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Naphthalene-Catalysed Lithiation of 2-(Chlorophenyl)-1,3-dioxolanes: Generation of Formyl- and Acetyl-phenyllithium Equivalents [1]†

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Abstract

The reaction of 2-(chlorophenyl)-1,3-dioxolanes **1** with an excess of lithium powder and a catalytic amount of naphthalene (5 mol %) in the presence of different carbonyl compounds [^{t}BuCHO, Et_2CO, $(CH_2)_5CO$, PhCOMe] as electrophiles (Barbier-type conditions) in THF at -78°C leads, after hydrolysis with water at the same temperature, to the expected products **3**, the corresponding intermediates involved being formyl- or acetyl-phenyl anion equivalents.

Keywords: lithiation; dioxolanes; catalysis; lithium and compounds

I. Introduction

Functionalised organolithium compounds [2,3] are interesting intermediates in synthetic organic chemistry because in their reaction with electrophiles polyfunctionalised molecules can be directly prepared. One problem to be overcome when these intermediates are very unstable is the lithiation step, which has to be carried out at low temperature in order to avoid decomposition of the organolithium. Recently, we discovered that the use of a catalytic amount of an arene [usually naphthalene or 4,4'-di-*tert*-butylbiphenyl (DTBB)] allows the lithiation to be performed under very mild reaction conditions [4,5]. Thus, using this methodology functionalised organolithium compounds can be easily prepared from halogenated [6][1] or non-halogenated [7][1] materials as well as from heterocyclic precursors [8][1][9]. Concerning this last type of compound, 2-(chloroalkyl) (**I**) [10-15] or 2-(chloro-

† This paper is dedicated to Professor A. R. Katritzky on occasion of his 70th birthday.

[1] For the last paper on this topic from our laboratory, see the corresponding reference indicated in the text.

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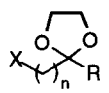
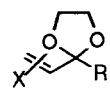
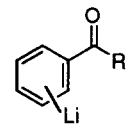
alkenyl) (**III**) [16,17] 1,3-dioxolanes have been successfuly used as precursors of masked saturated or unsaturated ω-lithio enolates of types **II** and **IV**. However, for substituted 2-phenyl-1,3-dioxolanes (**I** with R = Ph), during the lithiation step, the chlorine/lithium exchange competes with the benzylic opening of the heterocycle, even at low temperature [18]. In relation to our interest in functionalised aryllithium compounds [19], we report here the application of the mentioned arene-catalysed lithiation [4,5] to substituted 2-(chlorophenyl)-1,3-dioxolanes in order to generate the correponding formyl (**V**) and acetyl (**VI**) phenyllithium synthons.



I: X = Cl
II: X = Li



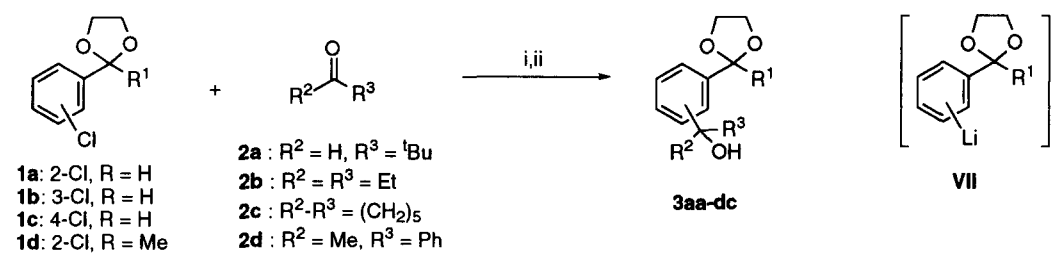
III: X = Cl
IV: X = Li



V: R = H
VI: R = Me

II. Results and discussion

The reaction of chlorophenyl-2,3-dioxolanes **1** and the corresponding electrophile **2** [^{t}BuCHO, Et_2CO, $(CH_2)_5CO$, PhCOMe; 1:1 molar ratio][2] with an excess of lithium powder (1:13 molar ratio) and a catalytic amount of naphthalene (1:0.2 molar ratio, 10 mol %) in THF at -78°C led, after hydrolysis with water at the same temperature, to the corresponding products **3** (Scheme 1 and Table 1). The reaction has to be carried out under Barbier conditions [20,21][2] in order to avoid decomposition of the corresponding intermediate of type **VII**: when the lithiation was performed in the absence of the electrophile (two-step process) an intractable mixture of compounds was obtained.



