unsaturated amide by using $AlCl_3$ as catalyst has been done by Koltunov (2004). Matsuura (1968) has used $ZnCl_2$ as catalyst in the hydrogenation process of coal.

Organometalic catalysts are used tremendously in the field of organic synthesis like in the hydrogenation reaction where Pd, Pt, Ru and Ni are widely used. However, they are expensive for us in Indonesia and most are highly toxic where needed to remove to a very low ppm levels before subjected to the pharmaceutical application. In this THPGV-0 hydrogenation reaction, the exploration of Iron (III) chloride ($FeCl_3$), Aluminium chloride ($AlCl_3$) dan Zinc chloride ($ZnCl_2$) as catalysts used in this type of hydrogenation is the aim of this project.

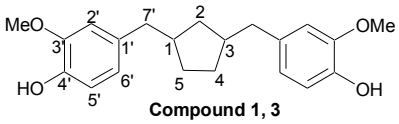
## 2. Experiment

PGV-0 was obtained from the laboratory of Synthetic Organic Chemistry, Faculty of Pharmacy, Universitas Gadjah Mada, Yogyakarta, Indonesia. PGV-0 was synthesised from vaniline and cyclopentanone at room temperature. All the data has been confirmed according to the publihed ones.

### *2.1 Synthesis of THPGV-0 by using Palladium/carbon (Pd/C) 10 %, Iron (III) chloride ($FeCl_3$), Aluminium chloride ($AlCl_3$) dan Zinc chloride ($ZnCl_2$)*

To round bottom flask, PGV-0 **(**250,0 mg; 0,710 mmol) in methanol (3 mL) was hydrogenated by hydrogen gas in baloon over different catalyst at room temperature for 2 - 7 hours (Palladium/carbon (Pd/C) 10 %, 2 hours; Iron (III) chloride ($FeCl_3$), 2 hours; Aluminium chloride ($AlCl_3$); 2 hours; Zinc chloride ($ZnCl_2$), 7 hours). The reaction was an autoindicator reaction where indicating by colour changing form yellow to colourless. Then the mixture was filtered and the solvent was evaporated by using rotavapor. The products were separated by flash column Chromatography and purified by recrystallisation. Structure elucidation was carried out by using spectroscopic method (IR, H-NMR, C-NMR, MS).

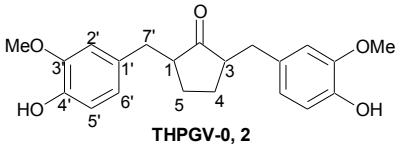
### *2.1 Structure elucidation of compound 1*




**Compound 1, 3**

White crystals 3.7 % (Pd/C), 0.4 % ($FeCl_3$), dan 0.3 % ($AlCl_3$), m.p. 106.8 – 108.4 °C (EtOH : $H_2O$ = 2 : 1), IR (vmaks, cm-1, KBr): 2939.5 and 2850.3 (C-H, *stretching*, aliphatic); 1440.9 ($CH_2$, *bending*); 798.2 (C-H, *bending*) $^1$H-NMR (500 MHz, ppm, $CDCl_3$): δ 6.81 (2H, dd, J = 1.95 Hz, J = 8.4 Hz, H6′-Ph x 2); δ 6.65 (2H, *d*, J = 1.95 Hz, H5'-Ph x 2); δ 6.64 (2H, d, J = 8.4 Hz, H2′-Ph x 2); δ 5.45 (2H, s, -OH x 2); δ 4.12(1H, dd, J = 14.24 Hz, J = 7.15 Hz, H7′-a); δ 3.86 (6H, *s*, -OCH3 x 2); δ 2.51 (1H, d*d*, J= 7.15 Hz, J = 2.6 Hz, H7′-b); δ 2.20 (1H, dd, J = 14.25 Hz, J = 7.15 Hz, H7′-d); δ 2.09-2.06(1H, m, H7′-c); δ 1.84-1.81(1H, m, H5-a), δ 1.79-1.77 (1H, *m*, H4-b); δ 1.72-1.67 (1H, *m*, H2-a); δ 1.44-1.41 (1H, *m*, H3); δ 1.33-1.28 (1H, *m*, H4-a); δ 1.27-1.24 (1H, *m*, H1); δ 1.22-1.19 (1H, *m*, H5); δ 0.91-0.83 (1H, *m*, H2): $^{13}$C-NMR (125 MHz, ppm, $CDCl_3$): δ 146.39 (s); δ 143.71 (s); δ 134.34 (s); δ 121.50 (d); 121.45 (d); δ 114.18 (d); δ 111.52 and 111.45 (d); δ 56.03 (q); δ 42.48 (d) 42.18 (d); δ 40.80 (t); 40.35 (t); δ 38.16 (t); 32.58 (t); 31.40 (t): MS (EI-MS, m/z) $C_{21}H_{26}O_4$ **:** 342 (18), 137 (100), 122 (10), 77 (4)

