Notes

(Trifluoroacetyl)benzotriazole: A Convenient Trifluoroacetylating Reagent

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Received August 7, 1996

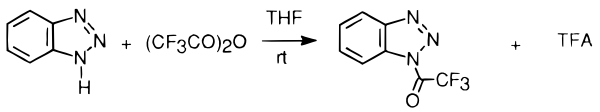
Trifluoroacetylation is important for the protection or activation of functional groups in organic and bioorganic synthesis.[1] Conventional reagents for trifluoroacetylation all have drawbacks or limitations: (i) trifluoroacetic anhydride[2] has a low boiling point (39.5 °C), is moisture sensitive, and is also too reactive for some applications; (ii) *S*-ethyl trifluorothioacetate forms the bad smelling by product ethanethiol;[3] (iii) ethyl trifluoroacetate reacts rather slowly;[4,5] (iv) *N*-(trifluoroacetyl)imidazole is highly moisture sensitive;[6] (v) 2-[(trifluoroacetyl)oxy]pyridine (TFAP)[7] is of somewhat mild reactivity; (vi) trifluoroacetyl triflate is extremely reactive;[8,9] (vii) *N*-(trifluoroacetoxy)succinimide[10] is unstable and has to be stored as a benzene solution in a freezer.

We recently reported formylbenzotriazole as a good formylating reagent,[11] and 1-(arylacetyl)benzotriazoles were used as precursors of arylketenes[12] for preparation of aryl benzyl sulfoxides[13] and symmetrical ketones.[12] We now present (trifluoroacetyl)benzotriazole (**1**) as a novel and facile trifluoroacetylating reagent for amines and alcohols.

(Trifluoroacetyl)benzotriazole (**1**) is easily synthesized in almost quantitative yield by reaction of trifluoroacetic anhydride with benzotriazole in dry THF. After the removal of the solvent, the solid product can be stored in the covered bottle for several weeks without decomposition and used directly for trifluoroacetylation. Compound **1** has not previously appeared in the literature despite its simple structure (Scheme 1).

Trifluoroacetylation of amines with trifluoroacetylbenzotriazole (**1**) was carried out by stirring a THF solution of trifluoroacetylbenzotriazole and amine (in a molar ratio of 1.1:1). The results are listed in Scheme 2 and Table 1. Primary and secondary, alkyl, and aryl amines all readily gave excellent yields of trifluoroacetamides **2** which were identified by NMR and comparison with

Scheme 1



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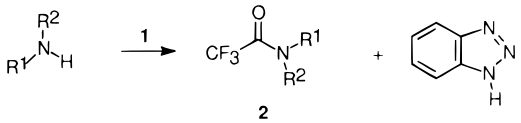
Scheme 2



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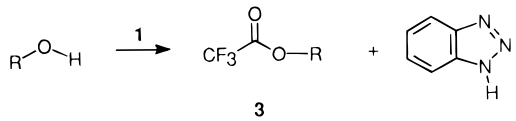
Scheme 3



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literature data. The byproduct benzotriazole can be removed easily by washing with sodium hydroxide aqueous solution. Amines of low nucleophilicity such as *p*-nitroaniline and sterically hindered amines such as *tert*-butylamine, all gave satisfactory yields when trifluoroacetylated with our reagent. The optically active (*R*)-(+)-2-phenylethylamine when reacted with (trifluoroacetyl)benzotriazole under the above conditions generated the corresponding optically pure trifluoroacetamide without racemization. 2-Amino-2-phenylethanol, containing both an amino and a hydroxy group in the molecule, was selectively trifluoroacetylated at the amino group. 4-(Phenylamino)aniline, containing a primary amino and a secondary amino group, with 1 equivalent of our reagent, underwent trifluoroacetylation selectively at the primary amino group.

