Synthesis of 3-chlorohimachal-7-one

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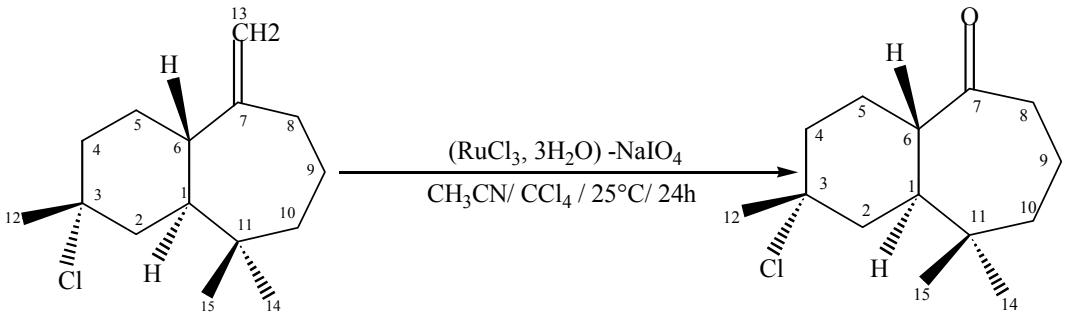
Abstract: The title compound, 3-chlorohimacha-3-one has been synthesized by catalytic oxidation with ruthenium trichloride and sodium periodate of 3-chlorhimachl-6,13-ene(1). .The structure of this new compound was confirmed by elemental analysis, IR, ^{1}H-NMR, ^{13}C-NMR and EI-Ms spectral analysis.

Keywords: himachalene; oxidation; catalytic; periodate; ruthenium trichloride.

Our work lies within the framework of the valorisation of the most abundant essential oils in Morocco, such as Cedrus atlantica. This oil is made up mainly (75%) of bicyclic sesquiterpenes hydrocarbons (himachalenes) (1, 2). The reactivity of these sesquiterpenes has been studied extensively by our team (3-7), in order to prepare new products having olfactive proprieties suitable for the perfume or cosmetics industry. In this paper, we report the synthesis of a novel compound (3-chlorohimachal-7-one) by catalytic oxidation with ruthenium trichloride and sodium periodate(8,9) of 3-chlorhimachl-6,13-ene(1)

The sodium periodate is prepared in situ with equimolecular quantity of soda NaOH (1g; 23mml)and periodic acid H_5IO_6(2.85g; 23mml), the mixture is stirred at 0°C. After 15min, 5ml of CCl_4, 10ml of H_3CCN and 186,06mg (0.24mml) of ruthenium trichloride (8,9) were added. The mixture was stirred during 15min ,and then 1.85g(7.7mml) of 3-chlorohimachal-6,13-ene **1** was added. The reaction was left under stirring at 25°C for 24 h, then 30 ml of distilled water was added and the reactional mixture was extracted with the dichloromethane . After filtration on silica gel column to eliminate RuO_4, the organic layer was recovered, died by Na_2SO_4 and evaporated under reduce pressure. The residue was purifedon silica gel column using hexane /ethyle acetate(98/2) as eluent to give 1.58 g (6.54mml) of **2** in 85% yield.



1 **2**

Milting point: 76-77°C (Hexane).

MS(EI, 70eV): 240 ($M^{+\cdot}$).

IR (KBr)ν_{max} cm^{-1}: 1725 (C=O).

^{1}H-NMR(300 MHz, $CDCl_3$) δ(ppm): 0.65, 0.82(2s, 6H, $(CH_3)_2$-11), 1.52(s, 3H, CH_3-3), 2.15 (m, 1H, H-1), 2.65(m, 1H,H-6).

^{13}C-NMR(75 MHz, $CDCl_3$), δ(ppm): 41.62(C-1), 40.48(C-2), 71.50(C-3), 38.76(C-4), 42.06(C-5), 54.12(C-6), 214.82(C-7), 43.37(C-8), 19.86(C-9), 27.11(C-10), 35.61(C-11), 34.33(C-12), 29.38(C-14), 21.78(C-15).

Anal.calc. for $C_{14}H_{23}ClO$: C,69.27, H, 9.48. Found: C, 69.25, H, 9.49.

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