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Reaction of 2-Perfluoroalkanoylcyclohexane-1,3-diones with Diazomethane

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Abstract—Treatment of 2-perfluoroalkanoyl-5.5-dimethylcyclohexane-1,3-diones with a solution of diazomethane in diethyl ether led to the formation of the corresponding enol ethers and 3-hydroxy-6,6-dimethyl-3-perfluoroalkyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-ones. The latter underwent dehydration on heating in boiling benzene in the presence of a catalytic amount of *p*-toluenesulfonic acid to give 6,6-dimethyl-3-perfluoroalkyl-6,7-dihydrobenzofuran-4(5*H*)-ones.

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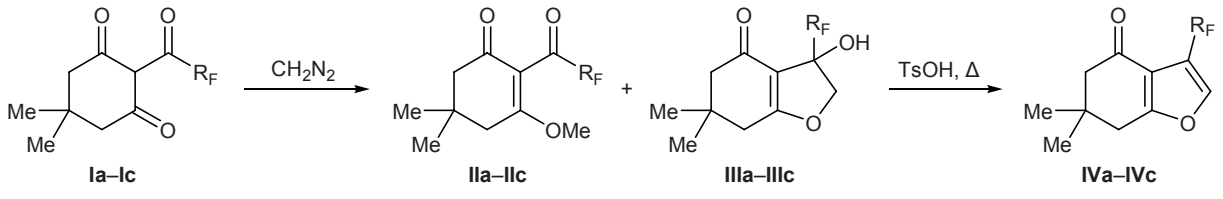
Polyfunctional 2-perfluoroalkanoylcyclohexane-1,3-diones attract much interest as promising building blocks for the synthesis of various biologically active compounds containing polyfluoroalkyl groups. Chemical transformations of cyclic β,β′-tricarbonyl compounds involve mainly electrophilic centers and lead to derivatives at the exo- and endocyclic carbonyl groups [1]. Reactions of their enol derivatives, such as enol ethers, chlorovinyl diketones, etc., with nitrogen-centered nucleophiles open a way to compounds that are regioisomeric to those available from β,β′-triketones. Enol ethers are more convenient intermediate products than chlorovinyl diketones in the synthesis of biologically active compounds; they ensure higher yields of the target products, and their reactions do not require the presence of bases for binding liberated hydrogen chloride [2].

Treatment with diazomethane is widely used to obtain enol methyl ethers from cyclic β-diketones of both cyclohexane and cyclopentane series [3]. However, reactions of diazomethane with 2-acetylcyclohexane-1,3-diones result in formation of complex mixtures of products, the main components of which are 6,7-dihydro- and 2,3,6,7-tetrahydrobenzofuran-4(5*H*)-ones, as well as 5,5-dimethyl-2-(2-oxopropyl)cyclohexane-1,3-dione, its methyl ether, and other compounds [4]. In these reactions diazomethane, like other nucleophiles, attacks β,β′-triketones of the cyclohexane series mainly at the side-chain carbonyl carbon atom.

The present work continues our systematic studies on the chemical transformations of fluorinated cyclic β,β′-triketones. It was aimed at studying the reaction of 2-perfluoroalkanoyl-5,5-dimethylcyclohexane-1,3-diones **Ia**–**Ic** with diazomethane with a view to obtain the corresponding enol ethers. The reactions were carried out by adding a solution of 1.5 equiv of diazomethane in diethyl ether to a solution of 5,5-dimethyl-2-perfluoroalkanoylcyclohexane-1,3-dione **Ia**–**Ic** in the

Scheme 1.