### *2.2 Tetrahydropentagamavunon-0 (THPGV-0)*

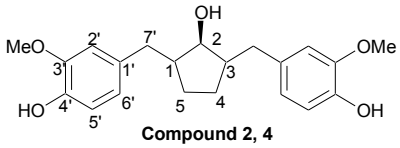



**THPGV-0, 2**

White crystals 40 % (katalis Pd/C) while with other catalyst, the products were not obtained (0 %), m.p. 122.4 – 123.7 °C (EtOH : $H_2O$ = 2 : 1): $^1$H-NMR (500 MHz, ppm, aceton): 7.52 (2H, *s*, -OH x 2); δ 6.75 (2H, *d*, J= 1.8 Hz, H2′-Ph x 2); δ 6.70 (2H, *m*, H5′-Ph x 2); δ 6.58 (2H, *m*, H6′-Ph x 2); δ 3.76 and 3.78 (6H, *s*, -OCH3 x 2); δ 2.97 and 2.85 (2H, *dd*, J= 4.25 and J=13.45 Hz, H7′-a) [lit. (Ritmaleni and Simbara, 2010; δ 2.97 and 2.85 (2H, *dd*, J= 4.25 and J=13.45 Hz, H7′-a)) δ 2.97 and 2.85 (2H, *dd*, J= 4.25 and J=13.45 Hz, H7′-a); δ 2.35 and 2,46 (2H, *dd*, J= 9.15 and J=13.45 Hz, H7′-b); δ 2.26 (2H, *dddd*, J= 4.30; J= 7.95; J= 9.15; and J=11.65; H2&5); δ 1.89 and 1.80 (2H, *dddd*, J=3.05; J= 5.50; J= 7.95; and 9.15 Hz, H3&4a); δ 1.39 and 1,55 (2H, *dddd*, J=3.05; J= 5.5; J= 9.15; and J= 11.60 Hz, H3&4b).

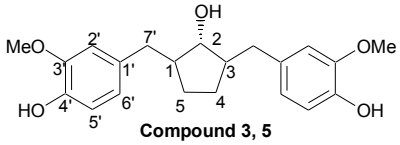
*2.3 Structure elucidation of compound 2*




**Compound 2, 4**

Colourless oil, 10.9 % (katalis Pd/C) while with other catalyst, the products were not obtained (0 %). IR (vmaks, cm-1, KBr): 3422.5 (O-H); 2940.9 (C-H, *stretching*, aliphatic); 1452.0 ($CH_2$, *bending*): $^1$H-NMR (500 MHz, ppm, CDCl3): δ 6,80 (2H, d, J = 7.75 Hz, H2′-Ph x 2); δ 6.70 (2H, dd, J = 7.75 Hz, J = 1.25, H6′-Ph x2); δ 6.67 (2H, d, J = 1.25 Hz, H5′-Ph x 2); δ 5.55 (2H, *s*, -OH-Ph x 2); δ 3.84 (6H, s, -$OCH_3$ x 2); δ 3.79 (1H, m, -OH); δ 2.75 (2H, *dd*, J = 13.6 Hz, J = 7.15 Hz, H7′-a and H7′-b); δ 2.59 (2H, *dd*, J = 13.6 Hz, J = 7.15 Hz, H7′-c and H7′-d); δ 2.08-2.11(2H, m, H4-a and H4-b); δ 1.73-1.77 (2H, m, H2 and H5); δ 1.53-1.56 (2H, m, H3-a and H3-b): $^{13}$C-NMR (125 MHz, ppm, $CDCl_3$) : δ 146.51 (s); δ 143.77 (s); δ 133.78 (s); δ 121.28 (d); δ 114.36 (d); δ 111.47 (d); δ 75.25 (d); δ 56.01 (q); δ 47,59 (t); δ 35,98 (t); δ 28,28 (t), MS (EI-MS, m/z) $C_{21}H_{26}O_5$ **:** 358 (40), 137 (100), 122 (13), 79 (18)

