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Simple access to novel β-hydroxy-β-trifluoromethyl imines☆

Jan Alexander Barten[a], Kazumasa Funabiki[b], Gerd-Volker Röschenthaler[a,*]

[a]*Institut für Anorganische and Physikalische Chemie, Universität Bremen, Leobener Strasse, D-28334 Bremen, Germany*
[b]*Department of Chemistry, Faculty of Engineering, Gifu University, Yanagido, Gifu 501-1193, Japan*

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Abstract

Selected imines reacted with three different trifluoromethyl group-containing ketones in a non-catalyzed manner at ambient temperature to give the corresponding β-hydroxy-β-trifluoromethyl imines in good to excellent yields. With 1,1,1-trifluoroacetone a 1:1 and a 2:1 reaction product was obtained. The reduction of 2-isopropylimino-4-phenyl-5,5,5-trifluoropentan-4-ol led to a 5:1 diastereomeric mixture of the corresponding amine, whose dominant form was found to be (*2S*, *4R*) 4-isopropylamino-4-phenyl-2-trifluoromethyl-butan-2-ol in the solid state. Hydrolysis in one case gave the respective β-hydroxy ketone. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Trifluoromethyl ketones; β-Hydroxy-β-trifluoromethyl imines; Hydrolysis; Reduction

1. Introduction

Recently the reaction of trifluoroacetaldehyde ethyl hemiacetal with several imines [1] and enamines [2] was reported to give the corresponding β-hydroxy-β-trifluoromethyl ketones in good yields after hydrolysis. Earlier, lithiated imines have been used and, e.g. hexafluoroacetone to obtain β-hydroxy-β-bis(trifluoromethyl) ketones or the respective aldehyde after an acidic workup [3,4]. β-Hydroxy-β-trifluoromethyl imines are precursors for β-hydroxy-β-trifluoromethyl ketones and α-trifluoromethyl-γ-amino alcohols. For the latter, to the best of our knowledge only four examples have been reported [5,6]. β-Fluoroalkyl β-amino and α-trifluoromethyl-β-amino alcohols however, are versatile bioactive compounds [7,8]. Here, we describe a simple and general new route to synthesize β-hydroxy-β-trifluoromethyl imines in good to excellent yields from various ketimines or aldimines and activated ketones, like trifluoroacetone, trifluoroacetophenone or 2-hydroxy-trifluoroacetophenone [9] *without* previous lithiation at ambient temperature.

2. Results and discussion

The acyclic imines **1–4** [10–12], the cyclic imines **5** [13], **6** [11] and *N*,*N*′-diisopropylidene-ethane-1,2-diamine [14–16] **7,** were prepared from the corresponding ketone and *iso*propylamine, ethylene diamine were allowed to react with an equimolar amount of the fluorinated ketones **8–10** (Scheme 1). In an enamine mediated addition (cf. [1,2]) non-moisture sensitive β-hydroxy-β-trifluoromethyl imines **11–18** were obtained in good to excellent yields as solids or analytically pure viscous liquids (Scheme 1, Table 1). (For compounds **17–21**, in the ^{1}H NMR ($CDCl_3$) spectra the HO resonances could not be observed. For **17**, ν (C=N) was found at 1666.5 cm^{-1}, δ_C (C=N) = 161.0).

Surprisingly, when aldimine **3** is reacted with trifluoroacetone in a 1:1 ratio in the NMR spectra, compound **15** along with traces of the disubstituted product **20** were found which led us perform the reaction in a 2:5 stoichiometry. In this case, a mixture of the mono- and disubstituted product (due to a second enamine mediated addition) was obtained, separated by recrystallization and sublimation to give a diastereomeric mixture of **20** in 28% yield. In compound **20** probably the intramolecular OH···N hydrogen bridge locks the adjacent CF_3 group above or below the resulting plane giving rise to at least two diastereomers each with two different resonances in the ^{19}F NMR spectra. Upon warming the intensities of one set of signals decreases.

Hydrolysis of compound **11** yielded the corresponding β-hydroxy ketone, 2-hydroxy-2-phenyl-1,1,1-trifluoropentan-

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* Corresponding author. Tel.: +49-421-218-2493; fax: +49-421-218-4267.
E-mail address: gvr@chemie.uni-bremen.de (G.-V. Röschenthaler).

