

(RESEARCH ARTICLE)



VANADIC ACID AS DEMETHYLATION CATALYST IN MANDELIN'S TEST FOR BRUCINE

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ABSTRACT

The Mandelin test for brucine (dimethoxy strychnine) and for strychnine are very different, giving blood-red and blue-violet colour, respectively. These results indicate that in brucine the methoxy groups are reacting. However, these ethers are commonly very stable, requiring strong experimental conditions for demethylation. Since brucine reacts in different form than strychnine, we can conclude that the methoxy groups in brucine intervene in some manner. Vanadic acid, coming from ammonium vanadate in acidic medium, can act as demethylation agent and catalyst. The reactive species is protonated vanadic acid. A coordination compound is formed with concomitant elimination of water. The oxonium intermediate can separate a methyl group with assistance of a bisulphate anion and formation of a vanadate. Acidolysis of this mixed ester forms a phenolic group.

The second methoxyl in brucine also forms a vanadate, but protonation of this group gives rise to a concerted reaction mechanism. A redox reaction form vanadous acid and a red o-benzoquinone.

Keywords: Acidolysis; Brucine; Coordination Compound; Demethylation Catalyst; Mandelin Test; Redox Reaction

1 INTRODUCTION

The reaction of brucine (2,3-dimethoxy-strychnine) is totally different to that presented by strychnine towards ammonium vanadate in sulphuric acid (Mandelin's test). This chemical department indicates that the methoxy groups in brucine must intervene in this reaction, probably by an initial demethylation. However, demethylation is a difficult reaction that requires an especial reagent and strong reaction conditions. Nevertheless, in our case there is instant reaction at room temperature, colour test conditions. It must be explained why and how this process occurs. It was deduced and verified that vanadic acid (formed in situ) is a catalyst for demethylation.

In the present communication the role of vanadic acid as demethylation catalyst is provided. This article is a follow up of our studies on reaction mechanism, [1-5].

2 STUDY METHOD AND PROCESS

This is an Organic Chemistry Theoretical Study. It is based on the chemical deportment of reagent and substrate. All is in accordance with the reaction medium and the catalyst present. The several steps leading to the final product are fully commented and the electron flow is also given.

3 ANTECEDENTS

The use of ammonium vanadate in sulphuric acid was introduced by the German pharmacologist Karl Friedrich Mandelin (1854-1906). He worked at the German University of Dorpat, now Tartu, in Estonia. He published his work in 1883, in German, in St. Petersburg, Russia [6], and in Germany [7], and his test has been registered in several analytical books, [8-10].

Mandelin reagent has intensive yellow or orange-brown colour. Mandelin test is used as a straightforward spot test for preliminary identification of alkaloids, and produces some of the most vivid, chemically distinct, colour reactions.

Kundrat in 1889 tested a number of alkaloids with Mandelin reagent. He obtained distinctive colours with thirty of the common alkaloids, [11, 12].

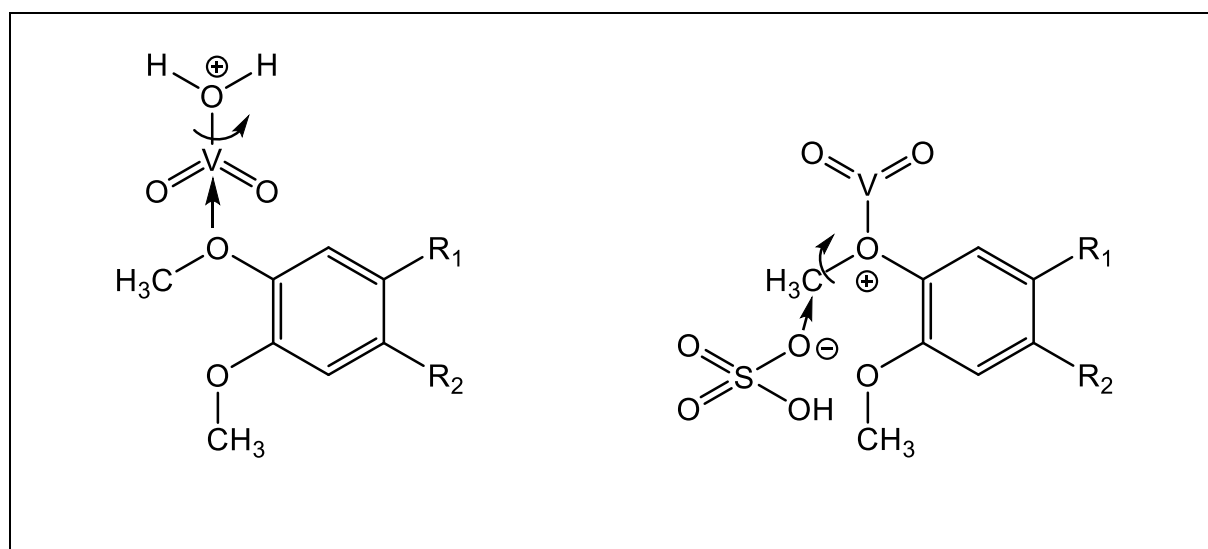
For the preparation of ammonium vanadate see references [13-15].

4 DISCUSSION

The reactive species in the Mandelin test for brucine is protonated vanadic acid, generated in situ by metathesis of ammonium vanadate with sulphuric acid. The first reaction step is coordination of the reactive species with an oxygen atom of a methoxy group, with concomitant separation of a water molecule from the reagent. The oxonium ion thus formed has an additional δ^+ from the two vanadium-oxygen double bonds (vanadium electronegativity is 1.6, and of oxygen is 3.5, Pauling scale [16]). This effect is also transmitted to the oxygen-methyl bond, making this bond labile, less firm. Elimination of a very unstable methyl carbonium ion is avoided by participation of a bisulphate anion in a push-pull synchronous reaction. So, there has been demethylation and formation of an organic vanadate.

