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SYNTHESIS OF α-MONOGLYCERIDES OF AROMATIC ACIDS

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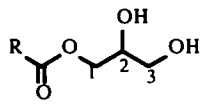
The synthesis of α-monoglycerides of aromatic acids has been performed by the transesterification of the methyl esters of the corresponding acids with glycerol. The structures of the compounds obtained have been confirmed by their IR, UV, and ^1H and ^{13}C NMR spectra.

Aromatic acids (phenolcarboxylic, hydroxycinnamic, and others) play an important role in the vital activity of plants and are frequently found in Nature in the form of esters with polyhydric alcohols and carbohydrates. [1]. Thus, for example, 1-O-galloylglycerol has been isolated from rhubarbs; it has also been obtained by synthesis [2]. 1-O-Feruloylglycerol, 1,2-di-O-feruloylglycerol and a number of derivatives of hydroxycinnamic acids and glycerol similar to them have been identified in *Lilium auratum* bulbs [3]. However, at the present time, the use of natural esters of aromatic acids with polyols is limited by their poor availability because of the difficulty of their isolation from plants (minor components) and the complexity of their synthesis (necessity for using protective groups), with the formation of by-products when acid chlorides and anhydrides are used [4, 5].

In continuation of investigations on the modification of organic carboxylic acids by polyhydric alcohols [6, 7], in the present paper we describe the synthesis of α-monoglycerides of aromatic acids by the transesterification of the methyl esters of the corresponding acids in the presence of an alkaline catalyst (KOH).



1-16

where R: **1** - C_6H_5-: **2** - C_6H_5-CH=CH-; **3** - C_6H_4OH-*o*; **4** - C_6H_5OH-*m*; **5** - C_6H_5OH-*p*; **6** - $C_6H_3(OH)_2$-2,4; **7** - $C_6H_2(OH)_3$-3,4,5; **8** - $C_6H_4NH_2$-*o*; **9** - $C_6H_4NH_2$-*m*; **10** - $C_6H_4NH_2$-*p*; **11** - (OCH_3-*m*, OH-*p*)C_6H_3-CH=CH-; **12** - C_6H_4Cl-*o*; **13** - $C_6H_3Cl_2$-2,4; **14** - 2,4-$Cl_2C_6H_3OCH_2$-; **15** - C_6H_4Br-*p*; **16** - $C_6H_4NO_2$-*p*

Of all the aromatic acid esters synthesized (**1**—**16**) only the α-monoglycerides of gallic and ferulic acids (**7**) an (**11**) were known previously [2, 3]; the synthesis and physicochemical characteristics of the other compounds are given in the present paper for the first time.

It is known that the reactivity of aromatic esters in the transesterification reaction depends on the polar influence of donor and acceptor substituents in the aromatic ring on the reaction center. In view of this, the yields of α-monoglycerides differed considerably (from 8—12% for the monoesters of *p*-amino- and *p*-hydroxybenzoic and gallic acids to 52—62% for the monoesters of *p*-nitrobenzoic and salicylic acids). The highest yield in the case of the monoester of salicylic acid can be explained by the fact that the OH group located in the *o*-position with respect to the carbonyl forms an intramolecular hydrogen bond which leads to an increase in the positive change on the C=O carbon and promotes the occurrence of the reaction.

The aromatic acid α-monoglycerides (**1**—**16**) were isolated from the reaction mixtures by column chromatography on silica gel L. The structures of the monoesters (**1**—**16**) were confirmed by their elementary analyses and by their IR, UV and ^1H and ^{13}C NMR spectra,

In the IR spectra of the monoglycerides the stretching vibrations of the C=C bonds of the aromatic ring were present in the 1450—1620 cm^{-1} region, and the C=O vibrations of the ester group at 1660—1760 cm^{-1}; the vibrations of associated OH

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groups were also present, in the 3100—3600 cm^{-1} region.

The UV spectra of the glycerol esters were determined by the chromophores of the initial aromatic acids. The extinctions of the absorption bands corresponded to the results calculated for monoesters.

