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Novel 1-Trifluoromethyl Substituted 1,2-Ethylenediamines and Their use for the Synthesis of Fluoroquinolones

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Abstract—A common approach to the synthesis of 1-trifluoromethyl-1,2-ethylenediamines was developed. Based on these original building blocks the new derivatives of *N*-substituted fluoroquinolones bearing the trifluoromethyl group were obtained. Through-space transmitted coupling constants $^{7}J_{F-F}$ between the trifluoromethyl group and F-8 were measured in the ^{19}F NMR spectra of all fluoroquinolones examined and assigned unequivocally by using $^{19}F\{^{19}F\}$ double resonance technique. © 2000 Elsevier Science Ltd. All rights reserved.

Introduction

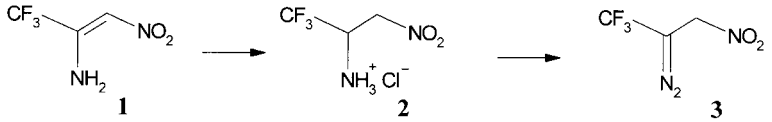
1,2-Ethylenediamines are known to be widely used for the synthesis of a variety of nitrogen-containing heterocyclic compounds of practical importance, such as pyrazines, piperazines, imidazolines and others.[1] Also many derivatives of aliphatic amines bearing α-polyfluoroalkyl substituent, such as aminoacids, aminoalcohols, etc., were shown to be of special importance due to profound biological activities.[2] 1-Trifluoromethyl substituted 1,2-ethylenediamines, which have hitherto been unknown, gained our attention as promising building blocks for the synthesis of new bi- and tri-cyclic derivatives of 6-fluoro-4-oxo-1,4-dihydroquinoline-3-carboxylic acids, a well-known class of the broad spectrum antibacterials.[3]

Results and Discussion

In this paper we wish to describe an approach to 1-trifluoromethyl-1,2-ethylenediamines based on using as starting materials 2-amino-1,1,1-trifluoro-3-nitro-2-propene (**1**), its hydrochloride (**2**), and 2-diazo-1,1,1-trifluoro-3-nitro-propane (**3**) (Scheme 1).[4] These starting materials, which have become available recently,[4] provide access to a variety of 1-trifluoromethyl substituted 1,2-diaminoethanes bearing two primary amino groups,[4d] and their *N*-acyl, *N*-alkyl or 1-cycloalkylimino substituted derivatives.

In the reaction of the *N*-benzoyl derivative of nitroamine **2** (compound **4**) with $LiAlH_4$ in absolute ether, reduction of the nitro group was accompanied, as expected, by conversion of the amide group as well, thus giving *N*-benzyl substituted 1-trifluoromethyl-1,2-diamine **5a** in a good yield (Scheme 2). When reacted with tosyl chloride, compound **4** was converted into the 2-*N*-tosyl derivative **5b**. It is worth noting that when compound **4** was treated with the 1:1 complex $LiAlH_4$-glycerol, reduction of the nitro group took place predominantly, yielding *N*-benzoyl-1-trifluoromethyl substituted 1,2-ethylenediamine **6**, together with a minor amount of *N*-benzyl 1,2-diaminoethane **5a** (Scheme 2).

Another type of functionalization of 1-trifluoromethyl



Scheme 1.

Keywords: fluoroquinolones; trifluoromethyl group; 1-trifluoromethyl-1,2-ethylenediamines.
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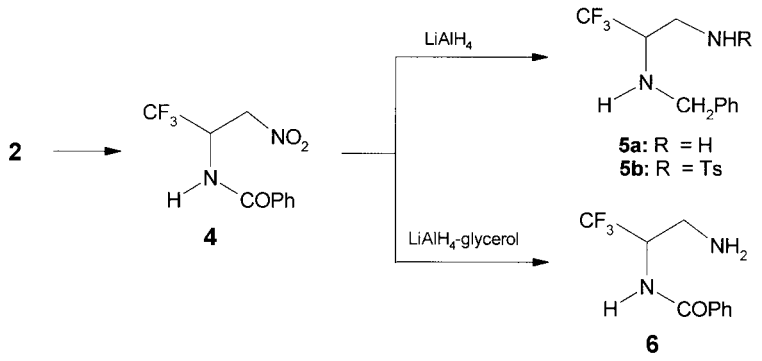