*2.3 Structure elucidation of compound 3*




**Compound 3, 5**

Colourless oil, 29.3 % (catalyst $FeCl_3$), 16.6 % (catalyst $AlCl_3$) while with other catalyst, the products were not obtained (0 %). IR (vmaks, cm-1, KBr): 2938.8 (C-H, *stretching*, aliphatic); 1430.5 ($CH_2$, *bending*); 795,2 (C-H, *bending*) $^1$H-NMR (500 MHz, ppm, $CDCl_3$): δ 6.81 (2H, d, J = 7.8 Hz, H2′-Ph x 2); δ 6.70 (2H, d, J = 1.95 Hz, H5′-Ph x 2); δ 6.67 (2H, dd, J = 7.8, J = 1.95 Hz, H6′-Ph x 2); δ 5.49 (2H, *s*, -OH x 2); δ 4.12 (1H, dd, J = 14.25 Hz, J = 7.15 Hz, H7′-b); δ 3.85 (6H, *m*, -$OCH_3$ x 2); δ 2.76-2.82 (1H, *m*, H7′-a); δ 2.61-2.65 (1H, m, H7′-c); δ 2.43-2.56 (2H, *m*, H2 and H5); δ 2.12-2.18 (1H, *m*, H7′-d); δ 1.88-1.94 (1H, m, H3-a); δ 1.67-1.75 (1H, *m*, H3-b); δ 1.46-1.52 1,25 (1H, *m*, H4-b); δ 1.19-1.23 (1H, *m*, H4-a): $^{13}$C-NMR (125 MHz, ppm, $CDCl_3$): δ 146.56 (s); δ 143.95 (s); 143.83 (s); δ 133.78 (s); 133.08 (s); δ 121.55 (s); 121.35 (d); δ 114,38 (d); 114.33 (d); δ 111.50 (d); 111.45 (d); δ 60,59 (d); δ 56,06 (q); 56,01 (q); δ 49.86 (d); 48.99 (d); δ 35,07 (t); δ 29.08 (t); 29.22 (t): MS (EI-MS, m/z) $C_{21}H_{26}O_5$ : 358 (40), 137 (100), 122 (13), 79 (18)

**3. Result and Discussion**

The hydrogenation reaction, that done in slightly different reaction condition, was done at room temperature for 2 – 7 hours and the results were monitored by TLC like figure below. Hydrogenation reaction by using Pd/C as catalyst gave four products, hydrogenation reaction by using $FeCl_3$ as catalyst gave two product, hydrogenation reaction by using $AlCl_3$ as catalyst gave two products and hydrogenation reaction by using $ZnCl_2$ as catalyst gave only one product. After all the reactions were stopped, only reaction with Pd/C that consumed all the PGV-0 and converted it into four different products and one of that is THPGV-0 (Rf = 0.60). On the other hand, the reaction with others were still remained with starting material PGV-0 although some PGV-0 converted to products which the same as those in the reaction with Pd/C as catalyst and these were coded as compound 1 (Rf = 0.69), compound 2 (Rf = 0.38) and compound 3 (Rf = 0.27).

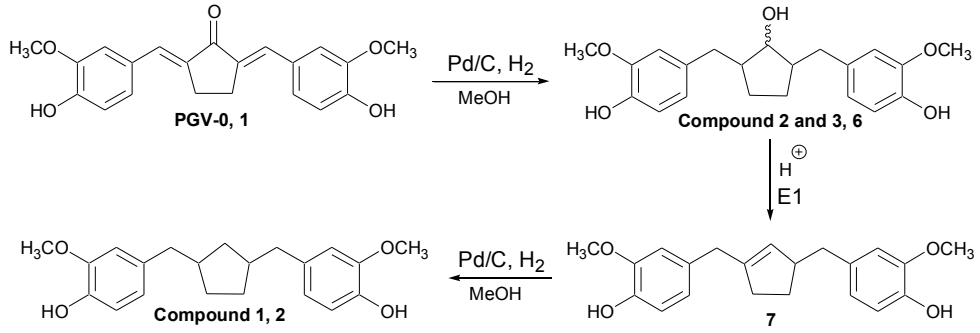
 

*3.1 Structure elucidation*

All the double bond on the benzylidene part of PGV-0 were succesfully hydrogenated. Althought not all PGV-0 converted to products indicating by a very small amount of product obtained but they showed that it does not have the double bond anymore. Ketone which can normally not be reduced to alcohol even to alkane, in this case it was. All the data were confirmed to spectra of IR, H-NMR, C-NMR and MS.

