COMPONENTS OF *Neopallasia pectinata**

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From the light petroleum extract of the above-ground part of *Neopallasia pectinata* (PALL.) POLYAKOV paraffins, sesquiterpenic hydrocarbons, a sesquiterpenic diol and a relatively considerable amount of eremophila-9(10),11(13)-dien-12-oic acid were isolated by chromatography. The mixture of plant phytosterols contained β-sitosterol, stigmasterol and campesterol.

Neopallasia pectinata (PALL.) POLYAKOV[1] (syn. *Artemisia pectinata* PALL.), belonging to the *Compositae* family, is a one-year plant occurring in Mongolia, East Siberia and Kazakstan. Its above-ground parts are characterized by considerable stickiness. No data on its components have been published. Since it is taxonomically very close to the genus *Artemisia*, we analysed its components extracted with light petroleum.

The analysis was carried out chromatographically on deactivated silica gel and individual fractions were rechromatographed. The hydrocarbon fraction was rechromatographed on silica gel impregnated with silver nitrate. So a mixture of paraffins was obtained in front fractions, which according to GLC analysis contained hydrocarbons C_{23}—C_{31} only. The main component was hydrocarbon C_{29}, the smallest components were hydrocarbons C_{31} and C_{27}. In the last fractions sesquiterpenic hydrocarbons were present. Using GLC coupled with mass spectrometry the presence of α-muurolene, calacorene and Δ_3-cadinene could be demonstrated. In addition to these substances another 12 substances were present in trace amounts.

In the following fractions wax esters were present. When submitted to alkaline hydrolysis, and the free acids obtained to esterification with diazomethane, the methyl esters formed were submitted to a chromatographic analysis. On comparison of their elution times with those of standards the presence of acids from $C_{12:0}$ to $C_{30:0}$ could be detected among which the $C_{20:0}$ and $C_{16:0}$ acids predominated. Unsaturated acids $C_{16:1}$, $C_{18:1}$ and $C_{18:2}$ were also present in trace amounts. The

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neutral fraction obtained after saponification was also analysed by GLC and on the basis of the comparison of elution times with standards of alkanols the presence of alkanol homologues from C_{16} to C_{28} with prevailing C_{18} could be detected.

In subsequent ester fractions a mixture of triglycerides was present. The acids present in them were identified by means of GLC in the form of corresponding methyl esters. The acids $C_{18:3}$ and $C_{18:2}$ were the main components. In the last chromatographic fractions of the lipoid fraction the presence of an as yet unidentified sesquiterpenic diol $C_{15}H_{20}O_2$ could be detected.

The fractions following the lipoid fraction represent more than 20% of the starting extract and they contained a substance of the composition $C_{15}H_{22}O_2$. According to spectral data it is a sesquiterpenic acid containing an exomethylene group in the α-position to the carboxyl group. Among the substances of this type described in literature only eremophila-9(10),11(13)-dien-12-oic acid has similar spectral characteristics – mainly ^{1}H-NMR data – which was found recently[2] in the roots of *Athanasia thodei* (*Compositae*). We were able to isolate this acid in a pure state for the first time. A considerable amount of this acid, present in light petroleum extract, probably affects the sticking properties of the leaves.

The last chromatographic fractions contained a mixture of phytosterols in addition to the acid mentioned, currently occurring in plants: β-sitosterol, stigmasterol and campesterol, as shown by GLC analysis.

EXPERIMENTAL

The infrared spectra were measured in chloroform on a Zeiss UR-20 (Jena) instrument, optical rotation was determined with an objective polarimeter Perkin–Elmer 141, the UV spectrum was measured on a JASCO spectrometer. Other conditions were the same as given in the preceding paper[3].

Chromatography: The extract (53 g) was obtained by extraction of 1500 g of dry above-ground parts of *N. pectinata* with light petroleum. The plant was collected in Dundga Ajmak (about 300 km southwest of Ulanbator) during the second part of July 1977. The herbarium specimen is deposited in the Botanical Institute, Mongolian Academy of Sciences, Ulanbator. The chromatography of the extract (50 g) was carried out on silica gel deactivated with 11% of water (2500 g). Elution was carried out with light petroleum with gradually increasing content of ether (from 2–20%). The volume of the fractions was 3 l.

