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GC-MS Characterization of Acetylated *O*-glucofuranosides: Direct Glucosylation of Volatile Alcohols from Unprotected Glucose

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Keywords
• direct glucosylation using $FeCl_3$
• glucofuranosides and glucopyranosides
• GC-MS spectra of glucofuranoside tetraacetates

O-Glucosylation of 1-pentanol, 1-octanol, 2-phenylethanol, benzyl alcohol, (±)-2-pentanol and (±)-menthol in 1,4-dioxane, using anhydrous $FeCl_3$, afforded anomeric mixture of the corresponding glucofuranosides as major and glucopyranosides as minor products in overall yields 20–52 %. Practical advantages of GC-MS for characterizing the prepared acetylated glucofuranosides are in the focus of this paper. Glucofuranoside tetraacetate spectra contain characteristic signals of glucone (acetylated glucose) along with fragments of the aglucone moiety. The mass range was 50–600 mass units and acetyl ion was not present in the spectra, which is of interest for differentiating glucofuranoside and glucopyranoside tetraacetates.

INTRODUCTION

Chemical synthesis of *O*-glycosides from unprotected glycosyl donors requires i) trapping of the glycosyl acceptor faster than the reaction with hydroxy groups of the donor, ii) control of the substrate intermediate(s) and/or products equilibrium, iii) control of the products α/β-anomery.[1] Direct enzymatic glycosylation is also an alternative to inherently circuitous chemical synthesis of glucopyranosides with protection and deprotection steps, but gives limited yields.[2,3]

After the original Fischer method of glycosylation of lower alcohols with unprotected carbohydrates using protic acid, Fischer and Lubineau[4] (1991) developed one-step conversion of glucose and galactose to the corresponding α/β-methyl glycofuranosides using ferric chloride. Later, Ferrières *et al.*[5] investigated the reactivity of a few long-chain alcohols toward unprotected and inactivated aldoses in different solvents, which afforded alkyl furanosides in high yields. It has been shown that the relative proportions of α/β-anomers produced by direct chemical glycosylation may be changed in the presence of alkaline-earth metal ions.[5,6] These glycofuranosides are becoming important as surfactants, liquid crystals and building blocks for glycofuranosyl donors in oligosaccharide synthesis.[7–9] Further, some water-soluble biodegradable derivatives of lignin as well as lignin model monomeric compounds were also prepared by this methodology.[10]

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Glycosylation from unprotected carbohydrates with Lewis acid has been insufficiently investigated, since Lewis acids were mainly used for promotion of glycosylation starting from protected or activated glycosyl donors.[11,12] Practical advantages of GC-MS for characterizing various acetylated glucosides prepared by direct chemical synthesis are in the focus of this paper. We have prepared glucofuranosides of (±)-menthol and, to our knowledge, no other monoterpenyl glycoside has been prepared so far using the above rapid and simple method. The spectra of acetylated glucofuranosides obtained in direct chemical synthesis with $FeCl_3$ are compared to the spectra of acetylated glucopyranosides from our previous papers.[13–15]

MATERIALS AND METHODS

Materials

D-Glucose was obtained from Kemika, Zagreb, Croatia. Silicagel plates (Kieselgel 60, 0.2 mm) were purchased from Merck, Darmstadt, Germany. Silicagel for flash chromatography (30–60 μm) was obtained from Mallinckrodt Baker B.V., Deventer, Netherlands. All other reagents, solvents, anhydrous $FeCl_3$ and Celite R-640® were of the highest grade available commercially from Fluka Chemie, Buchs, Switzerland. Secondary alcohols were used as racemic mixtures. 1,4-Dioxane was purified and dried before use by refluxing over excess of sodium.[16] Calcium chloride was dehydrated by heating at 215 °C for two hours.