In the ^1H NMR spectra of the compounds synthesized, as well as the signals of aromatic protons at 6.4—8.08 ppm, there were the signals of the protons of the glycerol residue: a two-proton doublet in the 4.6—4.72 ppm region corresponding to the methylene protons of an acylated primary hydroxy group, and a two-proton doublet in the 3.9—4.1 ppm region due to methylene protons present in the geminal position to a hydroxyl. The methine proton of the secondary hydroxy group of the glycerol molecule was represented by a multiplet at 4.10—4.55 ppm.

The ^{13}C NMR spectra confirmed that a primary hydroxy group of the glycerol molecule had been acylated, since carbon atoms (C-1) present in the α-position to an ester group undergo a downfield shift by 2.5—4.5 ppm and those in the C-2 position an upfield shift by 2—3 ppm in comparison with the chemical shifts of the carbon atoms of unsubstituted glycerol. The chemical shift of the C-3 carbon atom, present in the γ-position with respect to the other primary hydroxy group of glycerol had changed only insignificantly ($\Delta\delta = \pm 0.2$ ppm). The chemical shifts of the carbon atoms of the aromatic moieties agreed with the shifts of the initial methyl esters.

EXPERIMENTAL

IR spectra were recorded on a UR-20 instrument (compounds **1**, **2**, **12**, and **14** as films, and the others in KBr tablets), UV spectra on a Specord UV-VIS spectrophotometer, and ^1H NMR spectra on a Tesla-487 instrument (80 MHz) in C_5D_5N. ^{13}C NMR spectra were taken on a Bruker WP-80 instrument with a working frequency of 20.152 MHz in CD_3OD. The course of the reactions was monitored by TLC on Silufol UV-254 plates.

Synthesis of the α-Monoglycerides of Aromatic Acids (1—16). A mixture of 0.074 mole of the methyl ester of the appropriate acid with 20.2 g (0.22 mole) of glycerol was heated at 180—200°C for 6—14 h in an atmosphere of argon in the presence of 0.06 g (0.0011 mole) of KOH.

After cooling, the reaction products were extracted with ethyl acetate, the extract was washed free from glycerol with saturated sodium chloride solution and was dried over $MgSO_4$, and the solvent was distilled off. The α-monoglycerides (**1—16**) were isolated by column chromatography on silica gel L (0.04—0.1 mm) with elution by chloroform and chloroform—methanol (9:1).

This gave 4.46 g (31%) of substance (**1**), n_D^{20} 1.5360; d_4^{20} 1.6181; R_f 0.83 (chloroform—methanol (9:1). Found %: C 61.16; H 6.05; $C_{10}H_{12}O_4$. Calculated %: C 61.22; H 6.12.

UV spectrum (λ_{max}, nm): 229 (lg ε 4.06).

IR spectrum (ν, cm^{-1}): 1715 (C=O), 1600, 1585, 1500 (C=C), 3100-3600 (-OH).

^1H NMR (δ, ppm): 4.0 (2H, d, J = 4 Hz, CH_2OH, C-3), 4.13-4.49 (1H, m, CHOH, C-2); 4.64 (2H, d, J = 4 Hz, CH_2OCO-, C-1), 6.1 (2H, s, OH); 7.20 (3H, d, J = 8 Hz, Ar).

^{13}C NMR (δ, ppm): C-1 66.0 ($\Delta\delta$ = +3.16); C-2 70.0 (-2.2); C-3 63.0 (+0.16); arom. C (132.8, 129.9, 128.1); -COO- (166.7).

Substance **2**: 6.90 g (42%), n_D^{20} 1.5750, d_4^{20} 1.5910, R_f 0.33. Found %: C 64.91; H 6.35. $C_{12}H_{14}O_4$. Calculated %: C 64.86; H 6.31.

UV spectrum (λ_{max}, nm): 205, 217, 222, 278 (lg ε 3.98, 4.0, 3.94, 4.14).

IR spectrum (ν, cm^{-1}): 1705 (C=O), 1635, 1580, 1500, 1450 (C=C), 3100-3600 (-OH).

