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Tandem radical trifluoromethylation–nucleophilic cyclization of a glucose-derived ketene dithioacetal. Synthesis of 5-deoxy-5-*C*-trifluoromethyl-aldurono-6,3-lactones

Gaëlle Foulard, Thierry Brigaud, Charles Portella *

Laboratoire 'Réactions Sélectives et Applications', Associé au CNRS (UMR 6519), Université de Reims, Faculté des Sciences, BP 1039, 51687 Reims Cédex 2, France

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Abstract

A diastereoisomeric mixture of 5-*C*-trifluoromethylated analogs of D-glucuronolactone and L-iduronolactone was synthesized in a five-step procedure from 1,2-*O*-isopropylidene-α-D-glucofuranose. The key step involves a tandem radical trifluoromethylation—nucleophilic cyclization of a ketene dithioacetal derived from 1,2-*O*-isopropylidene-α-D-*xylo*-pentodialdo-1,4-furanose.

Keywords: Ketene dithioacetals; Radical trifluoroalkylation; Glucuronolactone; Iduronolactone

1. Introduction

Various changes are expected concerning the chemical and biological properties of target molecules by substitution of hydrogen atoms by fluorine or trifluoromethyl groups [1,2]. For this reason, many monofluoro and difluorosugars exhibiting interesting biological activities have been synthesized [3,4], but little is known about *C*-trifluoromethyl carbohydrates probably due to the lack of preparative methods. To our knowledge the few examples of *C*-trifluoromethyl sugars known were prepared either starting from a trifluoromethylated synthon [5–11] or through the nucleophilic trifluoromethylation of oxosugars with the Ruppert reagent [12–16] (CF_3SiMe_3) or a trifluoromethylated organometallic [17].

We report here the synthesis of 5-deoxy-5-*C*-(trifluoromethyl)aldurono-6,3-lactones by a tandem radical trifluoromethylation–nucleophilic cyclization of a sugar-derived ketene dithioacetal followed by an oxidative hydrolysis of the dithio-orthoester (Scheme 1).

Our targets were the 5-deoxy-5-*C*-trifluoromethyl-α-D-glucurono- and β-L-idurono-6,3-lactones. The former is a trifluoromethylated analog of glucuronolactone (antiarthritic and detoxificant [18–20]).

The glucose-derived ketene dithioacetal **1** was prepared from commercially available 1,2-*O*-isopropylidene-α-D-glucofuranose by periodic oxidation [21–24], followed by a Peterson reaction with the 2-lithio-2-trimethylsilyl-1,3-dithiane (Scheme 2).

We have recently reported the radical *F*-alkylation of the ketene dithioacetal **1** and its 3-*O*-protected derivative with *F*-alkyl iodide initiated with sodium dithionite [25]. The intermediate iodide was trapped in situ by the free 3-hydroxyl group to give the cyclic adduct. Thus **1** was treated with bromotrifluoromethane under pressure (7–10 bar) following the same pathway except that the sulfoxylate radical anion was generated from an HCO_2Na/SO_2 system [26] in DMF. The trifluoromethylation–cyclization occurred in 72% yield (Scheme 3) but showed a poor stereoselectivity as previously observed for a long-chain perfluoroalkyl analog [25]. A 60/40 ratio of two diastereomers was obtained. These diastereomers were separated by silica gel chromatography, but their configurations could not be determined unambiguously.

The oxidative hydrolysis of the dithio-orthoester **2** was carried out with 1,3-dibromo-5,5-dimethylhydantoin (DBH) in a THF/H_2O/acetone mixture. Calcium carbonate was added to avoid the acetal removal. The conversion was effective but the α-bromolactone **3** was obtained in addition to the expected D-glucuronolactone analog **4a** and L-iduronolactone analog **4b** even on using a slight excess of DBH. The GC monitoring of the reaction showed the formation of the bromolactone at the very beginning of the reaction. We thus decided to perform a two-step procedure to convert **2** into the

* Corresponding author. Fax: +33-2-6053166.