Glucosylation of Alcohols from Glucose Using $FeCl_3$

D-Glucose (20 mmol), appropriate alcohol (25 mmol), anhydrous $CaCl_2$ (20 mmol) and anhydrous $FeCl_3$ (20 mmol) were separately added into 1,4-dioxane. Since the reaction was performed in heterogeneous media, the mixtures were vigorously shaken in sealed vials at room temperature on a shaker. The reaction progress was TLC monitored. After 24 hours, inert Celite R-640® (3 g) was added and the reaction was stopped by adding aqueous Na_2CO_3 (in portions under stirring), until the solution was slightly alkaline (pH 8–9). Then, the reaction mixture was stirred for 15 min. Aqueous layer was separated in a centrifugal apparatus, decanted and the precipitate was extracted twice with water (20 mL) and once with ethanol (10 mL). The combined water-ethanol extracts were concentrated in a rotating evaporator under reduced pressure and further purified by flash chromatography on silicagel column applying a mixture of EtOAc, EtOH and conc. NH_3 (ψ = 6:3:1). Glucoside yields were determined by a gravimetric method, after purification by flash column chromatography.

Thin Layer Chromatography

Prepared glucosides were chromatographed on silicagel plates. A mixture of EtOAc, EtOH and conc. NH_3 (ψ = 6:3:1) and a mixture of chloroform and methanol (ψ = 5:1) were used as solvents for glucosides elution.[13] After developing the chromatograms, visualization was performed by spraying with 5 % vanillin in H_2SO_4, followed by heating at 125 °C for 10 minutes. Individual glucosides were detected as dark spots, except for violet glucosides of (±)-menthol.

Acetylation

Acetic anhydride (1 mL) was separately added to purified glucosides (10 mg), dissolved in dry pyridine (0.4 mL) and the mixture was heated at 100 °C. After 1 hour, the reaction mixture was cooled at room temperature and 20 mL of cold water with ice was added.[13,14] Aqueous layer was extracted with ether. The combined ether extracts were washed with saturated aqueous $NaHCO_3$, then with water and dried over anhydrous $MgSO_4$, concentrated to 0.5 mL, and 1 μL was used for GC-MS analysis.

Gas Chromatography – Mass Spectrometry Analysis

Duplicate analyses of acetylated glucosides were performed on a GC-MS Hewlett Packard (model 5890) with a mass selective detector (model 5971A). GC operating conditions:[13,14] column HP-101 (Dimethylpolysiloxane, Hewlett Packard, Vienna, Austria), 25 m x 0.2 mm i.d., film thickness 0.2 μm, column temperature programmed from 150 °C (isothermal for 4 minutes) to 220 °C (10 °C/min). Carrier gas helium, flow rate 1 mL/min. Injector temperature 250 °C; volume injected 1 μL; split ratio 1 : 50. MS conditions: ionization voltage 70 eV; ion source temperature 280 °C; mass range 50–600 mass units. Ratio of glucofuranoside/glucopyranoside tetraacetates and the anomeric ratio were determined from their peak areas on the same chromatogram.

RESULTS AND DISCUSSION

Glucofuranosides of various volatile alcohols were prepared by direct chemical glucosylation and acetylated for GC-MS analysis, Figure 1. TLC values of the reaction products in both solvents indicated that the main products were glucofuranosides with higher R_F values compared to the corresponding glucopyranosides from our earlier paper.[14] GC-MS analysis of acetylated products on HP-101 column also indicated that the main products were glucofuranosides, since tetraacetyl glucopyranosides were eluted with different retention times, as described in our previous papers.[13–15] Furanoside structures of the glucosides obtained under the same reaction conditions have been already determined by NMR spectroscopy in other papers.[5,10,12] It was also confirmed that in direct chemical synthesis with $FeCl_3$, the ring expansion to pyranosides is prevented by the presence of ferric ions in the reaction mixture.[4]

The obtained results are summarized in Table I with overall glucoside yields of 20–52 %. The reaction performance decreased when the alcohol chain length increased. The presence of phenyl substituent at the second carbon atom in the aliphatic chain did not represent any significant steric interference for glucosylation. Although menthol is a secondary monoterpene alcohol, the corre-

