

# International Journal of Engineering Sciences & Research Technology

(A Peer Reviewed Online Journal)  
Impact Factor: 5.164



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## ABSTRACT

The article studies the isolation of a phenolic alkaloid of 12-demethylregelin nature with the extraction of three percent leaves of *Colchicum kesselringii* stems with three percent acetic acid. The structure of the isolated alkaloid 12-demethylregelin was determined from spectral data and chemical transformations. It is shown that the selected alkaloid belongs to the group of homopaporphin series.

**KEYWORDS:** phenolic alkaloids, 12-demethylregelin, extraction, *Colchicum kesselringii*, homopaporphin, kesselringin, regecolin, O, O-diacetyl-12-demethylregelin.

## 1. INTRODUCTION

The Liliaceae family, which includes colchicine-containing plant species, is widely distributed throughout the globe. It has about 3000 species, mainly herbaceous plants, both annual and perennial, with bulb, tuber, corm or rhizome. A large number of them are of economic importance as medicinal, decorative, writing, fodder, insecticidal plants and cultivated [1].

Liliaceae produce a number of important physiologically active substances. Glucosides, saponins, alkaloids, polysaccharides, flavanoids, phytosterols, phytoncides, sterols, and other classes of organic compounds [2-4]. Moreover, the biosynthesis of a certain group of compounds is often characteristic of their individual genera or species.

Homopaporphyne alkaloids are unique compounds isolated from only a few species of plants of the lily family. Their main sources are two species of colchicum *Colchicum kesselringii* Rgl. and *Colchicum luteum*. [5]. Baker (sem (Liliaceae), growing in Central Asia. The main amount of homopaporphin alkaloids was isolated and studied by A. S. Sadykov et al. [6-7].

Our goal is to isolate alkaloids from the aerial parts of *C. kesselringii* Rgl., Which grows in Central Asia (Gissar district of the Republic of Tajikistan). To obtain the extract with alkaloids, 3% acetic acid was used. At the same time, 0.13% neutral alkaloids were obtained, of which colchicine was reliably identified. A mixture of alkaloids of the main phenolic nature found compounds that do not contain a tropolone ring.

As a result of studying the qualitative composition of the total alkaloids, it was found that the neutral alkaloids fraction in all plant species studied by us consists mainly of tropolone alkaloids (colchicine and others) and a small number of their photochemical isomers ( $\beta$ -lumicolchicine). The phenolic alkaloids fraction from the leaves and stems of *Kesselring colchicus* mainly includes 2-demethyl- $\beta$ -lumicolchicine, 3-demethylcolchicine predominates in corms and seeds. The fractions of phenolic alkaloids from corms and seeds of these plants also contain mainly 3-demethylcolchicine. The fraction of acidic substances in all plants consists exclusively of tropolon compounds of strong base a fraction in some plants [7, 8].

## 2. MATERIALS AND METHODS

The composition of the base  $C_{19}H_{25}O_4N$ , has t.m. 179-188<sup>0</sup>. 12-demethylregeline has absorption maxima in the UV spectrum at 248, 300 and 395 nm, in the IR spectrum there are absorption bands of the hydroxyl group ( $3465\text{ cm}^{-1}$ ), carbonyl group ( $1690\text{ cm}^{-1}$ ) and C = C bonds of the benzene ring ( $1470\text{ cm}^{-1}$ ).

[Alikulov, *et al.*, 8(10): October, 2019]  
ICTM Value: 3.00

Signals of the aromatic proton H-3 (1H, s, 6.45 ppm), the methoxyl group of the benzene ring (3H, s, 3.75 ppm), and the N-methyl group (3H) appear in the PMR spectrum of the base, s, 2.36 ppm)

In the mass spectrum of 12-demethylregelin, peaks of ions with  $m/e$  341 ( $M^+$ , 100%), 326, 298, 242, 241, 240, 228, 227, 213, 212, which are nitrogen-containing fragments, are detected. According to spectral data, 12-demethylregelin sharply differs from previously known isoquinoline bases, and it was not possible to attribute it to one of the groups of colchicids that we examined. According to spectral data, the compound was assigned to the group of homopaporphynses, the base of which is found in a number of colchicine-containing plants of the lily family, including in Kesselring colchicum.

UV spectra were recorded on an SF-4A spectrometer in methanol, IR spectra were recorded on a UR-10 double-beam spectrometer in KBr, and PMR spectra were recorded on a Varian XL-400 instrument in  $CDCl_3$ .

The individuality and authenticity of substances was controlled by BH methods. Radial BC on Filtrak paper was carried out using the following mobile phases: n-butyl alcohol - hydrochloric acid - water - 50: 7.5: 13.5 (system 1) and: p-butyl alcohol - 5%  $CH_3COOH$  (1: 1, organic part) (system 2). Stains of substances showed a modified Dragendorf reagent and iodine vapor.

### 3. Isolation of alkaloids

2.8 kg of dried crushed leaves of colchicum stems, harvested during flowering and flowering, were extracted five times with three percent acetic acid. The alkaloids fraction was obtained according to the previously described method.