Trifluoroacetylation of alcohols and phenols was performed by refluxing a THF solution of (trifluoroacetyl)-benzotriazole and an alcohol or phenol. Attempts to obtained the pure trifluoroacetate ester **3** by washing with aqueous sodium hydroxide solution to remove the byproduct benzotriazole failed due to rapid hydrolysis of the formed trifluoroacetate ester in aqueous sodium hydroxide solution. Thus distillation was applied to obtain the pure trifluoroacetate ester from the reaction mixture. Various alcohols and phenols were reacted with this reagent and all of them gave satisfactory yields. *p*-Nitrophenol, which failed to undergo trifluoroacetylation by TFAP,[7] was successfully trifluoroacetylated with our reagent (Scheme 3 and Table 2).

Comparison of (trifluoroacetyl)benzotriazole with literature trifluoroacetylating reagents demonstrates that our new reagent has several advantages. (1) Easy preparation: as already described, the preparation procedure is so simple that it took only 1 h to obtain this reagent. (2) Stable and easy to handle: the solid reagent can be stored in a covered bottle at room temperature for several weeks without decomposition and weighed out in air. (3) Forms a stable by product: trifluoroacetylations with our reagent gave the expected product along with benzotriazole, a weak acid ($pK_a = 8.3$) that will not induce side reactions. By contrast, trifluoroacetic anhy-

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S0022-3263(96)01537-X CCC: $14.00

Table 1. Preparation of Trifluoroacetamides 2

compd	R^1	R^2	reaction time (h)	solvent	temp (°C)	isolated yield (%)
2a	H	2-phenylethyl	4	THF	rt	100
2b	H	4-methylbenzyl	4	THF	rt	92
2c	H	2-methylbutyl	3	THF	rt	97
2d	H	*tert*-butyl	3	THF	rt	95
2e	H	1-(hydroxymethyl)benzyl	5	THF	rt	90
2f	H	2-naphthyl	3	THF	rt	93
2g	H	p-nitrophenyl	3	THF	rt	90
2h	H	2-isopropylphenyl	4	THF	rt	92
2i	H	4-(phenylamino)phenyl	5	THF	rt	92
2j		$-(CH_2)_5-$[a]	6	Et_2O	39	89
2k	Et	phenyl	6	Et_2O	39	85

[a] Piperidyl.

Table 2. Preparation of Trifluoroacetate Esters 3

compd	R	time (h)	isolated [a] yield (%)
3a	benzyl	3	95
3b	4-methylbenzyl	3	92
3c	cyclohexyl	3	80 (100)[b]
3d	pentyl	4	54 (100)[b]
3e	2,2-dimethylpropyl	3	79 (100)[b]
3f	4-chlorophenyl	6	94
3g	4-nitrophenyl	16	60[c]

[a] All reactions carried out in boiling THF. [b] GC yield. [c] NMR yield.

dride forms trifluoroacetic acid as a byproduct which can initiate several side reactions.[3] (4) Good selectivity: when a compound contains both amino and hydroxy groups, the reagent selectively trifluoroacetylated at the amino group. In a molecule with both primary amino and secondary amino groups, trifluoroacetylation only occurred at the primary amino group.

In a conclusion, (trifluoroacetyl)benzotriazole is a convenient trifluoroacetylation reagent due to its facile preparation, easy handling, and stability. We believe this novel trifluoroacetylation reagent will be widely applied in protection and activation of functional groups in organic and bioorganic synthesis.

Experimental Section

Melting points were measured on a Bristoline hotstage microscope and are uncorrected. ^{1}H NMR spectra were recorded at 300 MHz with tetramethylsilane (TMS) as an internal reference. ^{13}C NMR spectra were recorded at 75 MHz with the solvent peak ($CDCl_3$, $\delta = 77.0$ or DMSO, $\delta = 39.5$) as a reference. ^{19}F NMR spectra were recorded at 282 MHz with α,α,α-trifluorotoluene ($\delta = -63.00$) as an internal reference. THF was distilled from sodium benzophenone prior to use.