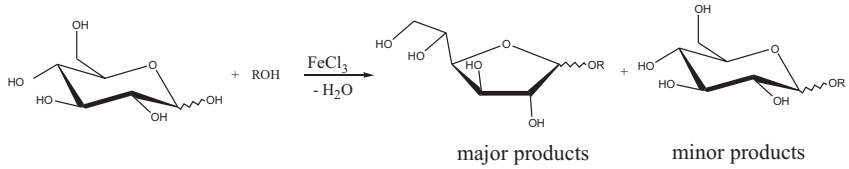

Figure 1. Direct glucosylation from unprotected glucose using $FeCl_3$.

sponding glucosides were prepared in relatively high yields (38 %). To the best of our knowledge, this is the first time that a monoterpene glycoside was prepared in direct chemical synthesis without an enzyme.

For this investigation, 1,4-dioxane was selected as a suitable solvent due to the slight solubility of reactant glucose, so no self-condensation of glucose would occur. In our preliminary investigations, ammonia was added into the reaction mixture to remove the promoter, but there were some problems with the separation of the obtained products, due to glucoside adsorption on the $Fe(OH)_3$ precipitate formed. Adsorption was less observable on $Fe_2(CO)_3$. To facilitate product extraction, inert Celite R-640® was added into the reaction media just before the start of precipitation with the addition of Na_2CO_3 aqueous solution.

Anomeric mixtures of glucofuranosides were obtained as the main products of direct glucosylation with $FeCl_3$, Table I. After acetylation of the reaction products, the corresponding glucoside teraacetates were analyzed by GC-MS for the first time. Molecular ions are rarely observed in EI MS. In general, the first detectable fragment ion in the high mass region corresponds to elimination of an acetoxy radical or acetic acid from molecular ion. Base peak is usually acetyl ion at *m/z* 43. We used the mass range from 50–600 mass units, so less intensive signals are visible since the dominant acetyl ion (*m/z* 43) is not present. This is important for better distinction between acetylated glucofuranoside and glucopyranoside spectra.[13] α/β-Anomers of acetylated glucofuranosides were not determined, only their ratio. Both anomers have similar spectra and they could be determined by different retention times of known standards.

Glucofuranoside tetraacetate spectra of 1-pentanol *m/z* 418 (M^+), (±)-2-pentanol *m/z* 418 (M^+, 2) and 1-oc-

TABLE I. Yields of the prepared glucosides and the ratio of glucofuranoside/glucopyranoside tetraacetates

No.	Glucosyl acceptor	Glucoside Yield/%[(c)]	Glucoside tetraacetates ratio[(a)]		
			furanoside: pyranoside	anomers[(b)] (furanosides)	α/β-anomers (pyranosides)
1.	1-pentanol	51.0	99 : 1	4.6 : 1	0 : 1
2.	(±)-2-pentanol	21.0	60 : 40	/	21 : 16
3.	1-octanol	20.0	60 : 40	2 : 1	1 : 99
4.	benzyl alcohol	29.0	95 : 5	1 : 1	0 : 1
5.	2-phenylethanol	52.0	99 : 1	1 : 99	0 : 1
6.	(±)-menthol	38.0	70 : 30	/	1.1 : 1.8

[(a)]Ratio of furanoside α/β-anomers is not determined for (±)-alcohols because of similarity of their mass spectra (4 furanoside and 4 pyranoside diastereomers for (±)-alcohols).
[(b)]Furanoside anomers are not determined, their ratio is presented.
[(c)]Overall yields of glucofuranosides and glucopyranosides.

