SYNTHESIS OF MONOISOPROPYLIDELAGOXYLIS AND STUDY OF PHYSICO-CHEMICAL PROPERTIES

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Annotation: *Lagochilin diterpenoid was isolated from Lagochilus inebrians according to the method known in the literature, and its monoisopropylidene derivative was synthesized on the basis of Lagochilin. physico-chemical properties and spectral properties were studied*

Keywords: *lagochilus inebrians, lagochilus pubescens, , infrared spectroscopy, lagoxiline, monoisopropylidene lagoxiline, thin layer chromatography*

INTRODUCTION.

The species Lagochilus has long been known for its healing properties, i.e. as a hemostatic agent, and it is one of the most popular, effective hemostatic medicinal plants of the East. Decoctions and infusions based on the Lagochilus plant have been used in folk medicine to stop various bleedings. The pharmacology of Lagochilus plant species was studied at the pharmacology departments of the Kuban, Samarkand, Andijan medical universities. Among them, aqueous and alcoholic decoctions of Lagochilus inebrians have been found to have physiologically active properties such as sedative, hypotensive, sedative, anti-shock, anti-radiation and anti-allergic (anti-allergic) in addition to hemostatic properties.

The most common species of Lagochilus plants is the Lagochilus inebrians plant. The main active ingredient of the Lagochilus plant is the diterpenoid lagochilin, which is a four-atom alcohol. The plant contains a small amount of lagoxilin, mainly in the form of various acetyl derivatives. When it is extracted with alkali, they are hydrolyzed to free lagoxilin. [1].

Based on the method known in the literature, in order to isolate Lagoxilin from the plant Lagochilus inebrians, the plant was crushed and sprayed with a 20% solution of alkali (sodium hydroxide), and after drying, it was extracted in a dichloroethane solvent. After the dichloroethane solution was concentrated by filtration, it was cooled in a refrigerator and the technical lagoxiline crystals were isolated. Technical lagoxilin was recrystallized and purified in acetone. The average yield of lagoxilin was around 1.7-1.8%. The chemical formula of lagoxilin is presented in Fig. 1.

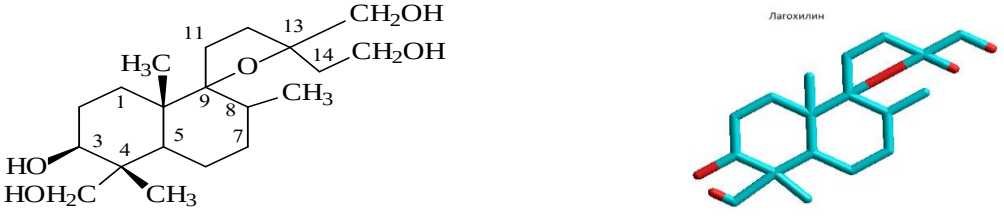
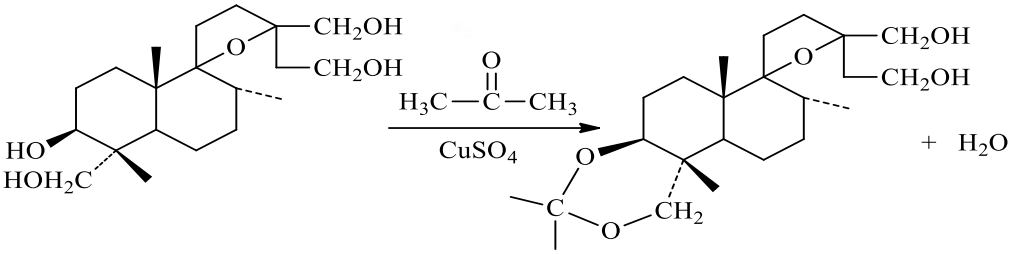


Figure 1. Chemical and conformational structure of lagoxiline

ANALYSIS OF OBTAINED RESULTS.

We will try to obtain monoisopropylidene lagoxilin based on Lagoxilin. For this, Lagoxilin is dissolved in acetone, distilled water and concentrated sulfuric acid are added to pH=3-4, the mixture is placed in a separatory funnel and extracted with cyclohexane 4 times from 100 ml. The cyclohexane solutions were combined and washed with 5% sodium bicarbonate solution. It is neutralized and the solvent is removed, and the solution is washed in a column with silica gel in the ether-benzene 15:1 system. We mix monoisopropylidenelagoxylin coldigy in ether-benzene 15:1 system and put it in the column. We mix the fractions that came out in the column and perform chromatography in the tetrachloromethane-acetone 7:5 system. We mix the fractions with the same Rf value. We pour 3/2 of the solvents into the refrigerator. White crystal monoisopropylidenelagoxylin is obtained by filtering it. it is recrystallized in ether, for this we heat monoisopropylidenelagoxylin with ether until it dissolves in a reflux condenser, pour the solvent into the refrigerator and form a white crystal, filter it, wash it with absolute ether, and dry it to obtain monoisopropylidenelagoxylin. The synthesis of 3,18-O-isopropylidenelagoxyline when lagoxiline reacts with acetone in the presence of anhydrous copper sulfate is shown in Scheme 1.