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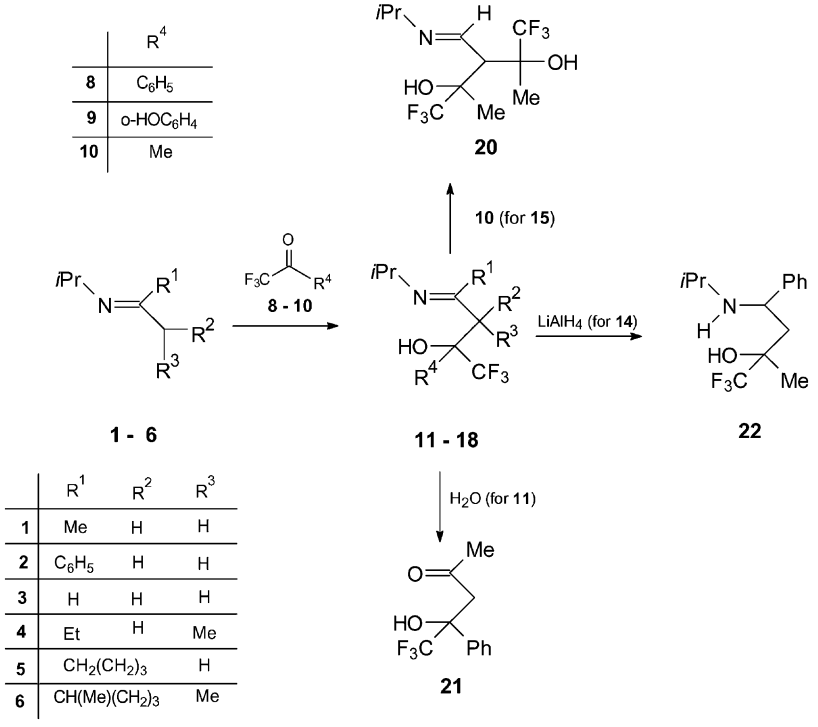


Scheme 1.

Table 1
Starting compounds, products and yields

Entry	R^1	R^2	R^3	Ketone	R^4	Product	Yield (%)
1	Me	H	H	8	C_6H_5	11	92
2	Me	H	H	9	*o*-HOC_6H_4	12	56
3	C_6H_5	H	H	8	C_6H_5	13	96
4	C_6H_5	H	H	10	Me	14	98
5	H	H	H	10	Me	15	76
6	Et	H	Me	8	C_6H_5	16	63
7	$CH_2(CH_2)_2CH_2$		H	8	C_6H_5	17	35
8	$CH(Me)(CH_2)_2CH_2$		Me	8	C_6H_5	18	100

4-one **21**, already synthesized by the lithiation of acetone and treatment with trifluoroacetophenone [17]. The reduction of compound **14**, having *one* chiral centre, using lithium aluminium hydride proceeds stereoselectively to furnish a 5:1 diastereomeric mixture of 4-isopropylamino-4-phenyl-2-trifluoromethyl-butan-2-ol **22A**,**B**.

The single crystal structure determination of the dominant diastereomer **22A** revealed four molecules in the unit cell. Very weak intermolecular hydrogen bonding N–H···O(1) 318.5 pm, and shorter intramolecular distances N(H)···H–O 266.3 pm were observed; N(1)–C(5) 145.6, C(2)–O(1) 141.9, C(5)–C(4) 153.1 and C(4)–C(2) 152.7 are single bonds [18]. The (*2S*, *4R*) configuration in the 4-*iso*propylamino-4-phenyl-2-trifluoromethyl-butan-2-ol molecule was confirmed Fig. 1.

Diimine **7** reacted similarly to the other imines and added two equivalents of trifluoroacetophenone to give the white solid diol **19** (Scheme 2).