TABLE II. Mass spectra and retention times of tetraacetyl glucosides obtained by acetylation of glucosylation products

Glucoside Tetraacetate of	Furanoside		Pyranoside	
	α/β-anomer		α-anomer	β-anomer
1-pentanol	55(38), 69(61), 71(58), 81(70), 85(43), 97(48), 98(100), 112(100), 115(50), 127(25), 141(42), 143(50), 157(40), 169(30), 171(65), 200(15), 242(10), 273(22), 274(2), 331(2)	55(38), 71(60), 73(71), 81(70), 85(48), 97(48), 98(100), 112(100), 115(61), 127(32), 141(82), 143(70), 157(40), 169(30), 171(45), 185(20), 200(18), 201(4), 242(4), 273(18)	/	55(38), 69(40), 81(100), 97(50), 98(100), 102(50), 103(60), 112(70), 115(86), 141(70), 157(55), 171(30), 225(10), 243(20), 273(8), 331(2), 337(2)
	$t_{ret.}$ = 14.83 min	$t_{ret.}$ = 15.01 min		$t_{ret.}$ = 15.30 min

TABLE II. continued

Glucoside Tetraacetate of	Furanoside		Pyranoside	
	α/β-anomer		α-anomer	β-anomer
1-octanol	55(58), 57(73), 71(55), 81(57), 98(80), 109(30), 112(100), 140(50), 145(45), 148(38), 169(30), 213(40), 242(10), 315(18), 331(8)	55(58), 57(73), 71(55), 81(57), 98(80), 109(30), 112(100), 140(50), 145(45), 148(38), 169(28), 213(40), 242(10), 315(18), 331(8)	/	55(53), 57(59), 81(100), 98(78), 112(55), 115(62), 157(50), 169(28), 243(12), 267(10), 289(2), 331(1), 423(1)
	$t_{ret.}$ = 23.1 min	$t_{ret.}$ = 23.5 min		$t_{ret.}$ = 25.8 min
benzyl alcohol	65(8), 81(2), 91(100), 97(10), 110(10), 115(3), 127(2), 139(15), 152(8), 157(4), 169(2), 331(2)	65(10), 81(2), 91(100), 97(10), 110(8), 115(8), 139(20), 152(8), 245(1), 321(1), 331(3)	/	55(2), 65(8), 79(8), 91(100), 97(11), 139(30), 152(8), 157(10), 169(6), 216(1), 331(2)
	$t_{ret.}$ = 24.7 min	$t_{ret.}$ = 25.11 min		$t_{ret.}$ = 27.42 min
2-phenylethanol	79(12), 81(10), 91(12), 98(10), 104(48), 105(100), 112(10), 127(10), 169(20), 242(1), 331(2)	79(10), 81(8), 91(18), 98(12), 104(50), 105(100), 112(10), 127(10), 169(22), 242(1), 331(2)	/	81(12), 91(18), 98(10), 104(48), 105(100), 115(11), 127(10), 169(18), 331(1)
	$t_{ret.}$ = 29.87 min	$t_{ret.}$ = 32.04 min		$t_{ret.}$ = 34.69 min
(±)-2-pentanol	α/β-glucofuranoside tetraacetate of (±)-2-pentanol			