Ia–Ic **IIa–IIc** **IIIa–IIIc** **IVa–IVc**

IIa–IIc $\xrightarrow{CH_2N_2}$ **IIIa–IIIc**

$R_F = CF_3$ (**a**), C_2F_5 (**b**), C_3F_7 (**c**).

same solvent on cooling to 0°C over a period of 15 min and further stirring the reaction mixture for 5 h at room temperature. The products were mixtures of the corresponding methyl enol ethers **IIa**–**IIc** and 3-hydroxy-6,6-dimethyl-3-perfluoroalkyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-ones **IIIa**–**IIIc** (Scheme 1) whose ratio depended on the length of the perfluoroalkyl group (1.4:1, 2.5:1, and 1.8:1, respectively, according to the ^{1}H NMR data). The product ratio also depended on the mode of addition of a solution of diazomethane. When the latter was added in one portion, compounds **IIIa**–**IIIc** were formed as the only products. Tetrahydrobenzofuranones **IIIa**–**IIIc** were also obtained in quantitative yield in the reactions with 5 equiv of diazomethane. Presumably, initially formed enol ethers **IIa**–**IIc** undergo further transformation into compounds **IIIa**–**IIIc** by the action of diazomethane. To verify this assumption, enol ethers **IIa**–**IIc** were treated with a solution of diazomethane in diethyl ether. In fact, we thus obtained 3-hydroxy-6,6-dimethyl-3-perfluoroalkyl-2,3,6,7-tetrahydrobenzofuranones **IIIa**–**IIIc** in 79–83% yield.

Our results showed that, unlike nonfluorinated β,β′-triketones of the cyclohexane series, attack by diazomethane on fluoroalkyl-containing cyclic β,β′-triketones **Ia**–**Ic** is directed at the enol oxygen atom to give enol ethers **IIa**–**IIc**, as well as on the carbonyl carbon atom in the perfluoroacyl group with formation of 2,3,6,7-tetrahydrobenzofuran-4(5*H*)-ones **IIIa**–**IIIc** having hydroxy and perfluoroalkyl groups at the same carbon atom. A probable factor responsible for preferential attack by diazomethane on the enol oxygen atom is that 5,5-dimethyl-2-perfluoroalkanoylcyclohexane-1,3-diones **Ia**–**Ic** are fairly strong vinylogous acids as are 2-acetylcyclopentane-1,3-diones which are known to react with diazomethane to give exclusively the corresponding enol ethers [4, 5]. The formation of hydroxy derivatives **IIIa**–**IIIc** rather than 6,7-dihydrobenzofuran-4(5*H*)-ones **IVa**–**IVc** [4] (as in reactions with nonfluorinated analogs) is explained by the presence of electron-withdrawing perfluoroalkyl group which hampers dehydration. 6,7-Dihydrobenzofuranones **IVa**–**IVc** were isolated in 78–82% yields by heating 3-hydroxy-2,3,6,7-tetrahydrobenzofuranones **IIIa**–**IIIc** for 4 h in boiling benzene in the presence of a catalytic amount of *p*-toluenesulfonic acid in a flask equipped with a Dean–Stark trap.

The structure of the isolated compounds was confirmed by IR and ^{1}H, ^{13}C, and ^{19}F NMR spectra and elemental analyses. The IR spectra of enol ethers **IIa**–**IIc** contained absorption bands typical of conjugated and unconjugated carbonyl groups (1645–1655 and 1720–1735 cm^{-1}) and double C=C bond (1590–1605 cm^{-1}). 2,3,6,7-Tetrahydrobenzofuranones **IIIa**–**IIIc** and 6,7-dihydrobenzofuranones **IVa**–**IVc** displayed in the IR spectra absorption bands due to conjugated carbonyl group (1640–1655 and 1685–1690 cm^{-1}, respectively) and double C=C bond (1610–1625 and 1565–1570 cm^{-1}, respectively). In the ^{1}H NMR spectra of **IIa**–**IIc**, protons in the methoxy group resonated as a singlet at δ 3.86–3.88 ppm. Nonequivalent methylene protons on C^2 and C^7 in compound **IIIa** resonated in the ^{1}H NMR spectrum as two pairs of doublets in the regions δ 4.46– 4.75 (^2J = 11.4 Hz) and 2.22–2.32 ppm (^2J = 16.4 Hz), respectively, whereas the spectra of **IIIb** and **IIIc** contained three pairs of doublets at δ 4.45–4.85 (^2J = 11.5 Hz), 2.36–2.42 (^2J = 17.9 Hz), and 2.21 – 2.33 ppm (^2J = 16.4 Hz) due to nonequivalent methylene protons on C^2, C^5, and C^7, respectively. The vinyl proton signals appeared in the spectra of **IVa**–**IVc** as multiplets at δ 7.66–7.69 ppm. In the ^{13}C NMR spectra of enol ethers **IIa**–**IIc** signals characteristic of OCH_3, $\mathbf{C}OR_F$, and C^1 carbon atoms were observed in the regions δ_C 56.42–56.88, 184.72–188.08, and 194.11–194.54 ppm. The ^{13}C NMR spectra of 2,3,6,7-tetrahydrobenzofuranones **IIIa**–**IIIc** displayed signals at δ_C 79.55–79.90 (C^2), 79.86–80.93 (C^3), and 194.31–194.66 ppm (C^4), while compounds **IVa**–**IVc** were characterized by signals at δ_C 144.18–144.54 (C^2), 113.38–115.15 (C^3), and 190.42–191.16 ppm (C^4).