Scheme 1. *Reagents and conditions*: i. Li, $C_{10}H_8$ (10%), THF, -78°C, 1 h; ii. H_2O, -78 to 20°C.

It is worthy to note that the process shown in Scheme 1 did not work when DTBB was used as the electron carrier agent instead of naphthalene: in this case together with the

[2] Barbier-type conditions. For reviews, see references [20,21].

chlorine/lithium exchange a reductive opening of the heterocycle took place , so a complex mixture of reaction products was obtained.

Table 1
Preparation of compounds **3**

Entry	Starting material	Electrophile	Product[a] No.	R^1	R^2	R^3	Yield (%)[b]	R_f[c]
1	**1a**	**2a**	**3aa**	H	H	^{t}Bu	37	0.58[d]
2	**1a**	**2b**	**3ab**	H	Et	Et	58	0.38
3	**1a**	**2c**	**3ac**	H	$(CH_2)_5$		32	0.24[e]
4	**1a**	**2d**	**3ad**	H	Me	Ph	53	0.26
5	**1b**	**2a**	**3ba**	H	H	^{t}Bu	39	0.63
6	**1b**	**2c**	**3bc**	H	$(CH_2)_5$		42	0.30
7	**1b**	**2d**	**3bd**	H	Me	Ph	28	0.42
8	**1c**	**2a**	**3ca**	H	H	^{t}Bu	59	0.62
9	**1c**	**2b**	**3cb**	H	Et	Et	55	0.28[f]
10	**1c**	**2c**	**3cc**	H	$(CH_2)_5$		37	0.30[f]
11	**1c**	**2d**	**3cd**	H	Me	Ph	41	0.42[e]
12	**1d**	**2a**	**3da**	Me	H	^{t}Bu	21	0.59
13	**1d**	**2b**	**3db**	Me	Et	Et	22	0.42
14	**1d**	**2c**	**3dc**	Me	$(CH_2)_5$		20	0.27

[a] All products **3** were >95% pure (GLC and 300 MHz ^{1}H NMR).

[b] Isolated yield after column chromatography (neutral silica gel, hexane/ethyl acetate) based on the starting material **1**.

[c] Silica gel, hexane/ethyl acetate: 1/1 unless otherwise noted.

[d] Silica gel, hexane/ethyl acetate: 7/3.

[e] Silica gel, hexane/ethyl acetate: 3/2.

[f] Silica gel, hexane/ethyl acetate: 4/1.

From the results included in Table 1 it can be deduced that the preparation of compounds **3** works, in general, with moderate yields, except in the case of the acetophenone derivatives, in which yields are poorer (Table 1, entries 12-14).

Starting dioxolanes **1** were prepared by reaction of the corresponding commercially available chlorinated carbonyl compounds with ethyleneglycol, following the literature procedure [22].

III. Conclusion

As a conclusion, we report here a new methodology for formyl and acetyl phenyl lithium synthons starting from simple precursors. Acidic hydrolysis of the obtained products **3** to afford the corresponding carbonyl derivatives is a trivial process.

IV. Experimental Section

IV.1. General

For general information see reference [23].

IV.2. Preparation of Starting Chlorinated Dioxolanes **1**.

