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Synthesis of 2,2,2-Trifluoroethylated Onium Salts of Nitrogen, Sulfur, and Phosphorus with (2,2,2-Trifluoroethyl)phenyliodonium Triflate

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Synopsis. Various 2,2,2-trifluoroethyl onium triflates were synthesized by treating tertiary amines, pyridines, quinoline, a triazole, sulfides, and a phosphine with (2,2,2-trifluoroethyl)phenyliodonium triflate. Sulfoxides and N-oxide were converted to the corresponding trifluoroethylated salts. As an application, CsF-treatment of (2,2,2-trifluoethyl)triphenylphosphonium triflate with an aldehyde was shown to produce a trifluoromethylated olefin.

Much attention has been directed to trifluoromethyl or 2,2,2-trifluoroethyl-containing organic compounds for developing new medicines, agricultural chemicals and other materials since trifluoromethyl and trifluoroethyl groups are considered to be importantly involved in biological activity and physical properties of materials in consideration of their own unique features.[1] The onium salts of nitrogen, sulfur, and phosphorus are important particularly for their use as reagents in synthetic organic chemistry.[2] No effective methods are presently available for preparing trifluoroethylated onium salts and only a few 2,2,2-trifluoroethylated salts are known.[3] We successfully developed (2,2,2-trifluoroethyl)phenyliodonium triflate (**1**), the smallest in a series of (1*H*,1*H*-perfluoroalkyl)phenyliodonium triflates (FMITS reagents), as the most reactive source of 2,2,2-trifluoroethyl cation.[4,5] The present study was conducted to establish an effective means for preparing 2,2,2-trifluoroethylammonium, pyridinium, quinolinium, sulfonium, and phosphonium triflates with **1**. One application of the phosphonium salt obtained is discussed.

Results and Discussion

Table 1 shows modes for the synthesis of various kinds of 2,2,2-trifluoroethyl onium triflates using **1**. Salt **1** reacted readily with tertiary amines, pyridine and its derivatives, quinoline and benzothiazole in dichloromethane at room temperature to give 2,2,2-trifluoroethyl onium triflates **2**—**10** in high yields. Both bulky 2,4,6-collidine or 2,6-lutidine and the less nucleophilic ethyl nicotinate were well-trifluoroethylated with **1** under the same conditions. Salt **1** was clearly shown as the most reactive in a series of (1*H*,1*H*-perfluoroalkyl)phenyliodonium triflates, since (1*H*,1*H*-perfluoropropyl, -perfluorobutyl and -perfluorooctyl)-phenyliodonium triflates (**15**, **16**, and **17**) failed to react with 2,4,6-collidine, due possibly to bulkiness about the pyridine nitrogen. Pyridine reacted with **15**, **16**, and **17** in dichloromethane to give the corresponding pyridinium triflates[6] in 93, 92, and 74% yields, respectively, but this required increase in reaction temperature and/or time. The high reactivity of **1** greatly facilitated converting sulfides to trifluoroethylsulfonium salts so that diphenyl and dimethyl sulfides smoothly reacted with **1** at room temperature to give the trifluoroethylsulfonium triflates, **11** and **12**, in high yields. The reaction of dibenzothiophene, however, was quite slow and the yield of **13** was low, owing possibly to the great decrease in the reactivity of the latter sulfide. Salts **11** and **13** are stable crystals, while those of **12** are unstable due to their highly hygroscopic nature. The reaction of triphenylphosphine with **1** was successfully conducted using benzene as the solvent to obtain trifluoroethylphosphonium triflate **14**, nonhygroscopic and stable crystals, in 95% yield. Previously, (2,2,2-trifluoroethyl)triphenylphosphonium iodide could be obtained only in 9% yield by treating triphenylphosphine with 2,2,2-trifluoroethyl iodide for 9 months at room temper-