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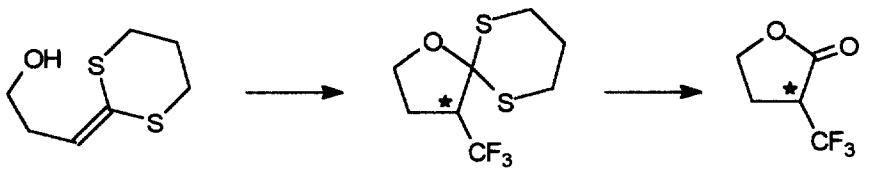
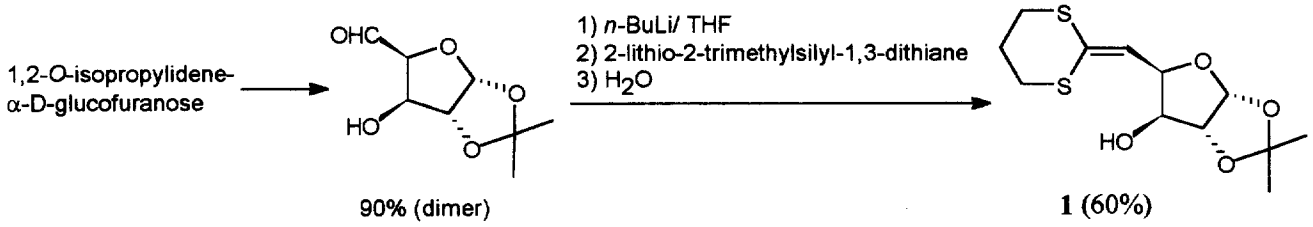
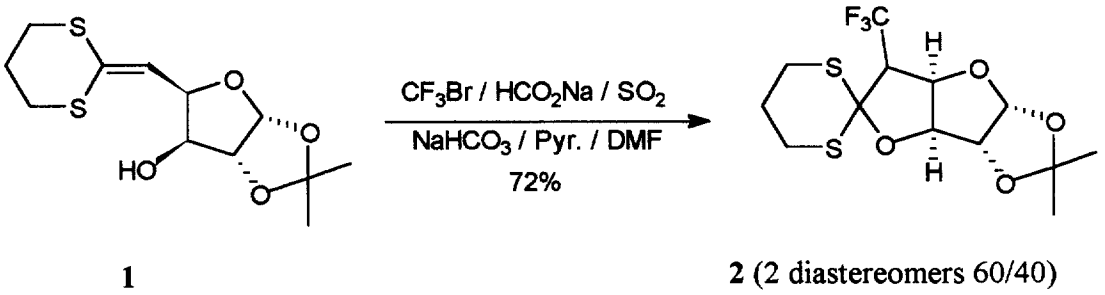


Scheme 1.



Scheme 2.



Scheme 3.

expected lactone **4** (Scheme 4). Treatment of **2** with a large excess of DBH (3 equiv.) gave a 90/10 mixture of **3** and **4** in good yield (84%), each being a mixture of diastereomers.

The crude product was then submitted to tributyltin hydride reduction of the carbon halogen bond to afford **4** in 92% yield as a 75/25 mixture of two diastereomers, the idurono analog being the major one. It should be noted that this ratio remained unchanged by treatment of the **4a/4b** mixture in acidic (PTSA) or basic (Et_3N) medium. No epimerization at C-5 occurred under these conditions and this ratio seems to reflect the thermodynamic equilibrium. Unfortunately, we were unable to separate the two lactones by silica gel chromatography or HPLC.

The D-glucurono and the L-idurono configurations were assigned to each lactone by correlation with literature data [27–29] (Table 1).

We have thus demonstrated that the tandem trifluoromethylation—cyclization reaction of ω-hydroxy ketene dithioacetal provides a new route to highly functionalized lactones. If the reaction is stereoselectively poor using the glucose derivative, we reported more stereoselective reactions with a mannose compound giving rise to various derivatives of 2-*C*-trifluoromethyl heptopyranose [30].