EXPERIMENTAL

The IR spectra were recorded in KBr on a UR-20 instrument. The NMR spectra were measured on a Bruker Avance-500 spectrometer from solutions in $CDCl_3$ using tetramethylsilane (^{1}H, 500 MHz; ^{13}C, 125 MHz) or trichlorofluoromethane (^{19}F, 470 MHz) as internal reference. The melting points were determined on a Boetius melting point apparatus. The progress of reactions was monitored, and the purity of products was checked, by TLC on Silufol UV-254 plates using diethyl ether as eluent. Initial 5,5-dimethyl-2-polyfluoroacylcyclohexane-1,3-diones **Ia**–**Ic** were synthesized according to the procedure reported in [6], and diazomethane was prepared from 2.06 g of *N*-nitrosomethylurea as described in [7]. Preparative thin-layer chromatography was performed on silica gel 60 HF_{254} plates (Aldrich) using diethyl ether–hexane (1 : 1) as eluent.

Reaction of 5,5-dimethyl-2-perfluoroalkanoylcyclohexane-1,3-diones Ia–Ic with diazomethane

(*general procedure*). *a*. A solution of 1 mmol of β,β′-triketone **Ia**–**Ic** in 10 ml of diethyl ether was cooled to 0°C, and 2.5 ml of a solution of diazomethane in diethyl ether was added under stirring over a period of 15 min. The mixture was stirred for 5 h at room temperature, the solvent was removed on a rotary evaporator, and the residue was subjected to preparative thin-layer chromatography to isolate compounds **IIa**–**IIc** and **IIIa**–**IIIc** as colorless crystals.

b. Following analogous procedures but adding a solution of diazomethane in one portion or using 5 equiv of diazomethane, we isolated only compounds **IIIa**–**IIIc** in, respectively, 83 or 85% (**IIIa**), 79 or 81% (**IIIb**), and 80 or 82% yield (**IIIc**). The products were recrystallized from diethyl ether–hexane.

3-Methoxy-5,5-dimethyl-2-(2,2,2-trifluoroacetyl)-cyclohex-2-en-1-one (IIa). Yield 58% (*a*), mp 77–80°C. IR spectrum, ν, cm^{-1}: 1735, 1655, 1605. ^1H NMR spectrum, δ, ppm: 1.14 s (6H, CH_3), 2.30 s (2H, CH_2), 2.53 s (2H, CH_2), 3.88 s (3H, OCH_3). ^{13}C NMR spectrum, δ_C, ppm: 28.26 (CH_3), 32.04 (C^5), 39.32 (C^4), 49.91 (C^6), 56.88 (OCH_3), 114.48 (C^2), 115.10 q (CF_3, ^1J = 291 Hz), 178.03 (C^3), 184.72 q (**C**OCF_3, ^2J = 38 Hz), 194.54 (C^1). ^{19}F NMR spectrum: δ_F –77.16 ppm, s (CF_3). Found, %: C 52.68; H 5.20. $C_{11}H_{13}F_3O_3$. Calculated, %: C 52.80; H 5.24.

