

Study of 2-methyl-2-cyclopenten-1-one reductive ozonolysis reaction.

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Abstract

Ozonolysis of α,β -unsaturated ketones are reactions able to yield anomalous products through mechanisms that are distinct when compared with typical alkene ozonolysis. The research of this kind of reaction, using 2-methyl-2-cyclopenten-1-one, has shown that 5-oxotetrahydrofuran-2-yl acetate (I) is the major product of it, indicating the occurrence of anomalous steps in its formation.

Key words: anomalous ozonolysis, 2-methyl-2-cyclopenten-1-one, NMR.

Introduction

In general, ozonolysis reactions are defined as those that promote the cleavage of σ and π alkene bonds through oxidation with ozone, leading to the formation of carbonyl compounds, depending on the treatment and the substitution degree of carbon atoms in double bond. α,β -unsaturated ketones, as some allylic compounds and indoles, are systems that are able to go through ozonolysis reactions in which the products not always have the desired structures¹. This kind of reaction is known as "Anomalous Ozonolysis", which is generally characterized by the occurrence of an additional cleavage, a C-C bond next to double one, leading to the formation of products with one less carbon atom than expected¹. Due to the versatility of these reactions, we propose this research aiming to study the products obtained in reductive ozonolysis through Mass Spectrometry-Gas Chromatography (GC-MS) and Nuclear Magnetic Resonance (NMR) techniques.

Results and Discussion

After the achievement of ozonolysis reaction, made by ozone bubbling for 210 minutes, dimethyl sulfide in excess was added to reaction mixture and magnetic stirring was performed for about 30 minutes. After it, reducer excess was removed using N_2 (g) purge and the solvent (CH_2Cl_2) was evaporated with a rotary evaporator. A GC-MS analysis of the esterified products, obtained with CH_2N_2 , indicated that three products were formed; the major one was responsible for about 42% of mixture composition. The products were isolated by column chromatography on silica gel, using hexane: ethyl acetate mixtures as eluents, and the major one was characterized by GC-MS, 1H NMR, ^{13}C NMR, DEPT 90 and DEPT 135. Comparison with data present in literature allowed us to deduce that the molecule in study was the **5-oxotetrahydrofuran-2-yl acetate (I)**, once its spectroscopic characterization is in

agreement with those described by Turrini [et al.]². All data and the correspondent structure of it are present in Chart 1 and Image 1, respectively.

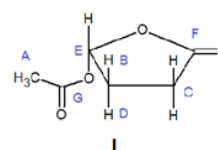


Image 1. Structural formula of 5-oxotetrahydrofuran-2-yl acetate.

Chart 1. Spectroscopic characterization of I.

Position	Experimental δ_H /ppm	Literature δ_H /ppm	Experimental δ_C /ppm	Literature δ_C /ppm
A	2,12 (s,3H)	2,1 (s,3H)	20,9	21,0
B	2,22-2,29 (m,1H)	2,2-2,3 (m,1H)	25,8	25,9
C	2,47-2,56 (m,2H)	2,4-2,6 (m,2H)	27,8	27,9
D	2,64-2,75 (m,1H)	2,6-2,8 (m,1H)	25,8	25,9
E	6,84 (m,1H)	6,6 (m,1H)	95,0	95,0
F	-----	-----	175,4	175,6
G	-----	-----	169,1	169,2

EI-MS: m/z= 43,0; 56,0; 72,0; 85,0; 100,0; 144,0.

Conclusions

The interpretation of all analysis involving separations (GC) and spectrometric techniques (MS, NMR) allowed us to conclude that 5-oxotetrahydrofuran-2-yl acetate is the main product of reductive ozonolysis reaction.

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¹ Bailey, P.S; *Ozonation in Organic Chemistry – Volume I*; Academic Press; **1978**; 147,149.

² Turrini, N.G; Hall,M; Faber,K; *Adv.Synth. Catal.*; **2015**; 357; 1868.