General Procedure [22].- A mixture of the corresponding commercially available chlorinated carbonyl compound (10 mmol), ethylene glycol (0.62 mL, 11 mmol) and a catalytic amount of *p*-toluenesulfonic acid (0.2 g, 1.1 mmol) in benzene (15 mL) was refluxed in a Dean-Stark apparatus for 12 h. Then, the solvent was evaporated (15 mm Hg and the residue was condensed (0.1 mm Hg) to give the title compounds. Yields, physical, spectroscopic and analytical data follow.

2-(2-Chlorophenyl)-1,3-dioxolane (**1a**) [24]: Yield 95%; colorless liquid, R_f 0.50 (hexane/ethyl acetate: 9/1); ν (film) 3071, 1594 (HC=C), 1095 cm^{-1} (C-O); δ_H 4.11 (4H, m, 2xCH_2O), 6.16 (1H, s, OCHO), 7.24-7.41 (4H, m, ArH); δ_C 65.4 (2C, 2xCH_2O), 100.7 (OCHO), 126.8, 127.5, 129.7, 130.3, 135.0 (ArC); *m/z* 186 (M+·+2, 7.5%), 185 (M+·+1, 24), 184 (M+·, 24), 183 (66), 149 (18), 141 (12), 139 (37), 125 (10), 119 (18), 112 (21), 111 (15), 91 (10), 89 (39), 77 (17), 76 (10), 75 (21), 74 (11), 73 (100), 63 (18), 51 (22), 50 (25), 45 (58), 43 (11).

2-(3-Chlorophenyl)-1,3-dioxolane (**1b**) [25]: Yield 95%; colorless liquid, R_f 0.37 (hexane/ethyl acetate: 9/1); ν (film) 3070, 1599 (HC=C), 1199, 1085 cm^{-1} (C-O); δ_H 4.06 (4H, m, 2xCH_2O), 5.78 (1H, s, OCHO), 7.35 (4H, m, ArH); δ_C 65.3 (2C, 2xCH_2O), 102.8 (OCHO), 126.6, 129.2, 129.6, 140.0, (ArC); *m/z* 186 (M+·+2, 7.9%), 185 (M+·+1, 26), 184 (M+·, 25), 183 (70), 149 (16), 141 (12), 139 (36), 125 (10), 119 (12), 112 (25), 111 (22), 91 (13), 89 (33), 77 (19), 75 (25), 74 (13), 73 (100), 63 (19), 51 (24), 50 (27), 45 (68), 43 (15) (Found: M+·, 184.0291. $C_9H_9ClO_2$ requires 184.0291).

2-(4-Chlorophenyl)-1,3-dioxolane (**1c**) [26]: Yield 94%; colorless liquid; R_f 0.40 (hexane/ethyl acetate: 9/1); ν (film) 3057, 1599 (HC=C), 1089 cm^{-1} (C-O); δ_H 4.01-4.15 (4H, m, 2xCH_2O), 5.77 (1H, s, OCHO), 7.35, 7.40 (4H, 2d, J = 8.5, ArH); δ_C 65.2 (2C, 2xCH_2O), 102.9 (OCHO), 127.8, 128.5, 130.8, 136.4 (ArC); *m/z* 186 (M+·+2, 11.4%), 185 (M+·+1, 35), 184 (M+·, 35), 183 (100), 149 (21), 141 (19), 139 (57), 125 (14), 119 (19), 114 (11), 113 (11), 112 (32), 111 (25), 91 (10), 89 (40), 77 (19), 75 (27), 74 (12), 73 (73), 63

(20), 51 (24), 50 (27), 50 (30), 45 (59), 43 (15).

2-(2-Chlorophenyl)-2-methyl-1,3-dioxolane (**1d**) [27]: Yield 96%; colorless liquid, R_f 0.26 (hexane); ν (film) 3069, 1592 (HC=C), 1036 cm^{-1} (C-O); δ_H 1.81 (3H, s, Me), 3.78, 4.07 (4H, m, 2xCH_2O), 7.23, 7.37 (4H, 2m, ArH); δ_C 25.2 (Me), 64.4 (2C, 2xCH_2O), 108.4 (OCO), 126.4, 127,6, 129.3, 131.4, (ArC); *m/z* 186 (M+-18, 5%), 184 (14), 183 (100), 141 (27), 139 (75), 111 (20), 103 (16), 87 (48), 77 (11), 75 (20), 51 (14), 43 (58).