3-Methoxy-5,5-dimethyl-2-(2,2,3,3,3-pentafluoropropanoyl)cyclohex-2-en-1-one (IIb). Yield 71% (*a*), mp 81–84°C. IR spectrum, ν, cm^{-1}: 1725, 1645, 1595. ^1H NMR spectrum, δ, ppm: 1.15 s (6H, CH_3), 2.29 s (2H, CH_2), 2.52 s (2H, CH_2), 3.86 s (3H, OCH_3). ^{13}C NMR spectrum, δ_C, ppm: 28.22 (CH_3), 32.12 (C^5), 38.78 (C^4), 49.81 (C^6), 56.50 (OCH_3), 106.36 t.q (CF_2, ^1J = 268, ^2J = 38 Hz), 115.15 (C^2), 118.08 q.t (CF_3, ^1J = 288, ^2J = 35 Hz), 177.27 (C^3), 188.08 t (**C**OC_2F_5, ^2J = 29 Hz), 194.21 (C^1). ^{19}F NMR spectrum, δ_F, ppm: –81.65 br.s (CF_3), –122.01 br.s (CF_2). Found, %: C 48.15; H 4.42. $C_{12}H_{13}F_5O_3$. Calculated, %: C 48.01; H 4.36.

2,2,3,3,4,4,4-Heptafluorobutanoyl-3-methoxy-5,5-dimethylcyclohex-2-en-1-one (IIc). Yield 64% (*a*), mp 65–68°C. IR spectrum, ν, cm^{-1}: 1720, 1650, 1590. ^1H NMR spectrum, δ, ppm: 1.15 s (6H, CH_3), 2.29 s (2H, CH_2), 2.52 s (2H, CH_2), 3.86 s (3H, OCH_3). ^{13}C NMR spectrum, δ_C, ppm: 28.23 (CH_3), 32.12 (C^5), 38.78 (C^4), 49.84 (C^6), 56.42 (OCH_3), 107.90 t.t (CF_2, ^1J = 269, ^2J = 32 Hz), 108.77 t.m (CF_2, ^1J = 267 Hz), 115.32 (C^2), 117.51 q.t (CF_3, ^1J = 288, ^2J = 34 Hz), 177.24 (C^3), 187.94 t (**C**OC_3F_7, ^2J = 29 Hz), 194.11 (C^1). ^{19}F NMR spectrum, δ_F, ppm: –80.94 m (CF_3), –119.09 m (CF_2), –126.26 m (CF_2). Found, %: C 44.61; H 3.79. $C_{13}H_{13}F_7O_3$. Calculated, %: C 44.58; H 3.74.

3-Hydroxy-6,6-dimethyl-3-trifluoromethyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one (IIIa). Yield 42% (*a*), mp 68–71°C. IR spectrum, ν, cm^{-1}: 1655, 1625. ^1H NMR spectrum, δ, ppm: 1.11 s (3H, CH_3), 1.12 s (3H, CH_3), 2.22 d (1H, ^2J = 16.4 Hz), 2.32 d (1H, ^2J = 16.4 Hz), 2.38 s (2H, CH_2), 4.46 d.m (1H, ^2J = 11.4 Hz), 4.75 d.m (1H, ^2J = 11.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: 28.09 (CH_3), 28.52 (CH_3), 34.27 (C^6), 37.96 (C^7), 50.88 (C^5), 79.55 (C^2), 80.93 q (C^3, ^2J = 33 Hz), 110.31 (C^{3a}), 124.64 q (CF_3, ^1J = 284 Hz), 181.86 (C^{7a}), 194.31 (C^4). ^{19}F NMR spectrum: δ_F –80.04 ppm, s (CF_3). Found, %: C 52.92; H 5.29. $C_{11}H_{13}F_3O_3$. Calculated, %: C 52.80; H 5.24.

