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THE USE OF ^{13}C-NMR IN THE DETERMINATION OF STRUCTURES: A CORRECTION OF THE STRUCTURE OF BORJATRIOL*

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Key Word Index—Diterpenoids; borjatriol; 8α,13-epoxy-labdane derivatives.

Abstract—^{13}C-NMR data of two borjatriol derivatives show that the carbocyclic hydroxyl function is placed on C-7, modifying the previous assignment on C-6.

As part of a study [1, 2] of the effects of substituents on ^{13}C-NMR of diterpenoids, the data on two derivatives (**1**, **2**) of borjatriol (6*S*,14*R*,15-trihydroxy-8α,13-epoxy-labdane) [3] have been obtained.

According to the data (Table 1), the hydroxyl group originally located on C-6 must instead be located at C-7. The former assignment was based principally on the ^{1}H-NMR spectrum of compound **2**. Two distinct signals at δ2.42 (1H, singlet) and δ2.55 (2H, AB quartet, J_{AB} = 14 Hz) were apparent. When the spectrum was measured at 100 MHz (previous ^{1}H-NMR spectra were obtained at 60 MHz) an eight peaked signal was obtained, the AB part of an ABX pattern, similar to the signal observed for the C-3 methylene group in flavanones [6]. Thus borjatriol is 7*S*,14*R*,15-trihydroxy-8α,13-epoxy-labdane (**3**).

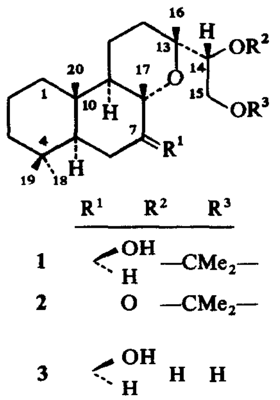

Table 1. Carbon-13 chemical shifts* of compounds **1** and **2**

Carbon atom	1	2	Carbon atom	1	2
1	38.8	39.0	11	14.1	14.3
2	18.5	18.3	12	31.4	30.5
3	41.9	41.7	13	73.5	74.9
4	33.2	33.7	14	82.6	81.9
5	56.2†	56.7	15	65.3	65.5
6	26.9	35.8	16	24.8	24.6
7	80.6	209.1	17	19.4	23.1
8	78.6	80.7	18	33.3	32.6
9	54.1†	59.0	19	21.3	20.7
10	37.0	37.0	20	15.9	15.2

* Assignments have been made taking into account published data [4, 5] and additivity rules. Assignments marked† could be reversed.

EXPERIMENTAL

The Fourier transform ^{13}C-NMR spectra were obtained on a Varian XL-100-12 WG spectrometer operating at 25.16 MHz (compound **1**) and on a Bruker Spectrospin at 15.08 MHz (compound **2**). The samples were examined as M soln in $CDCl_3$ using TMS as internal standard. Assignments were made with the aid of off-resonance and noise-decoupled ^{13}C-NMR spectra.

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