*3.2 Synthesis of THPGV-0 by using Pd/C as catalyst*

Hydrogenation on PGV-0 has been performed at room temperature in methanol for two hours and yield four compounds, one as THPGV-0 and others as side products. The mechanism for products' formation are followed the scheme below.

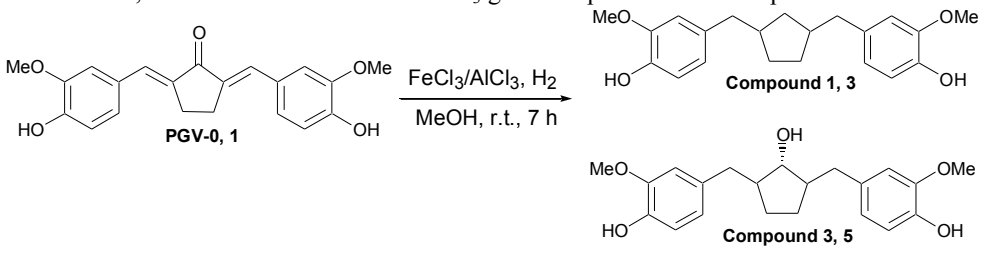



Scheme 2. Formation of compound 1 by using Pd/C as catalyst

For the side products, compound 1 is assigned as 1,3-bis(4′-hydroxy-3′-methoxy-benzyl)cyclopentane. This compound was obtained probably because of the catalytic reduction of PGV-0. It was not only reduced the α,β-unsaturated ketone to form the alkane but also reduce ketone to secondary alcohol as happened also in some cases when 10 % Pd/C used in protic solvent for hydrogenation. (Monarch Catalyst PVT LTD, 2013) As in the mechanism above, acid catalysed dehydration of secondary alcohol gave alkene **7** through the E1 mechanism. By catalytic hydrogeantion, the alkene **7** can be easily hydrogenated to alkane **2**. And compound **2** and **3** are assigned both as 2,5-bis(4′-hydroxy-3′-methoxy-benzyl)cyclopentanol. These two molecules are prochiral as cannot be assigned as R or S but known that hydrogenation happen from si-face (compound 2) and re-face (compound 3) of ketone.

*3.3 Synthesis of THPGV-0 by using $FeCl_3$ and $AlCl_3$ as catalysts*

For this type of reaction, when PGV-0 reacted with $FeCl_3$ gave compound 1 and 3 as products.

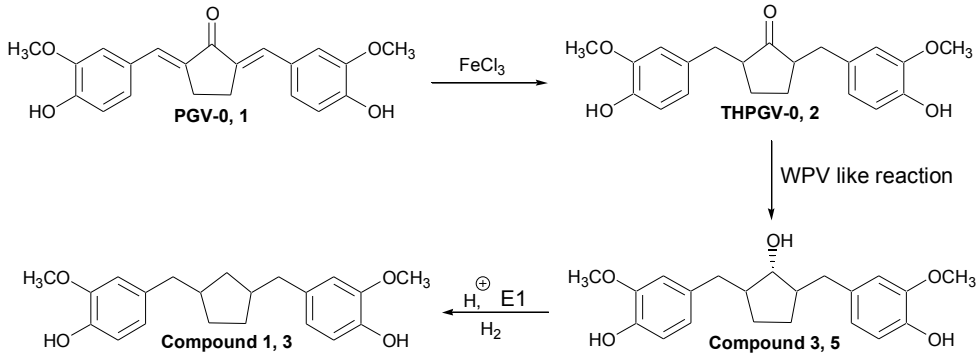



Scheme 3. Hydrogenation of PGV-0 by using $FeCl_3$ and $AlCl_3$ as catalyst

These two products are named as 1,3-bis(4′-hydroxy-3′-methoxy-benzyl)cyclopentane in 0.4 % yield and 2,5-bis(4′-hydroxy-3′-methoxy-benzyl)cyclopentanol. The last compound were obtained from the re-face attack of hydrogen in

