膦、胂叶立德的化学与应用26:含全氟烷基胂叶立德的合成及 水解反应研究

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摘要 报道溴化α-呋喃甲酰基)甲基三苯基申(1)在无水碳酸钾存在下,以无水二氯甲烷作溶剂,保持0-5℃下与2-全氟炔酸甲酯(2)反应,高产率地得到加合产物4-(α-呋喃甲酰基)-2-三苯基胂基-3-全氟烷基-3-丁烯酸甲酯(3)。加合产物3在V(甲醇):V(水)=9:1溶液中分别于室温、回流和封管120℃三种条件下反应,高产率地得到4-全氟烷基-6-(α-呋喃基)-2-吡喃酮(4)和4-(α-呋喃甲酰基)-3-全氟烷基-3-丁烯酸甲酯(5)。化合物4和5可以通过柱层析分离;化合物5为一对Z,E异构体,它们不能通过柱层析分离,但其比例可以由^1HNMR估算得到。研究还发现硅胶对该反应具有催化作用,提出并讨论了反应机理。关键词 胂叶立德 膦 水解 呋喃Ρ 吡喃酮P 碳酸钾 质子磁共振谱法 反应机理

Chemistry and applications of phosphonium and arsonium ylides 26: Synthesis of arsonium ylides containing perfluoroalkyl group and study on their hydrolysis

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Abstract In the presence of K~2CO~3, reaction of (α -furoyl) methyl- arsoniumbromide (1) with methyl 2-perfluoroalkynoates (2) in methylene chloride at 0-5 $^{\circ}$ C afforded the adduct --- methyl 4-(α -furoyl)-2-triphenylarsoranylidene-3-perfluoroalkyl-3-butenoates (3) in high yield. Hydrolysis of 3 in V(CH~3OH): V (H~2O)=9: 1 methanolic solution at room temperature, 70 $^{\circ}$ C and 120 $^{\circ}$ C in a sealed tube respectively, 4-perfluoroalkyl-6-(α -furoyl)-2-pyranones (4) and methyl 4-(α -furoyl)-3-perfluoroalkyl-3-butenoates (5) were obtained in excellent yield. Compounds 4 and 5 could be separated by column chromatography. 5 is a mixture of Z- and E- isomers which couldn't be separated by column chromatography, but the ratio of Z- and E- isomers could be estimated by $^{\circ}$ 1H NMR. The catalytic hydrolysis of compound 3 with silica gel and the mechanisms for the formation of products are also discussed in this paper.

Key words PHOSPHINE HYDROLYSIS FURAN P PYRANONE P POTASSIUM CARBONATE PROTON MAGNETIC RESONANCE SPECTROMETRY REACTION MECHANISM

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