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SYNTHESIS OF A NEW DERIVATIVE OF AMINOCOLCHAMINE WITH PROPENYLETHYNYLCARBINOL

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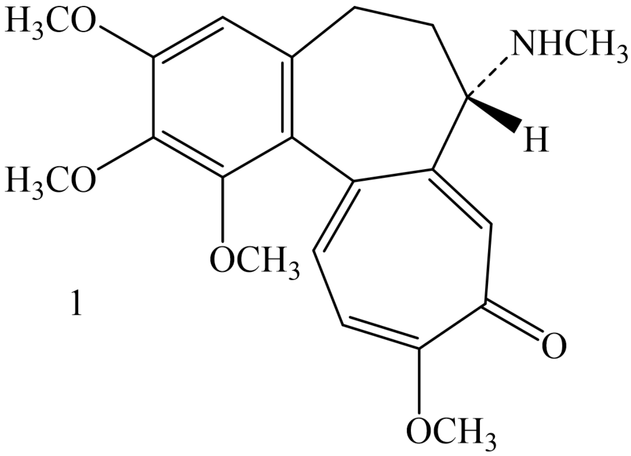
Abstract:synthesized 4-(aminocolchamino N/propenyl-1-butyne-2) carbinol. The structure of the synthesized compound was confirmed by IR and NMR spectral data.

Keywords: Aminocolchamine, propenylethynylcarbinol, 4-(aminocolchamino N/propenyl-1-butyne-2) carbinol.

Introduction

It is known that the introduction of groups containing an acetylene bond into the molecule of medicinal substances significantly reduces their toxicity. Since such work has not previously been carried out in the field of aminocolchamine alkaloids, we have synthesized a derivative of aminocolchamine (1) with propenylethynylcarbinol (2) [3]. The starting compounds for the synthesis of acetylene derivatives of aminocolchamine. (1)



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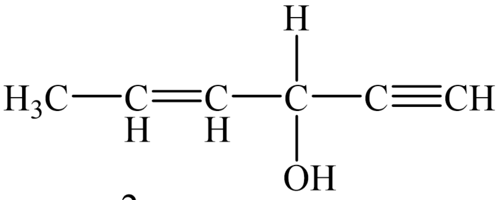


$H_3C-\underset{H}{C}=\underset{H}{C}-\overset{H}{\underset{OH}{C}}-C\equiv CH$

2

Experimental Section

a) Derivatives of Aminocolchamine with Propenylethynylcarbinol.

A sample of 1.0 g of aminocolchamine was dissolved in 17 ml of dried and freshly distilled dioxane. To the solution, 0.12 g of paraformaldehyde, 0.01 g of hydroquinone, and 0.03 g of zinc chloride were added. After this, an equimolar amount of propenylethynylcarbinol was added to the solution, and the contents of the flask were thoroughly mixed.

Table 1

Reaction conditions of propenylethynylcarbinol with aminocolchamine.

№	**Reagent**	**Calculated amount of reagent**	**Taken amount of reagent**	**Product yield (%)**
1	**Aminocolchamine**	**0.78**	**1.0**	**95**

The reaction mixture was heated in a glycerin bath with a reflux condenser at 80-100°C for 6-8 hours. The completion of the reaction was determined by thin-layer chromatography of the reaction mixture. After the practical completion of the reaction, insoluble substances in dioxane were separated by filtration, and the solvent (dioxane) was removed using a rotary evaporator. The residue was dissolved in 20-30 ml of chloroform, and the resulting very dark chloroform solution was extracted three times with 20 ml portions of 5% acetic acid.

The acetic acid extract contained unreacted aminocolchamine, which was isolated by alkalizing the acidic solution with ammonia and extracting it with chloroform.

The chloroform solution of the reaction product, after the removal of the starting aminocolchamine, was dried over anhydrous sodium sulfate. The sulfate was filtered off, and the filtrate was passed through a small layer (5-7 g) of aluminum oxide, which significantly lightened the dark extract. The solvent was evaporated, and the reaction product was dried in a vacuum desiccator.

The condensation reaction of aminocolchamine with acetylene compounds was carried out according to Mannich [4], in equimolar ratios of reagents.

The main starting compound, aminocolchamine (1), for the conducted syntheses was isolated from Colchicum luteum Baker, which grows in the Surkhandarya region.

