

A convenient preparative method for α -trifluoromethyl amines

Shoji Watanabe^{a,*}, Tsutomu Fujita^a, Masami Sakamoto^a, Hiroyuki Hamano^a,
Tomoya Kitazume^b, Takashi Yamazaki^b

^a Department of Applied Chemistry, Faculty of Engineering, Chiba University, Inageku, Yayoicho, Chiba 263, Japan

^b Department of Bioengineering, Tokyo Institute of Technology, Nagatsuta, Midoriku, Yokohama 227, Japan

Received 29 June 1996; revised 4 December 1996; accepted 12 December 1996

Abstract

α -Trifluoromethyl amines (IV) were prepared in three steps: preparation of α -trifluoromethyl ketones (I), their conversion to benzyloximes (II), and reduction of the oximes (II) with lithium aluminium hydride and sodium methoxide. For example, α -trifluoromethyltridecylamine was obtained from the reduction of trifluoromethyl dodecyl ketone benzyl oxime. © Elsevier Science S.A.

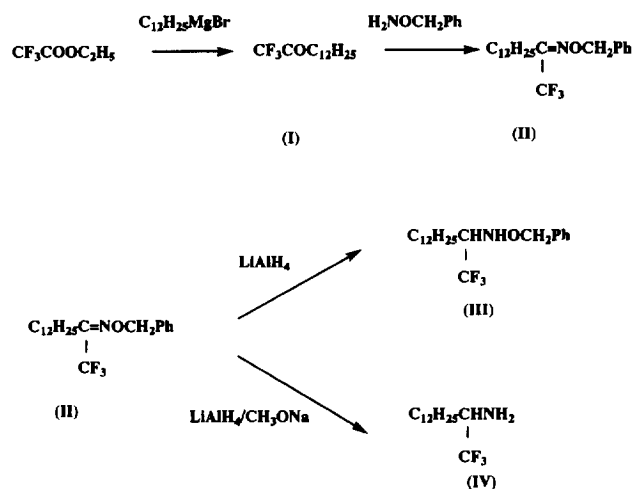
Keywords: Ethyl trifluoroacetate; Grignard reagent; Trifluoromethylketone; Benzyloxime; Reduction with LiAlH₄; Sodium methoxide; α -Trifluoromethylamine

1. Introduction

The utility of compounds containing trifluoromethyl groups as bioactive materials and synthetic intermediates for functionalized materials has received extensive attention in recent years [1,2]. We have reported syntheses of optically active γ -lactones as intermediates for liquid crystals [3] and trifluoromethylation of terpenoid carbonyl compounds [4]. However, a general preparative method for amines containing α -trifluoromethyl groups has not been described. Only the synthesis of 1,3-diamino alcohols from β -hydroxy ketones has been reported [5,6]. In this paper, we describe a convenient method for preparing various α -trifluoromethylamines from ethyl trifluoroacetate as the starting material.

2. Results and discussion

α -Trifluoromethylamines were prepared in three steps as shown in Scheme 1. As the first step, various α -trifluoromethyl ketones were prepared by the reaction of ethyl trifluoroacetate with Grignard reagents. Trifluoromethyl phenyl ketone is commercially available, but other trifluoromethyl ketones are not. A few methods for their preparation have been reported. The substitution reaction of the magnesium bromide salt of trifluoroacetic acid with a Grignard reagent was attempted [7]. The lithium salt of trifluoroacetic acid



was treated with a Grignard reagent to produce the desired ketones [8]. Chlorodifluoromethyl ketones can be prepared by the reactions of chlorodifluoroacetic acid with Grignard reagents under careful control of the reaction temperature ($< -5^\circ\text{C}$) [9,10]. We prepared trifluoromethyl ketones by reacting ethyl trifluoroacetate with various Grignard reagents. In this reaction, it is essential to keep the reaction temperature below -40°C and to use diethyl ether as the solvent. For example, the reaction of ethyl trifluoroacetate with dodecylmagnesium bromide in diethyl ether gave trifluoromethyl dodecyl ketone (I) in 54.5% yield. Other results are shown in Table 1.