Preparation of (Trifluoroacetyl)benzotriazole. To a solution of benzotriazole (11.9 g, 0.1 mol) in THF (dried, 100 mL) was added slowly trifluoroacetic anhydride (30.5 g, 0.15 mol) at room temperature under nitrogen. The formed mixture was stirred at rt for 0.5 h. After removal of the solvent, excess of anhydride, and byproduct trifluoroacetic acid, a white solid was obtained in quantitative yield.

1-(Trifluoroacetyl)benzotriazole (1): white solid, mp 89−91 °C; ^{1}H NMR ($CDCl_3$) δ 6.99−7.07 (m, 1 H), 7.09−7.17 (m, 1 H), 7.46−7.49 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 113.6, 115.0 (q, $^1J_{C-F}$ = 285.3 Hz, CF_3), 120.5, 127.5, 130.6, 131.5, 145.2, 154.2 (q, $^2J_{C-F}$ = 43.0 Hz, C=O). ^{19}F NMR ($CDCl_3$) δ 70.07. Anal. Calcd for $C_8H_4F_3N_3O$: C, 44.66; H, 1.87; N, 19.53. Found: C, 43.75; H, 2.37; N, 19.12.

General Procedure for Trifluoroacetylation of Amines. A solution of amine (5 mmol) and (trifluoroacetyl)benzotriazole (5.5 mmol, 1.18 g) in THF (dried, 20 mL) was stirred at room temperature for several hours (**2j** and **2k** were refluxed in dried ethyl ether). Then the mixture was washed with aqueous 2 N sodium hydroxide solution (2 × 15 mL) and extracted with ethyl acetate (2 × 20 mL). The combined organic layers were dried over sodium sulfate. After the removal of organic solvent, the crude product was obtained. Recrystallization or column chromatography of the crude compound gave pure product.

***N*-[(*R*)-(+)-α-Phenylethyl]-2,2,2-trifluoroacetamide (2a)**: beige solid; mp 95−97 °C [lit.[14] mp 97−98 °C]; ^{1}H NMR ($CDCl_3$) δ 1.55 (d, 3 H, J = 7.0 Hz), 5.06−5.16 (m, 1 H), 6.88 (br s, 1 H), 7.29−7.39 (m, 5 H); ^{1}H NMR (C_6D_6 + Eu) δ 1.02 (d, 3 H, J = 6.9 Hz), 5.20−5.40 (m, 1 H), 6.48 (br s, 1 H), 6.96−7.10 (m, 5 H); ^{13}C NMR ($CDCl_3$) δ 20.9, 49.8, 115.8 (q, $^1J_{C-F}$ = 286.2 Hz, CF_3), 126.1, 128.0, 128.9, 141.0, 156.3 (q, $^2J_{C-F}$ = 37.1 Hz, C=O).

***N*-[(*RS*)-α-Phenylethyl]-2,2,2-trifluoroacetamide (2a′)**: ^{1}H NMR (C_6D_6 + Eu) δ 1.07 (d, 3 H, J = 6.9 Hz), 1.08 (d, 3 H, J = 6.9 Hz), 5.20−5.40 (m, 1 H), 6.48 (br s, 1 H), 6.96−7.10 (m, 5 H).

***N*-(4-Methylbenzyl)-2,2,2-trifluoroacetamide (2b)**: white solid; mp 100−102 °C; ^{1}H NMR ($CDCl_3$) δ 2.33 (s, 3 H), 4.40 (s, 1 H), 4.42 (s, 1 H), 7.02 (br s, 1 H), 7.10−7.19 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 21.0, 43.6, 115.9 (q, $^1J_{C-F}$ = 285.8 Hz, CF_3), 127.9, 129.6, 132.9, 138.0, 157.2 (q, $^2J_{C-F}$ = 36.8 Hz, C=O). Anal. Calcd for $C_{10}H_{10}F_3NO$: C, 55.30; H, 4.64; N, 6.45. Found: C, 55.35; H, 4.59; N, 6.41.