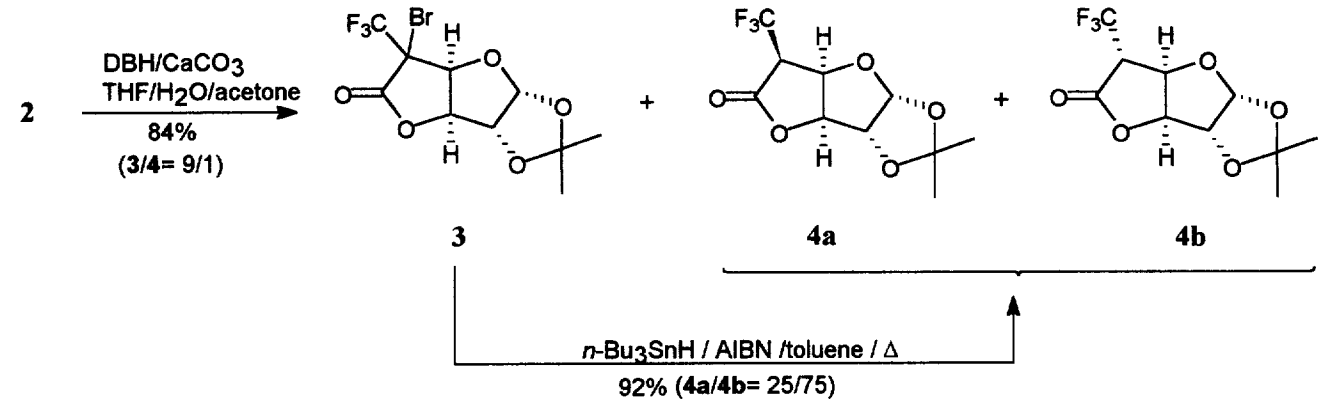
2. Experimental section

2.1. General methods

Melting points are uncorrected. FT-IR spectra were run on a MIDAS apparatus. ^1H, ^{19}F, and ^{13}C NMR spectra were recorded on a BRUKER AC-250 spectrometer. All chemical shifts are reported in ppm. against internal tetramethylsilane for ^1H and ^{13}C NMR spectra and $CFCl_3$ for ^{19}F NMR spectra. MS data were obtained on a Fison VG autospec apparatus at 70 eV in the electron impact mode. Elemental analyses were performed with a Perkin-Elmer CHN 2400 apparatus. All reactions were monitored by TLC (Merck F 254) or GC. GC analyses were performed on a HP 5890 chromatograph equipped with a polydimethylsiloxane HP ultra I column and a flame ionization detector. Silica gel Merck 9385 (40–63 μm) was used for flash chromatography.

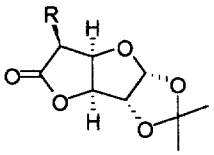
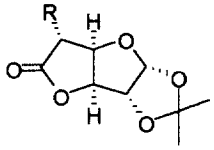
2.1.1. 2-(5-Deoxy-1,2-O-isopropylidene-α-D-xylo-furanose-5-ylidene)-1,3-dithiane (1)

A solution of *n*-BuLi (1.6 M in hexane, 15 ml, 2.4 10^{-2} mol) was slowly added under argon at −50°C to a solution of 1,2-*O*-isopropylidene-α-D-*xylo*-pentodialdo-1,4-furanose (4.10 g, 2.18 10^{-2} mol) in anhydrous THF (25 ml). After 20 min stirring, a solution of 2-lithio-2-trimethylsilyl-1,3-dithiane was slowly added at −50°C to the reaction mixture (2-lithio-2-trimethylsilyl-1,3-dithiane was previously prepared by addition at −30°C of a 1.6 M solution of *n*-BuLi (17.7 ml, 2.83 10^{-2} mol) to a solution of 2-trimethylsilyl-1,3-dithiane (5.45 g, 2.83 10^{-2} mol) in THF (25 ml) and stirring 30 min). The mixture was stirred overnight at room temperature and was neutralized with a 2 N HCl solution and a saturated NH_4Cl solution. After extraction with CH_2Cl_2, the organic layer was dried over $MgSO_4$ and the solvent was



Scheme 4.

Table 1
NMR correlation

Product		δ H_1 ($^3J_{1,2}$)	δ H_2 ($^3J_{2,3}$)	δ H_3 ($^3J_{3,4}$)	δ H_4 ($^3J_{4,5}$)
	R = OTf [14,15]	6.06 (3.7)	4.86 (0.5)	4.93 (3.1)	5.07 (4.0)
	R = CF_3 (**4a**)	6.03 (3.7)	4.84 (0)	4.86 (3.3)	5.17 (4.5)
	R = OBz [14]	6.01 (3.7)	4.89 (0.5)	4.94 (3.4)	5.23 (0.5)
	R = CF_3 (**4b**)	5.99 (3.4)	4.88 (0)	4.94 (3.4)	5.10 (0)