Lagoxiline 3,18-O-isopropylidenelagoxiline **Scheme 1. Scheme of the synthesis of 3,18-O-isopropylidenelagoxyline**

The chemical and conformational formulas of monoisopropyledenlagoxylin are presented in Figures 2

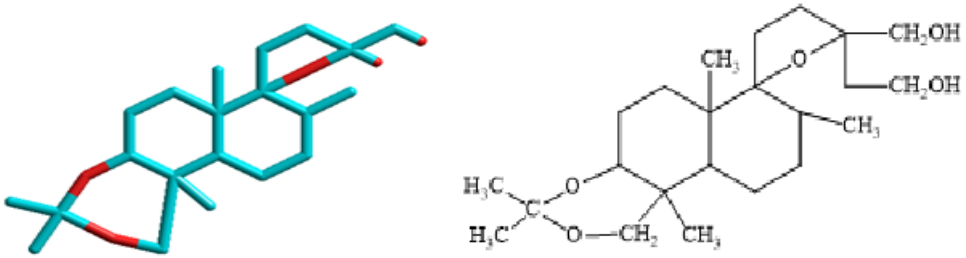


Fig 2. Chemical and conformational structure of monoisopropyladenlagoxyline

The IR spectrum of monoisopropylidenelagoxylin is presented in Figure 3

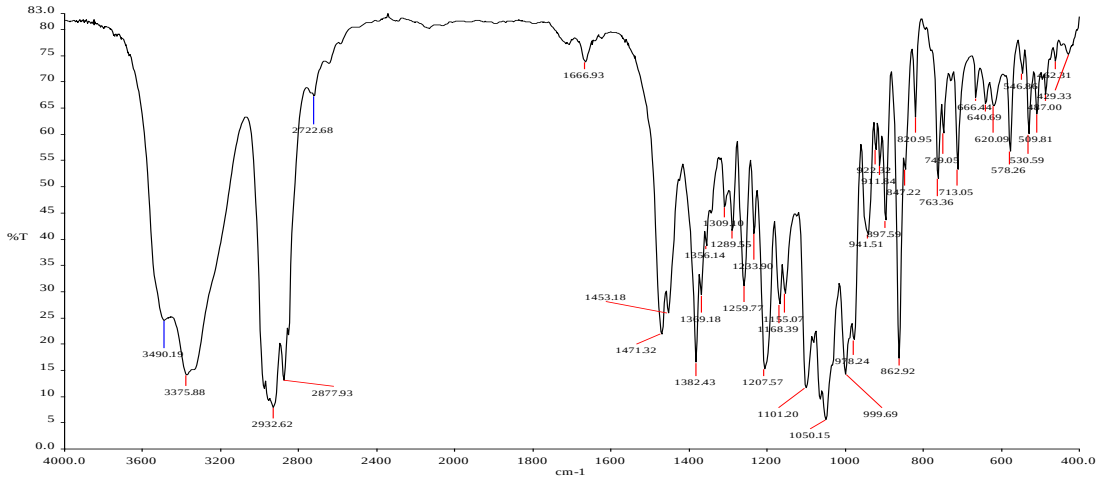


Figure 3. IR spectrum of monoisopropylidenelagoxylin

In the IK-spectrum of monoisopropylidene lagoxyline, the valence vibration frequencies of the ON groups in the molecule were observed in the intensive state at 3490, 3375 cm–1, the valence vibration frequencies of the CH_3, CH_2 groups appeared at 2932, 2878 cm–1, SN3 at 1471, 1453, 1382, 1369 cm–1 , CH_2, CH groups deformational vibration frequencies were observed, symmetric valence vibrations of epoxide ring at 1207 cm-1, frequencies characteristic of their asymmetric vibrations were observed at 941 cm-1, and deformational vibrations of this ring were observed at 863 cm-1. The valence vibration frequencies of C-O-C, C-OH bonds in the molecule at 1101-1050 cm–1 observed in the intense state are presented in Table 1.

Table 1

Physico-chemical parameters of Lagoxilin and Monoisopropyledenlagoxilin

№	Substances	Gross Formula and mol. weight	$T._{liquid}$. C^0	R_f^* (system)	$[\alpha]_D$ 0,5% water:ethanol 1:1	IR spectrum $см^{-1}$
1.	LG White crystal	$C_{20}H_{36}O_5$ 356	167-168	0,15 (I)	-	1053(-O-); 2938,(CH_3) 3336,(OH)
3	MIPL White crystal	$C_{23}H_{40}O_5$ 396	150-151	0,72 (III)	+12	1666(-O-);2932,(CH_3) 3490,(OH)

I. Tetrachloromethane-acetone 7:5 III.. Chloroform-acetone 9:1,

In short, the results of studying the composition and structure of the obtained compounds showed that ulra is an individual compound [1-2].

CONCLUSION

Monoisopropyledenlagoxylin was synthesized. physico-chemical properties and spectral properties were studied.

LITRATURE:

1.Акопов И.Э. Кровоостанавливающие растения. Ташкент «Медицина», 296,1981.

2.Акопов И.Э. О гемостатической эффективности лагохилина и лагохилина ацетата. В кн: Проблемы гемокоагуляции в экспериментальной и лечебно-профилактической работе. Под. ред. Акопова И.Э. Краснодар, 12, 1976.

3.Исламов Р. Дис... на степ.уч.степени канд.хим.наук., Ташкент, 142 с., 1988

4.Зайнутдинов У.Н. Дитерпеноиды растений рода Lagochilus // Дисс. докт. хим. наук. Т.1993. С.253.

5.Монография. Зайнутдинов. У.Н. Маулянов. С.А. Исламов. А.Х. // Lagochilus авлодига мансуб ўсимликлар кимёси, биологияси ва физиологик хоссалари.// Тошкент«Университет» 2019 Б.194

6.Монография. Исломов А.Х, Матчанов А.Д, Қурбанова А.Дж, Комилов Қ.У. //Лагохилиннинг ацетил ҳосилаларининг глицирризин кислотаси, унинг тузлари билан комплекс бирикмалари ва гемостатик фаоллиги // “Lesson Press”МЧЖ нашриёти, Тошкент-2022., 108 б.