* Corresponding author.

Table 1
Preparation of trifluoromethyl and difluoromethyl ketones

Esters	RMgX	Trifluoromethyl ketones (CF ₃ COR) ^a		
	R	b.p. (°C/mmHg)	Yield (%)	¹⁹ F NMR δ (ppm)
CF ₃ COOEt	C ₈ H ₁₇	73/18	62.5	–2.0 (s)
CF ₃ COOEt	C ₉ H ₁₉	60–63/2	55.5	–3.9 (s)
CF ₃ COOEt	C ₁₀ H ₂₁	78–79/4–5	52.0	–2.0 (s)
CF ₃ COOEt	C ₁₂ H ₂₅	95–96/2	48.5	–0.83 (s)
CF ₃ COOEt	C ₆ H ₅	60–62/3	23	^b
CHF ₂ COOEt	C ₆ H ₁₃	71–73/35	64	–46.3 (d, <i>J</i> _{FH} = 50.0 Hz)
CHF ₂ COOEt	C ₈ H ₁₇	94–96/22	80	–47.0 (d, <i>J</i> _{FH} = 52.8 Hz)
CHF ₂ COOEt	C ₁₀ H ₂₁	94–97/3	51	–51.6 (d, <i>J</i> _{FH} = 53.4 Hz)
CHF ₂ COOEt	CH ₂ Ph	82–83/10	77	–46.0 (d, <i>J</i> _{FH} = 50.0 Hz)

^a IR spectra of these ketones showed the absorptions of carbonyl group at ca. 1770–1740 cm^{–1}. Satisfactory microanalyses (C, ±0.04%; H, ±0.4%) were obtained for each of the mentioned products. These compounds showed one spot in thin layer chromatography with Kiesel gel (Merck Art 5735) using a mixture of hexane and ethylacetate (10: 1, v/v) as a solvent, and one peak in gas chromatography (column: Silicone DC-200 on Celite 545, 3 m).

^b This compound was identified with an authentic sample.

In the second step, benzyl oximes of type (II) were obtained from the reactions of ketones (I) with benzylhydroxylamine hydrochloride.

The third step was the reduction of the oximes to amines. Since the syntheses of 1,3-diamino alcohols from β-hydroxy ketone benzyloximes have been reported [5,6], we examined analogous reductions of our benzyloximes (II) possessing the trifluoromethyl group. Reduction of compound (II) with lithium aluminium hydride alone gave α-trifluoromethyl *N*-benzyl-oxtridecylamine (III) instead of the desired α-trifluoromethyl tridecylamine (IV). The reduction of compound (II) with lithium aluminium hydride and sodium methoxide afforded α-trifluoromethyltridecylamine (IV) in 60.5% yield. It is suspected that the addition of sodium methoxide accelerates the reduction of lithium aluminium hydride [5]. The structures of compounds (III) and (IV) were determined by spectral methods as shown in Section 3. Reduction of other oximes were carried out in the same way and the results are shown in Table 2.

3. Experimental details

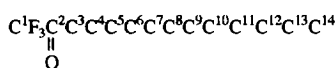
3.1. General procedure

Ethyl trifluoroacetate was commercially available. NMR spectra were recorded at 60 or 200 MHz for ¹H NMR and 56.4 MHz or 470 MHz for ¹⁹F NMR in CDCl₃. ¹⁹F NMR chemical shifts are reported in parts per millions (ppm) relative to trifluoroacetic acid (δ 0.00) as an external standard, low field positive. ¹³C NMR spectra were recorded by using a GSX-500 spectrometer. Yields were those of the products actually isolated.