	71(40), 81(38), 98(70), 109(38), 115(58), 126(50), 140(30), 143(100), 145(40), 157(38), 169(70), 200(20), 242(8), 273(20), 331(50), 418 (**M$^+$,** 2)	71(40), 81(40), 98(70), 109(40), 115(58), 126(50), 140(30), 143(100), 145(40), 157(38), 169(70), 200(20), 242(8), 273(18), 331(50), 418 (**M$^+$,** 2)	71(30), 98(100), 109(38), 115(90), 126(38), 140(30), 143(60), 145(42), 157(50), 169(80), 200(24), 242(22), 273(10), 331(20), 418(**M$^+$**, 2)	81(44), 98(100), 109(42), 115(90), 126(48), 140(38), 143(100), 145(42), 157(50), 169(80), 200(30), 242(18), 273(10), 331(36), 418(**M$^+$**, 2)
	$t_{ret.}$ = 12.56 min	$t_{ret.}$ = 12.78 min	$t_{ret.}$ = 12.86 min	$t_{ret.}$ = 12.96 min
	α/β-glucopyranoside tetraacetate of (±)-2-pentanol			
	81(28), 98(95), 109(38), 115(100), 126(28), 140(36), 145(37), 157(60), 169(40), 200(37), 242(18), 287(8), 331(27), 364(5), 418(**M$^+$**, 3)	81(38), 98(100), 109(50), 115(100), 126(30), 140(42), 145(40), 157(50), 169(50), 200(30), 242(28), 286(10), 331(24), 418(**M$^+$**, 3)	81(38), 98(70), 109(30), 115(100), 140(40), 145(24), 157(80), 169(59), 200(40), 242(30), 271(2), 331(20), 418 (**M$^+$**, 2)	/
	$t_{ret.}$ = 13.21 min	$t_{ret.}$ = 13.29 min	$t_{ret.}$ = 13.54 min	
(±)-menthol	α/β-glucofuranoside tetraacetate of (±)-menthol			
	55(38), 69(33), 81(38), 83(78), 95(20), 97(18), 101(100), 109(10), 115(8), 127(20), 139(22), 143(10), 203(22), 229(8), 331(2), 427(5)	55(60), 69(40), 81(58), 83(90), 95(30), 97(20), 101(100), 109(18), 115(18), 127(34), 139(38), 143(18), 203(28), 229(18), 331(2), 427(8)	55(50), 69(38), 81(40), 83(90), 95(22), 97(24), 101(100), 109(10), 115(8), 127(30), 139(28), 143(26), 203(30), 229(10), 331(1), 427(8)	/
	$t_{ret.}$ = 17.88 min	$t_{ret.}$ = 18.38 min	$t_{ret.}$ = 18.54 min	
	α/β-glucopyranoside tetraacetate of (±)-menthol			
	55(50), 69(48), 81(58), 83(100), 97(38), 109(30), 115(25), 138(48), 157(30), 169(58), 203(10), 287(2), 289(2), 331(10)	55(60), 81(58), 83(100), 97(38), 109(30), 138(50), 169(48), 200(8), 242(10), 284(10), 331(2)	55(60), 69(50), 81(52), 83(100), 97(40), 109(20), 115(38), 138(40), 157(38), 169(42), 200(10), 242(10), 289(4), 331(10)	55(60), 69(50), 81(50), 83(100), 97(40), 109(20), 115(38), 138(30), 157(38), 169(28), 200(22), 242(28), 289(4), 331(2)
	$t_{ret.}$ = 24.03 min	$t_{ret.}$ = 25.21 min	$t_{ret.}$ = 27.82 min	$t_{ret.}$ = 30.44 min