3-Hydroxy-6,6-dimethyl-3-perfluoroethyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one (IIIb). Yield 29% (*a*), mp 58–61°C. IR spectrum, ν, cm^{-1}: 1640, 1610. ^1H NMR spectrum, δ, ppm: 1.11 s and 1.12 s (3H each, CH_3), 2.21 d (1H, ^2J = 16.4 Hz), 2.33 d (1H, ^2J = 16.4 Hz), 2.36 d (1H, ^2J = 17.9 Hz), 2.42 d (1H, ^2J = 17.9 Hz), 4.45 d.t (1H, ^2J = 11.5, ^3J = 2.7 Hz), 4.85 d.m (1H, ^2J = 11.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 28.11 (CH_3), 28.57 (CH_3), 34.21 (C^6), 38.01 (C^7), 50.90 (C^5), 79.90 (C^2), 81.59 t (C^3, ^2J = 26 Hz), 109.97 (C^{3a}), 113.72 t.q (CF_2, ^1J = 261, ^2J = 35 Hz), 119.09 q.t (CF_3, ^1J = 287, ^2J = 36 Hz), 181.57 (C^{7a}), 194.66 (C^4). ^{19}F NMR spectrum, δ_F, ppm: –80.18 br.s (CF_3), –120.86 d.m (1F, $^2J_{FF}$ = 275.5 Hz), –125.28 d.m (1F, $^2J_{FF}$ = 275.2 Hz). Found, %: C 48.17; H 4.41. $C_{12}H_{13}F_5O_3$. Calculated, %: C 48.01; H 4.36.

3-Hydroxy-6,6-dimethyl-3-(perfluoropropyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one (IIIc). Yield 36% (*a*), mp 67–70°C (from diethyl ether–hexane). IR spectrum, ν, cm^{-1}: 1655, 1620. ^1H NMR spectrum, δ, ppm: 1.13 s and 1.14 s (3H each, CH_3), 2.23 d (1H, ^2J = 16.4 Hz), 2.36 d (1H, ^2J = 16.4 Hz), 2.37 d (1H, ^2J = 18.0 Hz), 2.43 d (1H, ^2J = 18.0 Hz), 4.47 d.m (1H, ^2J = 11.6 Hz), 4.84 d.m (1H, ^2J = 11.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 28.04 (CH_3), 28.52 (CH_3), 34.11 (C^6), 38.01 (C^7), 50.94 (C^5), 79.86 (C^2), 82.52 t (C^3, ^2J = 26 Hz), 109.87 t.m (CF_2, ^1J = 268 Hz), 110.09 (C^{3a}), 115.45 t.t (CF_2, ^1J = 260, ^2J = 28 Hz), 117.67 q.t (CF_3, ^1J = 288, ^2J = 34 Hz), 181.62 (C^{7a}), 194.57 (C^4). ^{19}F NMR spectrum, δ_F, ppm: –81.48 m (3F, CF_3), –117.51 d.m (1F, $^2J_{FF}$ = 280.9 Hz), –120.99 d.m (1F, $^2J_{FF}$ = 281.6 Hz), –123.26 d.m (1F, $^2J_{FF}$ = 293.5 Hz), –126.65 d.m (1F, $^2J_{FF}$ = 293.5 Hz). Found, %: C 44.67; H 3.79. $C_{13}H_{13}F_7O_3$. Calculated, %: C 44.58; H 3.74.

Reaction of 3-methoxy-5,5-dimethyl-2-perfluoroalkanoylcyclohex-2-en-1-ones IIa–IIc with diazomethane (*general procedure*). A solution of 1 mmol of enol ether **IIa–IIc** in 10 ml of diethyl ether was cooled to 0°C, 2.5 ml of a solution of diazomethane was added under stirring over a period of 15 min, and the mixture was stirred for 5 h at room temperature. The solvent was removed on a rotary evaporator, and the residue was subjected to preparative thin-layer chromatography to isolate compounds **IIIa–IIIc** as colorless crystals in 81, 83, and 79% yield, respectively.