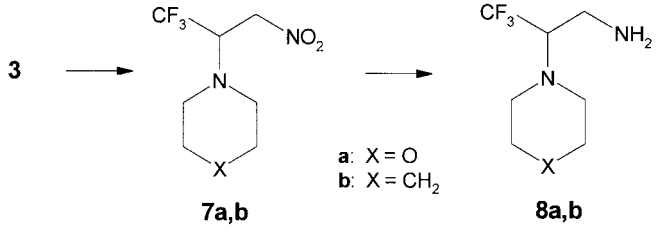
Scheme 2.

substituted 1,2-ethylenediamines is based on substitution of the diazo group in 2-diazo-1,1,1-trifluoro-3-nitropropane (**3**) by cycloalkylimines. It is known that secondary amines are able to cause displacement of the diazo group in aliphatic diazo compounds, although these reactions take place only in the presence of copper, palladium or rhodium complexes, catalyzing insertion of the generated carbenes into the N–H bond of secondary amines.[5] We have found that 2-diazo-1,1,1-trifluoro-3-nitropropane (**3**) can easily be transformed into 2-cycloalkylimino-1,1,1-trifluoro -3-nitropropanes **7a**,**b** by action of morpholine and piperidine without the use of any catalysts (Scheme 3). It has been suggested that a plausible reason for such behavior is that compound **3** is able to generate carbene species from the corresponding anion derived from **3** under basic conditions.[6] Reduction of the nitro group in compounds **7a**,**b** with $LiAlH_4$ afforded the corresponding diamines **8a**,**b** (Scheme 3).

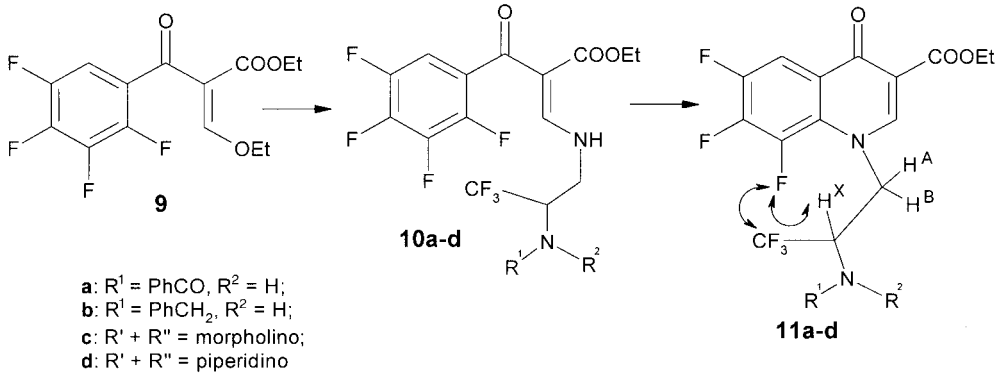
1-Trifluoromethyl substituted 1,2-diamines **5a**,**6** and **8a**,**b** appear to be versatile and useful building blocks, as illustrated by their conversion by action of ethyl 3-ethoxy-2-(2,3,4,5-tetrafluorobenzoyl)acrylate **9**[7] into new derivatives of fluoroquinolones **11a**–**d** (Scheme 4). Nucleophilic displacement of the ethoxy group in **9** by amines **5a**, **8a**,**b** is presumed to give the corresponding benzoylacrylates **10b**–**d** followed by simultaneous intramolecular amino-defluorination reaction leading to quinolones **11b**–**d** (Scheme 4). The exception is the reaction of the *N*-benzoyl compound **6** with ethoxyacrylate **9**, which requires the presence of *N*-methyl-morpholine in the reaction mixture in order to convert the intermediate **10a** into quinolone **11a**.

^{1}H NMR Spectral data for fluoroquinolones **11a**–**d** are characterized by a considerable difference in chemical shifts of H^A and H^B signals which are nonequivalent due to the presence of the neighboring asymmetric carbon, as well as H^A and H^B protons coupled to F^8 with coupling constants $^5J_{HF}$ of 2–4 Hz (Table 1).

