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MINOR PHOSPHOLIPIDS OF THE COTTON PLANT OF VARIETY 159-F

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Continuing a study of the phospholipids (PL's) of the cotton plant of variety 159-F "Elit," 1974 harvest [1], by preparative TLC on type KSK silica gel in the chloroform–methanol–water (65:25:4) and chloroform–methanol–25% ammonia (14:6:1) systems we have isolated the homogeneous minor PL's (MPL's) x_2, x_3, and x_4 and lysophosphatidylcholine (lyso-PC).

In the products of the acid hydrolysis of the MPL's, in addition to fatty acids (FA's) we detected glycerol in the case of x_2 and x_3, ethanolamine and glycerol in the case of x_4, and choline and glycerol in the case of the lyso-PC. All the MPL's belong to the glycerophospholipid group:

Acid	x_2	x_3	x_4			Lyso-PC
			Total	O-acyl	N-acyl	
12:0	2,6	10,7	3,8	1,1	2,4	—
14:0	1,8	—	—	—	—	—
16:0	25,7	37,2	25,3	27,1	25,4	24,1
18:0	4,2	—	3,5	—	6,0	2,0
18:1	7,4	15,0	16,0	16,6	17,6	21,7
18:2	58,3	31,1	51,4	55,2	48,6	52,2
ΣS	34,3	47,9	32,6	28,2	33,8	26,1
ΣU	65,7	52,1	67,4	71,8	66,2	73,9

The IR spectra of all the MPL's had the characteristic absorption of CH, CH_2, CH_3, P–O–C and C=O (ester) groups and, in addition, in the case of x_4 there were the bands of a $C(=O)NHR$ group (1540 and 1640 cm^{-1}) and in the case of the lyso-PC the bands of P=O, $N^+(CH_3)_3$, and OH groups (1250, 975, and 3400-3200 cm^{-1}, respectively). As a standard we synthesized a N-acylphosphatidylethanolamine (N-acyl-PE) from phosphatidylethanolamine and margaroyl chloride [2]. The identity of the IR spectra, chromatographic mobilities, and the products of alkaline and acid hydrolysis of the N-acyl-PE and x_4 showed that x_4 is a N-acyl-PE.

We determined the N- and O-acyl groups in x_4 by Bomstein's method [3] (see above). As compared with other varieties of the cotton plant [4, 5], the total fatty-acid composition of the N-acyl-PE from the variety 159-F was more unsaturated. The fatty acids of the O- and N-acyl groups were distributed qualitatively and quantitatively almost identically, with the exception of the 18:0 acid attached to the amino group.

In its qualitative and quantitative composition, the lyso-PC of the cotton plant of variety 159-F differs little from that of other varieties [5].

On the basis of chromatographic mobilities, qualitative reactions, IR spectra, and the products of acid hydrolysis it may be assumed that x_2 and x_3 are polyglycerophosphatides.

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A NEW QUINOID PIGMENT FROM Lithospermum erythrorhizon

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In a study of the composition of the pigments from the roots of the plant Lithospermum erythrorhizon Sieb. et Zucc., family Boraginaceae (Maritime Territory) a new quinone of unknown structure was detected. This substance was first isolated by repeated chromatography on silica gel with subsequent purification on Sephadex LH-20 [1].

Subsequently, a different method of isolation was used: the total hexane extract from 500 g of air-dry roots was evaporated, and the residue was dissolved in chloroform and chromatographed on neutral alumina (activity grade III). The naphthoquinoid pigments were retained completely by the alumina, and the substance under investigation was eluted from the column in the form of an orange zone. After further purification on KSK silica gel in the hexane–ether (6 : 1) system, 90 mg of orange yellow needles of (I) were obtained with mp 72-74°C (hexane), $[\alpha]_D^{20} -81°$.

On the basis of the results of elementary analysis and the mass spectrum (M^+ 354), the composition $C_{21}H_{22}O_5$ was proposed for (I). When it was subjected to TLC on Silufol plates in the petroleum ether–diethyl ether (10 : 3.5) system it had R_f 0.42.

The pigment was unstable in acid and alkaline media. Under the action of the Ehrlich reagent [2], a change in color from orange to dark green was observed. Alkaline hydrolysis gave β,β-dimethylacrylic acid, which was identified with the aid of GLC. Absorption spectrum: $\lambda_{max}^{C_2H_5OH}$ 222, 244, 287, 444 nm (log ε 4.41; 4.38; 4.25; 3.67).

The IR spectrum had the absorption bands of quinoid carbonyls (1657 and 1670 cm^{-1}), of an ester carbonyl (1720 cm^{-1}), of methyl and methylene groups (2840-3020 cm^{-1}) and of the CH vibrations of an aromatic ring (3060 cm^{-1}), and also the characteristic absorption band of the vibrations of CH groups of an α-substituted furan ring (3160 cm^{-1}). The NMR spectra were taken on a Bruker HX-90E instrument with a working frequency of 22.63 MHz for ^{13}C nuclei and 90 MHz for ^1H in $CDCl_3$ (δ scale) with TMS as internal standard.

^1H NMR spectrum (s, singlet; d, doublet; t, triplet; m, multiplet): 1.61 (s, 3H, $3H_{15}$); 1.68 (s, 3H, $3H_{16}$); 1.89 (d, 3H, $3H_4'$, J = 0.9 Hz); 2.16 (d, 3H, $3H_5'$, J = 1.2 Hz); 2.56 (t, 2H, $2H_{12}$); 5.07 (t, 1H, $1H_{13}$); 5.76 (t, 1H, $1H_{11}$, J = 6.7 Hz); 5.68 (m, 1H, $1H_2'$); 6.74 (d, 2H, $1H_3$ and $1H_4$, degenerate AB spectrum); 7.03 (s, 1H, $1H_1$); 7.46 (s, 1H, $1H_9$), and 7.52 (s, 1H, $1H_8$).

In the ^{13}C NMR spectrum the signals of 21 carbon atoms were detected, five of which were quaternary and three of which belonged to carbonyl groups. The assignment of the signals of the carbon atoms was performed by the method of selective decoupling from the protons.

On comparing the ^{13}C and ^1H NMR spectra for (I) with the analogous spectra of esters of shikonin [1], it was found that the signals corresponding to the isopentenyl part of the chain coincided completely. The chemical shifts of H_{11}, and also of C_{11}, confirmed the position of the ester substituent at this carbon atom [1]. The C_2 and the C_5 chemical shifts (187.0 and 185.1 ppm, respectively) are characteristic for a monosubstituted

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