LETTERS TO THE EDITOR

ESTABLISHMENT OF THE STRUCTURE OF 2,7-DIMETHYLDECAHYDRO-4-QUINOLONES

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UDC 547.831.8:541.621.2:543.422.25

The signal of the angular 9-H proton in the PMR spectra of two stereoisomeric [1] 2,7-dimethyldecahydro-4-quinolones recorded at 300 MHz is observed at δ 2.6-2.9 ppm in the form of a triplet (11.0 Hz) of doublets ($\sim$3.7 Hz). This sort of splitting of the signal of the proton attached to 9-C is possible only in the case of trans fusion of the rings. The 2-H and 3-CH_2 protons form a spin subsystem, which at this frequency can be examined within the AMX approximation. The J_{vic} values of 6.6 and 1.8 Hz found by means of spin decoupling indicate an equatorial orientation of the proton attached to 2-C (and consequently an axial orientation of the 2-CH_3 group in ketone I), while the J_{vic} values of 11.6 and 2.9 Hz correspond to an axial orientation of the proton attached to 2-C (and consequently an equatorial orientation of the 2-CH_3 group of ketone II). The 7-H signal, the half-width of which amounts to 11 Hz for both compounds in the case of suppression of spin–spin coupling with the 7-CH_3 group ($\{2\text{-}H_a, 7\text{-}CH_3\}$ triple resonance was used in the case of ketone II in order to reduce overlapping of the signals of the 3-H_a and 7-H protons), is observed at 2.13 ppm. This value attests to an axial orientation of the 7-CH_3 group.

The signals of the 2-H, 9-H, and 3-CH_2 protons in the spectra of ketones I and II at 100 MHz are also resolved at weak field, while the signal of the 7-H proton is hidden in the region of methylene exaltation. The addition of tris(1,1,1,2,2,3,3-heptafluoro-7,7-dimethyl-4,6-octanedionato)europium [$Eu(FOD)_3$] in a reagent-to-compound molar ratio of 0.14 makes it possible to isolate the individual signal of the 7-H proton of ketone II. Measurement of the half-width of this signal also gives 11 Hz. We were unable to isolate the signal of the proton attached to 7-C with this reagent in the case of ketone I because of the considerable broadening of the signals that the reagent causes.

PMR spectra, δ, ppm (300 MHz, 0.2 M solutions in $CDCl_3$): ketone I: 3.77 (1H, m, 2-H_e), 1.17 (3H, d, $J = 7.0$ Hz, 2-CH_3), 2.71 and 2.24 (2H, two q, $J_{vic} = 6.6$ and 1.8 Hz; $J_{gem} = -13.2$ Hz, 3-CH_2), 2.13 (1H, br s, 7-H_e), 1.00 (3H, d, $J = 7.3$ Hz, 7-CH_3), 3.02 (1H, m, $\Sigma J = 25.8$ Hz, $J_{9,10} = 11.0$ Hz, 9-H), 1.98 (1H, m $\Sigma J = 26$ Hz, 10-H), 1.45-1.76 (6H, m, 5-CH_2, 6-CH_2, 8-CH_2); ketone II: 3.09 (1H, m, 2-H_a), 1.22 (3H, d, $J = 6.2$ Hz, 2-CH_3), 2.17 (1H, t, $J_{vic} = 11.6$ Hz, 3-H_a), 2.39 (1H, q, $J_{vic} = 2.9$ Hz, $J_{gem} = -13.2$ Hz, 3-H_e), 2.13 (1H, br s, 7-H_e), 0.99 (3H, d, $J = 7.2$ Hz, 7-CH_3), 2.74 (1H, m, $\Sigma J = 25.7$ Hz, $J_{9,10} = 11.0$ Hz, 9-H), 1.96 (1H, m, $\Sigma J = 26$ Hz, 10-H), 1.25-1.80 (6H, m, 5-CH_2, 6-CH_2, 8-CH_2).

LITERATURE CITED

1. A. A. Akhrem, L. I. Ukhova, and A. N. Sergeeva, Izv. Akad. Nauk Belorussk. SSR, Ser. Khim., No. 4, 46 (1975).

Institute of Bioorganic Chemistry, Academy of Sciences of the Belorussian SSR, Minsk 220600. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 11, p. 1569, November, 1977. Original article submitted February 7, 1977.