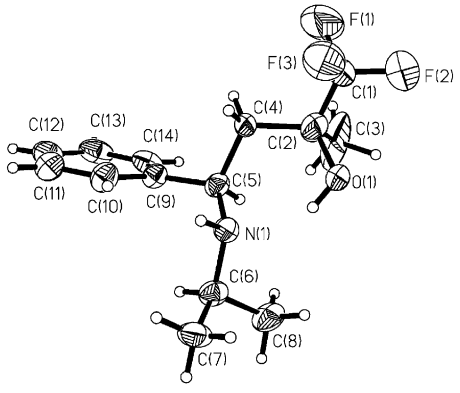
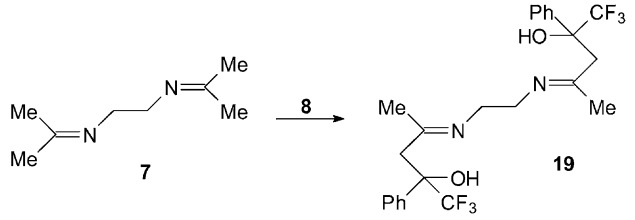


Fig. 1. Selected bond lengths (pm) and bond angles (°) for compound 22A (thermal elipsoids with 50% probability): C(1)–C(2) 145.2(16), C(2)–O(1) 141.9(9), C(2)–C(4) 152.7(10), C(2)–C(3) 169.4(18), C(5)–N(1) 145.6(8), C(5)–C(9) 152.1(8), N(1)–C(6) 149.5(9), C(9)–C(14) 139.3(9); O(1)–C(2)–C(1) 105.3(9), O(1)–C(2)–C(4) 112.6(7), C(1)–C(2)–C(4) 111.7(8), C(4)–C(2)–C(3) 107.6(8), C(2)–C(4)–C(5) 113.1(6), N(1)–C(5)–C(9) 116.5(5), N(1)–C(5)–C(4) 109.4(5), C(14)–C(9)–C(10) 119.0(6).



Scheme 2.

3. Conclusion

In a straightforward enamine mediated C–C bond formation fluorinated ketones add to some ketimines and one aldimine to furnish the respective β-imino alcohols precursors for fluorinated β-amino alcohols and β-hydroxy ketones (shown in one case each). Further studies are in progress.

4. Experimental

NMR spectra were obtained on a Bruker AC 80 instrument operating at 75.39 MHz (^{19}F, internal standard CCl_3F) and a Bruker DPX-200 spectrometer operating at 200.13 MHz for ^{1}H (TMS), 188.31 MHz for ^{19}F ($CFCl_3$) and 50.32 MHz ^{13}C (TMS). The IR spectra of compound **17** was recorded on a Perkin–Elmer Aragon 500 FT-IR between KCl plates. MS spectra were obtained on a Varian MAT CH7A instrument at 70 eV. All reactions and manipulations were conducted under an atmosphere of dry nitrogen. The X-ray structural study was carried out on a Siemens P4 diffractometer using graphite monochromated Mo Kα radiation ($\lambda = 71.073$ pm). The structure was solved by direct methods and anisotropically refined based on F^2 using the SHELX-97 program package [19]. The C–H hydrogen atoms were placed in calculated positions, assigned common isotropic thermal parameters and allowed to ride on their parent atoms. Crystal data for **22A**, colorless crystals, $C_{14}H_{20}F_3NO$, $M = 275.31$, tetragonal, $P4_3$, $a = b = 1046.8(2)$, $c = 1303.1(2)$ pm, $\alpha = \beta = \gamma\ 90^\circ$, $V = 1.4279(4)$ nm^3, final R values ($I > 2\sigma(I)$), $R_1 = 0.0940$, $wR_2 = 0.2341$, R values (all reflections) $R_1 = 0.0973$, $wR_2 = 0.0.2405$; crystal size $1.0 \times 0.7 \times 0.6$ mm^3 with $Z = 4$, reflections measured 2030, unique reflections 1591 ($R_{int} = 0.0403$). The structure was refined as a meroedric twin. A possible disorder at the CF_3 group was not resolved. CCDC deposit number CCDC 171580. See http://www.rsc.org/suppdata/.

4.1. Typical procedure

To a solution of 10 mmol imine in 10 ml diethylether 10 mmol trifluoromethylated ketone in 10 ml diethylether were added at room temperature. The reaction mixture was stirred for 1 h and all volatile substances were removed under reduced pressure. The remaining colorless solids (entries 1, 2, 5–7) were recrystallized from petroleum ether. The remaining oils (entries 3, 4 and 8) were obtained analytically pure.