In the ^{19}F NMR spectra of all fluoroquinolones examined (Table 2) the long-range coupling constants ^7J(F-8, F–CF_3) were observed and assigned unequivocally by using the



Scheme 3.



Scheme 4.

Table 1. ^{1}H NMR spectral data for fluoroquinolones **11a**–**d** in DMSO-d_6

Compound	^{1}H Chemical shifts, δ (ppm) and coupling constants (Hz)					
	H^A	H^B	H^X	H^2	H^5	Other signals
11a	5.2 m	4.53 m	5.3 m	8.73 s	8.00 ddd $^{3}J_{H^5F^6}$=10.5 $^{4}J_{H^5F^7}$=8.5 Hz $^{5}J_{H^5F^8}$=2.1 Hz	9.18 d (NH, J=9.5 Hz)
11b	4.78 ddd $^{2}J_{AB}$=14.7 Hz $^{3}J_{AX}$=3.1 Hz $^{5}J_{H^BF^8}$=3.1 Hz	3.80 ddd $^{2}J_{AB}$=14.7 Hz $^{3}J_{BX}$=110 Hz $^{5}J_{H^BF^8}$=2.8 Hz	3.42 m	8.29 s	8.11 ddd $^{3}J_{H^5F^6}$=10.1 Hz $^{4}J_{H^5F^7}$=8.1 Hz $^{5}J_{H^5F^8}$=2.8 Hz	3.98 and 3.70 (NCH_2, ^{2}J=12.9 Hz)
11c	4.81 ddd $^{2}J_{AB}$=14.8 Hz $^{3}J_{AX}$=2.1 Hz $^{5}J_{H^AF^8}$=2.1 Hz	4.57 ddd $^{2}J_{AB}$=14.8 Hz $^{3}J_{BX}$=10.7 Hz $^{5}J_{H^BF^8}$=3.7 hz	3.93 m	8.55 s	8.01 ddd $^{3}J_{H^5F^6}$=10.5 Hz $^{4}J_{H^5F^7}$=8.5 Hz $^{5}J_{H^5F^8}$=2.1 Hz	1.30 t (3H, J=7.1 Hz) and 4.26 m (2H, OEt), 2.55 and 3.05 (2 NCH_2), 3.42 and 3.48 (2 OCH_2)
11d	4.77 ddd $^{2}J_{AB}$=14.8 Hz $^{3}J_{AX}$=2.2 Hz $^{5}J_{H^AF^8}$=2.2 Hz	4.55 ddd $^{2}J_{AB}$=14.8 Hz $^{3}J_{BX}$=11.2 Hz $^{5}J_{H^BF^8}$=3.6 Hz	3.80 m	8.48 s	8.01 ddd $^{3}J_{H^5F^6}$=10.5 Hz $^{4}J_{H^5F^7}$=8.5 Hz $^{5}J_{H^5F^8}$=2.2 Hz	1.28 t (3H, J=6.9 Hz) and 4.25 m (2H, OEt), 2.43 and 2.98 (2 NCH_2), 1.39 m (3 CH_2)

double resonance $^{19}F\{^{19}F\}$ decoupling technique, whilst the corresponding ^{6}J(F-8, C–CF_3) couplings were not observed in the ^{13}C NMR spectra (see experimental part). The presence of $^{7}J_{F-F}$ coupling and the absence of $^{6}J_{C-F}$ coupling support the hypothesis of a through-space pathway for the transmission of the spin information between the aromatic fluorine atom C^8–F and the trifluoromethyl group which is due to the geometrical proximity of the interacting nuclei.[8]

Conclusion

In conclusion we point out that a new effective method for obtaining of 1-trifluoromethyl-1,2-ethylenediamines from 2-amino-1,1,1-trifluoro-3-nitropropanes can be useful for the synthesis of a variety of biologically active compounds or intermediates, as illustrated in this paper by the synthesis of a number of new fluoroquinolones. The presence of the long-range coupling ^{7}J(F-8, F–CF_3) in the ^{19}F NMR spectra of all fluoroquinolones demonstrates the existence of through-space interaction between aromatic fluorine atom C^8–F and the trifluoromethyl group.