***N*-(2-Methylbutyl)-2,2,2-trifluoroacetamide (2c)**: yellow oil [lit.[15] bp 115 °C/11 mmHg]; ^{1}H NMR ($CDCl_3$) δ 0.93 (d, 3 H, J = 6.9 Hz), 0.94 (t, 3 H, J = 7.2 Hz), 1.14−1.29 (m, 1 H), 1.38−1.49 (m, 1 H), 1.65−1.73 (m, 1 H), 3.13−3.20 (m, 1 H), 3.26−3.32 (m, 1 H), 8.02 (br s, 1 H); ^{13}C NMR ($CDCl_3$) δ 10.6, 16.4, 26.6, 34.3, 45.5, 116.0 (q, $^1J_{C-F}$ = 285.5 Hz, CF_3), 157.8 (q, $^2J_{C-F}$ = 36.5 Hz, C=O).

***N*-*tert*-Butyl-2,2,2-trifluoroacetamide (2d)**: yellow solid; mp 44 °C [lit.[16] mp 44−45 °C]; ^{1}H NMR ($CDCl_3$) δ 1.33 (s, 9 H); ^{13}C NMR ($CDCl_3$) δ 27.8, 52.7, 115.6 (q, $^1J_{C-F}$ = 287.3 Hz, CF_3), 156.2 (q, $^2J_{C-F}$ = 35.6 Hz, C=O).

***N*-(2-Phenylethan-1-ol)-2,2,2-trifluoroacetamide (2e)**: white solid; mp 128−130 °C; ^{1}H NMR (DMSO-d_6) δ 4.10−4.15 (m, 2 H), 5.36−5.38 (m, 1 H), 5.53 (br s, 1 H), 7.71−7.78 (m, 5 H), 10.23 (br s, 1 H); ^{13}C NMR (DMSO-d_6) δ 56.5, 63.8, 116.1 (q, $^1J_{C-F}$ = 286.2 Hz, CF_3), 126.9, 127.5, 128.4, 139.2, 156.3 (q, $^2J_{C-F}$ = 35.6 Hz, C=O). Anal. Calcd for $C_{10}H_{10}F_3NO_2$: C, 51.50; H, 4.32; N, 6.01. Found: C, 51.56; H, 4.28; N, 6.72.

***N*-(2-Naphthyl)-2,2,2-trifluoroacetamide (2f)**: mauve solid; mp 146 °C [lit.[17] mp 146−148 °C]; ^{1}H NMR ($CDCl_3$) δ 7.43−7.52 (m, 3 H), 7.74−7.85 (m, 3 H), 8.18 (br s, 1 H), 8.16−8.28 (m, 1 H); ^{13}C NMR ($CDCl_3$) δ 115.8 (q, $^1J_{C-F}$ = 287.0 Hz, CF_3), 118.3, 119.5, 126.1, 127.0, 127.7, 127.9, 129.3, 131.5, 132.4, 133.4, 155.0 (q, $^2J_{C-F}$ = 36.3 Hz, C=O).

***N*-(4-Nitrophenyl)-2,2,2-trifluoroacetamide (2g)**: yellow solid; mp 152 °C [lit.[7] mp 151−152 °C]; ^{1}H NMR (DMSO-d_6) δ 7.91−7.95 (m, 2 H), 8.23−8.26 (m, 2 H), 11.74 (br s, 1 H); ^{13}C NMR (DMSO-d_6) δ 115.5 (q, $^1J_{C-F}$ = 286.7 Hz, CF_3), 121.0, 124.7, 142.4, 144.0, 155.0 (q, $^2J_{C-F}$ = 37.6 Hz, C=O).