3.2. Reaction of ethyl trifluoroacetate with dodecylmagnesium bromide

To a solution of ethyl trifluoroacetate (14.2 g, 0.1 mol) in diethyl ether (110 ml), dodecylmagnesium bromide pre-

pared from dodecyl bromide (24.9 g, 0.1 mol) and magnesium (2.4 g, 0.1 mol) in diethyl ether (200 ml) was added slowly at –78 °C under an atmosphere of nitrogen. After 5 h stirring at –40 °C, the mixture was quenched with 1 N HCl and then oily materials were extracted with diethyl ether. The ethereal extract was washed with brine, and then dried over MgSO₄. On removal of the solvent, fractional distillation afforded the following fractions; (1), b.p. ≈ 82 °C/2 mmHg, (1.5 g); (2), b.p. 82–90 °C/2 mmHg, (16.9 g). Fraction (2) was chromatographed on a silica gel column using *n*-hexane and ethyl acetate (9:1 v/v) to give pure trifluoromethyl dodecyl ketone (I) (14.5 g, yield 54.5%). This eluate showed one peak on gas chromatography and one spot on thin layer chromatography. The compound exhibited the following spectral data; IR (cm^{–1}) (liq. film): 1765 (–C=O); ¹⁹F NMR δ: –0.830 (3F, s, CF₃); ¹H NMR δ: 0.883 (3H, t, *J* = 6.78 Hz, CH₃), 1.26 (18H, s, –CH₂– × 9), 1.68 (2H, m, –CH₂–), 2.71 (2H, t, *J* = 7.32 Hz, –COCH₂–); ¹³C NMR δ: 14.16 (14-C), 29.46 (9-C), 36.46 (3-C), 115.69 (q, *J*_{CF} = 292.2 Hz, 1-CF₃), 191.72 (q, *J*_{CF} = 34.7 Hz, 2-CO) ppm.



3.3. Preparation of benzyl oxime (II) of trifluoromethyl dodecyl ketone (I)

A mixture of trifluoromethyl dodecylketone (I, 0.60 g, 0.00225 mol), benzylhydroxylamine hydrochloride (0.432 g, 0.00271 mol), pyridine (2 ml) and ethanol (30 ml) was refluxed for 4 h. The reaction mixture was extracted with diethyl ether. The ether extracts were washed with water, dried over anhydrous sodium sulfate, and concentrated. The residue was chromatographed over a silica gel column using a mixture of *n*-hexane and ethyl acetate as a solvent to give 0.52 g (62.3% yield) of the benzyl oxime (II) of the compound (I) (an oily product). This product