^1H NMR (δ, ppm): 4.0 (2H, d, J = 4 Hz, C-3); 4.1-4.48 (1H, m, C-2); 4.68 (2H, d, J = 4 Hz, C-1); 5.58 (2H, s, OH); 6.45 (1H, d, J = 15 Hz, CH=); 7.7 (1H, d, J = 15 Hz, CH=); 7.08-7.4 (5H, Ar).

^{13}C NMR (δ, ppm): C-1 65.6 ($\Delta\delta$ = +2.76); C-2 70.0 (-2.2); C-3 63.0 (+0.16); arom. C (135.28; 130.2; 128.6; 127.4); -COO- (167.1).

Substance **3**: 9.73 g (62%), mp 70-71°C, R_f 0.38. Found %: C 56.57; H 5.73. $C_{10}H_{12}O_5$. Calculated %: C 56.60; H 5.66.

UV spectrum (λ_{max}, nm): 208, 239, 308 (lg ε 4.67, 3.96, 3.67).

IR spectrum (ν, cm^{-1}): 1680 (C=O), 1610, 1585, 1485 (C=C), 3100-3600 (-OH).

^1H NMR (δ, ppm): 4.62 (2H, d, J = 4 Hz, C-1); 4.14-4.48 (1H, m, C-2); 4.0 (2H, d, J = 4 Hz, C-3); 5.83 (2H, s, OH); 6.6-7.7 (4H, m, Ar); 10.95 (1H, s, phenolic OH).

^{13}C NMR (δ, ppm): C-1 65.99 (Δδ = +3.15); C-2 69.8 (-2.4); C-3 62.8 (-0.04); arom. C (161.1; 135.3; 129.8; 118.8, 117.0, 112.2); COO- (169.7).

Substance **4**: 5.18 g (33%), mp 90-91°C, R_f 0.15. Found %: C 56.47; H 5.70. $C_{12}H_{14}O_4$. Calculated %: C 56.60; H 5.66.

UV spectrum (λ_{max}, nm): 211, 239, 301 (lg ε 4.56, 4.04, 3.66).

IR spectrum (ν, cm^{-1}): 1720 (C=O), 1590, 1460 (C=C), 3100-3600 (OH).

^{1}H NMR (δ, ppm): 4.65 (2H, d, J = 4 Hz, C-1); 4.10-4.48 (1H, m, C-2); 3.95 (2H, d, J = 4 Hz, C-3); 5.5 (2H, s, OH); 7.01 (2H, d, J = 4 Hz, Ar).

^{13}C NMR (δ, ppm): C-1 65.6 (Δδ = +2.76); C-2 69.8 (-2.4); C-3 62.8 (-0.04); arom. C (156.7; 130.6; 128.9; 120.5, 116.0); -COO- (166.7).

Substance **5**: 1.41 g (9%), mp 155-157°C, R_f 0.32. Found %: C 56.52; H 5.71. $C_{10}H_{12}O_5$. Calculated %: C 56.60; H 5.66.

UV spectrum (λ_{max}, nm): 202, 210, 260 (lg ε 4.22, 4.14, 4.20).

IR spectrum (ν, cm^{-1}): 1690 (C=O), 1610, 1580, 1460 (C=C), 3100-3600 (-OH).

^{1}H NMR (δ, ppm): 4.6 (2H, d, J = 4 Hz, C-1); 4.10-4.41 (1H, m, C-2); 3.91 (2H, d, J = 4 Hz, C-3); 6.9 (2H, d, J = 8 Hz, Ar); 7.95 (2H, d, J = 8 Hz, Ar).

^{13}C NMR (δ, ppm): C-1 65.8 (Δδ = +2.96); C-2 69.9 (-2.3); C-3 62.9 (+0.06); arom. C (162.6; 132.08; 121.48; 115.73); -COO- (167.6).

Substance **6**: 4.38 g (26%), mp 150-152°C, R_f 0.35. Found %: C 52.68; H 5.31. $C_{10}H_{12}O_6$. Calculated %: C 52.63; H 5.26.