Experimental

General procedures

Solvents were purified according to standard procedures. ^{1}H NMR spectra were measured in solution of DMSO-d_6 and $CDCl_3$ on a Bruker WM-250 (250 MHz) and a Tesla BS-587 A (80 MHz) spectrometers, ^{19}F NMR spectra—on a Tesla BS-587 A (75.3 MHz) spectrometer. All signals are given in ppm (δ) with tetramethylsilane as an internal standard for ^{1}H and hexafluorobenzene for ^{19}F. Double resonance experiments $^{1}H\{^{19}F\}$ and $^{19}F\{^{19}F\}$ were performed on a Tesla BS-587A supplied with a specially designed decoupler operating on ^{19}F frequency.[9]

Microanalyses were carried out on a CHNS-O model EA-1102 elemental analyzer and were found to be in good

Table 2. ^{19}F NMR spectral data for fluoroquinolones **11a**–**d** in DMSO-d_6

Compound	^{19}F Chemical shifts, δ (ppm) and coupling constants (Hz)			
	F^6	F^7	F^8	CF_3
11a	26.22 ddd $^{3}J_{F^6F^7}$=23.4 Hz $^{3}J_{F^6H^5}$=10.5 Hz $^{4}J_{F^6F^8}$=4.9 Hz	11.19 ddd $^{3}J_{F^7F^6}$=23.4 Hz $^{3}J_{F^7F^8}$=19.5 Hz $^{4}J_{F^7H^5}$=8.5 Hz	19.9 dm $^{3}J_{F^8F^7}$=19.5 Hz	90.18 dd $^{3}J(CF_3, H^X)$=7.8 Hz $^{7}J(CF_3, F^8)$=5.9 Hz
11b	26.12 ddd $^{3}J_{F^6F^7}$=23.4 Hz $^{3}J_{F^6H^5}$=10.1 Hz $^{4}J_{F^6F^8}$=4.9 Hz	10.08 ddd $3J_{F^7F^6}$=23.2 Hz $^{3}J_{F^7F^8}$=19.5 Hz $^{4}J_{F^7H^5}$=8.1 Hz	20.76 m	90.49 dd $^{3}J(CF_3, H^X)$=6.0 Hz $^{7}J(CF_3, F^8)$=6.0 Hz
11c	25.08 $^{3}J_{F^6F^7}$=23.4 Hz $^{3}J_{F^6H^5}$=10.5 Hz $^{4}J_{F^6F^8}$=4.9 Hz	10.32 ddd $^{3}J_{F^7F^6}$=23.4 Hz $^{3}J_{F^7F^8}$=19.5 Hz $^{4}J_{F^7H^5}$=8.5 Hz	21.95 m	96.36 dd $^{3}J(CF_3, H^X)$=8.8 Hz $^{7}J(CF_3, F^8)$=8.8 Hz
11d	25.06 ddd $^{3}J_{F^6F^7}$=23.2 Hz $^{3}J_{F^6H^5}$=10.5 Hz $^{4}J_{F^6F^8}$=4.9 Hz	10.36 ddd $^{3}J_{F^7F^6}$=23.2 Hz $^{3}J_{F^7F^8}$=19.5 Hz $^{4}J_{F^7H^5}$=8.5 Hz	21.99 m	96.60 dd $^{3}J(CF_3, H^X)$=9.2 Hz $^{7}J(CF_3, F^8)$=9.2 Hz

agreement with the calculated values. The compounds **1**–**3**[4] and **9**[7] were prepared as described previously.

2-Benzoylamino-1,1,1-trifluoro-3-nitro-propane (4). Suspension of **2** (13.90 g, 72 mmol), benzoyl chloride (12.08 g, 86 mmol) and dry pyridine (12.7 ml, 160 mmol) in 1,4-dioxane (50 ml) was stirred at 40°C for 6 h. After cooling of the mixture to room temperature, 50 ml cold water was added and the crystalline residue was filtered off, washed with 50 ml of hot water and dried. Yield 15.20 g (92%). Mp 158°C [Lit.[10] 158–159°C].

