

## SINTESIS METIL-3,5-DIKLORO-2-HIDROKSIBENZOAT DAN TURUNAN 2-HIDROKSIBENZAMIDA DARI METIL SALISILAT

Heni Sulistyawati  
12/339526/PPA/03953

### INTISARI

Telah dilakukan sintesis metil-3,5-dikloro-2-hidroksibenzoat dan turunan 2-hidroksibenzamida dari bahan dasar metil salisilat. Senyawa metil-3,5-dikloro-2-hidroksibenzoat disintesis melalui reaksi klorinasi metil salisilat menggunakan gas  $\text{Cl}_2$ . Hasil klorinasi metil salisilat selanjutnya direaksikan dengan 4-nitroanilin menggunakan katalis NaOMe dan pelarut toluena dengan pemanasan pada suhu  $50\text{ }^{\circ}\text{C}$  menghasilkan senyawa 3,5-dikloro-2-hidroksi-N-(4'-nitrofenil)benzamida (**A**). Metil salisilat direaksikan dengan amonia, dimetilamina dan dietilamina dengan pengadukan selama 36 jam pada temperatur kamar, secara berurutan menghasilkan senyawa 2-hidroksibenzamida (**B**), 2-hidroksi-N,N-dimetilbenzamida (**C**) dan 2-hidroksi-N,N-dietilbenzamida (**D**). Metil salisilat direaksikan dengan anilin menghasilkan senyawa 2-hidroksi-N-fenilbenzamida (**E**). Senyawa **E** disintesis dengan 4 cara yaitu ke-1: dilakukan dengan pengadukan selama 24 jam, ke-2: refluks dalam pelarut toluena selama 9 jam menggunakan katalis NaOMe, ke-3: menggunakan *microwave* selama 10, 600, 1200 dan 1800 detik, ke-4: pemanasan pada suhu  $60\text{ }^{\circ}\text{C}$  menggunakan katalis asam (*amberlite*) dengan pelarut dan tanpa pelarut metanol. Senyawa 2-hidroksi-N-(4'-nitrofenil)benzamida (**F**) disintesis dengan merefluks metil salisilat dengan 4-nitroanilin dalam pelarut toluena selama 48 jam menggunakan katalis NaOMe. Senyawa 2-hidroksibenzohidrazida (**G**) dan 2-hidroksi-N'-fenilbenzohidrazida (**H**) disintesis dengan merefluks metil salisilat dan amina (hidrazin hidrat dan fenil hidrazin) dalam pelarut metanol selama 5 jam. Produk hasil sintesis dikarakterisasi dengan spektrometer FTIR, GC-MS dan  $^1\text{H-NMR}$ .

Hasil penelitian menunjukkan bahwa senyawa metil-3,5-dikloro-2-hidroksibenzoat telah berhasil disintesis dengan rendemen 9,92%. Senyawa **B**, **C**, **D** dan **G** telah berhasil disintesis dengan rendemen berturut-turut sebesar 61,44%, 30,14%, 29,68% dan 35,59%. Sedangkan senyawa **A**, **E** dan **F** belum berhasil disintesis karena amina yang digunakan bersifat kurang reaktif sehingga tidak dapat berperan sebagai nukleofilik yang kuat. Senyawa **H** berhasil disintesis akan tetapi kurang optimal, dengan persentase luas puncak pada data GC adalah 0,25%.

Kata kunci: Metil salisilat, klorinasi, 2-hidroksibenzamida

## **SYNTHESIS OF METHYL-3,5-DICHLORO-2-HYDROXYBENZOATE AND 2-HYDROXYBENZAMIDE DERIVATIVES FROM METHYL SALICYLATE**

Heni Sulistyawati  
12/339526/PPA/03953

### **ABSTRACT**

Synthesis of methyl-3,5-dichloro-2-hydroxybenzoate and 2-hydroxybenzamide derivatives from methyl salicylate have been carried out. Methyl-3,5-dichloro-2-hydroxybenzoate synthesized through chlorination reaction of methyl salicylate using  $\text{Cl}_2$ . Product of chlorination from methyl salicylate reacted with 4-nitroaniline using NaOMe as catalys and toluene as solvent and heated at temperature  $50\text{ }^\circ\text{C}$  to produce 3,5-dichloro-2-hydroxy-N-(4'-nitrophenyl)benzamide (**A**). Methyl salicylate reacted with ammonia, dimethylamine and diethylamine under stirring for 36 hours in room temperature, to produce 2-hydroxybenzamide (**B**), 2-hydroxy-N,N-dimethylbenzamide (**C**) and 2-hydroxy-N,N-diethylbenzamide (**D**), respectively. Reaction of methyl salicylate with aniline produce 2-hydroxy-N-phenylbenzamide (**E**). The compound **E** can be synthesized with 4 methods, (1) stirring for 24 hours, (2) refluxing in toluene for 9 hours using NaOMe as catalyst, (3) reacting with microwave in various times (10, 600, 1200 and 1800 second) and (4) heating the compound at  $60\text{ }^\circ\text{C}$  using amberlite catalyst, under solvent free condition and methanol solvent. The 2-hydroxy-N-(4'-nitrophenyl)benzamide (**F**) synthesized from methyl salicylate with 4-nitroaniline by refluxing in toluene for 48 hours and using NaOMe as catalyst. The 2-hydroxybenzohidrazide (**G**) and 2-hydroxy-N'-phenylbenzohidrazide (**H**) synthesized by refluxing methyl salicylate and amine (hydrazine and phenylhydrazine) in methanol for 5 hours. All products were characterized by using FTIR, GC-MS and  $^1\text{H-NMR}$  spectrometer.

The result obtained from this research showed that the methyl-3,5-dichloro-2-hydroxybenzoate has been successfully synthesized as 9.92% yield. The compound **B**, **C**, **D** and **G** have been synthesized and obtained 61.44%, 30.14%, 29.68% and 35.59% yield, respectively. The compound **A**, **E** and **F** have not successfully synthesized, due to the less reactivity of amine as nucleophilic, while the compound **H** has been synthesized less successfully, result as shown by GC-0,25%.

Keyword : Methyl salicylate, Chlorination, 2-hydroxybenzamide