UV spectrum (λ_{max}, nm): 208, 226, 261, 298 (lg ε 4.49, 4.07, 4.19, 3.91).

IR spectrum (ν, cm^{-1}): 1660 (C=O), 1620, 1520, 1500, 1460 (C=C), 3100-3600 (-OH).

^{1}H NMR (δ, ppm): 4.6 (2H, d, J = 4 Hz, C-1); 4.10-4.41 (1H, m, C-2); 3.98 (2H, d, J = 4 Hz, C-3); 6.41 (1H, d, J = 8 Hz, Ar); 6.6 (1H, d, J = 2 Hz, Ar); 7.7 (1H, d, J = 8 Hz).

^{13}C NMR (δ, ppm): C-1 65.4 (Δδ = +2.56); C-2 69.8 (-2.4); C-3 62.8 (-0.04); arom. C (164.0; 163.3; 131.4; 108.0, 104.2, 102.5); -COO (169.7).

Substance **7**: 2.17 g (12%), mp 178-181°C, R_f 0.16 (chloroform-methanol, 4:1). Found %: C 49.25; H 4.98. $C_{10}H_{12}O_7$. Calculated %: C 49.18; H 4.92.

UV spectrum (λ_{max}, nm): 218, 276 (lg ε 4.54, 4.12).

IR spectrum (ν, cm^{-1}): 1700 (C=O), 1535, 1455 (C=C), 3100-3600 (OH).

^{1}H NMR (δ, ppm): 4.6 (2H, d, J = 4 Hz, C-1); 4.0-4.54 (1H, m, C-2); 3.92 (2H, d, J = 4 Hz, C-3); 7.5 (2H, s,Ar).

^{13}C NMR (δ, ppm): C-1 65.4 (Δδ = +2.56); C-2 70.0 (-2.2); C-3 62.8 (-0.04); arom. C (146.4; 139.23; 121.57; 109.82); -COO- (167.09).

Substance **8**: 7.49 g (48%), mp 87-88°C, R_f 0.4. Found %: C 56.71; H 6.04; N 6.52. $C_{10}H_{13}NO_4$. Calculated %: C 56.87; H 6.16, N 6.63.

UV spectrum (λ_{max}, nm): 220, 248, 340 (lg ε 4.36, 3.81, 3.61).

IR spectrum (ν, cm^{-1}): 1690 (C=O), 1620, 1595, 1560 (C=C), 3000-3340 (-OH), 3360, 3470 ($-NH_2$).

^{1}H NMR (δ, ppm): 4.7 (2H, d, J = 5 Hz, C-1); 4.28-4.5 (1H, m, C-2); 4.04 (2H, d, J = 5 Hz, C-3); 5.8 (4H, s, 2OH, NH_2); 6.5 (1H, d, J = 7 Hz, Ar, H-6); 6.85 (1H, d, J = 7 Hz, Ar, H-5); 7.1 (1H, d, J = 7 Hz, Ar, H-4); 7.96 (1H, d, J = 7 Hz, Ar, H-3).

^{13}C NMR (δ, ppm): C-1 65.4 (Δδ = +2.56); C-2 70.0 (-2.2); C-3 63.0 (+0.16); arom. C (150.9; 133.8; 130.8; 116.6, 115.4, 110.0); -COO- (168.0).

Substance **9**: 3.90 g (25%), mp 141-142.5°C, R_f 0.32. Found %: C 56.73; H 6.02, N 6.55. $C_{10}H_{13}NO_4$. Calculated %: C 56.87; H 6.16, N 6.63.

UV spectrum (λ_{max}, nm): 222, 247, 321 (lg ε 4.29, 3.83, 3.20).

IR spectrum (ν, cm^{-1}): 1720 (C=O), 1600, 1490 (C=C), 3100-3560 (-OH).

^{1}H NMR (δ, ppm): 4.72 (2H, d, J = 5 Hz, C-1); 4.13-4.53 (1H, m, C-2); 4.0 (2H, d, J = 5 Hz, C-3); 5.60 (4H, s, 2OH, NH_2); 7.02-7.65 (4H, m, Ar).