evaporated under reduced pressure. The residue was washed with a small amount of diethylether and filtered to give **1** (3.79 g, 60%) as a white solid: mp = 171.5°C; ^{1}H NMR δ 1.33 (s, 3H, CH_3), 1.54 (s, 3H, CH_3), 1.80 (m, 1H, OH), 2.12–2.23 (m, 2H, $SCH_2CH_2CH_2S$), 2.80–3.06 (m, 4H, SCH_2), 4.16 (m, 1H, H_3), 4.55 (d, 1H, $^3J_{1,2}$ = 3.8 Hz, H_2), 5.14 (dd, 1H, $^3J_{3,4}$ = 2.9 Hz, $^3J_{4,5}$ = 7.8 Hz, H_4), 5.92 (d, 1H, $^3J_{4,5}$ = 7.8 Hz, H_5), 5.94 (d, 1H, $^3J_{1,2}$ = 3.8 Hz, H_1); ^{13}C NMR δ 24.25 ($SCH_2CH_2CH_2S$), 26.8 and 26.3 ($C(CH_3)_2$), 29.3 and 29.0 (SCH_2), 76.2 (C_3), 78.1 (C_4), 85.1 (C_2), 104.4 (C_1), 111.7 ($C(CH_3)_2$), 122.8 (C_5), 135.4 (C_6); IR (KBr) 3428, 1582, 1221, 1163, 1076, 1003 cm^{-1}; MS *m/e* (%) 290 (M^+, 9), 160 (100), 59 (34); Anal. Calcd for $C_{12}H_{18}O_4S_2$: C, 49.63; H, 6.25. Found: C, 49.42; H, 6.14%.

2.1.2. 6,3-Anhydro-5-deoxy-1,2-O-isopropylidene-6,6-[propylenebis(sulfanediyl)]-5-C-trifluoromethyl-α-D-gluco- and β-L-ido-furanose (2)

A solution of **1** (1.48 g, 5.1 mmol) in DMF (12 ml) was introduced in the autoclave reactor with sodium hydrogencarbonate (10.2 mmol), pyridine (10.2 mmol), sulfur dioxide (3–4 M) in solution in DMF (10.2 mmol) and sodium formate (30.3 mmol). The apparatus was purged and submitted to a 8–10-bar pressure of CF_3Br at room temperature with vigorous stirring. After 8 h the pressure was released and the solvent was evaporated under vacuum. The crude mixture was diluted with CH_2Cl_2 and washed with water. The aqueous layer was extracted three times with CH_2Cl_2. The combined organic layers were washed with brine and dried over $MgSO_4$ and the solvent was evaporated to give **2** (72%) as a 60/40 mixture of two diastereomers which were separated by silica gel chromatography (petroleum ether/AcOEt 9/1).

Minor diastereomer. White solid: mp = 93°C; ^{1}H NMR δ 1.35 (s, 3H, CH_3), 1.49 (s, 3H, CH_3), 1.96–2.19 (m, 2H, $SCH_2CH_2CH_2S$), 2.74–2.83 (m, 2H, SCH_2), 2.96 (dq, 1H, $^3J_{HF}$ = 9.0 Hz, $^3J_{4,5}$ = 5.0 Hz, H_5), 3.29–3.52 (m, 2H, SCH_2), 4.72 (d, 1H, $^3J_{1,2}$ = 3.8 Hz, H_2), 4.80 (d, 1H, $^3J_{3,4}$ = 4.2 Hz, H_3), 5.16 (t, 1H, $^3J_{3,4}$ – – $^3J_{4,5}$ = 4.6 Hz, H_4), 6.12 (d, 1H, $^3J_{1,2}$ = 3.8 Hz, H_1); ^{13}C NMR δ 24.03 (CH_2), 26.85 (CH_3), 27.26 (SCH_2), 27.35 (CH_3), 28.91 (SCH_2), 60.31 (q, $^2J_{CF}$ = 28.9 Hz, *C*H–CF_3), 81.57, 84.08 and 86.99 (C_2, C_3 and C_4), 91.90 (S–C–S), 107.96 (C_1), 112.97 ($C(CH_3)_2$), 123.26 (q, $^1J_{CF}$ = 280 Hz, CF_3); ^{19}F NMR δ –60.05 (d, 3F, $^3J_{HF}$ = 9.0 Hz, CF_3); MS *m/e* (%) 358 (M^+, 78), 343 (M – 15, 17), 301 (5), 285 (7), 269 (28), 241 (8), 227

(26), 213 (21), 173 (51), 129 (11), 106 (100); Anal. Calcd for $C_{13}H_{17}O_4F_3S_2$: C, 43.57; H, 4.78. Found: C, 43.81; H 4.40%.