***N*-(2-Isopropylphenyl)-2,2,2-trifluoroacetamide (2h)**: brown solid; mp 84−86 °C; ^{1}H NMR ($CDCl_3$) δ 1.17 (d, 6 H, J = 6.9 Hz), 2.95 (heptet, 1 H, J = 6.9 Hz), 7.12−7.41 (m, 4 H), 8.23 (br s, 1 H); ^{13}C NMR ($CDCl_3$) δ 22.8, 28.1, 116.0 (q, $^1J_{C-F}$ = 280.0 Hz, CF_3), 125.2, 126.1, 126.5, 127.9, 131.1, 142.1, 155.8 (q, $^2J_{C-F}$

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= 28.5 Hz, C=O). Anal. Calcd for $C_{11}H_{12}F_3NO$: C, 57.14; H, 5.23; N, 6.06. Found: C, 57.37; H, 5.24; N, 5.92.

***N*-[4-(Phenylamino)phenyl]-2,2,2-trifluoroacetamide (2i)**: black solid; mp 140−142 °C; ^{1}H NMR (DMSO-d_6) δ 6.82−6.86 (m, 1 H), 7.12−7.24 (m, 6 H), 7.57−7.59 (m, 2 H), 8.25 (br s, 1 H), 11.10 (br s, 1 H); ^{13}C NMR (DMSO-d_6) δ 116.0 (q, $^1J_{C-F}$ = 286.6 Hz, CF_3), 116.8, 119.8, 122.4, 128.4, 129.2, 141.3, 143.3, 154.1 (q, $^2J_{C-F}$ = 36.2 Hz, C=O). Anal. Calcd for $C_{14}H_{11}F_3N_2O$: C, 60.00; H, 3.96; N, 10.00. Found: C, 60.36; H, 3.93; N, 10.17.

***N*-(Trifluoroacetyl)piperidine (2j)**: oil [lit.[18] bp 77 °C/15 mmHg]; ^{1}H NMR ($CDCl_3$) δ 1.58−1.76 (m, 6 H), 3.55 (t, 2 H, J = 5.4 Hz), 3.62 (t, 2 H, J = 5.2 Hz); ^{13}C NMR ($CDCl_3$) δ 23.7, 25.0, 26.0, 44.1, 46.4, 116.4 (q, $^1J_{C-F}$ = 286.5 Hz, CF_3), 154.8 (q, $^2J_{C-F}$ = 35.0 Hz, C=O).

***N*-Phenyl-*N*-ethyl-2,2,2-trifluoroacetamide (2k)**: oil [lit.[15] bp 120 °C/11 mmHg]; ^{1}H NMR ($CDCl_3$) δ 1.17 (t, 3 H, J = 7.1 Hz), 3.79 (q, 2 H, J = 7.1 Hz), 7.21−7.24 (m, 2 H), 7.39−7.42 (m, 3 H); ^{13}C NMR ($CDCl_3$) δ 11.7, 46.6, 116.3 (q, $^1J_{C-F}$ = 286.7 Hz, CF_3), 128.1, 128.7, 129.1, 138.7, 156.0 (q, $^2J_{C-F}$ = 35.2 Hz, C=O).

General Procedure for Trifluoroacetylation of Alcohol. A solution of alcohol or phenol (5 mmol) and (trifluoroacetyl)-benzotriazole (5.5 mmol, 1.18 g) in THF (dried, 20 mL) was refluxed for several hours. Then the mixture was directly distilled at normal pressure to remove the solvent, or by vacuum to give pure product.

Benzyl trifluoroacetate (3a): yellow oil; bp 97−99 °C/12 mmHg [lit.[7] 175 °C]; ^{1}H NMR ($CDCl_3$) δ 5.30 (s, 2 H), 7.27−7.41 (m, 5 H); ^{13}C NMR ($CDCl_3$) δ 69.6, 114.6 (q, $^1J_{C-F}$ = 284.4 Hz, CF_3), 128.6, 128.7, 129.2, 133.3, 157.5 (q, $^2J_{C-F}$ = 42.2 Hz, C=O).

