Chlorination of Achillin

Oleg V. Alebastrov, Sergazy M. Adekenov, G. K. Buketova, Arman T. Kulyjasov

Institute of Phytochemistry MS-AS RK 470032 Karaganda, Erzhanova str., a/b 19, Kazakhstan

Achillin (1), the major guaianolide from *Achillea micrantha Willd*. was first chlorinated in MeOH. The chlorination was conducted according to literature procedure[1].



The products of chlorination were separated by flash-chromatography. The eluents - the mixture of hexane - ethylacetate (with increase of percentage of last). Two compounds were isolated. The first compound (**2**) has m.p. - 160-162°C (CHCl₃) yield - 63,7 %. IR-spectrum (cm⁻¹): 1775 - the lactone carbonyle, 1710 - the carbonyl group and 1620 - the double bond of cyclopentenone. UV: λ_{max} 241 nm (lgɛ 3,98) - cyclopentenone and weak band - the carbonyl group of γ -lactone at 337nm (lgɛ 1,77). NMR (CDCl₃), δ : 6,15 (1H, s, H-3); 3,49 (1H, J=10 Hz, d, H-5); 4,86 (1H, J=10Hz, t, H-6); 1,24 (3H, J=7 Hz, d, H-13); 3,88 (1H, J=10 Hz, d, H-14); 4,02 (1H, J=10 Hz, d, H-14'); 2,25 (3H, s, H-15).

The second one (**3**) has m.p. 184-185°C (CHCl₃), yield 36,3 %. IR-spectrum (cm⁻¹): 1787 - the carbonyl group of lacton cycle, 1725 - the carbonyl group, 1630 - the double band of cyclopentenone, UV: λ_{max} 244 nm (lgɛ 3,88). NMR ¹H (CDCl₃), δ : 6,12 (1H, s., H-3); 3,66 (1H, J-8 Hz, d, H-5); 4,62 (1H, J=8 Hz, t., H-6); 1,18 (3H, J=8 Hz, d, H-13); 3,40 (1H, I=10 Hz, d, H-14); 3,52 (1H, J=10 Hz, d, H-14'); 2,22 (3H, s, H-15).

On the basis of spectral data the structures (2) and (3) were proposed for chlorinated products of achillin.

Reference

1. Takayanagi H., Ogura H., Mc Murry T.B. // Chem. Pharm. Bull. 1990. V. 38. N 3. P. 581-584.