3-Amino-2-benzylamino-1,1,1-trifluoropropane (5a). A solution of the compound **4** (1.81 g, 7 mmol) in dry ether (40 ml) was added dropwise to a suspension of $LiAlH_4$ (1.59 g, 42 mmol) in dry ether, and the reaction mixture was stirred for 8 h. After addition of 20% NaOH (10 ml) the organic phase was separated, and the solvent was evaporated to give a yellow oil which was fractionally distilled under vacuum, yielding 0.99 g (65%) of **5a**, as a colorless oil, bp 104–105°C/2 mm. The compound was used immediately for the syntheses of **5b** and **11b**.

2-Benzylamino-1,1,1-trifluoro-3-tosylaminopropane (5b). A solution of tosyl chloride (1.71 g, 9 mmol) in pyridine was added dropwise to a solution of **5a** (0.99 g, 4.5 mmol) in pyridine under cooling (0–5°C). The reaction mixture was stirred for 2 h at ambient temperature, and after that 2% HCl (100 ml) was added. A precipitate of **5b** was filtered off and recrystallized from ethanol to yield 0.92 g (58%) of **5b** as a white solid. Mp 108–109°C. ^{1}H NMR: δ 7.68 (d, 2H, Ts, J=8.2 Hz), 7.31 (c, 5H, Ph), 7.27 (d, 2H, Ts, J=8.2 Hz), 5.07 (dd, 1H, $CH_2N\mathit{H}$, J=8.2, 3.3 Hz), 3.93 (d, 1H, $C\mathit{H}_2$Ph, J=13.2 Hz), 3.37 (d, 1H, $C\mathit{H}_2$Ph, J=13.2 Hz), 3.41–2.75 (m, 4H, $CHN\mathit{H}$, $CHCH_2$), 2.41 (s, 3H, CH_3). ^{19}F NMR: δ 88.28 (d, CF_3, J_{HF}=7.3 Hz). Anal. calcd for $C_7H_{13}F_3N_2O_2S$: C 54.8, H 5.1, N 7.5. Found: C 55.1, H 5.2, N 7.5.

3-Amino-benzoylamino-1,1,1-trifluoropropane (6). Glycerol (5.74 g, 62 mmol) was added to suspension of $LiAlH_4$ (2.36 g, 62 mmol) in dry ether (50 ml) and was stirred for 1 h, then compound **4** (5.38 g, 21 mmol) was added. After stirring for 6 h 20% NaOH was added and organic phase was separated and evaporated. Oily residue was treated with hexane and filtered to give 0.36 g (67%) of 6. Mp 74–74.5°C. ^{1}H NMR: δ 7.91–7.79 (m, 2H, Ph), 7.58–7.43 (m, 3H, Ph), 7.37 (br. s, 1H, $N\mathit{H}$COPh), 4.78 (m, 1H, $C\mathit{H}^XCF_3$), 3.30 (dd, 1H, $C\mathit{H}^AH^BNH_2$, J_{AB}=13.8, J_{AX}=3.8 Hz), 2.97 (ddq, 1H, $CH^A\mathit{H}^BNH_2$, J_{AB}=13.8, J_{BX}=5.1, J_{HF}=1.0 Hz), 1.36 (br.s, 2H, NH_2). ^{19}F NMR: δ 87.52 (d, CF_3, J_{HF}=7.3 Hz). Anal. calcd for $C_{10}H_{11}F_3N_2O$: C 51.7, H 4.8, N 12.1. Found: C 51,6, H 4.7, N 11.8. Evaporation of hexane from the filtrate gave 36 mg of **5a** (for characteristics of **5a** see the procedure given above).