tanol *m/z* 460 (M^+) contain abundant peaks *m/z* 112, 98 and 143. Fragment *m/z* 143 originated from *m/z* 331 by elimination of one *O*-acetyl group (when *m/z* 272 was obtained) and three acetyl groups, Figure 2. Glucofuranoside-tetraacetate ion (*m/z* 331) is also present. Peak *m/z* 213 in the spectrum of 1-octyl glucofuranoside tetraacetate, probably originated from elimination of two *O*-acetyl groups from *m/z* 331. Ion *m/z* 171 (in the spectrum of 1-pentyl glucofuranoside tetraacetate) was obtained by elimination of one acetyl and one *O*-acetyl group from *m/z* 272. Ion $C_2H_3(OAc)_2^+$, *m/z* 145 can be attributed to the elimination reactions of the side chain on C-4 furanosides. Peak *m/z* 98 was obtained by elimination of one *O*-acetyl group from *m/z* 157 ($C_3H_3(OAc)_2^+$, furanoside ring fragmentation), and further elimination of one acetyl group led to *m/z* 55. This peak is abundant in all spectra and it can also originate from the aglucone part. Characteristic ions from the aglucone part are present in a mass range up to *m/z* 70 for 1-pentyl and 2-pentyl, and *m/z* 84 for 1-octyl aglucone. They are not so specific, so they could be used for identification of a particular aglucone part in the series of aliphatic alcohols. Identification of particular glucofuranoside tetraacetate of aliphatic alcohols is based on their different retention times, Table I. α/β-Anomer ratios of 1-pentyl glucofuranosides were not determined due to similar mass spectra. Namely, eight diastereomers can originate from direct glucosylation of racemic 2-pentanol (4 furanosides and 4 pyranosides). No rearrangement of 1-pentanol to 2-pentanol was noted (due to acidic media) by comparison of retention times and mass spectra of 1-pentyl and 2-pentyl glucoside tetraacetates. Different peaks among the mass spectra of glucofuranoside and glucopyranoside tetraacetates with aliphatic aglucone were: i) for furanosides abundant *m/z* 112, 143 and 183, more intensive *m/z* 273 and 171, *m/z* 213 and 315 (not present in 1-octyl glucopyranoside te-

