

## 论文

## 胆甾醇衍生的几种含硫化合物的合成

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## 摘要:

1. 由 $3\beta$ -溴代- $\Delta^5$ -胆甾烯与 $\beta$ -氨基乙醇反应,分离得到三种产物: $3\beta$ -(2'-羟乙基氨基)- $\Delta^5$ -胆甾烯; $3\alpha$ -(2'-羟乙基氨基)- $\Delta^5$ -胆甾烯与6-(2'-羟乙基氨基)-3:5-环胆甾烷。2.  $3\beta$ -(2'-羟乙基氨基)- $\Delta^5$ -胆甾烯经亚硫酸氯作用后,与异硫脲反应可获得 $3\beta$ -(2'-异硫脲代乙氨基)- $\Delta^5$ -胆甾烯。继续水解得 $3\beta$ -(2'-巯乙基氨基)- $\Delta^5$ -胆甾烯。它们可分别视为N取代的半胱胺或 $\beta$ -氨基异硫脲的衍生物。3. 由 $3\beta$ -巯基- $\Delta^5$ -胆甾烯与 $\beta$ -溴代乙胺作用,制得 $3\beta$ -(2'-氨基乙基巯基)- $\Delta^5$ -胆甾烯。后者可视为S取代的半胱胺衍生物。 $3\beta$ -(3'-邻苯二甲酰亚胺丙基代巯基)- $\Delta^5$ -胆甾烯与 $3\beta$ -(3',4',5'-三甲氧基苯甲酰巯基)- $\Delta^5$ -胆甾烯也由类似方法合成。

关键词:

## SYNTHESIS OF SOME SULFUR CONTAINING STEROIDS DERIVED FROM CHOLESTEROL

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## Abstract:

Some sulfur containing steroids derived from cholesterol were synthesized for the study of their protective effect against ionizing radiation on mammals. On refluxing cholesteryl bromide with ethanolamine followed by suitable treatment three isomeric products were isolated in the form of  $3\beta$ -(2'-hydroxyethylamino)- $\Delta^5$ -cholestene ( $I_a$ ),  $3\alpha$ -(2'-hydroxyethylamino)- $\Delta^5$ -cholestene hydrochloride ( $II_b$ ), and 6-(2'-hydroxyethylamino)-3:5-cyclocholestane hydrochloride ( $III_b$ ) from the reaction mixture. However, when the two were allowed to react at 200°C in a bomb, only  $I_a$  and  $II_b$  were obtainable. The *i*-structure is assigned to  $III_b$  on the basis of its dextrorotatory power, negative bromine unsaturation test, and ready conversion to cholesteryl aniline on treatment with aniline. Both  $I_a$  and  $II_a$  when reacted with *p*-nitrobenzoyl chloride, were easily converted to the corresponding N, O-di-(*p*-nitrobenzoyl) derivatives, which showed maxima at 1730—1720  $cm^{-1}$  and 1640  $cm^{-1}$  in the infrared region, characteristic for the ester C=O and amide C=O groups respectively. Furthermore, neither  $I_b$  nor  $II_b$  gave cholesteryl aniline on treatment with aniline, so both  $I$  and  $II$  must be  $C_3$ -substituted 2'-hydroxyethylamino derivatives of  $\Delta^5$ -cholestene, but differing in configuration. By the difference in the rotatory power,  $I$  and  $II$  are assigned as  $3\beta$ - and  $3\alpha$ -orientations respectively, according to Bernstein's rule.  $I_a$ , when treated with thionyl chloride, gave  $3\beta$ -(2'-chloroethylamino)- $\Delta^5$ -cholestene hydrochloride (IV), which on reacting with thiourea, yielded  $3\beta$ -(2'-isothiuroniumethylamino)- $\Delta^5$ -cholestene hydrochloride (V). V gave  $3\beta$ -(2'-mercaptoethylamino)- $\Delta^5$ -cholestene hydrochloride (VI) on alkaline hydrolysis followed by acidification with hydrochloric acid. VI showed positive iodine-azide test and absorption band at 2510  $cm^{-1}$  in the infrared region, both characteristic for sulfhydryl group. Thiocholesterol, on treatment with  $\beta$ -bromoethylamine in ethanolic sodium ethoxide solution, gave  $3\beta$ -(2'-aminoethylmercapto)- $\Delta^5$ -cholestene (VII), which was isolated in the form of nicotinic acid salt. Similarly,  $3\beta$ -(3'-phthalimidopropylmercapto)- $\Delta^5$ -cholestene (VIII) was obtained by the reaction of thiocholesterol with  $\gamma$ -phthalimidopropylbromide in ethanolic sodium ethoxide solution. Thiocholesterol, when treated with 3, 4, 5-trimethoxybenzoyl chloride gave  $3\beta$ -(3', 4', 5'-trimethoxybenzoylmercapto)- $\Delta^5$ -cholestene (IX).

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