Hydrocarbons: A part of the fraction 1–4 (light petroleum; 60 mg) was chromatographed on silica gel impregnated with silver nitrate (3 g). Elution with light petroleum gave hydrocarbons (35 mg) in the first fractions. Their mixture was analysed by gas chromatography on a Pye 104 gas chromatograph provided with FID; for working conditions see[3]. The identification was carried out with a standard mixture of n-paraffins (C_{10-26}). The last eluted fractions (20 mg) were analysed on a GLC apparatus Packard–Becker 427 with FID; operating conditions: column length 1·85 m, i.d. 0·24 cm, Gas-Chrom Q (80/100) as carrier, impregnated with 3% of SE-30; programmed temperature from 80–300°C (3° per min). Conditions of GLC/MS:

A Pye instrument provided with FID; column length 1·5 m, i.d. 4 mm, carrier Gas-Chrom Q wetted with 3% of OV-17, carrier gas helium.

Waxes: A part (400 mg) of the combined fractions 5—10 (light petroleum–ether 2—6%; 10·65 g) was chromatographed on deactivated silica gel (45 g). The first fractions eluted with light petroleum (60 mg) corresponded to a mixture of hydrocarbons (according to TLC in 1% ether in light petroleum); the remaining fractions (300 mg) were combined. IR spectrum: 1720 cm^{-1}. Saponification of a part (200 mg) was carried out in boiling 5% methanolic potassium hydroxide for 40 min. The isolated acids were esterified with ethereal diazomethane solution and analysed by GLC (Packard–Becker 427 chromatograph with FID; column length 185 cm, i.d. 0·15 cm, carrier Gas-Chrom Q (80—100 mesh) impregnated with 5% of Silar 10 C, carrier gas nitrogen, programmed temperature from 140° to 240°C/3°C per min). The identification was carried out by comparison with a standard mixture of methyl esters of fatty acids and by means of mass spectrometry which displayed intensive fragments at 270, 298, 326, 354, 372, 410 and 428 mass units. The neutral fraction was also analysed by GLC (the same apparatus, the support impregnated with 3% SE-30, programmed temperature from 180° to 310°C, at a 3°C/min rate). The identification was carried out with a standard mixture of alcohols C_{16}—C_{28}.

Fats: A part (4·0 g) of the combined fractions 11—13 (light petroleum–ether 8—10%; 5·8 g) was chromatographed on silica gel (400 g) with 8% ether in light petroleum. The first fractions (0·6 g, IR spectrum: 1748, 1162 cm^{-1}) were saponified with methanolic potassium hydroxide. After esterification of the acid material with diazomethane the product was analysed by means of GLC. GLC conditions: apparatus Packard–Becker 427, column length 185 cm, i.d. 0·24 cm, support Gas Chrom Q 80—100 mesh impregnated with 5% of Silar 10 C; programmed temperature from 140°C to 240°C (3°C per min), carrier gas nitrogen.

Sesquiterpenic diol: The more polar fractions from the preceding chromatography afforded a substance with m.p. 75—76°C (ether) which had the bands 3628 and 3470 cm^{-1} in its IR spectrum. According to mass spectrometry its mass was 232·1461.

Sesquiterpenic acid: The combined fractions 13—16 (light petroleum–ether (10—14%, 5 g) were rechromatographed on silica gel (500 g) with 6% of ether in light petroleum, under addition of 3% of acetic acid. The middle chromatographic fraction was a substance (4·2 g) with m.p. 72°C, $[\alpha]_D^{20}$ −2·71° (*c* 0·48 in chloroform); IR spectrum: 950, 1626, 1695, 1663 cm^{-1}; UV spectrum: λ_{218} 3080 (*c* 0·00071, methanol). Methylation with diazomethane gave a methyl ester with the mass 248·1773; for $C_{16}H_{24}O_2$ calculated: 248·1776.

Phytosterols: The combined fractions 17—20 (light petroleum–ether 16—20%, 6·5 g) were rechromatographed on silica gel (650 g). The first fractions (4·0 g) were eluted with a mixture of 6% ether in light petroleum to which 3% of acetic acid were added. They still contained the sesquiterpenic acid. Further fractions (0·64 g) contained a product the IR spectrum of which showed the presence of OH groups (3610, 3430 cm^{-1}). According to gas chromatography (on Packard–Becker 427 chromatograph, column length 185 cm, i.d. 0·24 cm, support Gas-Chrom Q (80—100 mesh) impregnated with 3% of SE-30, temperature 260°C, carrier gas nitrogen) and comparison with a standard mixture it contained campesterol, stigmasterol and β-sitosterol.

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