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The preparation of marmesin benzoate has been described, and it has been shown [1] to be identical with natural felamedin [2]. A difference in the melting points and angles of rotation of the substances (absence of optical activity in the synthetic sample) was explained by the authors in terms of racemization taking place during the process of benzoylating marmesin in benzene. At the same time, the marmesin benzoate obtained in very low yield by the benzoylation of marmesin in the presence of pyridine and separated from the unchanged material by preparative TLC was identical with natural felamedin [2].

The reaction of marmesin with benzoyl chloride in pyridine at 65-70°C and subsequent crystallization from methanol gave us 74% of marmesin benzoate with the composition $C_{21}H_{18}O_5$, mp 135-136°C (Kofler), $[\alpha]_D^{17}$ -106.3° (c 0.9; chloroform), identical according to its IR spectrum [2] with natural felamedin. UV spectrum; λ_{max} , nm (log ϵ): 226 (4.35), 248 (3.67), 259 (3.58), 300 (3.78), 336 (4.23). Rf 0.20 [alumina, activity grade II, neutral, petroleum ether (bp 40-60°C)-diethyl ether (1:1)].

In a similar manner, we obtained an almost quantitative yield of nodakenetin benzoate, $C_{21}H_{18}O_5$, mp 135-136°C, $[\alpha]_D^{17}+104.9$ °. The IR and UV spectra of the compounds obtained coincided completely with those of marmesin benzoate; R_f 0.20 (same system).

The racemate obtained by dissolving equal amounts of the benzoates of marmesin and nodakenetin in methanol followed by crystallization had mp 147-148.5°C and an IR spectrum (Fig. 1) differing from that of the two antipodes. The melting points and IR spectra of the two antipodes and of the racemate differed completely from the benzoylation product described previously [1].

A check of the procedure for obtaining marmesin benzoate [1] showed that the reaction product consists of at least five components with the following R_f values (in the same system): 0 (marmesin), 0.20 (marmesin benzoate), 0.30 (5'-isopropenyl-4',5'-dihydropsoralen), 0.50 (anhydromarmesin), and 0.55 (unidentified compound).

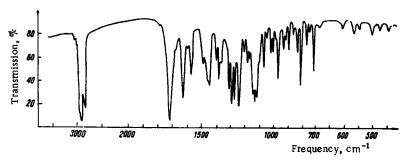


Fig. 1. IR spectrum of a racemic mixture of the benzoates of marmesin and nodakenetin (mull in paraffin oil).

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LITERATURE CITED

- 1. V. A. Krivut, L. G. Avramenko, M. E. Perel'son, and G. K. Nikonov, Khim. Prirodn. Soedin., 526 (1970).
- 2. I. Ognyanov and D. Bocheva, Planta Medica, <u>17</u>, No. 1, 65 (1969).