Table 1. Syntheses of Various 2,2,2-Trifluoroethyl Onium Triflates with **1**

Run[a)]	Substrate	Time[b)] / min	Product	Yield/%
1	$PhCH_2NMe_2$	30	$PhCH_2N^+(CH_2CF_3)Me_2\,^-OTf$ (**2**)	89
2	$PhNMe_2$	30	$PhN^+(CH_2CF_3)Me_2\,^-OTf$ (**3**)	89
3	Pyridine	10	*N*-(CF_3CH_2)-Pyridinium triflate (**4**)	83
4	3,5-Lutidine	10	*N*-(CF_3CH_2)-3,5-Lutidinium triflate (**5**)	83
5	2,6-Lutidine	10	*N*-(CF_3CH_2)-2,6-Lutidinium triflate (**6**)	77
6	2,4,6-Collidine	10	*N*-(CF_3CH_2)-Collidinium triflate (**7**)	63
7	Ethyl nicotinate	10	*N*-(CF_3CH_2)-3-Ethoxycarbonylpyridinium triflate (**8**)	93
8	Quinoline	10	*N*-(CF_3CH_2)-Quinolinium triflate (**9**)	87
9	Benzothiazole	10	*N*-(CF_3CH_2)-Benzothiazolinium triflate (**10**)	84
10	Ph_2S	120	$Ph_2S^+{-}CH_2CF_3\,^-OTf$ (**11**)	90
11	Me_2S	120	$Me_2S^+{-}CH_2CF_3\,^-OTf$ (**12**)	92
12	Dibenzothiophene	5 days	*S*-(CF_3CH_2)-Dibenzothiophenium triflate (**13**)	31
13	Ph_3P	60	$Ph_3P^+{-}CH_2CF_3\,^-OTf$ (**14**)	95

a) Dichloromethane was used as a solvent for Runs 1—12 and benzene for Run 13. All the reactions were carried out at room temperature. b) Minute, unless otherwise noted.

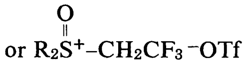
ature with northern exposure.[3b)]

Diphenyl sulfoxide and dimethyl sulfoxide were each found to react smoothly with **1**. Isolating the resulting salts in pure form, however, was difficult owing to their extremely hygroscopic nature. The ^{19}F NMR spectra of the crude products showed a peak of the trifluoroethyl group at −72.8 ppm for the product from diphenyl sulfoxide and at −73.9 ppm for dimethyl sulfoxide. These chemical shifts appeared in considerably high magnetic fields compared with those of sulfonium salts **11** (−60.0) and **12** (−61.1). However, structural assignments for *O*- or *S*-trifluoroethyl salts **18** or **19** could not

$R_2S{=}O + \mathbf{1} \longrightarrow R_2S^+\text{-}OCH_2CF_3\ {}^-OTf$

a; R=Ph **18, b**

b; R=Me



$$\text{or } R_2\overset{O}{\overset{\|}{S}}{}^+\text{-}CH_2CF_3\ {}^-OTf$$

19a,b

be determined. Pyridine *N*-oxide smoothly reacted with **1** under the same conditions to give *N*-(2,2,2-trifluoroethoxy)pyridinium triflate (**20**) in a quantitative yield.

As an application of trifluoroethyl onium salts in synthetic organic chemistry, phosphonium triflate **14** was treated with benzaldehyde using cesium fluoride as a base in *N,N*-dimethylformamide at room temperature to give *β*-(trifluoromethyl)styrene in 43% yield (*E*-/*Z*-isomer=46/54). When sodium hydride was used as the base, there was no product formation.

Experimental

Melting points were uncorrected. ^{1}H and ^{19}F NMR spectra were recorded with a Varian EM 390 NMR and a Hitachi R-20 B NMR spectrometer, respectively. Unless otherwise noted, acetone-d_6 was used as a solvent. ^{19}F NMR chemical shifts were reported in ppm downfield from trichlorofluoromethane as an internal standard. The IR spectra were measured on a JASCO A-202 diffraction grating infrared spectrometer. Mass spectra were recorded on a Hitachi RMU-6MG spectrometer at 70 eV. (2,2,2-Trifluoroethyl)phenyliodonium triflate (**1**) was prepared according to a reported method.[4c)] Cesium fluoride was dried at 140 °C for 3—12 h in vacuo.

