

Percobaan Sintesis Senyawa 4(4fluorofenil)2,6dimetil1,4dihidropiridin 3,5dikarbaldehida dan 2,6dimetil4fenil1,4 dihidropiridin3,5-dikarbaldehida = Experimental Synthesis of 4(4fluorophenyl)2,6dimethyl1,4dihydropyridine-3,5dicarbaldehyde and 2,6dimethyl4phenyl1,4dihydropyridine3,5-dicarbaldehyde

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Abstrak

Senyawa turunan 1,4-dihidropiridin dikembangkan hingga saat ini karena memiliki aktivitas yang cukup banyak diantaranya memberikan efek vasodilatasi otot polos dan jantung, antioksidan, antituberkulosis, antitumor, dan antimikroba. Senyawa turunan 1,4-dihidropiridin masih perlu dilakukan penelitian lebih lanjut terhadap gugus-gugus substituenya untuk memperoleh aktivitas yang optimal. Hal tersebut karena 1,4-dihidropiridin menunjukkan aktivitas antimikroba yang dapat meningkat dengan adanya substitusi aromatik pada C4 serta substitusi gugus penarik elektron pada C3 dan C5. Oleh karena itu dilakukan sintesis dan elusidasi senyawa turunan 1,4-dihidropiridin yang tersubstitusi gugus fenil dan fluorofenil pada posisi 4. Sintesis dilakukan dalam 2 tahap yaitu untuk menghasilkan senyawa pertama yaitu 4(4fluorofenil)2,6dimetil1,4dihidropiridin3,5dikarbaldehida dan senyawa kedua yaitu 2,6dimetil4fenil1,4dihidropiridin3,5dikarbaldehida. Sintesis tahap 1 melalui reaksi Hantzsch menggunakan metode refluks selama kurang lebih 4-5 jam. Monitoring reaksi menggunakan kromatografi lapis tipis. Pemurnian kedua senyawa dilakukan dengan kromatografi kolom dan rekristalisasi. Hasil elusidasi struktur menyatakan bahwa hasil sintesis tahap 1 senyawa pertama yaitu 3,5diethyl 2,6dimetil4fenil1,4dihidropiridin3,5dikarboksilat dan senyawa kedua yaitu 3,5diethyl 4(4fluorofenil)2,6dimetil1,4dihidropiridin3,5dikarboksilat. Nilai rendemen yang didapatkan dari senyawa pertama dan kedua berturut-turut yaitu 51,50 % dan 39,25%. Profil senyawa yang disintesis pada tahap pertama senyawa pertama yaitu serbuk putih kekuningan dengan titik lebur $150 \pm 2^\circ\text{C}$ dan senyawa dua yaitu serbuk berwarna putih dengan titik lebur $158 \pm 2^\circ\text{C}$. Percobaan sintesis tahap 2 dilakukan dengan reaksi reduksi DIBAL-H dengan pelarut tetrahidrofur dan diklorometana serta dilanjutkan percobaan dengan reduktor NaBH_4 . Senyawa tahap 2 dinyatakan tidak terbentuk karena profil elusidasi yang sama dengan senyawa tahap 1.

.....Compounds of 1,4-dihydropyridine derivatives were developed until now because they have quite a lot of activities including smooth muscle and heart vasodilation effects, antioxidants, antituberculosis, antitumor, and antimicrobial. The 1,4-dihydropyridine derivative compound still needs further research on its substituent groups to obtain optimal activity. This is because 1,4-dihydropyridine shows antimicrobial activity that can increase with aromatic substitution at C4 and substitution of electron-withdrawing groups at C3 and C5. Therefore, the synthesis and elucidation of 1,4-dihydropyridine derivative compounds substituted with phenyl and fluorophenyl groups at position 4 were carried out. The synthesis was carried out in 2 stages, to produce the first compound, namely 4-(4-fluorophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarbaldehyde and the second compound, namely 2,6-dimethyl-4-phenyl-1,4-dihydropyridine-3,5-dicarbaldehyde. Stage 1 synthesis via Hantzsch was reacted by reflux for 4-5 hours. The compound was monitored by thin-layer chromatography. Purification of both compounds was carried

out by column chromatography and recrystallization. The results of structural elucidation stated that in stage 1 the first compound was 3,5-diethyl 2,6-dimethyl-4-phenyl-1,4-dihydropyridine-3,5-dicarboxylate and the second compound was 3,5-diethyl 4-(4-fluorophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate. The yield values obtained from the first and second compounds were 51,50% and 39,25%, respectively. The profile of the first compounds synthesized in the first stage was a yellowish white powder with a melting point of $150 \pm 2^{\circ}\text{C}$ and compound two was a white powder with a melting point of $158 \pm 2^{\circ}\text{C}$. Stage 2 experiments were carried out with the reduction reaction of DIBAL-H with tetrahydrofuran and dichloromethane solvents and continued experiments with NaBH_4 reductant. The stage 2 compound was declared not formed due to the same elucidation profile as the stage 1 compound.