The water molecule separated from the reactive species can be added to the meta-vanadate, and hydron interchange produces hydrolysis of the ester with formation of a phenol group. These two steps show the catalytic role of vanadic acid since it has been recuperated after demethylation.

In the other methoxy group in brucine there is also demethylation and formation of a vanadate, Figure 1. However, protonation of the ester originates an internal reaction, that is, a concerted mechanism involving a C-C double bond in the benzene ring and



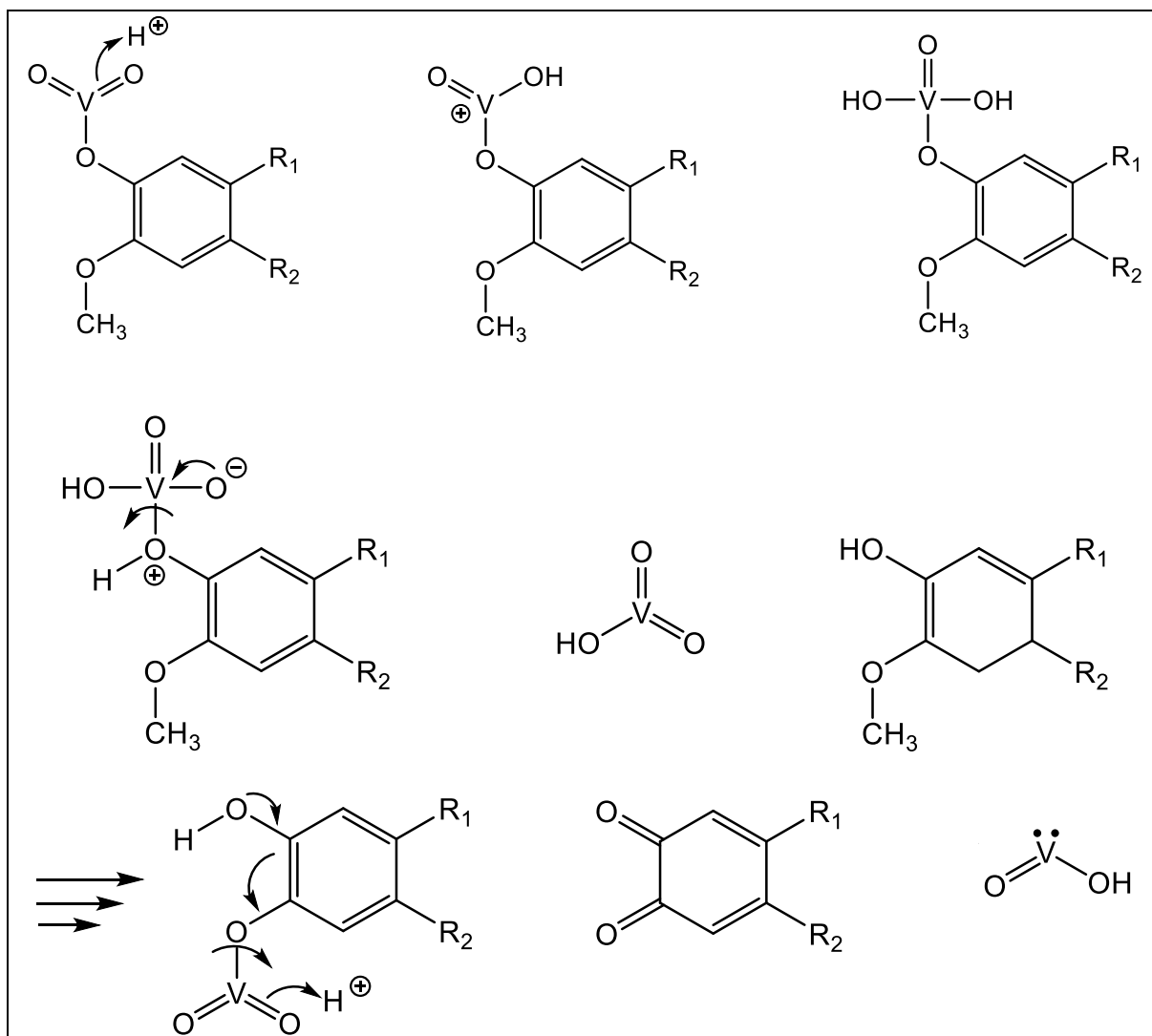


Figure 1: Demethylation of brucine by catalysis of vanadic acid and oxidation of the intermediate phenolic vanadate to an o-benzoquinone

participation of the electrodotic property [17-19] of the phenol group. That is, a redox process occurs. Vanadium is reduced to vanadium III (vanadous acid) and the organic compound is oxidised to a red ortho-benzoquinone. See reference 20. This internal process is faster than the external hydrolysis and therefore the hydrolysis doesn't occur now.

This way the Mandelin test for brucine has been cleared, showing the catalytic role of vanadic acid.

5 CONCLUSION

Mandelin test for brucine gives an intense blood-red, whereas strychnine produces a blue-violet colour. These very different analytical results with two compounds whose only difference lie in the extra two methoxy groups present in brucine, indicated reactivity in the apparent stable alkyl-aryl-ethers. We have found the role of vanadic acid, generated in situ, as demethylation catalyst. Also is described a redox reaction that occur in an intermediate phenolic vanadat

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

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Disclosure of conflict of interest

There is no conflict of interest to declare.

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