4-Methylbenzyl trifluoroacetate (3b): yellow liquid; bp 112−114 °C/14 mmHg [lit.[7] bp 184−185 °C]; ^{1}H NMR ($CDCl_3$) δ 2.35 (s, 3 H), 5.29 (s, 2 H), 7.17−7.28 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 21.1, 69.6, 114.6 (q, $^1J_{C-F}$ = 284.3 Hz, CF_3), 128.9, 129.5, 130.4, 157.4 (q, $^2J_{C-F}$ = 41.9 Hz, C=O).

Cyclohexyl trifluoroacetate (3c): colorless liquid; bp 142−144 °C/760 mmHg [lit.[7] bp 145 °C]; ^{1}H NMR ($CDCl_3$) δ 1.18−1.82 (m, 10 H), 4.85−4.94 (m, 1 H); ^{13}C NMR ($CDCl_3$) δ 23.1, 24.9, 30.8, 77.5, 114.7 (q, $^1J_{C-F}$ = 283.9 Hz, CF_3), 156.9 (q, $^2J_{C-F}$ = 41.2 Hz, C=O).

Pentyl trifluoroacetate (3d): colorless liquid; bp 60 °C/15 mmHg [lit.[7] bp 140−141 °C]; ^{1}H NMR ($CDCl_3$) δ 0.93 (t, 3 H, J = 6.4 Hz), 1.36−1.38 (m, 4 H), 1.74−1.78 (m, 2 H), 4.35 (t, 2 H, J = 6.6 Hz); ^{13}C NMR ($CDCl_3$) δ 13.6, 22.1, 27.6, 27.8, 68.2, 114.7 (q, $^1J_{C-F}$ = 283.9 Hz, CF_3), 157.6 (q, $^2J_{C-F}$ = 41.6 Hz, C=O).

2,2-Dimethylpropyl trifluoroacetate (3e): colorless liquid; bp 96 °C; ^{1}H NMR ($CDCl_3$) δ 1.00 (s, 9 H), 4.05 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 25.9, 31.15, 76.9, 114.7 (q, $^1J_{C-F}$ = 284.0 Hz, CF_3), 157.5 (q, $^2J_{C-F}$ = 41.6 Hz, C=O); ^{19}F NMR ($CDCl_3$) δ −75.38 [lit.[18] ^{19}F NMR δ −7.85 by using α,α,α-trifluoroacetophenone as internal reference peak].

4-Chlorophenyl trifluoroacetate (3f): colorless liquid; bp 45 °C/14 mmHg [lit.[7] 84−85 °C /40 mmHg]; ^{1}H NMR ($CDCl_3$) δ 7.13−7.16 (m, 2 H), 7.37−7.40 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 114.5 (q, $^1J_{C-F}$ = 284.0 Hz, CF_3), 121.9, 130.0, 133.0, 147.8, 155.7 (q, $^2J_{C-F}$ = 43.8 Hz, C=O).

4-Nitrophenyl trifluoroacetate (3g): unstable for purification; ^{1}H NMR ($CDCl_3$) δ 7.44−7.48 (m, 2 H), 8.30−8.38 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 114.2 (q, $^1J_{C-F}$ = 284.0 Hz, CF_3), 121.6, 125.4, 146.1, 153.2, 154.8 (q, $^2J_{C-F}$ = 44.3 Hz, C=O); ^{19}F NMR ($CDCl_3$) δ −74.96 [lit.[19] ^{19}F NMR δ −7.33 by using α,α,α-trifluoroacetophenone as internal reference peak].

Acknowledgment. We thank the University Paul-Sabatier of Toulouse (France) for the financial support of Dr. Delphine Semenzin.

Supporting Information Available: The NMR spectra of compound **1** (5 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

JO961537R

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