Major diastereomer. White solid: mp = 83°C; ^{1}H NMR δ 1.31 (s, 3H, CH_3), 1.45 (s, 3H, CH_3), 1.88–2.17 (m, 2H, $SCH_2CH_2CH_2S$), 2.62–2.78 (m, 2H, SCH_2), 2.95 (dq, 1H, $^3J_{HF}$ = 10.0 Hz, $^3J_{4,5}$ = 3.1 Hz, H_5), 3.19–3.49 (m, 2H, SCH_2), 4.74 (d, 1H, $^3J_{1,2}$ = 3.4 Hz, H_2), 4.74 (d, 1H, $^3J_{3,4}$ = 4.6 Hz, H_3), 5.05 (dd, 1H, $^3J_{3,4}$ = 4.6 Hz, $^3J_{4,5}$ = 3.1 Hz, H_4), 5.99 (d, 1H, $^3J_{1,2}$ = 3.4 Hz, H_1); ^{13}C NMR δ 23.8 (CH_2), 26.4 (SCH_2), 26.6 and 27.2 (2x CH_3), 28.8 (SCH_2), 63.8 (q, $^2J_{CF}$ = 28.0 Hz, *C*H–CF_3), 83.1, 83.5 and 85.8 (C_2, C_3 and C_4), 92.3 (S–C–S), 107.1 (C_1), 112.6 (*C*(CH_3)$_2$), 123.9 (q, $^1J_{CF}$ = 279 Hz, CF_3); ^{19}F NMR δ −64.16 (d, 3F, $^3J_{HF}$ = 10.0 Hz, CF_3); IR (KBr) 2998, 2934, 1391, 1377, 1273 (vs), 1173 (vs), 1130 (vs), 1030 (vs), 905, 710 cm^{-1}; MS *m/e* (%) 358 (M^+, 100), 283 (6), 269 (9), 241 (5), 227 (9), 213 (16), 173 (33), 157 (28), 119 (68); Anal. Calcd for $C_{13}H_{17}O_4F_3S_2$: C, 43.57; H 4.78. Found: C, 43.33; H, 4.36.

2.1.3. 5-Bromo-5-deoxy-1,2-O-isopropylidene-5-C-trifluoromethyl-D-glucurono-6,3-lactone and 5-bromo-5-deoxy-1,2-O-isopropylidene-5-C-trifluoromethyl-L-idurono-6,3-lactone (3)

A solution of **2** (0.396 g; 1.10 mmol) in 15 ml of a THF/H_2O/acetone (1/2/1) mixture was reacted during 3 h at 0–10°C with DBH (0.948 g, 3.31 mmol) in the presence of $CaCO_3$ (0.772 g, 7.73 mmol). The mixture was poured into a CH_2Cl_2/H_2O mixture and the aqueous layer was extracted three times with CH_2Cl_2. The combined organic layers were washed with a saturated solution of NH_4Cl, dried over $MgSO_4$ and the solvent was evaporated. The crude product (0.549 g) was submitted to silica gel chromatography (petroleum ether/AcOEt 85/15) to give **3** (0.292 g, 76%) as a 55/45 mixture of diastereomers and **4** (0.025 g, 8%).

3 (major diastereomer): ^{1}H NMR δ 1.38 (s, 3H, CH_3), 1.55 (s, 3H, CH_3), 4.84 (d, 1H, $^3J_{1,2}$ = 3.4 Hz, H_2), 5.11 (m, 2H, H_3 and H_4), 6.06 (d, 1H, $^3J_{1,2}$ = 3.4 Hz, H_1); ^{13}C NMR δ 26.46 and 27.06 (C(*C*H_3)$_2$), 52.26 (q, $^2J_{CF}$ = 33.5 Hz, *C*H–CF_3), 80.58, 83.61, 83.98 (C_2, C_3, C_4), 107.87 (C_1), 114.29 (*C*(CH_3)$_2$), 121.08 (q, $^1J_{CF}$ = 281 Hz, CF_3), 164.87 (C=O); ^{19}F NMR δ −66.95 (s, CF_3); IR (KBr) 2998, 2942 (w), 1804 (vs), 1389, 1321, 1302, 1262, 1196 (vs), 1167 (vs), 1071, 1024 (vs), 982 (vs), 897, 822, 685, 523 cm^{-1}.

3 (minor diastereomer; representative peaks are described): ^{1}H NMR δ 1.38 (s, 3H, CH_3), 1.55 (s, 3H, CH_3), 4.95 (d, 1H, $^3J_{1,2}$ = 3.7 Hz, H_2), 5.03 (s, 2H, H_3 and H_4), 6.04 (d, 1H, $^3J_{1,2}$ = 3.7 Hz, H_1); ^{13}C NMR δ 58.59 (q, $^2J_{CF}$ = 31.5 Hz, *C*H–CF_3), 122.01 (q, $^1J_{CF}$ = 281 Hz, CF_3); ^{19}F NMR δ −70.85 (s, CF_3); MS *m/e* (%): 333 (M+1–15, 97), 331 (M−1-15, 100), 291 (40), 289 (40), 251 (4), 209 (24), 190 (5), 151 (10); Anal. Calcd for $C_{10}H_{10}O_5F_3Br$: C, 34.61; H, 2.90. Found: C, 34.63; H, 2.56%.