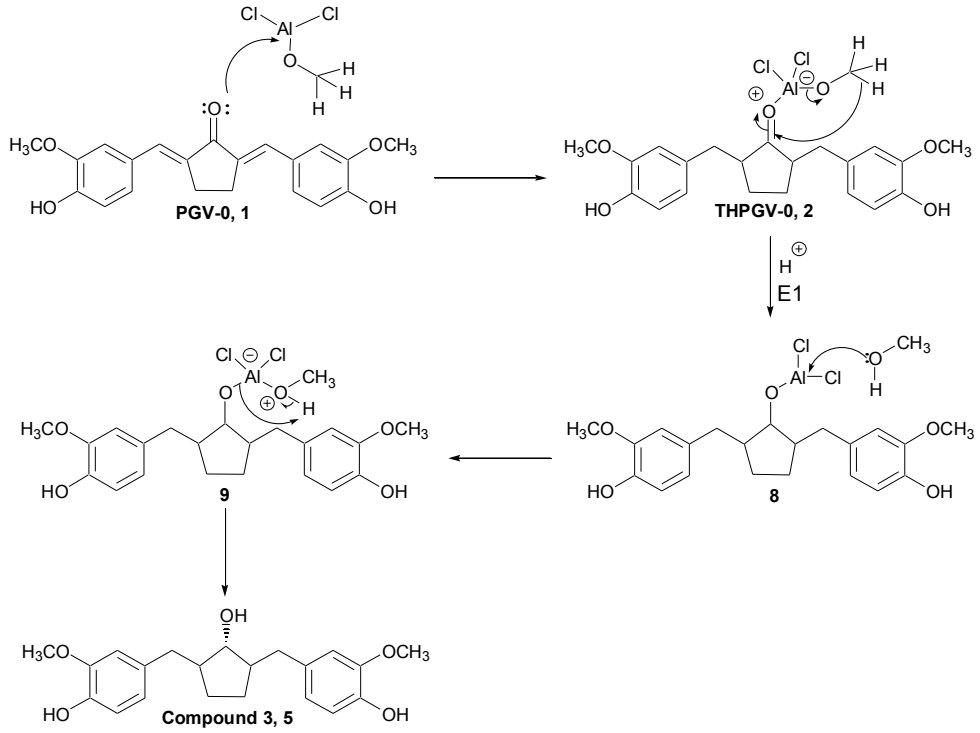
 

29.3 % yield and this is the main product in this reaction condition. Phua et al. (2009) used $FeCl_3$ alone as catalyst (5 mol %) for hydrogenation but no reaction was obtained. But, Mazin et. al. in 1984 has succesfully hydrogenated N-benzylideneaniline to N-benzylaniline by $H_2$ by using Iron as catalyst precursor as reviewed by Bolm et al. (2004). As proposed mechanism in section 3.2, this result showed that the benzylidene part on PGV-0 was reduced following the literature result informing ketone. Afterward, the ketone was reduced to the secondary alcohol eventually underwent the Meerwein-Pendorf-Verley (MPV) like reaction. The alcohol, like happened in the reaction with Pd/C, will be reduced to alkane.




Scheme 4. Formation of compound 1 and 3 by using $FeCl_3$ as catalyst

No difference with reaction by using $FeCl_3$, the reaction with $AlCl_3$ also produced two spots by TLC judgement, compound 1 and 3, the main product in this reaction condition is 2,5-bis(4′-hydroxy-3′-methoxy-benzyl)cyclopentanol in 16.6 % yield which the result from the re-face attack of hydrogen and 1,3-bis(4′-hydroxy-3′-methoxy-benzyl)cyclopentane in very tiny amount, 0.3 % yield. This was obtained probably as a result of Meerwein-Pendorf-Verley (MPV) like reaction. The MPV proposed mechanism could be explained like the scheme below. $AlCl_3$ and methanol complex is coordinated to the ketone funcyional group of PGV-0, hydrogen is transfered through the catalytic cycle and the product is formed as secondary alcohol.