4.2. 2-Isopropylimino-4-phenyl-5,5,5-trifluoropentane-4-ol (*11*)

The mp is 30 °C. ^{1}H NMR (200.13 MHz, TMS, $CDCl_3$): $\delta = 0.91$ (*i*Pr, 6H, d, $^{3}J_{HH}$ 6.2 Hz), 1.76 (CH_3, 3H, s), 2.92 (CH_2, 2H, AB-system, J_{AB} 17.0 Hz), 3.46 (*i*Pr, 1H, sep, $^{3}J_{HH}$ 6.2 Hz), 7.33 (Ph, 5H, m), 8.57 (OH, 1H, s). ^{19}F NMR (188.31 MHz, $CFCl_3$, $CDCl_3$): $\delta = -83.8$ (CF_3, s). MS: *m/e* (%) = 273 (M^+, 36), 258 (M^+-CH_3, 35), 204 (M^+-CF_3, 62), 84 (*i*PrNCCH_3^+, 100). Analysis: $C_{14}H_{18}F_3NO$ (MG 273.30), calcd.: C 61.51, H 6.64, F 20.87; found: C 61.46, H 6.80, F 21.00.

4.3. 2-Isopropylimino-4-phenyl-5,5,5-trifluoropentan-4-ol (*12*)

The mp is 68 °C. ^{19}F NMR ($CDCl_3$): $\delta = -63.1, -74.5, -75.9, -82.4$. MS: *m/e* (%) = 289 ($M^+$, 28), 231 ($M^+-$*i*Pr –$CH_3$, 59), 220 ($M^+$–$CF_3$, 37), 120 ($C_6H_4OHCO^+$, 100).

4.4. 1-Isopropylimino-1,3-diphenyl-4,4,4-trifluorobutan-2-ol (*13*)

^{1}H-NMR ($CDCl_3$): $\delta = 0.93$ (*i*Pr, 6H, d, $^{3}J_{HH}$ 6.2 Hz), 3.21 (CH_2, 2H, AB-system, J_{AB} 16.8 Hz), 3.28 (CH *i*Pr, 1H, sep, $^{3}J_{HH}$ 6.2 Hz), 7.34 (Ph, 5H, m), 8.55 (OH, 1H, s). ^{19}F MR ($CDCl_3$): $\delta = -83.6$ (CF_3, s). MS: *m/e* (%) = 335 (M^+, 100), 320 (M^+-H_3, 8), 292 (M^+-*i*Pr, 9), 277 (M^+-CH_3, –Pr, 11), 266 (M^+-CF_3, 26). Analysis: $C_{19}H_{20}F_3NO$ (MG 335.37), calcd.: C 68.05, H 6.01, F 16.99; found: C 68.15, H 6.11, F 16.95.

4.5. 1-Isopropylimino-1-phenyl-2-trifluoromethyl-butan-2-ol (*14*)

^{1}H NMR ($CDCl_3$): $\delta = 1.01$ (CH_3 *i*Pr, 6H, d, $^{3}J_{HH}$ 6.2 Hz), 1.44 (CH_3, 3H, s), 2.70 (CH_2, 2H, AB-system, J_{AB} 17.2 Hz), 3.49 (CH *i*Pr, 1H, sep, $^{3}J_{HH}$ 6.2 Hz), 7.22 (Ph, 5H, m), 8.23 (OH, 1H, s). ^{19}F NMR ($CDCl_3$): $\delta = -85.7$ (CF_3, s). MS: *m/e* (%) = 272 (M^+, 15), 258 (M^+-CH_3, 14), 204 (M^+-CF_3, 6), 160 (M^+-CF_3,–*i*Pr, 54), 104 ($PhCO^+$, 100). Analysis: $C_{14}H_{18}F_3NO$ (MG 273.30), calcd.: C 61.53, H 6.64, F 20.85; found: C 61.35, H 6.78, F 21.30.

4.6. 4-Isopropylimino-2-trifluoromethyl-butan-2-ol (*15*)

The mp is 86 °C. ^{1}H NMR ($CDCl_3$): $\delta = 1.01$ (CH_3, *i*Pr, 6H, d, $^{3}J_{HH}$ 6.2 Hz), 1.27 (CH_3, 3H, s), 2.54 (CH_2, 2H, m), 3.32 (CH *i*Pr, 1H, sep, $^{3}J_{HH}$ 6.2 Hz), 6.51 (OH, 1H, s), 7.94 (=CH, 1H, m). ^{19}F NMR ($CDCl_3$): $\delta = -86.3$ (CF_3, s). MS: *m/e* (%) = 197 (M^+-H, 6), 182 (M^+-CH_3, 38), 128 (M^+-CF_3, 13), 70 (CF_3H, 100). Analysis: $C_8H_{14}F_3NO$ (MG 197.20), calcd.: C 48.73, H 7.12, F 28.93; found: C 48.71, H 7.05, F 28.90.