Preparation, of 2,2,2-Trifluoroethyl Onium Triflates with 1. General Procedure; Into a solution of 1 mmol of a substrate (shown in Table 1) in 2 ml of dry dichloromethane was added 436 mg (1 mmol) of **1** under an argon atmosphere. The reaction mixture was stirred for the time shown in Table 1 at room temperature. The results are summarized in Table 1. In the case of triphenylphosphine as a substrate, benzene was used as a solvent. Isolation of products **2**—**10** was carried out by preparative thin-layer chromatography on silica gel. Products **11**—**14** each appeared as crystals upon adding pentane to the reaction mixture, and were collected by filtration. Analytically pure samples of **2** and **4** could not be obtained, probably due to changing hydrate formation for the former and the oily nature for the latter. For **12**, only ^{1}H and ^{19}F NMR were measured, since pure crystals of **12** were not obtained because of lability.

Benzyldimethyl(2,2,2-trifluoroethyl)ammonium triflate (**2**): mp 99—100 °C; ^{1}H NMR δ=3.48 (6H, s, 2×CH$_3$), 4.65 (2H, q, J=9 Hz, CH$_2$CF$_3$), 5.00 (2H, s, CH$_2$), 7.43—7.86 (5H, m); ^{19}F NMR δ=−60.8 (3F, t, J=9 Hz, CF$_3$), −77.6 (3F, s, SCF$_3$); Mass m/z 218 (M$^+$ −OTf). *N,N*-Dimethyl-*N*-(2,2,2-trifluoroethyl)anilinium triflate (**3**): mp 82—84 °C; ^{1}H NMR δ=4.10 (6H, s, 2×CH$_3$), 5.25 (2H, q, J=9 Hz, CH$_2$), 7.56—7.83 (3H, m), 8.06—8.26 (2H, m); ^{19}F NMR δ=−62.3 (3F, t, J=9 Hz, CF$_3$), −78.0 (3F, s, SCF$_3$); Mass m/z 204 (M$^+$ −OTf). Found; C, 36.84; H, 3.87; N, 4.14%. Calcd for $C_{11}H_{13}F_6NO_3S \cdot 1/2H_2O$; C, 36.47; H, 3.89; N, 3.87%. *N*-(2,2,2-Trifluoroethyl)pyridinium triflate (**4**):[7)] oil; ^{1}H NMR δ=5.81 (2H, q, J=8 Hz, CH$_2$), 8.28—8.53 (2H, m), 8.80—9.40 (3H, m); ^{19}F NMR δ=−70.7 (3F, t, J=8 Hz, CF$_3$), −77.9 (3F, s, SCF$_3$); IR (neat) 3070, 1640, 1500, 1400, 1260, 1170, 1030, 840, 780, 695, 640, 600, 520 cm^{-1}; Mass m/z 162 (M$^+$ −OTf). *N*-(2,2,2-Trifluoroethyl)-3,5-lutidinium triflate (**5**): mp 162—164 °C (1,2-dichloroethane); ^{1}H NMR δ=2.70 (6H, s, 2×CH$_3$ 5.72 (2H, q, J=8 Hz, CH$_2$), 8.60 (1H, br.s), 9.02 (2H, br.s); ^{19}F NMR δ=−70.4 (3F, t, J=8 Hz, CF$_3$), −77.9 (3F, s, SCF$_3$); Mass m/z 190 (M$^+$ −OTf). Found: C, 35.31; H, 3.33, N, 4.07%. Calcd for $C_{10}H_{11}F_6NO_3S$: C, 35.40; H, 3.27; N, 4.13%. *N*-(2,2,2-Trifluoroethyl)-2,6-lutidinium triflate (**6**): mp 188—189 °C (1,2-dichloroethane); ^{1}H NMR δ=2.97 (6H, s, 2×CH$_3$), 5.68 (2H, q, J=8 Hz, CH$_2$), 7.8—8.6 (3H, m); ^{19}F NMR δ=−65.4 (3F, t, J=8 Hz, CF$_3$), −77.6 (3F, s, CF$_3$S); Mass m/z 190 (M$^+$ −OTf). Found: C, 35.31; H, 3.33, N, 4.07%. Calcd for $C_{10}H_{11}F_6NO_3S$; C, 35.40; H, 3.27; N, 4.13%. Found; C, 