2.1.4. Reduction of (3) into (4a) and (4b)

The compound **3** (0.292 g, 0.84 mmol, a 55/45 ratio of two diastereomers) was introduced into a round bottom flask equipped with a condenser. Toluene (5.5 ml), tri-*n*-butyltin hydride (0.26 ml, 0.97 mmol) and AIBN (14 mg, 0.08 mmol) were added and the mixture was stirred 2 h at 70–80°C under argon. After cooling to room temperature, the solvent was evaporated. The crude product was purified by dissolving in CH_3CN and washing the CH_3CN layer three times with petroleum ether to get rid of the tin (II) residues. The polar layer was evaporated to give a **4a/4b** (25/75) mixture (0.206 g, 92%) as a white solid which can be recrystallized in toluene.

2.1.5. 5-Deoxy-1,2-O-isopropylidene-5-C-trifluoromethyl-α-D-glucurono-6,3-lactone (4a)

^{1}H NMR δ 1.37 (s, 3H, CH_3), 1.54 (s, 3H, CH_3), 3.52 (dq, 1H, $^3J_{HF}$ = 8.0 Hz, $^3J_{4,5}$ = 4.6 Hz, H_5), 4.84 (d, 1H, $^3J_{1,2}$ = 3.7 Hz, H_2), 4.86 (d, 1H, $^3J_{3,4}$ = 3.3 Hz, H_3), 5.17 (dd, 1H, $^3J_{3,4}$ = 3.3 Hz, $^3J_{4,5}$ = 4.5 Hz, H_4), 6.03 (d, 1H, $^3J_{1,2}$ = 3.7 Hz, H_1); ^{13}C NMR δ 26.45 and 26.92 (C(*C*H_3)$_2$), 49.87 (q, $^2J_{CF}$ = 30.8 Hz, *C*H–CF_3), 81.64, 83.86, 76.91 (C_2, C_3, C_4), 106.96 (C_1), 113.60 (*C*(CH_3)$_2$), 122.18 (q, $^1J_{CF}$ = 278 Hz, CF_3), 166.48 (C=O); ^{19}F NMR δ −63.15 (d, 3F, $^3J_{HF}$ = 7.5 Hz, CF_3).

2.2. 5-Desoxy-1,2-O-isopropylidene-5-C-trifluoromethyl-β-L-idurono-6,3-lactone (4b)

^{1}H NMR δ 1.37 (s, 3H, CH_3), 1.54 (s, 3H, CH_3), 3.46 (q, 1H, $^3J_{HF}$ = 9.9 Hz, H_5), 4.88 (d, 1H, $^3J_{1,2}$ = 3.4 Hz, H_2), 4.94 (d, 1H, $^3J_{3,4}$ = 3.4 Hz, H_3), 5.10 (d, 1H, $^3J_{3,4}$ = 3.4 Hz, H_4), 5.99 (d, 1H, $^3J_{1,2}$ = 3.4 Hz, H_1); ^{13}C NMR δ 26.41 and 26.95 (C(*C*H_3)$_2$), 52.15 (q, $^2J_{CF}$ = 28.9 Hz, *C*H–CF_3), 78.32, 82.08, 85.33 (C_2, C_3, C_4), 106.03 (C_1), 113.41 (*C*(CH_3)$_2$), 122.30 (q, $^1J_{CF}$ = 280 Hz, CF_3), 166.85 (C=O); ^{19}F NMR δ −66.74 (d, 3F, $^3J_{HF}$ = 7.4 Hz, CF_3); IR (KBr) 3000, 2940, 1800 (vs), 1389, 1254 (vs), 1171 (vs), 1123 (vs), 1074, 1026 (vs), 899, 826, 691 cm^{-1}; MS *m/e* (%) 253 (M−15, 100), 211 (98), 191 (7), 165 (6), 152 (15), 132 (9), 123 (8); Anal. Calcd for $C_{10}H_{11}O_5F_3$ (**4a/4b** mixture): C, 44.79; H, 4.13. Found: C, 45.20; H, 3.81%.

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