Table 2
Reduction of benzoyloximes of α -trifluoromethylketones

Benzoyloximes R-C=NOCH ₂ Ph CF ₃	Reducing agent	Products ^b		¹⁹ F NMR	¹ H NMR δ (ppm)	¹³ C NMR δ (ppm)
		R-CH-NHCH ₂ Ph CF ₃	R-CH-NH ₂ CF ₃			
		Yield (%) ^a	Yield (%) ^a			
R = C ₆ H ₁₇	LAH	61.3	-	3.4545 (d, $J_{FH} = 6.1$ Hz)	0.813 (3H, t, $J = 6.60$ Hz, CH ₃) 1.191 (12H, s, -CH ₂ × 6) 1.504 (2H, m, -CH ₂ -) 3.10 (1H, m, -CF ₃ CH-) 4.64 (2H, AB quartet, $J_{HH} = 11.48$ Hz, -OCH ₂ Ph) 5.58 (1H, d, $J = 4.12$ Hz, -NH) 7.32 (5H, s, aromatic protons)	14.145, 22.69, 25.58, 25.83, 29.23, 29.34, 31.86, 62.26 (q, $J_{CF} = 20.9$ Hz, CF ₃ CH) 77.69 (OCH ₂ Ph) 126.01 (q, $J_{CF} = 28.2$ Hz, CF ₃) 137.36, 128.64, 128.83, 128.01 (aromatic carbons)
C ₉ H ₁₉	LAH	60.0	-	1.4825 (d, $J_{FH} = 7.63$ Hz)	0.884 (3H, t, $J = 7.07$ Hz, CH ₃) 1.260 (14H, s, -CH ₂ × 7) 1.530 (2H, m, -CH ₂ -) 3.27 (1H, m, CF ₃ CH-) 4.71 (2H, AB quartet, $J_{HH} = 11.71$ Hz, -OCH ₂ Ph) 5.61 (1H, d, $J = 4.11$ Hz, -NH) 7.34 (5H, s, aromatic protons)	14.09, 22.66, 25.56, 25.79, 29.26, 29.28, 29.32, 31.86, 62.221 (q, $J_{CF} = 33.0$ Hz, CF ₃ CH-) 76.91 (OCH ₂ Ph) 126.2 (q, $J_{CF} = 28.1$ Hz, CF ₃) 127.97, 128.39, 128.58, 137.31 (aromatic carbons)
C ₁₀ H ₂₁	LAH	65.1	-	0.671 (d, $J_{FH} = 7.63$ Hz)	0.886 (3H, t, $J = 7.09$ Hz, CH ₃) 1.262 (16H, s, CH ₂ × 8) 1.531 (2H, m, -CH ₂ -) 3.28 (1H, m, CF ₃ CH) 4.72 (2H, AB quartet, $J_{HH} = 11.68$ Hz, -OCH ₂ Ph) 5.60 (1H, d, $J = 4.31$ Hz, -NH) 7.33 (5H, s, aromatic protons)	14.08, 22.93, 25.65, 25.81, 29.46, 29.38, 29.32, 31.53, 31.96, 62.24 (q, $J_{CF} = 28.0$ Hz, CF ₃ CH) 77.03 (OCH ₂ Ph) 126.03 (q, $J_{CF} = 28.2$ Hz, CF ₃) 127.81, 128.63, 128.71, 137.21 (aromatic carbons)
C ₁₂ H ₂₅	LAH	69.3	-	2.978 (d, $J_{FH} = 6.10$ Hz)	0.88 (3H, t, $J = 6.77$ Hz, CH ₃) 1.26 (20H, s, -CH ₂ × 10) 1.55 (2H, m, -CH ₂ -) 3.28 (1H, m, CF ₃ CH-) 4.71 (2H, AB quartet, $J_{HH} = 11.54$ Hz, -OCH ₂ Ph) 5.60 (1H, d, $J = 4.22$ Hz, -NH) 7.33 (5H, s, aromatic protons)	14.557, 23.16, 26.08, 26.12, 26.15, 26.19, 26.278, 29.81, 30.08, 30.12, 32.398 (3-C) 62.75 (q, $J_{CF} = 27.3$ Hz, 2-C), 77.48 (15-C), 126.4 (q, $J_{CF} = 26.9$ Hz, 1-CF ₃), 128.85 (18, 18'-C), 129.04 (17, 17'-C), 137.86 (16-C)
C ₆ H ₅	LAH	50.2	-	3.653 (d, $J_{FH} = 6.1$ Hz)	4.50 (1H, m, CF ₃ -CH) 4.69 (2H, AB quartet, $J_{HH} = 11.3$ Hz, -OCH ₂ Ph) 5.79 (1H, d, $J = 4.63$ Hz, -NH) 7.36 (5H, s, aromatic protons) 7.39 (5H, s, aromatic protons)	66.43 (NOC-Ph) 66.71 (q, $J = 29.3$ Hz, CF ₃ C) 126.4 (q, $J_{CF} = 28.1$ Hz, 1-CF ₃) 128.725, 128.695, 129.397, 128.404, 132.141, 128.273, 136.928, 128.069 (aromatic carbons)
C ₈ H ₁₇	LAH, CH ₃ ONa	-	80	-1.664 (d, $J_{FH} = 7.62$ Hz)	0.883 (3H, t, $J = 6.60$ Hz, CH ₃) 1.29 (12H, broad s, -CH ₂ × 6) 1.40 (2H, m, -CHCH ₂) 1.70 (2H, m, -NH ₂) 3.10 (1H, broad m, CF ₃ CH)	14.12, 22.71, 25.70, 29.30, 29.42, 29.84, 31.89, 53.76 (q, $J_{CF} = 28.5$ Hz, CF ₃ CH) 126.89 (q, $J_{CF} = 28.1$ Hz, CF ₃)
C ₉ H ₁₉	LAH, CH ₃ ONa	-	76	-3.641 (d, $J_{FH} = 7.63$ Hz)	0.882 (2H, t, $J = 6.83$ Hz, CH ₃) 1.266 (14H, broad s, -CH ₂ × 7) 1.57 (2H, m, -CHCH ₂) 1.70 (2H, m, -NH ₂) 3.11 (1H, broad m, CF ₃ CH)	14.03, 22.62, 25.59, 29.26, 29.29, 29.36, 29.50, 29.74, 31.84, 53.67 (q, $J_{CF} = 28.0$ Hz, CF ₃ CH) 128.79 (q, $J_{CF} = 27.7$ Hz, CF ₃)