^{13}C NMR (δ, ppm): C-1 65.7 (Δδ = +2.86); C-2 69.9 (-2.3); C-3 63.0 (+0.16); arom. C (147.2; 130.6; 129.01; 120.16, 119.3, 116.08); -COO- (167.2).

Substance **10**: 1.25 g (8%), mp 115-117°C, R_f 0.41. Found %: C 56.79; H 6.10, N 6.58. $C_{10}H_{13}NO_4$. Calculated %: C 56.87; H 6.16, N 6.63.

UV spectrum (λ_{max}, nm): 221, 295 (lg ε 3.95, 4.23).

IR spectrum (ν, cm^{-1}): 1690 (C=O), 1610, 1530, 1450 (C=C), 3000-3430 (-OH), 3450 ($-NH_2$).

^1H NMR (δ, ppm): 4.05 (2H, d, J = 5 Hz, C-3); 4.18-4.6 (1H, m, C-2); 4.7 (2H, d, J = 5 Hz, C-1); 6.25 (4H, s, 2OH, NH_2); 6.78 (2H, d, J = 9 Hz, Ar), 8.0 (2H, d, J = 9 Hz, Ar)..

^{13}C NMR (δ, ppm): C-1 65.5 (Δδ = +2.66); C-2 70.2 (-2.0); C-3 63.0 (+0.16); arom. C (153.2; 131.6; 117.4; 113.6); -COO- (167.6).

Substance **11**: 3.57 g (18%), mp 47-49°C, R_f 0.51. Found %: C 58.15; H 5.81. $C_{13}H_{16}O_6$. Calculated %: C 58.21; H 5.97.

UV spectrum (λ_{max}, nm): 218, 238, 318, 327 (lg ε 4.25, 4.17, 4.16, 4.27).

IR spectrum (ν, cm^{-1}): 1720 (C=O), 1610, 1525 (C=C), 3050-3650 (-OH).

^1H NMR (δ, ppm): 3.6 (3H, s, $-OCH_3$); 4.05 (2H, d, J = 5 Hz, C-3); 4.18-4.5 (1H, m, C-2); 4.7 (2H, d, J = 5 Hz, C-1); 6.5 (1H, d, J = 16 Hz, CH=); 6.73-7.1 (3H, Ar), 7.7 (1H, d, J = 16 Hz, CH=).

^{13}C NMR (δ, ppm): C-1 65.5 (Δδ = +2.66); C-2 70.08 (-2.12); C-3 63.02 (+0.18); arom. C (151.48; 149.36; 127.34; 122.53, 115.08, 112.72); -CH= (145.09, 116.36), -COO- (167.47).

Substance **12**: 7.83 g (46%), n_D^{20} 1.5370, d_4^{20} 1.7335, R_f 0.4. Found %: C 52.10; H 4.80, Cl 15.46. $C_{10}H_{11}O_4Cl_4$. Calculated %: C 52.06; H 4.80, Cl 15.40.

UV spectrum (λ_{max}, nm): 229, 282 (lg ε 4.13, 3.24).

IR spectrum (ν, cm^{-1}): 1720 (C=O), 1596, 1480 (C=C), 3100-3600 (-OH), 680, 740 (C-Cl).

^1H NMR (δ, ppm): 4.69 (2H, d, J = 4 Hz, C-1); 4.13-4.5 (1H, m, C-2); 4.0 (2H, d, J = 5 Hz, C-3); 7.0-7.74 (4H, Ar).

^{13}C NMR (δ, ppm): C-1 66.4 (Δδ = +3.56); C-2 69.8 (-2.4); C-3 63.0 (+0.16); arom. C (133.1; 132.6; 131.2; 130.6, 129.9, 126.7); -COO- (165.7).

Substance **13**: 9.80 (50%), mp 68-70°C, R_f 0.4. Found %: C 45.32; H 3.81, Cl 26.70. $C_{10}H_{10}O_4Cl_2$. Calculated %: C 45.28; H 3.77, Cl 26.79.

UV spectrum (λ_{max}, nm): 208, 240 (lg ε 4.53, 4.03).