35.30; H, 3.33; N, 4.02%. *N*-(2,2,2-Trifluoroethyl)-2,4,6-collidinium triflate (**7**): mp 146—148 °C (chloroform); ^{1}H NMR δ=2.59 (3H, s, CH$_3$), 2.96 (6H, s, 2×CH$_3$), 5.68 (2H, q, J=8 Hz, CH$_2$);7.89 (2H, br.s); ^{19}F NMR δ=−65.6 (3F, t, J=8 Hz, CF$_3$), −77.3 (3F, s, SCF$_3$); Mass m/z 204 (M$^+$ −OTf). Found: C, 37.10; H, 3.62; N, 3.86%. Calcd for $C_{11}H_{13}F_6NO_3S$: C, 37.40; H, 3.71; N, 3.96%. *N*-(2,2,2-Trifluoroethyl)-3-(ethoxycarbonyl)pyridinium triflate (**8**): mp 88—89 °C (1,2-dichloroethane); ^{1}H NMR δ=1.47 (3H, t, J=7 Hz, CH$_3$), 4.45 (2H, q, J=7 Hz, CH$_2$), 6.04 (2H, q, J=8 Hz, CH$_2$CF$_3$), 8.50—8.77 (1H, m), 9.30—9.66 (2H, m), 9.89 (1H, br.s, 2-H); ^{19}F NMR δ=−70.5 (3F, t, J=8 Hz, CF$_3$), −78.0 (3F, s, SCF$_3$); IR (KBr) 1740 (COO) cm^{-1}; Mass m/z 234 (M$^+$ −OTf). Found; C, 34.34; H, 2.89; N, 3.57%. Calcd for $C_{11}H_{11}F_6NO_5S$; C, 34.47; H, 2.89; N, 3.65%. *N*-(2,2,2-Trifluoroethyl)quinolinium triflate (**9**): mp 103—105 °C (1,2-dichloroethane); ^{1}H NMR δ=6.35 (2H, q, J=8 Hz, CH$_2$), 8.05—8.98 (5H, m), 9.52—9.86 (2H, m); ^{19}F NMR δ=−68.6 (3F, t, J=8 Hz, CF$_3$), −77.9 (3F, s, SCF$_3$); Mass m/z 212 (M$^+$ −OTf). Found: C, 39.69; H, 2.32; N, 3.71%. Calcd for $C_{12}H_9F_6NO_3S$: C, 39.89; H, 2.51; N, 3.88%. *N*-(Trifluoroethyl)benzothiazolinium triflate (**10**): mp 133—135 °C (1,2-dichloroethane); ^{1}H NMR δ=6.06 (2H, q, J=8 Hz, CH$_2$), 8.0—8.4 (2H, m), 8.7—8.9 (2H, m), 10.9 (1H, br.s, 2-H); ^{19}F NMR δ=−69.0 (3F, t, J=8 Hz, CF$_3$), −77.9 (3F, s, SCF$_3$); Mass m/z 218 (M$^+$ −OTf). Found: C, 32.40; N, 1.75; N, 3.65%. Calcd for $C_{10}H_7F_6NO_3S$: C, 32.70; H, 1.92; N, 3.81%. (2,2,2,-Trifluoroethyl)diphenylsulfonium triflate (**11**): mp 138—140 °C (acetonitrile–ether); ^{1}H NMR (acetonitrile-d_3) δ=5.21 (2H, q, J=9 Hz, CH$_2$), 7.66—7.98 (6H, m), 7.98—8.23 (4H, m); ^{19}F NMR (acetonitrile-d_3) δ=−60.0 (3F, t, J=9 Hz, CF$_3$), −78.0 (3F, s, SCF$_3$); Mass m/z 186 (Ph$_2$S$^+$). Found: C, 42.93; H, 2.93%. Calcd for $C_{15}H_{12}F_6O_3S_2$: C, 43.06; H, 2.89%. (2,2,2-Trifluoroethyl)dimethylsulfonium triflate (**12**): ^{1}H NMR (acetonitrile-d_3) δ=3.10 (6H, s, 2×CH$_3$), 4.30 (2H, q, J=9 Hz, CH$_2$); ^{19}F NMR (acetonitrile-d_3) δ=−61.1 (3F, t, J=9 Hz, CF$_3$), −78.1 (3F, s, SCF$_3$). (2,2,2-Trifluoroethyl)dibenzothiophenium trilfate (**13**): mp 168 °C (acetonitrile–ether); ^{1}H NMR (acetonitrile-d_3) δ=4.80 (2H, q, J=9 Hz, CH$_2$), 7.78—8.11 (4H, m), 8.34—8.48 (4H, m); ^{19}F NMR (acetonitrile-d_3) δ=−59.4 (3F, t, J=9 Hz, CF$_3$), −78.4 (3F, s, SCF$_3$); Mass (SIMS method) m/z 267 (M$^+$ −OTf), 184 (267−CH$_2$CF$_3$). Found: C, 43.05; H, 2.69%. Calcd for $C_{15}H_{10}F_6O_3S_2$: C, 43.27; 2.42%. (2,2,2-Trifluoroethyl)-

