

炔基苯甲酰胺衍生物的液相合成

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摘要 液相合成兼容了固相合成可快速简便地对产物进行分离纯化以及溶液相合成可在均相条件下进行反应和用常规手段对反应中间体进行分析检测的优点,用聚乙二醇(PEG)4000作为可溶性聚合物支持体,通过醚键将[1, 3, 5]氯三嗪连接在PEG上制备成PEG支持的新型的可溶性聚合物试剂I. 该试剂在N-甲基吗啉的作用下与对碘苯甲酸反应生成相应的活性酯中间体II, 该中间体继而在Pd(II) / Cu(I)共催化下与端炔发生Sonogashira偶联反应得到另一中间体III, 在伯胺或仲胺的存在下进行胺解反应得到炔基苯甲酰胺衍生物IV. 由于PEG具有柔韧的长链而不会对链端连接的反应中心的活性产生影响,因此该反应在均相体系中进行,不仅反应条件温和,产率良好,而且反应还具有良好的选择性. 实验发现,芳香胺和非芳香胺均可获得良好的反应结果,但芳香胺所需的反应时间较长;伯胺和仲胺都是良好的胺解试剂,但使用仲胺时产率较低;N原子亲核试剂比O原子亲核试剂具有更大的反应性. 此外,PEG 4000支持的[1, 3, 5]氯三嗪试剂没有强烈的刺激性,可长期储存使用.

关键词 聚乙二醇 苯甲酰胺 P 三嗪 P 氯化物 钯 铜

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Liquid-Phase Synthesis of Alkynyl Benzamide Derivatives

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Abstract Based on the strategy of liquid-phase synthesis, polyethylene glycol (PEG) 4000 was used to load [1,3, 5] chlorotriazine by etherification, forming a novel soluble polymer-supported reagent I which reacted with 4-io-dobenzoic acid in the presence ofN- methylmorpholine to generate the activated ester n. From the ester n, the intermediate IIIwas produced by the Sonogashira coupling reaction with terminal alkynes under the co-catalysts of Pd(OAc)2 and CuI. These final products of alkynyl benzamide derivatives IV were obtained by the cleavage of support with primary or second amines. Due to the long and flexible chain of PEG, PEG supported substrates could maintain high reactivity so that the reactions involved could be carried out under mild conditions in good yields. Both aliphatic and aromatic amines were effective, but aromatic amine took longer time to yield the corresponding amide. Both primary and secondary amines were readily aminolyzed, but secondary amine gave relatively low yield. It appeared that the aminolysis had good selectivity since the intermediate IIIwas more readily attacked by N-nucleophiles than by O- nucleophiles. Besides, PEG 4000 supported reagent I could be more conveniently operated than [1,3,5]-chlorotriazine without the strong irritation.

Key words POLYGLYCOL BENZAMIDE P TRIAZINE P CHLORINE COMPOUNDS PALLADIUM COPPER

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