1,1,1-Trifluoro-2-morpholino-3-nitropropane (7a). To a solution of morpholine (2.48 g, 29 mmol) in benzene (25 ml) was added dropwise the diazo compound **3** in benzene (25 ml). After stirring for 3 h the mixture was evaporated and the residual yellow oil was fractionated under vacuum to give 4.97 g (90%) of **7a**. Bp 83–84°C/5 mm. ^{1}H NMR: δ 4.66 (dd, 1H, $C\mathit{H}^AH^BNO_2$, J_{AB}=12.8, J_{AX}=10.4 Hz), 4.55 (dd, 1H, $CH^A\mathit{H}^BNO_2$, J_{AB}=12.8, J_{AX}=4.3 Hz), 4.01 (dqd, 1H, $C\mathit{H}^XCF_3$, J_{AX}=10.4, J_{BX}=4.3, J_{XF}=8.0 Hz), 3.63 (m, 4H, CH_2OCH_2), 3.00–2.73 (m, 4H, CH_2NCH_2). ^{19}F NMR: δ 94.59 (d, CF_3, J_{XF}=8.0 Hz). Anal. calcd for $C_7H_{11}F_3N_2O_3$: C 36.8, H 4.9, N 12.3. Found: C 36.8, H 4.8, N 12.1.

1,1,1-Trifluoro 3-nitro-2-piperidinopropane (7b) was obtained similarly as an unstable yellow oil from the diazo compound 3 and piperidine. Yield 61%. Bp 75–76°C/5 mm. ^{1}H NMR: δ 4.66 (dd, 1H, $C\mathit{H}^AH^BNO_2$, J_{AB}=12.8, J_{AX}=10.4 Hz), 4.50 (dd, 1H, $CH^A\mathit{H}^BNO_2$, J_{AB}=12.8, J_{AX}=4.3 Hz), 3.99 (dqd, 1H, $C\mathit{H}^XCF_3$, J_{AX}=10.4, J_{BX}=4.3, J_{XF}=8.2 Hz), 2.88–2.64 (m, 4H, CH_2NCH_2), 1.50 (m, 6H, $(CH_2)_3$). ^{19}F NMR: δ 94.46 (d, CF_3, J_{HF}=8.2 Hz).

1,1,1-Trifluoro-3-amino-2-morpholinopropane (8a) was obtained from **7a** according to the standard procedure used for preparation of **5a**. White hygroscopic oil. Yield 56%. Bp 71–72°C/5 mm. ^{1}H NMR: δ 3.68 (m, 4H, CH_2OCH_2), 2.99 (dqd, 1H, $C\mathit{H}^XCF_3$, J_{AX}=8.9, J_{BX}=5.2, J_{XF}=8.2 Hz), 2.97–2.67 (m, 4H, CH_2NCH_2), 2.91 (dd, 1H, $C\mathit{H}^AH^BNH_2$, J_{AB}=12.8, J_{AX}=8.9 Hz), 2.86 (dd, 1H, $CH^A\mathit{H}^BNH_2$, J_{AB}=12.8, J_{BX}=5.2 Hz), 1.51 (br. s, 2H, NH_2). ^{19}F NMR: δ 94.33 (d, CF_3, J_{HF}=8.2 Hz). Anal. calcd for $C_7H_{13}F_3N_2O$: C 42.4, H 6.6, N 14.1. Found: C 42.9, H 6.6, N 14.1.

1,1,1-Trifluoro-3-amino-2-piperidinopropane (8b) was obtained from **7b** according to the standard procedure used for preparation of **5a**. Colorless hygroscopic oil. Yield 31%. Bp 49–50°C/2 mm. ^{1}H NMR: δ 2.96 (dqd, 1H, $C\mathit{H}^XCF_3$, J_{AX}=9.5, J_{BX}=4.3, J_{XF}=8.5 Hz), 2.91 (br. m, 2H, CH_2NCH_2), 2.87 (dd, 1H, $C\mathit{H}^AH^BNH_2$, J_{AB}=12.8, J_{AX}=9.5 Hz), 2.80 (dd, 1H, $CH^A\mathit{H}^BNH_2$, J_{AB}=12.8, J_{BX}=4.3 Hz), 2.54 (br. m, 2H, CH_2NCH_2), 1.53 (m, 8H, $(CH_2)_3$, NH_2). ^{19}F NMR: δ 94.29 (d, CF_3, J_{HF}=8.5 Hz). Anal. calcd for $C_8H_{15}F_3N_2$: C 49.0, H 7.7, N 14.3. Found: C 49.0, H 7.7, N 14.2.