4.7. 4-Isopropylimino-3-methyl-2-phenyl-1,1,1-trifluoro-heptan-2-ol (**16**)

The mp is 30 °C. ^{1}HNMR ($CDCl_3$): $\delta = 0.71$ (CH_3, 3H, d, $^3J_{HH}$ 6.7 Hz), 1.10 (CH_3 Et, 3H, t, $^3J_{HH}$ 7.2 Hz), 1.24 (CH_3 *i*Pr, 6H, d, $^3J_{HH}$ 6.2 Hz), 2.17 (CH_2 Et, 2H, q, $^3J_{HH}$ 7.2 Hz), 3.09 (CH, 1H, q, $^3J_{HH}$ 6.7 Hz), 3.82 (CH *i*Pr, 1H, sep, $^3J_{HH}$ 6.2 Hz), 7.33 (Ph, 5H, m), 8.88 (OH, 1H, s). ^{19}F NMR ($CDCl_3$): $\delta = -78.9$ (CF_3, s). Analysis: $C_{16}H_{22}F_3NO$ (MG 301.35), C 63.75, H 7.36, F 18.92, found: C 63.28, H 7.43, F 19.25.

4.8. 1-Isopropylimino-2-(1′-hydroxy-1′-phenyl-2′,2′,2′-trifluoroethyl)-cyclohexane (**17**)

The mp is 66 °C. ^{1}H NMR ($CDCl_3$): $\delta = 1.20$ (CH_3 *i*Pr, 6H, d; $^3J_{HH}$ 6.2 Hz), 1.54 (CH_2, 8H, m), 2.26 (CH, 1H, m), 3.04 (CH *i*Pr, 1H, sep, $^3J_{HH}$ 6.2 Hz), 7.25 (Ph, 5H, m). ^{19}F NMR (C_6D_6): $\delta = -77.9$ (CF_3, s). MS: *m/e* (%) = 313 (M^+, 35), 270 (M^+-iPr, 17), 244 (M^+-CF_3, 28), 202 (M^+-CF_3, $-i$Pr, 13), 139 ($M^+-PhCOCF_3$, 100). Analysis: $C_{17}H_{22}F_3NO$ (MG 313.36); calcd.: C 65.15, H 7.08, F 18.19; found: C 64.95, H 7.13, F 17.91.

4.9. 2,6-Dimethyl-2-(1′-hydroxy-1′-phenyl-2′,2′,2′-trifluoroethyl)-1-isopropylimino-cyclohexane (**18**)

^{1}H NMR ($CDCl_3$): $\delta = 1.07$ (CH_3, 12H, m), 1.88 (CH_2, CH, 7H, m), 3.82 (CH *i*Pr, 1H, sep, $^3J_{HH}$ 6.2 Hz), 7.70 (Ph, 5H, m). ^{19}F NMR ($CDCl_3$): $\delta = -75.7$ (CF_3, s). MS: *m/e* (%) = 341 (M^+, 22), 284 (M^+-CH_3,$-i$Pr, 52), 273 (M^+-CF_3, 100). Analysis: $C_{19}H_{26}F_3NO$ (MG 341.42); calcd.: C 66.84, H 7.68, F 16.69; found: C 67.35, H 7.79, F 16.30.

4.10. Ethylen-bis-[2-hydroxy-4-imino-2-phenyl-1,1,1-trifluoro-pentane] (**19**)

In 10 ml diethylether 15 mmol **7** were dissolved and 30 mmol trifluoroacetophenone were added. All volatile substances were removed under reduced pressure to give a white solid recrystallized from petroleum ether. Yield: 50%, mp 128 °C. The data for ^{1}H NMR ($CDCl_3$): $\delta = 1.7$ (CH_3, 6H, s), 2.9 (CH_2, 4H, s), 3.2 (NCH_2, 4H, s), 7.3 (Ph, 5H, m). ^{19}F NMR ($CDCl_3$): $\delta = -84.0$ (CF_3, s). MS: *m/e* (%) = 488 (M^+, 7), 418 (M^+-CF_3H, 8), 314 ($M^+-PhCOCF_3$, 14), 244 ($M^+/2$, 100), 176 ($M^+/2$ $-CF_3$, 32). Analysis: $C_{24}H_{26}F_6N_2O_2$ (MG 488.47); C 59.01, H 5.36, F 23.34; found: C 58.87, H 5.40, F 23.22.