triphenylphosphonium trilfate (**14**): mp 208 °C (acetonitrile–ether); ^{1}H NMR (acetonitrile-*d*$_3$) δ=4.42—4.96 (2H, m, CH$_2$), 7.60—8.00 (15H, m, 3×Ph); ^{19}F NMR (acetonitrile-*d*$_3$) δ=−52.5 (3F, dt, *J*=7, 9 Hz, CF$_3$), −78.0 (3F, s, SCF$_3$); ^{31}P NMR (acetonitrile+acetonitrile-*d*$_3$, Ph$_3$P as an standard) δ=22.6 (q, *J*=7 Hz); Mass *m*/*z* 220 (M$^+$ −OTf). Found: C, 51.05; H, 3.46%. Calcd for $C_{21}H_{17}F_6O_3PS$: C, 51.02; H, 3.47%.

Reaction of Pyridine with (1*H*,1*H*-Perfluoropropyl, -Perfluorobutyl, or -Perfluorooctyl)phenyliodonium Triflate (15, 16, or 17). The procedure was the same as that mentioned above. The reaction time and temperature were as follows: 30 min, room temperature for **15** and **16**; 1 h, reflux temperature for **17**. After each reaction, the solvent was evaporated and the residue recrystallized from 1,2-dichloroethane. Spectral data and elemental analysis of new compounds are as follows. *N*-(1*H*,1*H*-Perfluoropropyl)pyridinium triflate: mp 84—86 °C; ^{1}H NMR δ=6.05 (2H, t, *J*=15 Hz, CH$_2$), 8.30—8.66 (2H, m), 8.90—9.50 (3H, m); ^{19}F NMR δ=−77.6 (3F, s, SCF$_3$), −82.7 (3F, s, CF$_3$), −119.3 (2F, t, *J*=15 Hz, CF$_2$); Mass *m*/*z* 262, 242. Found: C, 29.89; H, 1.77; N, 3.80%. Calcd for $C_9H_7F_8NO_3S$: C, 29.93; H, 1.95; N, 3.88%. *N*-(1*H*,1*H*-Perfluorobutyl)-pyridinium triflate: mp 126—127 °C; ^{1}H NMR δ=6.02 (2H, t, *J*=15 Hz, CH$_2$), 8.33—8.60 (2H, m), 8.90—9.50 (3H, m); ^{19}F NMR; δ=−77.6 (3F, s, SCF$_3$), −79.7 (3F, t, *J*=15 Hz, CH$_2$CF$_2$), −116.3 (2F, m, CF$_2$), −125.3 (2F, m, CF$_2$); Mass *m*/*z* 212, 192. Found: C, 29.07; H, 1.73, N, 3.28%. Calcd for $C_{10}H_7F_{10}NO_3S$: C, 29.21; H, 1.72; N, 3.41%.