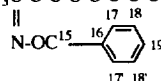
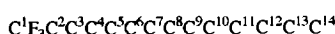
(continued)

$C_{10}H_{21}$	LAH, CH_3ONa	71	-3.641 (d, $J_{FH} = 7.63$ Hz)	0.882 (3H, t, $J = 7.07$ Hz, CH_3), 1.266 (16H, s, $-CH_2 \times 8$) 1.545 (2H, m, $-CH_2-$) 1.702 (2H, m, NH_2) 3.10 (1H, m, CF_3CH)	14.071, 22.66, 25.28, 28.39, 29.028, 29.324, 29.390, 29.530, 29.554, 31.874 53.70 (q, $J_{CF} = 28.0$ Hz, CF_3CH) 126.83 (q, $J_{CF} = 112.0$ Hz, CF_3CH)
$C_{12}H_{25}$	LAH, CH_3ONa	60.5	-3.603 (d, $J_{FH} = 7.63$ Hz)	0.884 (3H, t, $J = 6.60$ Hz, CH_3), 1.267 (20H, s, $-CH_2 \times 10$) 1.58 (2H, m, $-CH_2-$) 1.70 (2H, m, $-NH_2$) 3.10 (1H, m, CF_3CH)	14.05 (14-C), 22.87, 25.63, 29.70, 29.72, 29.73, 29.75, 29.77, 29.78, 29.79, 29.80, 31.90 (3-C), 53.69 (q, $J_{CF} = 28.35$ Hz, 2-C), 126.81 (q, $J_{CF} = 280.15$ Hz, 1-C)
C_6H_5	LAH, CH_3ONa	35.2	-1.383 (d, $J_{FH} = 7.63$ Hz)	4.40 (1H, m, CF_3CH) 2.50 (2H, m, NH_2) 7.35 (2H, s, aromatic protons) 7.38 (3H, s, aromatic protons)	66.51 (q, $J = 28.3$ Hz, CF_3C) 125.3 (q, $J = 281.5$ Hz, CF_3) 128.41, 132.13, 128.28, 136.93
<i>p</i> - $CH_3C_6H_5$	LAH, CH_3ONa	52.9	0.4875 (d, $J_{FH} = 7.63$ Hz)	2.35 (3H, s, CH_3) 2.52 (2H, m, $-NH_2$) 4.40 (1H, m, CF_3CH) 7.40 (4H, AB quartet, $J_{HH} = 9.35$ Hz, aromatic protons)	20.9, 66.52 (q, $J = 282.2$ Hz, CF_3C) 125.2 (q, $J = 282.5$ Hz, CF_3) 128.28, 128.42, 132.23, 136.83

^a Yields were isolated yields.