VI.2.Preparation of Compounds **3**.

General Procedure.- To a green suspension of lithium powder (90 mg, 13 mmol) and naphthalene (23 mg, 0.2 mmol) in THF (5 mL) was slowly added (*ca.* 1 h) a solution of the corresponding substrate **1** (1 mmol) and the electrophile (1 mmol) in THF (3 mL) at -78°C and it was stirred 1 h at the same temperature. The resulting mixture was then hydrolysed with water (10 mL) and after warming up to room temperature it was extracted with ethyl acetate (3x10 mL), the organic layer dried over anhydrous sodium sulfate and evaporated (15 mm Hg). The resulting residue was the purified by column chromatography (neutral silica gel, hexane/ethyl acetate) to give the title compounds. Yields and R_f values are included in Table 1; physical, spectroscopic and analytical data follow.

2-[2-(1-Hydroxy-2,2-dimethylpropyl)phenyl]-1,3-dioxolane (**3aa**): colorless liquid; ν (film) 3461 (OH), 1604 (C=C), 1082 cm^{-1} (C-O); δ_H 0.97 (9H, s, 3xMe), 3.99-4.21 (4H, m, 2xCH_2O), 4.92 (1H, broad s, C*H*OH), 6.16 (1H, s, OCHO), 7.32, 7.56 (4H, 2m, ArH); *m/z* 218 (M+-18, 0.3%), 179 (29), 159 (16), 135 (100), 79 (10), 77 (16), 45 (21) (Found: M+-18, 218.1307. $C_{14}H_{18}O_2$ requires 218.1307).

2-[2-(1-Ethyl-1-hydroxypropyl)phenyl]-1,3-dioxolane (**3ab**): colorless liquid; ν (film) 3397 (OH), 3088, 3062, 3030, 1601 (HC=C), 1118 cm^{-1} (C-O); δ_H 0.84, 0.94 (6H, 2t, J = 7.5, 2xMe), 1.27, 1.39, 1.55, 1.73 (4H, 4m, 2xC*H*$_2$Me), 3.40, 3.49 (2H, 2m, CH_2O), 3.72 (2H, broad s, CH_2O), 4.26 (1H, s, OCHO), 7.27-7.37 (4H, m, ArH); δ_C 7.3, 8.0 (2xMe), 26.7, 27.4 (2x*C*H$_2$Me), 62.0 (2C, 2xCH_2O), 85.8 (OCHO), 127.8, 128.0, 128.4, 138.0 (ArC); *m/z* 221 (M+-15, 0.1%), 152 (49), 147 (13), 107 (75), 105 (11), 92 (17), 91 (54), 79 (34), 77 (24), 69 (12), 57 (25), 45 (100), 43 (20) (Found: M+-15, 221.1178. $C_{13}H_{17}O_3$ requires 221.1177).

2-[2-(1-Hydroxycyclohexyl)phenyl]-1,3-dioxolane (**3ac**): colorless liquid; ν (film) 3387 (OH), 3063, 3025, 1497 (HC=C), 1117, 1060 cm^{-1} (C-O); δ_H 1.06-1.94 (10H, m, 5xring CH_2), 3.41, 3.52, 3.69 (4H, 3m, 2xCH_2O), 4.88 (1H, s, OCHO), 7.31 (4H, m, ArH); *m/z* 231 M+-17, 1%), 151 (13), 170 (85), 169 (20), 155 (15), 153 (17), 152 (100), 143 (17), 142 (97), 141 (60), 129 (52), 128 (32), 108 (11), 105 (28), 99 (51), 92 (25), 91 (78), 81 (56), 79 (43), 77 (36), 73 (44), 65 (12), 57 (18), 55 (32), 51 (17), 45 (34), 44 (20), 43 (51) (Found: M+-17, 231.1380. $C_{15}H_{19}O_2$ requires 231.1385).

