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On the mechanism of the interaction of narceine with the Froehde-Buckingham reagent

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Abstract

Narceine is a little-known opium alkaloid. In this communication the mechanism of the interaction of this alkaloid with the Froehde-Buckingham reagent is provided. Protonated molybdic acid reacts with an oxygen of the methylenedioxy group present in the narceine molecule. There is elimination of a water molecule from the acid, and an oxonium ion is formed. Neutralisation is accomplished by reaction of a water molecule at the methylene group with simultaneous breaking of the five-member ring. The molybdic ester thus formed reacts with a hydrion, inducing separation of water and molybdenum dioxide, (reduction step to Mo-IV). The electron deficient oxygen attracts electrons from the benzene ring, and an ortho-quinone is formed (oxidation step) via elimination of formaldehyde. In other words, a synchronous mechanism took place involving five electron-shifts.

Keywords: Froehde-Buckingham reagent; Methylenedioxy group; Molybdenum dioxide; Molybdic acid; Oxonium ion; Reactive intermediates; Redox reaction

1. Introduction

There are many colour tests for identification of morphine, however, other opioids like narceine have not been considered. Fröhde used ammonium molybdate in sulphuric acid for morphine detection, [1-3]. Buckingham extended the use of this reagent to many other compounds [4-6], but narceine was not included, as well as in the list of other different compounds presented by Wikipedia [7]. It was Bruylants who presented a list of colours observed by reaction of other opium alkaloids with the Froehde reagent, [8-10].

Narceine presents a brown colouration, turning green, then blue.

In this communication the mechanism of this reaction is provided. Each step of this route is fully commented and the electron flow is given. This paper is a follow up our studies on reaction mechanism, [11-15].

2. Antecedents

There are two studies on the mechanism of the Froehde reaction with morphine, [16, 17]. In the first approach there is use of the concept of back donation which can be found in known books on mechanism of oxidation of organic compounds, [18]. In the second paper this concept was left out as unnecessary and other reaction mechanism was provided.

The structure of narceine, Figure 1, diversely to morphine, has no phenolic group, but it presents a carboxylic acid that can form a mixed ester with molybdic acid. However, this ester cannot go further reaction. Besides there are three

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methoxy groups and a methylenedioxy group. It has been found that in the presence of these two types of groups, the more reactive is the methylenedioxy group. [19-20].

The reaction of narceine with Froehde reagent is treated in the next section.

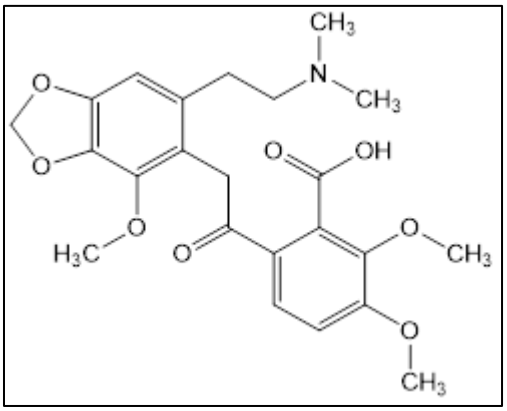
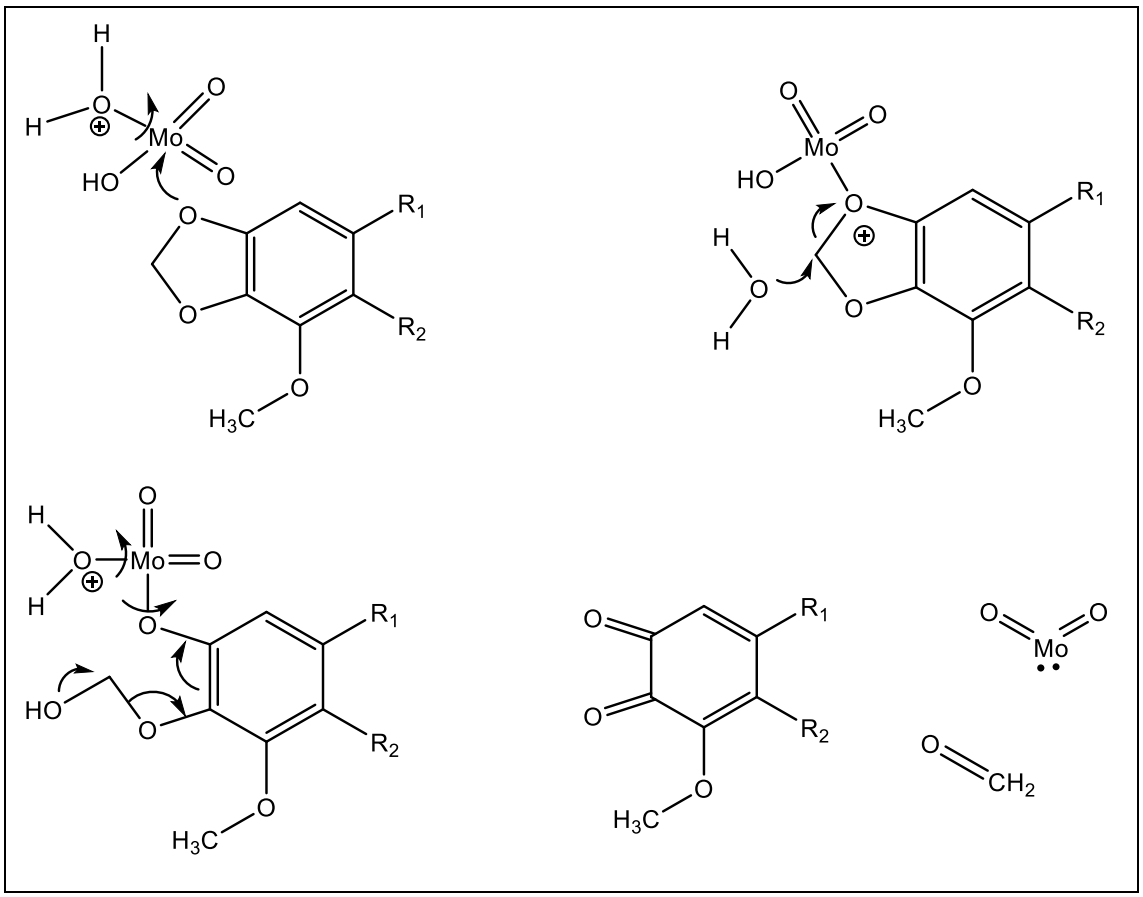