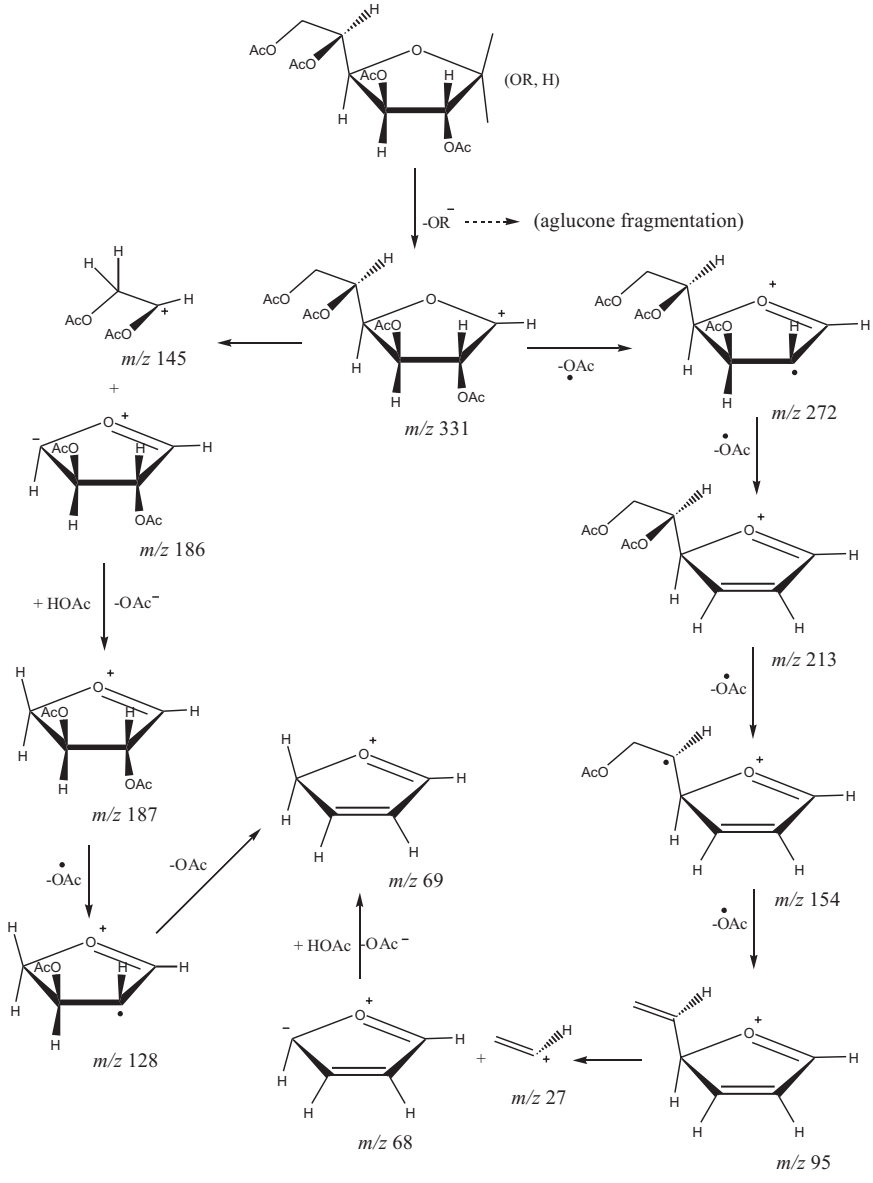

Figure 2. Fragmentation of glucofuranoside tetraacetates (70 eV) according to the fragmentation of glucopyranosides[17] and the obtained spectra.

traacetate), *m/z* 331 more abundant in 2-pentyl glucofuranoside tetraacetate; ii) for pyranosides *m/z* 81 more intensive, *m/z* 115 intensive, *m/z* 243 not observed in 1-octyl glucofuranoside tetraacetate.

Spectrum of benzyl glucofuranoside tetraacetates *m/z* 438 (M^+) contains remarkably abundant tropilium ion *m/z* 91 from the aglucone part. Peaks *m/z* 139, 152 and 331 are present in all spectra. No marked differences between the spectrum of benzyl glucopyranoside and glucofuranoside tetraacetate were noted. Spectra of 2-phenylethyl glucofuranoside tetraacetates *m/z* 452 (M^+) contain parent ion *m/z* 105 ($C_8H_7^+$) from the aglucone part. Less intensive fragments are *m/z* 104 ($C_8H_8^+$), 79, 169 and 141. No marked differences in the spectrum of 2-phenylethyl glucofuranosides and glucopyranosides were observed.

The spectra of glucofuranoside tetraacetates of (±)-menthol *m/z* 486 (M^+) contain abundant peaks *m/z* 101 and 83. Peaks *m/z* 55, 203 and 139 follow in intensity. In the reaction of direct glucosylation with (±)-menthol, eight diastereomers can be produced (seven peaks were detected in the chromatogram, Table I). Peaks that are different in the spectra of menthyl glucofuranosides and menthyl glucopyranosides are: i) for furanosides abundant *m/z* 101, *m/z* 203; ii) for pyranosides *m/z* 242, abundant signals *m/z* 157, 169 and 115.

CONCLUSION

Glucofuranosides of different volatile alcohols were synthesized by direct chemical glucosylation using $FeCl_3$. Further work is needed to identify the corresponding glucofuranoside α/β-anomers, especially for racemic glucosyl acceptors. Product characterization was done by GC-MS analysis of the prepared tetraacetyl glucosides. Fragments characteristics of the aglucone moiety were present in all spectra. Mass range 50–600 mass units enable better differentiation of glucofuranoside and glucopyranoside spectra.

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SAŽETAK

GC-MS karakterizacija acetiliranih *O*-glukofuranozida: izravna glukozilacija isparljivih alkohola iz nezaštićene glukoze

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O-glukozilacija 1-pentanola, 1-oktanola, 2-feniletanola, benzil-alkohola, (±)-2-pentanola i (±)-mentola u 1,4-dioksanu, rabeći bezvodni $FeCl_3$, dala je odgovarajuće glukofuranozide kao glavne i glukopiranozide kao sporedne produkte s ukupnim prinosima 20–52 %. Praktične prednosti GC-MS za karakterizaciju pripravljenih glukofuranozida su u žarištu ovoga rada. Spektri glukofuranozid tetraacetata sadrže karakteristične signale glukona (acetilirane glukoze) zajedno s fragmentima aglukonskoga dijela. Interval praćenja masa bio je 50–600 masenih jedinica i acetil-ion nije bio prisutan u spektrima, što je od interesa za razlikovanje glukofuranozid i glukopiranozid tetraacetata.