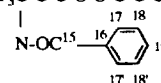
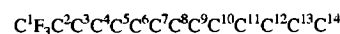
^b These compounds showed one spot in thin layer chromatography with Kieselgel 60 (Merck Art. 5735) using a mixture of hexane and dichloromethane (10:1, v/v) or a mixture of hexane and ethylacetate (1:1, v/v) as a solvent. Satisfactory microanalyses ($N, \pm 0.15\%$) were obtained for each of the mentioned products.

showed one spot ($R_f = 0.7$) in thin layer chromatography on Kieselgel 60 (Merck Art 5735) using a mixture of hexane and dichloromethane (95:5, v/v) as a solvent. This compound exhibited the following spectral data: IR (cm^{-1}) (liq. film): 1636 ($-C=N-O$), 752.1 and 697.1 (mono substituted); ^{19}F NMR δ : +8.234 (3F, s, CF_3); 1H NMR δ : 0.884 (3H, t, $J = 6.76$ Hz, CH_3-), 1.248 (18H, s, $-CH_2 \times 9$), 1.55 (2H, m, $-CH_2-$), 2.42 (2H, m, $-N=C-CH_2-$), 5.197 (2H, s, $-CH_2-ph$), 7.343 (5H, s, aromatic protons); ^{13}C NMR δ : 14.12 (14-C), 29.688 (9-C), 31.979 (3-C), 77.561 (15-C), 121.027 (q, $J_{CF} = 274.7$ Hz, 1- CF_3), 128.265 (19-C), 128.333 (18 and 18'-C), 128.505 (17 and 17'-C), 136.631 (16-C), 150.011 (q, $J_{CF} = 31.5$ Hz, 2- $C=N$) ppm. Other oximes were prepared in the similar way.



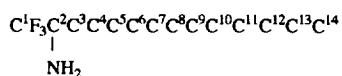
3.4. Reduction of the benzyl oxime (II) of trifluoromethyl dodecyl ketone (I) with lithium aluminium hydride

A solution of the oxime (II) (1.08 g, 0.0029 mol) in dry diethyl ether (20 ml) was added dropwise to a suspension of $LiAlH_4$ (0.34 g) in dry diethyl ether (30 ml) under nitrogen at $0^\circ C$. The mixture was stirred for 4 h at $0^\circ C$ and then hydrolyzed with a saturated solution of aqueous NH_4Cl . It was extracted with ether, the ether extracts washed with water, dried over anhydrous sodium sulfate and concentrated. The residue was chromatographed over a silica gel column using a mixture of hexane and dichloromethane as a solvent to give 0.75 g (69.3% yield) of α -trifluoromethyl *N*-benzyl-oxyltridecylamine (III) (an oily product). This product showed one spot ($R_f = 0.55$) in thin layer chromatography on Kiesel gel 60 (Merck Art 5735) using a mixture of hexane and dichloromethane (10:1 v/v) as a solvent. The compound exhibited the following spectral data: IR (cm^{-1}) (liq. film): 3,276 ($-NH$), 747 and 698 (mono substituted); ^{19}F NMR δ : +2.978 (3F, d, $J = 6.10$ Hz, CF_3); 1H NMR δ : 0.882 (3H, t, $J = 6.77$ Hz, CH_3-), 1.261 (20H, s, $-CH_2 \times 10$), 1.55 (2H, m, $-CH_2-$), 3.28 (1H, m, CF_3-CH-), 4.707 (2H, AB quartet, $J_{HH} = 11.54$ Hz, $-O-CH_2-Ph$), 5.6025 (1H, d, $J = 4.22$ Hz, $-NH$), 7.334 (5H, s, aromatic protons); ^{13}C NMR δ : 14.557 (14-C), 26.278 (9-C), 32.3985 (3-C), 62.7525 (q, $J_{CF} = 27.3$ Hz, 2-C), 77.4810 (15-C), 126.4 (q, $J_{CF} = 269.2$ Hz, 1- CF_3), 128.433 (19-C), 128.858 (18, 18'-C), 129.042 (17, 17'-C), 137.861 (16-C) ppm. Other oximes were reduced in a similar way.



