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].

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\begin{align*}
\text{(1)} & \quad \rightarrow \\
\text{(2)} & : 10\% \text{ Cl} \\
\text{(3)} & : 10\% \text{ Cl}
\end{align*}
\]

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: \( \lambda_{\text{max}} \) 241 nm (lgε 3,98) - cyclopentenone and weak band - the carbonyl group of \( \gamma \)-lactone at 337nm (lgε 1,77). NMR (CDCl₃), \( \delta \): 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: \( \lambda_{\text{max}} \) 244 nm (lgε 3,88). NMR \( ^1 \text{H} \) (CDCl₃), \( \delta \): 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