As a result, we synthesized 4-(aminocolchamino N/propenyl-1-butyne-2) carbinol (3) (Table 2) [5].

The obtained compounds are light yellow powders with similar Rf values. At the same time, their chromatographic mobility differs significantly from the starting aminocolchamine, exhibiting higher Rf values.

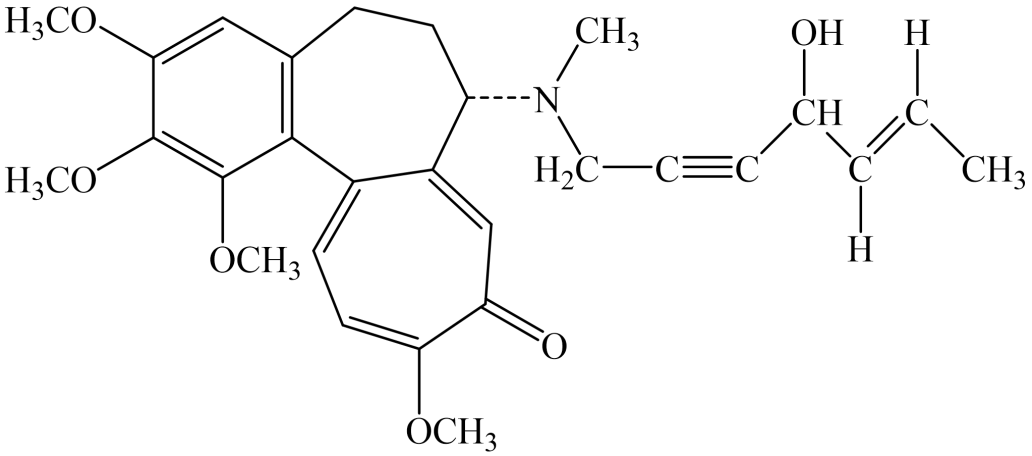
A characteristic feature of all acetylene derivatives is the presence of a two-proton doublet from the bridging N-CH_2 group in their ^{1}H NMR spectra, appearing in the region of 3.32–3.38 ppm. The bridging OCH_3 group present in compounds 4-5 forms a narrow two-proton doublet in the region of 4.53–4.70 ppm.

The structure of the synthesized compounds was confirmed by IR and ^{1}H NMR spectral data. In the IR spectra of compounds with an ester group (3-4), absorption bands of the carbonyl group appear at 1735–1730 cm^{-1}.

The aminocolchamine fragments of the synthesized compounds do not differ significantly in their ^{1}H NMR spectra:

- N-methyl group signals appear at 2.20–2.22 ppm,
- Methoxy groups at 3.66–3.70 ppm (at C-1) and 3.82–3.85 ppm (at C-2, C-3, and C-10),
- Proton H-4 at 6.54–6.61 ppm,
- H-8 at 7.90–7.96 ppm,
- H-11 at 6.78–6.85 ppm,
- H-12 at 7.17–7.22 ppm.

The synthesized acetylene derivative.



4-(Aminocolchamino N/propenyl-1-butyne-2) carbinol (3).

IR spectrum: 1210, 1140, 1730, 2600, 3000, 3410, 3540 cm^{-1}.

^{1}H NMR spectrum:
1.60, 1.85 (CH_2),
2.05 (CH_3),
2.26 (NCH_3),
3.58; 3.85 × 2, 3.83 (3H × 4, ss, 4OCH_3),
5.16 (OH),
6.48 (H-4),
6.94 (H-11),
7.24 (H-12 and H-8) ppm.

Conclusions

A new aminocolchamine derivative with propenylethynylcarbinol was synthesized. The structure of the synthesized compound was confirmed by ^{1}H NMR and IR spectral data.

This work presents the synthesis of a new aminocolchamine derivative with propenylethynylcarbinol- 4-(aminocolchamino N/propenyl-1-butyne-2) carbinol**.** The structure of the synthesized compound was confirmed by IR and ^{1}H NMR spectroscopy.

The synthesis process involves a condensation reaction of aminocolchamine with acetylene-containing compounds, conducted using the Mannich reaction**.** The obtained compounds exhibit characteristic spectral properties, including the presence of an acetylene bond, which reduces their toxicity. Spectral analysis confirms the structural features and the high chromatographic mobility of the new compounds.

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