2-[2-(1-Hydroxy-1-phenylethyl)phenyl]-1,3-dioxolane (**3ad**): colorless liquid; ν (film) 3387 (OH), 3088, 3060, 3029, 1602, 1498 cm^{-1} (HC=C); δ_H 1.38 (3H, s, Me), 3.30, 3.55 (4H, 2m, 2xCH_2O), 4.47 (1H, s, OCHO), 7.11, 7.32 (9H, 2m, ArH); δ_C 24.0 (Me), 61.5 (COH), 76.6 (2C, 2xCH_2O), 89.1 (OCHO), 125.7, 127.0, 127.5, 127.7, 7.8, 128.3, 137.2, 144.4, 145.3 (ArC); *m/z* 208 (M+-62, 0.2%), 152 (18), 151 (25), 121 (25), 107 (38), 105 (10), 79 (23), 77 (18), 45 (19), 43 (100) (Found: M+-62, 208.0860. $C_{15}H_{12}O$ requires 208.0888).

2-[3-(1-Hydroxy-2,2-dimethylpropyl)phenyl]-1,3-dioxolane (**3ba**): colorless liquid; ν (film) 3450 (OH), 3070, 3038,1599 cm^{-1} (HC=C); δ_H 0.92 (9H, s, 3xMe), 1.84 (1H, broad s, OH), 4.09 (4H, m, 2xCH_2O), 4.42 (1H, broad s, C*H*OH), 5.81 (1H, s, OCHO), 7.36 (4H, m, ArH); δ_C 25.9 (3C, 3xMe), 35.6 (*C*Me_3), 65.3 (2C, 2xCH_2O), 82.3 (CHOH), 103.8 (OCHO), 125.4, 125.7, 127.7, 128.4, 137.2 (ArC); *m/z* 237 (M+-1, 0.2%), 236 (M+, 0.4), 180 (31), 179 (100), 135 (19), 79 (23), 77 (20), 73 (76), 57 (22), 45 (46), 43 (18) (Found: M+, 236.1409. $C_{14}H_{20}O_3$ requires 236.1412).

2-[3-(1-Hydroxycyclohexyl)phenyl]-1,3-dioxolane (**3bc**): colorless liquid; ν (film) 3376 (OH), 3062, 3025, 1606 (HC=C), 1116 cm^{-1} (C-O); δ_H 1.06-1.75 (10H, m, 5xring CH_2), 3.41, 3.50, 3.73 (4H, 3m, 2xCH_2O), 4.12 (1H, s, OCHO), 7.25-7.38 (4H, m, ArH); δ_C 21.2, 21.4, 25.8, 32.3, 34.2 (5xring CH_2), 61.9, 70.7 (2xCH_2O), 73.5 (COH), 89.1 (OCO), 127.8, 127.9, 128.4, 137.9, 158.7 (ArC); *m/z* 249 (M+-1, 3%), 105 (11), 91 (13), 77 (12), 73 (100), 55 (17), 45 (27), 44 (10), 43 (14).

2-[3-(1-Hydroxy-1-phenylethyl)phenyl]-1,3-dioxolane (**3bd**): colorless liquid; ν (film) 3417 (OH), 3058, 1598 (HC=C), 1107 cm^{-1} (C-O); δ_H 1.95 (3H, s, Me), 4.00-4.16 (4H, m, 2xCH_2O), 5.78 (1H, s, OCHO), 7.21-7.42 (9H, 2m, ArH); δ_C 30.9 (Me), 65.3 (2C, 2xCH_2O), 76.2 (COH), 103.8 (OCHO), 123.8, 125.1, 125.9, 126.1, 127.0, 127.05, 128.2, 128.3, 148.2 (ArC); *m/z* 252 (M+-18, 10%), 180 (16), 179 (13), 178 (17), 165 (12), 152 (15), 151 (17), 121 (14), 107 (24), 105 (31), 91 (10), 89 (11), 79 (15), 77 (33), 73 (34), 51 (19), 45 (29), 44 (34), 43 (100) (Found: M+-18, 252.1160. $C_{17}H_{16}O_2$ requires 252.1150).