Preparation of 18a,b or 19a,b and 20. The procedure was the same as that described above using diphenyl sulfoxide, dimethyl sulfoxide, and pyridine N-oxide as substrates. The reaction time and temperature were as follows: 4 h, room temperature for **18a** or **19a**; 5 h, room temperature for **18b** or **19b**; 3 h, room temperature for **20**. The spectral data and elemental analaysis are shown in the following: **18a** or **19a**: ^{1}H NMR (acetonitrile-*d*$_3$) δ=5.15 (2H, q, *J*=9 Hz, CH$_2$), 7.35—8.13 (10H, m, 2×Ph); ^{19}F NMR (acetonitrile-*d*$_3$) δ=−72.8 (3F, t, *J*=9 Hz, CF$_3$), −78.0 (3F, s, SCF$_3$). **18b** or **19b**: ^{1}H NMR (acetonitrile-*d*$_3$) δ=3.41 (6H, s, 2×CH$_3$), 4.82 (2H, q, *J*=9 Hz, CH$_2$); ^{19}F NMR (acetonitrile-*d*$_3$) δ=−73.9 (3F, t, *J*=9 Hz, CF$_3$), −78.0 (3F, s, SCF$_3$). Salt **20** was purified by washing the oily product, which was obtained by adding pentane to the reaction solution, with pentane. **20**: oil; ^{1}H NMR (acetonitrile-*d*$_3$) δ=5.26 (2H, q, *J*=9 Hz, CH$_2$), 8.06—8.40 (2H, m, 4-H), 8.58—8.80 (1H, m, 3-H), 9.00—9.28 (2H, m, 2-H); ^{19}F NMR (acetonitrile-*d*$_3$) δ=−72.0 (3F, t, *J*=9 Hz, CF$_3$), −78.0 (3F, s, SCF$_3$); Mass *m*/*z* 178 (M$^+$ −OTf). Found: C, 28.36; H, 2.23; N, 4.16%. Calcd for $C_8H_7F_6NO_4S \cdot 1/2H_2O$: C, 28.58; H, 2.40; N, 4.17%.

Reaction of 14 with Benzaldehyde. Into a mixture of 5 mmol of dry cesium fluoride in 5 ml of dry *N*,*N*-dimethylformamide were added 1 mmol of **14** and 1 mmol of benzaldehyde. The reaction mixture was stirred at room temperature for 8 h, poured into water, and extracted with pentane. The pentane layer was washed with water, dried with magnesium sulfate, and concentrated. The product yields were determined by ^{19}F NMR analyses of the concentrated solution. Structural assignments were carried out by spectral analyses of the isolated products. β-(Trifluoromethyl)styrene: ^{19}F NMR (chloroform-*d*) δ=−58.3 (d, *J*=9.0 Hz, CF$_3$, *Z*-isomer), −64.1 (dd, *J*=7.0, 2.0 Hz, CF$_3$, E-isomer).

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7) It was described that salt **4** was formed when 2,2,2-trifluoroethanol was treated with trifluoromethanesulfonyl fluoride in the presence of pyridine, but **4** was not fully assigned.[3a)]