

ω -氯氟烷基醇, 烯烃及环氧化物的合成

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摘要 本文报道在引发剂存在下, ω -氯氟烷基碘与烯丙基化合物($\text{CH}_2=\text{CH}-\text{CH}_2\text{X}$, $\text{X}=\text{OH}, \text{OAc}$)及烯基化合物 $\text{CH}_2=\text{CH}-\text{OAc}$ 发生自由基加成反应, 生成相应的加成产物 $\text{Cl}(\text{CF}_2)_n\text{CH}_2\text{CHICH}_2\text{OH}$ ($2a\sim d$), $\text{Cl}(\text{CF}_2)_n\text{CH}_2\text{CHICH}_2\text{OAc}$ ($3a\sim d$)和 $\text{Cl}(\text{CF}_2)_n\text{CH}_2\text{CHIOAc}$ ($4a\sim d$), 产率较好. $2a\sim d$ 用 LiAlH_4 脱碘生成 $\text{Cl}(\text{CF}_2)_n\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ ($5a\sim d$), 反应条件温和. $2a\sim d$ 与 $\text{KOH}-\text{CH}_3\text{OH}$ 反应, 主要得到醇 $\text{Cl}(\text{CF}_2)_n\text{CH}=\text{CHCH}_2\text{OH}$ ($6a\sim c$), 若 $2a\sim d$ 与 NaOH -水溶液反应则得到环氧丙烷化合物. 在少量 HOAc 存在下, 异丙醇溶剂中, 锌粉与 $2a\sim d$ 和 $3a\sim d$ 反应得到消除产物 $\text{Cl}(\text{CF}_2)_n-\text{CH}_2\text{CH}=\text{CH}_2$ ($8a\sim d$). $4a\sim d$ 与锌反应, 再经 $\text{KOH}-\text{CH}_3\text{OH}-\text{H}_2\text{O}$ 水解得到 $\text{Cl}(\text{CF}_2)_n(\text{CH}_2)_2\text{OH}$ ($10a\sim d$).

关键词 [醇](#) [环氧化物](#) [加成反应](#) [全氟烷基碘化物](#)

分类号 [0621](#)

Synthesis of ω -chloroperfluoroalkylated alcohols, olefines and epoxides

GUO CAIYUN, WANGSHANDI

Abstract The free radical addition of $\text{Cl}(\text{CF}_2)_n\text{I}$ ($n=2,4,6,8$) $1a$ to allylic compounds ($\text{CH}_2=\text{CH}-\text{CH}_2\text{X}$, $\text{X}=\text{OH}, \text{OAc}$) and vinyl acetate in the presence of initiator gave the corresponding adducts $\text{Cl}(\text{CF}_2)_n\text{CH}_2\text{CHICH}_2\text{OH}$ ($2a\sim d$), $\text{Cl}(\text{CF}_2)_n\text{CH}_2\text{CHICH}_2\text{OAc}$ ($3a\sim d$) and $\text{Cl}(\text{CF}_2)_n\text{CH}_2\text{CHIOAc}$ ($4a\sim d$) with good yields. The deiodination of $2a\sim d$ to $\text{Cl}(\text{CF}_2)_n\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ ($5a\sim d$) by LiAlH_4 went smoothly. The alcohol $\text{Cl}(\text{CF}_2)_n\text{CH}=\text{CHCH}_2\text{OH}$ $6a\sim c$ was formed from the reaction of $2a\sim d$ with KOH -methanol, however, the epoxide was obtained with aqueous NaOH . Zinc reduction of $2a\sim d$ and $3a\sim d$ in isopropanol in the presence of small amounts of acetic acid afforded the elimination products $\text{Cl}(\text{CF}_2)_n-\text{CH}_2\text{CH}=\text{CH}_2$ ($8a\sim d$). Contrarily the reduction of $4a\sim d$ with Zinc, followed by hydrolysis with KOH -methanol-water gave clearly $\text{Cl}(\text{CF}_2)_n(\text{CH}_2)_2\text{OH}$ ($10a\sim d$).

Key words [ALCOHOL](#) [EPOXIDE](#) [ADDITION REACTION](#)

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