3.5. Reduction of the oxime (II) with lithium aluminium hydride in the presence of sodium methoxide

A solution of the oxime (II) (0.933 g, 0.0025 mol) in THF (15 ml) was added drop by drop to a suspension of LiAlH_4 (1.42 g, 0.0375 mol) and CH_3ONa (1.62 g, 0.030 mol) in dry THF (135 ml) under nitrogen at -78°C . The mixture was stirred for 6 h at -78°C and for 12 h at 0°C . The mixture was then hydrolyzed with saturated aqueous Na_2SO_4 solution, extracted with diethyl ether and worked up in the same manner as before. The residue was chromatographed over silica gel column using a mixture of hexane and methanol as a solvent to give 0.404 g (60.5% yield) of α -trifluoromethyltridecylamine (IV). This product showed one spot ($R_f=0.2$) in thin layer chromatography on Kiesel gel 60 (Merck Art 5235) using a mixture of hexane and ethylacetate (50:50 v/v) as a solvent, and exhibited the following spectral data: IR (cm^{-1}) (liq. film): 3400, 3320 (NH_2 -); ^{19}F NMR δ : -3.603 (3F, d, $J_{\text{FH}}=7.63$ Hz, CF_3); ^1H NMR δ : 0.884 (3H, t, $J=6.60$ Hz, CH_3), 1.267 (20H, s, $-\text{CH}_2-\times 10$), 1.58 (2H, m, $-\text{CH}_2-$), 1.70 (2H, m, $-\text{NH}_2$), 3.10 (1H, m, CF_3-CH); ^{13}C NMR δ : 14.053 (14-C), 31.901 (3-C), 53.6885 (q, $J_{\text{CF}}=28.35$ Hz, 2-C), 126.8145 (q, $J_{\text{CF}}=280.15$ Hz, 1-C) ppm. Other oximes were reduced in a similar manner.



Acknowledgements

The authors are indebted to Mr. Yuhzoh Komatsu of Tokyo Institute of Technology for measurements of many spectra.

References

- [1] R. Filler and Y. Kobayashi (Eds.), *Biomedical Aspects of Fluorine Chemistry*, Kōdansha and Elsevier Biomedical, Amsterdam, 1982.
- [2] J.T. Welch and S. Eswarakrishnan (Eds.), *Fluorine in Bioorganic Chemistry*, Wiley, New York, 1991.
- [3] S. Watanabe, Y. Sakai, M. Takeda, T. Kitazume and T. Yamazaki, *J. Fluorine Chem.* 67 (1994) 149.
- [4] S. Watanabe, T. Fujita, M. Sakamoto, Y. Mino and T. Kitazume, *J. Fluorine Chem.* 73 (1995) 21.
- [5] K. Narasaka, Y. Ukaji and S. Yamazaki, *Bull. Chem. Soc. Jpn.* 59 (1986) 525.
- [6] J.T. Lin, T. Yamazaki and T. Kitazume, *J. Org. Chem.* 52 (1987) 3211.
- [7] T. Ishizuka, H. Miura and H. Nohira, *Nippon Kagaku Kaishi*, (1990) 1171 (in Japanese).
- [8] H. Hamada, M. Shiromoto, M. Funahashi, T. Itoh and K. Nakamura, *J. Org. Chem.* 61 (1996) 2332.
- [9] I.L. Knunyants, G.G. Yakobson, *Syntheses of Fluoroorganic Compounds*, Springer, New York, 1985, p. 37, and references cited therein.
- [10] Z.-M. Qie, D.J. Burton, *J. Org. Chem.* 60 (1995) 5570.