Dehydration of 3-hydroxy-6,6-dimethyl-3-perfluoroalkyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-ones IIIa–IIIc (*general procedure*). *p*-Toluenesulfonic acid, 20 mg, was added to a solution of 0.4 mmol of benzofuran **IIIa–IIIc** in 40 ml of benzene, and the mixture was heated for 4 h under reflux in a flask equipped with a Dean–Stark trap. The mixture was washed with water (2×10 ml) and dried over $MgSO_4$, the solvent was removed on a rotary evaporator, and the residue was recrystallized from diethyl ether–hexane to isolate compounds **IVa–IVc**.

6,6-Dimethyl-3-trifluoromethyl-6,7-dihydrobenzofuran-4(5*H*)-one (IVa). Yield 79%, mp 35–38°C. IR spectrum, ν, cm^{-1}: 1685, 1570, 1455. ^1H NMR spectrum, δ, ppm: 1.15 s (6H, CH_3), 2.42 s (2H, CH_2), 2.77 s (2H, CH_2), 7.69 m (1H, 2-H). ^{13}C NMR spectrum, δ_C, ppm: 28.37 (CH_3), 35.05 (C^6), 37.20 (C^7), 52.17 (C^5), 115.15 q (C^3, ^2J = 39 Hz), 116.45 (C^{3a}), 121.55 q (CF_3, ^1J = 267 Hz), 143.21 q (C^2, ^3J = 6 Hz), 167.94 (C^{7a}), 191.16 (C^4). ^{19}F NMR spectrum, δ_F, ppm: –60.07 s (CF_3). Found, %: C 56.78; H 4.72. $C_{11}H_{11}F_3O_2$. Calculated, %: C 56.90; H 4.77.

6,6-Dimethyl-3-perfluoroethyl-6,7-dihydrobenzofuran-4(5*H*)-one (IVb). Yield 78%, mp 39–42°C. IR spectrum, ν, cm^{-1}: 1685, 1565, 1450. ^1H NMR spectrum, δ, ppm: 1.14 s (6H, CH_3), 2.41 s (2H, CH_2), 2.79 s (2H, CH_2), 7.66 m (1H, 2-H). ^{13}C NMR spectrum, δ_C, ppm: 28.29 (CH_3), 34.83 (C^6), 37.33 (C^7), 52.53 (C^5), 111.35 t.q (CF_2, ^1J = 251, ^2J = 40 Hz), 113.38 t (C^3, ^2J = 30 Hz), 117.10 (C^{3a}), 118.80 q.t (CF_3, ^1J = 286, ^2J = 38 Hz), 144.18 t (C^2, ^3J = 10 Hz), 168.09 (C^{7a}), 190.45 (C^4). ^{19}F NMR spectrum, δ_F, ppm: –84.38 br.s (CF_3), –109.02 br.s (CF_2). Found, %: C 51.14; H 3.99. $C_{12}H_{11}F_5O_2$. Calculated, %: C 51.07; H 3.93.

6,6-Dimethyl-3-perfluoropropyl-6,7-dihydrobenzofuran-4(5*H*)-one (IVc). Yield 82%, mp 59–62°C. IR spectrum, ν, cm^{-1}: 1690, 1565, 1445. ^1H NMR spectrum, δ, ppm: 1.15 s (6H, CH_3), 2.42 s (2H, CH_2), 2.80 s (2H, CH_2), 7.67 m (1H, 2-H). ^{13}C NMR spectrum, δ_C, ppm: 28.32 (CH_3), 34.88 (C^6), 37.43 (C^7), 52.63 (C^5), 108.61 t.m (CF_2, ^1J = 265 Hz), 113.63 t (C^3, ^2J = 30 Hz), 113.69 t.t (CF_2, ^1J = 253, ^2J = 33 Hz), 117.36 (C^{3a}), 118.00 q.t (CF_3, ^1J = 288, ^2J = 35 Hz), 144.54 t (C^2, ^3J = 10 Hz), 168.11 (C^{7a}), 190.42 (C^4). ^{19}F NMR spectrum, δ_F, ppm: –80.50 m (CF_3), –106.08 m (CF_2), –125.57 m (CF_2). Found, %: C 47.12; H 3.41. $C_{13}H_{11}F_7O_2$. Calculated, %: C 47.00; H 3.34.

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