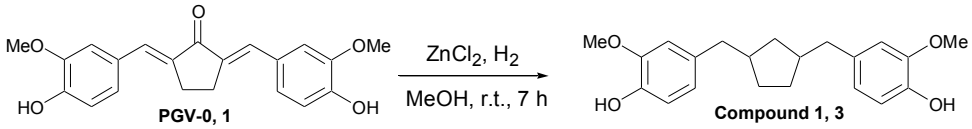

Scheme 5. Proposed mechanism of MPV-like reaction

This result was in-line with published report by Nakazawa and Itazaki (2011) where α,β-unsaturated ketone was hydrogenated by metal iron complex to unsaturated and saturated alcohol. It means the ketone group can be reduced to alcohol also.

*3.4 Synthesis of THPGV-0 by using $ZnCl_2$ as catalyst*

$ZnCl_2$ is rarely used as catalyst for hydrogenation reaction. Once, Zinc chloride anhydrous was used as an efficient and new catalyst for conversion of ketones and aldehydes to corresponding *gem*-dihydroperoxides by aqueous hydrogen peroxide (30%) at room temperature with excellent yields and notable reaction times. (Khosrazi & Kazemi, 2012) When $ZnCl_2$ was applied to this kind of hydrogenation, this reaction condition not only can reduce the α,β-unsaturated carbonyl but also can reduce the carbonyl group on PGV-0 resulting the alkane. The reaction mechanism is still a mystery. 1,3-(4′-hydroxy-3′-methoxy-benzyl)cyclopentana could not be isolated yet and this is the only product obtained.




Scheme 6. Hydrogenation of PGV-0 by using $ZnCl_2$

All the catalysts performed that they can work as catalyst to reduce PGV-0 to 1,3-(4′-hydroxy-3′-methoxy-benzyl)cyclopentane. The mechanism is proposed as hydrogenation on alkene went first and continued by reduction of ketone to alkane in which the alcohols were intermediate. Good Lewis acid contacted with hydrogen to work as catalyst for this kind of hydrogenation reaction. In this reaction, 10 mol % $FeCl_3$ and $AlCl_3$ were that worked as catalyst. However, the reaction did not lead to the completion. The reason is still unclear but might be due to the lower concentration of catalysts used. $ZnCl_2$ also can work as catalyst although only small amount of compound 1 formed and this can not be isolated yet.

## 4. Conclusion

The use of different catalyst in the synthesis of THPGV-0 has shown that only palladium on carbon that can form THPGV-0. $FeCl_3$, $AlCl_3$ and $ZnCl_2$, when reacted with hydrogen, they all reduced the double bond on benzylidene part and ketone of PGV-0 and these are the same compounds as side products when hydrogenation catalysed by Pd/C .

## 5. Acknowledgement

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**Supporting information**

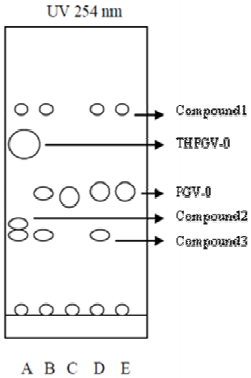


Figure 1. TLC stained by $KMnO_4$, $CHCl_3$: EtOAc (5:1) A = Hydrogenation of PGV-0 by using Pd/C as catalyst, B = Hydrogenation of PGV-0 by using $FeCl_3$ as catalyst, C = PGV-0 (starting material), D = Hydrogenation of PGV-0 by using $AlCl_3$ as catalyst, E = Hydrogenation of PGV-0 by using $ZnCl_2$ as catalyst.

 

Table 1. Yield, Rf and melting point of hydrogentaion products

| **Compound No.** | **% Yield** | | | | | **Rf** | **m.p. (°C)** |
|---|---|---|---|---|---|---|---|
| | **Pd/C** | **$FeCl_3$** | **$AlCl_3$** | **$ZnCl_2$** | **Form** | | |
| 1 | 3.7 | 0.4 | 0.3 | tiny | White crystals | 0.69 | 106.8 – 108.4 |
| (THPGV-0) | 40 | - | - | - | White crystals | 0.60 | 122.4 – 123.7 |
| PGV-0 | PGV-0 | PGV-0 | PGV-0 | PGV-0 | Yellow powder | 0.45 | - |
| 2 | 10.9 | - | - | - | Colourless Oil | 0.38 | - |
| 3 | tiny | 29.3 | 16.6 | - | Colourless Oil | 0.27 | - |

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