IR spectrum (ν, cm^{-1}): 1715 (C=O), 1480, 1590 (C=C), 760, 640 (C-Cl), 3200-3600 (-OH).

^1H NMR (δ, ppm): 4.69 (2H, d, J = 5 Hz, C-1); 4.12-4.5 (1H, m, C-2); 4.0 (2H, d, J = 5 Hz, C-3); 5.8 (2H, s, OH); 7.11-7.71 (3H, Ar).

^{13}C NMR (δ, ppm): C-1 66.5 (Δδ = +3.66); C-2 69.8 (-2.4); C-3 63.0 (+0.16); arom. C (138.0; 134.4; 132.6; 130.4, 128.3, 126.9); -COO- (164.5).

Substance **14**: 8.73 g (40%), n_D^{20} 1.5660, R_f 0.60. Found %: C 44.68; H 4.00, Cl 24.00. $C_{11}H_{12}O_5Cl_2$. Calculated %: C 44.74; H 4.07, Cl 24.06.

UV spectrum (λ_{max}, nm): 224, 232, 288, 296 (lg ε 4.12, 4.14, 3.54, 3.47).

IR spectrum (ν, cm^{-1}): 1760 (C=O), 1450, 1492 (C=C), 3170-3640 (-OH), 780 (C-Cl).

^1H NMR (δ, ppm): 3.9 (2H, d, J = 5 Hz, $-OCH_2$); 4.08 (2H, d, J = 5 Hz, C-3); 4.2-4.45 (1H, m, C-2); 4.6 (2H, d, J = 5 Hz, C-1), 4.85 (2H, s, OH), 7.05-7.3 (3H, Ar).

^{13}C NMR (δ, ppm): C-1 66.45 (Δδ = +3.61); C-2 70.02 (-2.18); C-3 63.13 (+0.29); arom. C (129.93; 127.85; 126.87; 124.06, 115.48); -COO- (168.9).

Substance **15**: 9.56 g (47%), mp 72-74°C, R_f 0.45. Found %: C 43.68; H 4.12, Br 28.96. $C_{10}H_{11}O_4Br$. Calculated %: C 43.63; H 4.00, Br 29.10.

UV spectrum (λ_{max}, nm): 245 (lg ε 4.23).

IR spectrum (ν, cm^{-1}): 1715 (C=O), 1590, 1485 (C=C), 690 (C-Br), 3100-3600 (-OH).

^1H NMR (δ, ppm): 4.65 (2H, d, J = 4 Hz, C-1); 4.10-4.49 (1H, m, C-2); 3.98 (2H, d, J = 4 Hz, C-3); 5.75 (2H, s, -OH); 7.35 (2H, d, J = 8 Hz, Ar), 7.80 (2H, d, J = 8 Hz, Ar).

^{13}C NMR (δ, ppm): C-1 66.2 (Δδ = +3.36); C-2 69.9 (-2.3); C-3 63.0; arom. C (131.4; 130.9; 128.9; 127.7); -COO- (165.80).

Substance **16**: 9.27 g (52%), mp 105-107°C, R_f 0.4. Found %: C 49.83; H 4.50, N 5.73. $C_{10}H_{11}NO_6$. Calculated %: C 43.79; H 4.56, N 5.81.

UV spectrum (λ_{max}, nm): 260 (lg ε 4.19).

IR spectrum (ν, cm^{-1}): 1725 (C=O), 1605, 1445 (C=C), 1355, 1520 (Ar-NO_2), 3200-3550 (-OH).

^1H NMR (δ, ppm): 4.72 (2H, d, J = 4 Hz, C-1); 4.18-4.55 (1H, m, C-2); 4.02 (2H, d, J = 4 Hz, C-3); 5.7 (2H, s, OH);

8.08 (4H, s, Ar).

^{13}C NMR (δ, ppm): C-1 67.2 (Δδ = +4.36); C-2 69.8 (-2.24); C-3 63.0 (+0.16); arom. C (150.70; 135.80; 130.40; 123.30); -COO- (164.8).

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