2-[4-(1-Hydroxy-2,2-dimethylpropyl)phenyl]-1,3-dioxolane (**3ca**): colorless liquid; δ_H 0.92 (9H, s, 3xMe), 4.10 (4H, m, 2xCH_2O), 4.41 (1H, broad s, C*H*OH), 5.80 (1H, s, OCHO), 7.33, 7.43 (4H, 2d, J = 8.2, ArH); δ_C 25.8 (3C, 3xMe), 35.6 (*C*Me_3), 66.3 (2C, 2xCH_2O), 82.2 (CHOH), 103.7 (OCHO), 125.7, 127.6, 128.3, 129.0, 143.3 (ArC); *m/z* 236 (M+, 2%), 180 (20), 179 (58), 107 (23), 79 (20), 77 (14), 73 (100), 57 (17), 45 (34), 43 (10) (Found: M+, 236.1413. $C_{14}H_{20}O_3$ requires 236.1412).

2-[4-(1-Ethyl-1-hydroxypropyl)phenyl]-1,3-dioxolane (**3cb**): colorless liquid; ν (film) 3444 (OH), 1698, 1682 (C=C), 1114, 1082 cm^{-1} (C-O); δ_H 0.75 (6H, m, 2xMe), 1.81 (4H, m, 2xCH_2Me), 4.01-4.17 (4H, m, CH_2O), 5.80 (1H, s, OCHO), 7.36 (4H, m, ArH); δ_C 7.7 (2C, 2xMe), 35.0 (2C, 2xCH_2Me), 60.4 (CHO), 65.3 (2C, 2xCH_2O), 103.7 (OCHO), 125.6,

126.1, 135.7, 146.9 (ArC); *m/z* 236 (M+, 0.4%), 208 (11), 207 (86), 135 (35), 117 (15), 91 (10), 77 (11), 73 (100), 57 (68), 45 (30), 43 (19) (Found: M+, 236.1412. $C_{14}H_{20}O_3$ requires 236.1422).

2-[4-(1-Hydroxycyclohexyl)phenyl]-1,3-dioxolane (**3cc**): colorless liquid; ν (film) 3434 (OH), 1606 (C=C), 1271 cm-1 (C-O); δ_H 1.25-1.80 (10H, m, 5xring CH_2), 4.01-4.14 (4H, 3m, 2xCH_2O), 5.81 (1H, s, OCHO), 7.45, 7.52 (4H, 2d, J = 8.5, ArH); δ_C 22.1, 25.5, 38.8, (5xring CH_2), 65.3 (2C, 2xCH_2O), 73.15 (COH), 103.6 (OCHO), 124.7, 126.3, 136.2 (ArC); *m/z* 230 (M+-18, 19%), 229 (22), 186 (14), 185 (16), 158 (19), 157 (15), 156 (13), 143 (16), 129 (33), 128 (22), 115 (17), 91 (23), 77 (22), 73 (73), 64 (11), 51 (15), 45 (28), 44 (100) (Found: M+-18, 230.1292. $C_{15}H_{18}O_2$ requires 230.1307).

2-[4-(1-Hydroxy-1-phenylethyl)phenyl]-1,3-dioxolane (**3cd**): colorless liquid; ν (film) 3424 (OH), 3089, 3059, 3027, 1643, 1601 cm-1 (HC=C); δ_H 1.94 (3H, s, Me), 4.02-4,15 (4H, 2m, 2xCH_2O), 5.79 (1H, s, CHO), 7.23-7.43 (9H, 2m, ArH); *m/z* 270 (M+, 0.7%), 255 (16), 252 (60), 251 (61), 208 (20), 207 (39), 193 (16), 181 (15), 180 (100), 179 (59), 178 (72), 177 (10), 176 (12), 165 (59), 152 (14), 149 (18), 119 (16), 105 (23), 104 (16), 103 (32), 96 (11), 94 (12); 89 (41), 88 (14), 82 (18), 77 (29), 76 (22), 73 (62), 63 (12), 51 (24), 45 (46), 44 (17), 43 (33) (Found: M+-18, 252.1161. $C_{17}H_{16}O_2$ requires 252.11503).