The number of fractions of alkaloids were:

Alkaloids of a neutral nature - 3.64 g (0.13%)

Phenolic alkaloids - 0.56 g (0.02%)

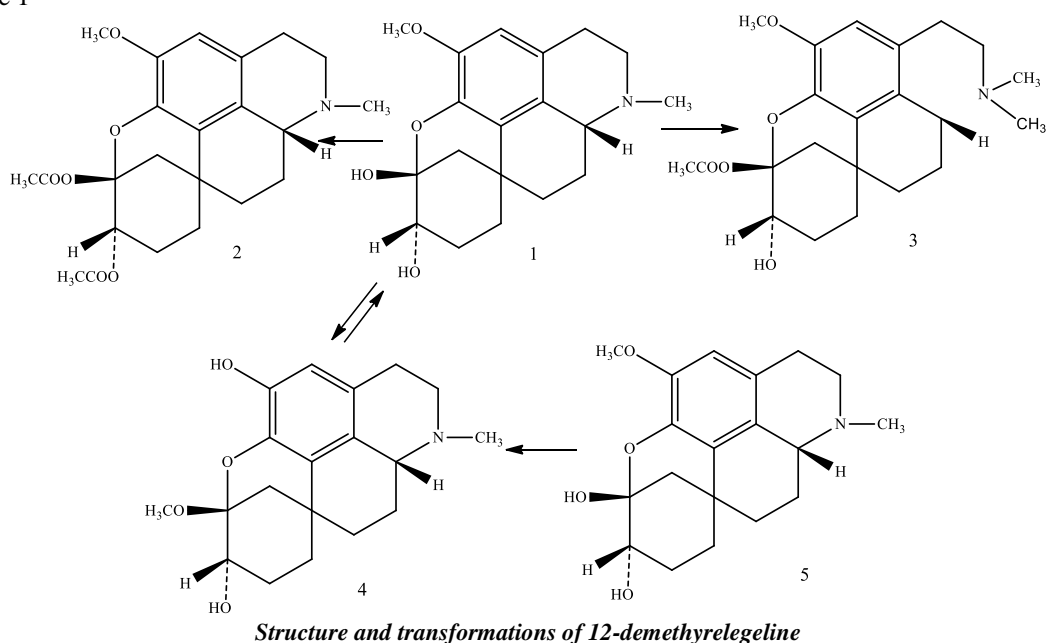
Alkaloids of an acidic nature - 0.84 g (0.03%)

Alkaline insoluble bases - 4.72 g (0.17%)

Phenol-based alkaloids - 2.76 g (0.10%)

The sum of fractions of alkaloids - 12.52 g (0.45%)

Scheme 1



#### 4. RESULTS AND DISCUSSION

Regeline (5, scheme) ( $R_f$  0.67, system 1) in the fraction of phenol-basic alkaloids was identified by its authentic sample. Unknown compounds with  $R_f$  0.16, 0.56, 0.75 were found in the base fraction.

Isolation of 12-demethylregeline. (1, scheme) In the mother liquor after crystallization of regeline, in addition to this alkaloid ( $R_f$  0.67, system 2), compounds with  $R_f$  0.16, 0.56 and 0.75 are contained. The first two of them were chromatographically identified with isoregelinone and isorecolin, respectively, while treating this mixture with water, 0.06 g of a substance with  $R_f$  0.75 was isolated, which differs in physical constants and spectral data from known alkaloids. This new base turned out to be 12-demethylrecolin. T.ml. 220-2220 from the water.

O, O-diacetyl-12-demethylregeline (2, Scheme). 0.03 g of the base was dissolved in 2 ml of freshly distilled acetic anhydride, and one drop of concentrated sulfuric acid was added to the solution. After 2 hours, after completion of the reaction, the excess of acetic anhydride was removed by adding a small amount of methanol to the reaction mixture and evaporation. Then the reaction product was dissolved in water. The aqueous solution was made basic with ammonia and extracted with chloroform.

IR spectrum: 1750, 1740  $\text{cm}^{-1}$  (2OCOCH<sub>3</sub>).

PMR spectrum: 2.02; 2.00 ppm (2OCOCH<sub>3</sub>).

O, O-diacetyl derivative of 12-demethylregelin was isolated.

O, O, N-triacetyl-12-demethylregelin (3, scheme). To a solution of 0.03 g of base in 2 ml of acetic anhydride was added 1 g of freshly melted acetic acid sodium and the mixture was heated one day at 45-500. Excess acetic anhydride was removed by adding methanol and evaporating the mixture, the remaining substance was dissolved in water and extracted with chloroform.

O, O, N-triacetyl derivatives of 12-demethylregelin were isolated.

IR spectrum: 1645 (N-COCH<sub>3</sub>), 1750 (2OCOCH<sub>3</sub>)  $\text{cm}^{-1}$ .

O-methyl-12-demethylregeline (4, Scheme). 0.10 g of regeline was dissolved in 3 ml of methanol and an excess of a saturated solution of diazomethane in petroleum ether was added to the solution with shaking. After completion of the reaction, the solvent was distilled off, and the remaining substance was dissolved in chloroform and water. Separating the chloroform and water layers of the mixture, the chloroform part was washed twice more with water, dried over sodium sulfate and the solvent was distilled off.

O-methyl-12-demethylregeline (5, Scheme), identical to regeline and O-methylregeline, was isolated.

12-demethylregeline is distinguished by regequolchine arrangement of the hydroxyl group of ring D [8].

#### 5. CONCLUSION.

- As a result of the studies, alkaloids fractions of a neutral, acidic and phenolic nature and alkali-insoluble bases were obtained with extraction of crushed leaves of colchicum stems with three percent acetic acid.
- 12-demethylregelin was isolated from phenol-basic fractions and identified by paper chromatography. The structure of 12-demethylregelin was determined from spectral data and chemical transformations. The isolated alkaloid belongs to the group of homoporphyrin series

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