Figure 1 Structure of narceine

3. Discussion

The Buckingham's reagent for alkaloids is a freshly prepared solution of 1 g ammonium molybdate in 16 g of concentrated pure sulphuric acid. Heat is applied until the solution is clear. It was named sulpho-molybdate of ammonia and yields different colour with various alkaloids.



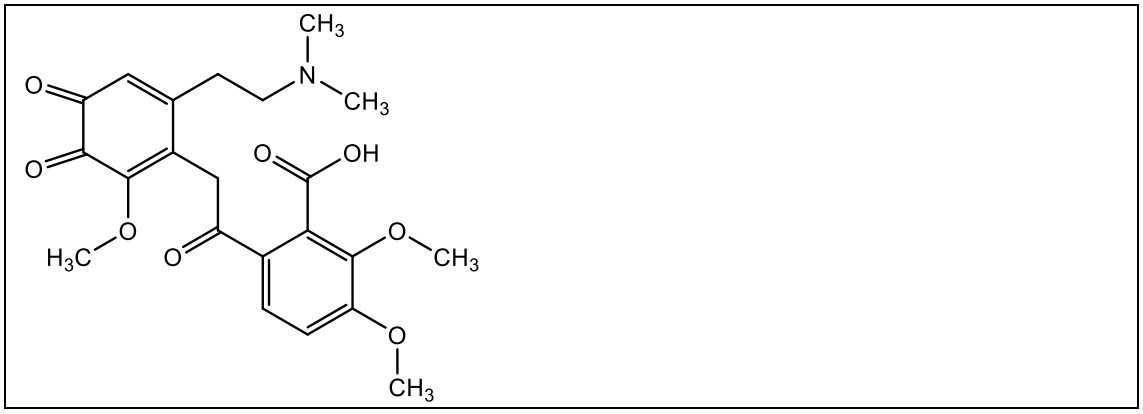


Figure 2 Mechanism of oxidation of narceine with Froehde-Buckingham reagent

The electrodotic property [21] of an oxygen atom in the methylenedioxy group combines with the reactive species, protonated molybdic acid. Figure 2. A water molecule is eliminated and an oxonium ion is formed, which is neutralised by reaction of a water molecule at the methylene group, breaking the five-member ring.

A mixed ester has been formed. Protonation of this organo-inorganic molybdate separates water and a concerted mechanism occurs: molybdenum dioxide is produced (reduction to Mo-IV), and the organic molecule is oxidised to an ortho-quinone via five electron shifts.

The ortho-quinones are red [22], but molybdenum blue is a mixture of oxides derived from molybdenum dioxide, [23].

4. Conclusion

Bruylants presented the results obtained with several opioids employing the Froehde-Buckingham reaction. Narceine gave blue colour. In this communication the mechanism of this reaction is provided. Protonated molybdic acid reacts with the methylenedioxy group in narceine, followed by rupture of the five-member ring by a water molecule in order to neutralise an oxonium ion. Protonation of a molybdic ester separates water and molybdenum dioxide, then an ortho-quinone is formed with elimination of formaldehyde.

Compliance with ethical standards

Acknowledgments

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Disclosure of conflict of interest

There is no conflict of interest to declare

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