

论文

氯霉素之合成研究(七)二氯乙酸甲酯合成方法的研究

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摘要:

研究了应用氰化物为接触剂,使三氯乙醛(可能是其加成物,三氯乙醛缩甲醇)与无水碳酸钠在甲醇中的混悬液作用脱氯化氢,一步生成二氯乙酸甲酯。反应温度60—64℃;三氯乙醛滴加速度必须尽可能地快,使整个反应在半小时内结束;加水分出油层;洗涤,干燥,分馏。收率能达到70%以上。但当反应液中氰化物浓度减低时,三氯乙醛水解分裂生成氯仿的趋势即逐渐增加。由于,当先将三氯乙醛与部分甲醇制成三氯乙醛缩甲醇,然后再加到无水碳酸钠和含有氰化钾的甲醇中反应时,结果也同样地好;同时由于,这个反应速度很快,而在反应所用条件下三氯乙醛缩甲醇分解出游离三氯乙醛的可能性极小;我们提出了反应机制的新解释。

关键词:

STUDIES RELATED TO THE SYNTHESIS OF CHLORAMPHENICOL. VII STUDIES ON THE SYNTHESIS OF METHYL DICHLORACETATE

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Abstract:

Cyanide catalysed dehydrohalogenation of chloral (probably chloral methanolate) by a suspension of anhydrous sodium carbonate in methanol was exploited as the simplest way for a one step synthesis of methyl dichloracetate. Chloral (1 mol.) was dropped as rapidly as possible into a suspension of anhydrous sodium carbonate (0.52 mol.) in methanol (110 ml.) containing 4 per cent its weight of potassium cyanide (5.9 g) when an energetic reaction set in almost instantly. The temperature of the reaction was kept at 60-64°. By finishing the reaction off in within about half an hour's time then cooled rapidly down to 20°, the oily ester layer was separated, washed with water, dried and fractionated. A yield of over 70% theory was obtained. However, hydrolytic fission of chloral into chloroform was shown to be competitive, when the concentration of cyanide ion present was reduced. Since equally good results could be obtained by first forming chloral methanolate, and then adding it to anhydrous sodium carbonate in methanol containing potassium cyanide; and since the reaction was rapid, and decomposition of chloral methanolate into chloral and methanol seemed improbable under the experimental conditions used, a new explanation of the mechanism of reaction was suggested as following:

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