Ethyl 1-(2-benzoylamino-3,3,3-trifluoropropyl)-6,7,8-trifluoro-4-oxoquinoline-3-carboxylate (11a). A mixture of **6** (0.75 g, 32 mmol) and 9 (1.04 g, 32 mmol) in toluene (15 ml) was refluxed for 6 h. After addition of *N*-methylmorpholine (0.49 g, 48 mmol) the reaction mixture was refluxed for 14 h, cooled and hexane was added. White solid of **11a** was filtered off, washed with 5 ml of hot isopropanol, and dried in air. Yield 0.57 g (36%). Mp 156–157°C. Anal. calcd for $C_{22}H_{16}F_6N_2O_4$: C 54.3, H 3.3, N 5.8. Found: C 54.0, H 3.3, N 5.6. ^{1}H and ^{19}F NMR spectral data are given in Tables 1 and 2. ^{13}C NMR in DMSO-d_6: δ 14.15 (CH_3), 50.29 (CH, $^2J_{CF}$=30.6, $^5J_{CF}$=4.7 Hz), 53.57 (NCH_2, $^4J_{CF}$=14.2 Hz), 60.13 (OCH_2), 108.88 (C-5, $^2J_{CF}$=19.0 Hz), 109.74 (C-3), 124.12 (CF_3, $^1J_{CF}$=283 Hz), 124.69 (C-8a, $^2J_{CF}$=5.3 Hz), 126.04 (C-4a), 127.37 (C^{ortho}), 128.42 (C^{meta}), 132.22 (C^{para}), 132.55 (C^{ipso}), 141.41 (C-8, $^1J_{CF}$=238, $^2J_{CF}$=14,5 Hz), 141.75 (C-7, $^1J_{CF}$=255, $^2J_{CF}$=17,5, $^2J_{CF}$=15,3 Hz), 147.60 (C-6, $^1J_{CF}$=250, $^2J_{CF}$=10,6 Hz), 152.54 (C-2), 163.34 (O–C=O), 167.37 (N–C=O), 169.96 (C-4).

Ethyl 1-(2-benzylamino-3,3,3-trifluoropropyl)-6,7,8-trifluoro-4-oxoquinoline-3-carboxylate (11b). A mixture of **5a** (0.409 mg, 1.8 mmol) and 9 (0.60 g, 1.8 mmol) in

toluene was refluxed for 2 h. Toluene was evaporated and the residue obtained was recrystallized from a mixture of isopropanol and water (8:1). Yield 0.68 g (81%). Mp 120–121°C. Anal. calcd for $C_{22}H_{118}F_6N_2O_3$: C 55.9, H 3.8, N 5.9. Found: C 55.7, H 3.8, N 5.9. ^{1}H and ^{19}F NMR spectral data are given in Tables 1 and 2.

Ethyl 1-(2-morpholino-3,3,3-trifluoropropyl)-6,7,8-trifluoro-4-oxoquinoline-3-carboxylate (11c) was obtained as a white solid from **8a** and **9**. Yield 74%. Mp 202–203°C. Anal. calcd for $C_{19}H_{18}F_6N_2O_4$: C 50.4, H 4.0, N 6.2. Found: C 50.2, H 4.1, N 6.1. ^{1}H and ^{19}F NMR spectral data are given in Tables 1 and 2.

Ethyl 1-(2-piperidino-3,3,3-trifluoropropyl)-6,7,8-trifluoro-4-oxoquinoline-3-carboxylate (11d). A mixture of **8b** (0.23 g, 12 mmol) and **9** (0.37 g, 12 mmol) in toluene (10 ml) was refluxed for 2 h, toluene was evaporated and the residue was recrystallized from isopropanol-water (8:1). Yield 0.38 g (72%). Mp 169–17°C. Anal. calcd for $C_{20}H_{20}F_6N_2O_3$: C 53.3, H 4.5, N 6.2. Found: C 53.1, H 4.5, N 6.1. ^{1}H and ^{19}F NMR spectral data are given in Tables 1 and 2.

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