4.11. 3-Isopropylmethyliden-2,4-bis(trifluoromethyl)-pentan-2,4-diol (**20**)

To a solution of 50 mmol trifluoroacetone in 20 ml diethylether were added 20 mmol aldimine **3** in 20 ml diethylether at 0 °C and stirred for 1 h. After removal of the solvent the remaining white solid was recrystallized from *n*-hexane/THF 2:1. Selected data for: ^{1}H NMR ($CDCl_3$): $\delta = 1.08$ (CH_3 *i*Pr, 6H, d, $^3J_{HH}$ 6.2 Hz), 1.54 (CH_3, 6H, s), 3.23 (CH *i*Pr, 1H, sep, $^3J_{HH}$ 6.2 Hz), 4.77 (CH, 1H, s), 7.80 (=CH, 1H, s). ^{19}F NMR ($CDCl_3$): $\delta = -79.5$, -86.3, -89.2, -89.7 (CF_3, s). MS: *m/e* (%) = 309 (M^+, 11), 294 (M^+-CH_3, 100), 240 (M^+-CF_3, 37), 196 ($M^+-HOC(CF_3)_2$, 14), 44 ($C_3H_7^+$, 85). Analysis: HRMS (for $C_{11}H_{17}F_6NO$): calcd.: 309.11636; found: 309.11710.

4.12. 2-Hydroxy-2-phenyl-1,1,1-trifluoro-pentan-2-on (**21**)

To a solution of 5 mmol **11** in 10 ml ethanol 5 ml HCl (20%) was added and refluxed for 1 h. Diethylether (3*x* 20 ml) was added, the organic layer separated and dried over $MgSO_4$, filtered and the solvent removed under reduced pressure. A white solid remained. Yield 56%, mp 51 °C. Selected data for: ^{1}H NMR ($CDCl_3$): $\delta = 2.08$ (CH_3, 3H, s), 3.24 (CH_2, 2H, s), 7.32 (Ph, 5H, m). ^{19}F NMR ($CDCl_3$): $\delta = -84.4$ (CF_3, s). MS: *m/e* (%) = 232 (M^+, 21), 175 ($M^+-CH_3COCH_2$, 11), 163 (M^+-CF_3, 46), 105 ($C_6H_5CO^+$, 100).

4.13. 4-Isopropylamino-4-phenyl-2-trifluoromethyl-butan-2-ol (**22A**,**B**)

Compound **11** (8 mmol) in 20 ml diethylether was added dropwise at 0 °C to a suspension of 18 mmol $LiAlH_4$ in 30 ml diethylether. Afterwards, water is added to the mixture and the residue is filtered and washed with diethylether. The organic layer was separated, dried over $MgSO_4$, and the solvent was removed under reduced pressure to leave a white solid recrystallized from *n*-hexane. The data for two diastereomers **A**:**B** = 5:1): ^{1}H NMR ($CDCl_3$): $\delta = 0.95$(**B**), 1.12(**A**) (*i*Pr, 6H, d,$^3J_{HH}$ 6.2 Hz), 1.27 (**B**), 1.50(**A**) (CH_3, 3H, s), 1.92(**B**), 2.14(**A**) (CH_2, 2H, ABX-system), 2.65(**B**), 2.71(**A**) (*i*Pr, 1H, sep,$^3J_{HH}$ 6.2 Hz), 4.24(**B**), 4.26(**A**) (PhCH, 1H, m), 7.20(**B**), 7.25(**A**) (Ph, 5H, m). ^{19}F NMR ($CDCl_3$): $\delta = -83.1$(**B**), -88.8(**A**) (CF_3, s), MS: *m/e* (%) = 276 ($M^+ + H$, 18), 260 (M^+-CH_3, 22), 217 (M^+-CH_3,$-i$Pr, 12), 148 ($M^+-HOCCH_3CF_3CH_2$, 100). Analysis: $C_{14}H_{20}F_3NO$ (MG 2 75.31); calcd.: C 61.07, H 7.32, F 20.70; found: C 61.01, H 7.13, F 20.60.

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