2-[2-(1-Hydroxy-2,2-dimethylpropyl)phenyl]-2-methyl-1,3-dioxolane (**3da**): colorless liquid; ν (film) 3421 (OH), 3061, 1686 cm-1 (HC=C); δ_H 0.92 (9H, s, 3xMe), 1.72 (3H, s, Me), 3.75, 3.88, 4.03 (5H, 3m, 2xCH_2O, C*H*OH), 5.81 (1H, s, C*H*OH), 7.27, 7.63 (4H, 2m, ArH); *m/z* 235 (M+-15, 0.5%), 193 (34), 175 (62), 173 (11), 149 (40), 132 (17), 131 (100), 103 (22), 87 (15), 77 (19), 57 (11), 45 (11), 43 (49) (Found: M+-15, 235.1334. $C_{14}H_{19}O_3$ requires 235.1324).

2-[2-(1-Ethyl-1-hydroxypropyl)phenyl]-2-methyl-1,3-dioxolane (**3db**): colorless liquid; ν (film) 3419 (OH), 3059, 3028, 1611 (HC=C), 1118 cm-1 (C-O); δ_H 0.81, 0.88 (6H, 2t, J = 7.5, 2xCH_2*Me*), 1.48 (2H, q, J = 7.5, C*H*$_2$Me), 1.71 (5 H, m, C*H*$_2$Me, Me), 3.26 (1H, ddd, J = 9.5, 5.0, 3.7, CH*H*O), 3.47 (1H, ddd, J = 9.5, 6.4, 3.7, CH*H*O), 3.77 (2H, m, CH_2O), 7.37 (4H, m, ArH); δ_C 8.6, 9.0, 19.85 (3xMe), 26.5, 26.7 (2xCH_2Me), 62.5, 63.5 (2xCH_2O), 77.9 (COH), 84.8 (OCO), 127.1, 127.7, 128.2, 141.5 (ArC); *m/z* 232 (M+-18, 0.2%), 133 (22), 121 (36), 105 (23), 91 (64), 87 (30), 77 (18), 57 (20), 55 (13), 51 (11), 44 (28), 43 (100) (Found: M+-18, 232.1463. $C_{15}H_{20}O_2$ requires 232.1463).

2-[2-(1-Hydroxycyclohexyl)phenyl]-2-methyl-1,3-dioxolane (**3dc**): colorless liquid; ν (film) 3431 (OH), 1649 cm-1 (C=C); δ_H 1.39-1.91 (13H, m, 5xring CH_2, Me), 3.27-3.79 (4H, m, 2xCH_2O), 7.19-7.46 (4H, m, ArH); δ_C 24.3, 25.7, 31.0, (5xring CH_2), 38.9 (Me), 63.6, 74.4 (3C, 2xCH_2, COH), 84.2 (OCO), 125.0, 126.5, 128.2, 141.0, 143.3 (ArC); *m/z* 263 (M+-1, 1,2%), 262 (M+, 6.5), 219 (11), 202 (10), 201 (63), 200 (37), 185 (24), 161 (10), 158 (29),

157 (100), 147 (20), 145 (16), 144 (13), 131 (14), 129 (22), 128 (19), 117 (10), 115 (27), 91 (169, 87 (23), 77 (18), 55 (14), 51 (13), 45 (12), 43 (92) (Found: M+, 262.1578. $C_{16}H_{22}O_3$ requires 262.1568).

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VI. References

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