N-METHYLHEXADECANAMIDE IN THE OIL OF COTTON SEEDS OF VARIETY TASHKENT-1

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The seed oil (50 g) of the cotton plant of variety Tashkent-1 (containing 0.45% of nitrogen) was chromatographed on a column of KSK/100 mesh silica gel in system 1 (hexane–ether (8 : 2 by volume)). The compositions of the eluates were checked on Silufol in the same system. Two fractions were obtained: (I) (47 g – carbohydrates, unoxidized triglycerides, oxidized triglycerides, and fatty acids) and (II) (2 g – oxidized triglycerides, sterols, methylamides, and oxidized fatty acids) with a nitrogen content of about 1.9%.

The total substances of fraction (II) were transesterified with methanol in the presence of sodium methanolate. From the ether-soluble reaction products in a thin layer of KSK/150 mesh silica gel in system 1 we isolated "start" substances containing about 3.75% of nitrogen (methyl esters of oxidized fatty acids, oxidized fatty acids, methylamides). In a thin layer of KSK silica gel with 20% of $AgNO_3$ in system 2 (benzene–chloroform–ether (50 : 50 : 15 by volume)) we obtained four zones of substances from the total "start" substances. In the fastest-migrating zone 1 we detected the methylamide of palmitic acid (16 : 0) – $CH_3(CH_2)_{14}CONHCH_3$, mp 82.0-83.5°C (according to the literature [1]: 85.5°C); 5.03% N; mol. wt. 269 (mass spectrum).

IR spectrum, taken on a UR-10 instrument, ν_{max}^{film}, cm^{-1}: 3310 s (free NH_2 group), 3100 m (bound NH group [2]), 2920 s, 2855 s, 1465 m, 720-760 m ($-CH_2-$), 2960 s, 2870 s, 1375 m, 1440 m (CH_3-), 1640 v.s ($-CO-$, amide band I), 1560 s ($-NH-$, amide band II), 1290 m, 1250 w, 1230 w, 1205 w, 1170 m.

Mass spectrum, taken on a MKh-1303 instrument fitted with a glass system for direct introduction into the ion source, at 140°C and with an ionizing voltage of 3.5 V, m/e (rel. %): 41 (20), 43 (38), 58 (60), 73 (100), 86 (75), 100 (60), 114 (55), 128 (70), 142 (60), 156 (22), 170 (15), 184 (21), 198 (21), 212 (7), 226 (12), 240 (16), 254 (6), M+269 (40), 297 (1.3). The presence of a homolog – the methylamide of stearic acid with a molecular weight of 297 – as impurity made no impression on the spectrum of the 16 : 0 methylamide.

Hydrolysis of the 16 : 0 methylamide by Gatterman's method [3] gave the 16 : 0 fatty acid, which was identified by paper chromatography [4], and also, after methylation with diazomethane with the formation of the 16 : 0 methyl ester, by gas–liquid chromatography (UKh-2 instrument with a 4 mm × 2.5 m copper column filled with 15% of Reoplex 400 on Chromaton N-AW-HMDS). The amount of the 16.0 methylamide in the oil was about 0.04%.

According to mass spectroscopy, zone 2 contained the methylamides of oleic acid (M^+ 295) and of linoleic acid (M^+ 293).

To exclude the possibility of the formation of artifacts, in place of the oil we took "chromatographically pure triglycerides" after column chromatography and transesterification with methanol. No methylamides were detected in the ether-soluble transesterification products (IR spectrum and thin-layer chromatography in system 2). There is no information on natural methylamides in the literature available to us.

LITERATURE CITED

1. A. L. Markman, Khimiya Lipidov, No. 1, 36 (1963).
2. M. Kates, Techniques of Lipidology, American Elsevier, New York (1972).
3. Houben-Weyl, Methoden der organischen Chemie, Georg Thieme Verlag, Stuttgart.
4. É. I. Gigienova and A. U. Umarov, Technical and Economic Information, "Methods of Analysis and Control of Production in the Chemical Industry" Series [in Russian], NIITÉKhIM, No. 10 (1973), p. 9.

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