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Hydroxyl-directed trifluoromethylation of hydrazones

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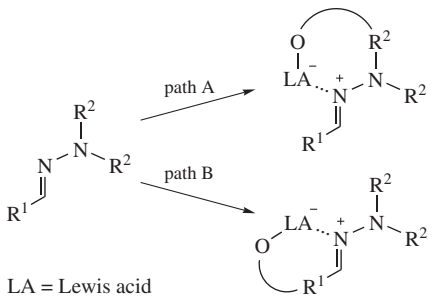
The trifluoromethylation of hydrazones derived from salicyl aldehydes and *N*-aminopiperidine and *N*-aminomorpholine through the generation of chelate difluoroboron complexes, which subsequently interact with Me_3SiCF_3 in the presence of sodium acetate, has been developed.

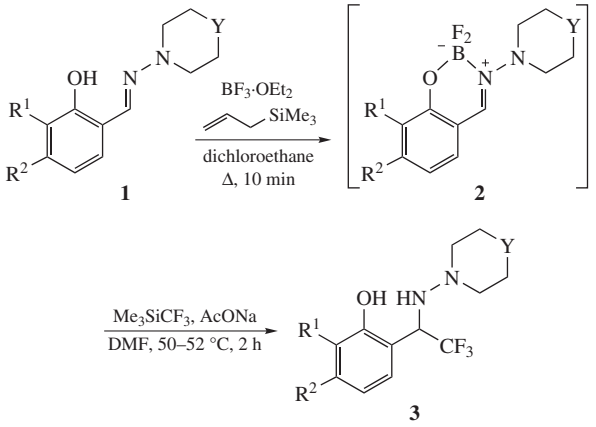
Nucleophilic trifluoromethylation reactions using the Ruppert–Prakash reagent (Me_3SiCF_3) have become the subject of intense investigations in recent years.[1] As electrophilic substrates, carbonyl compounds have been studied comprehensively, and methods for the addition of CF_3 carbanion to aldehydes, ketones and esters are very well developed.[1,2] At the same time, the scope of nucleophilic trifluoromethylation of the C=N bond is narrow, which is associated with low reactivity of azomethine fragment.[2(b),3,4] Importantly, the latter process leads to amines bearing the CF_3 group at the α-position, and such compounds have gained considerable attention due to potential pharmaceutical applications.[5] In this regard, besides nucleophilic trifluoromethylation, several approaches have been reported for the synthesis of these substances from CF_3 substituted imines by nucleophilic addition[6–8] and 1,3-proton shift reactions.[9]

Hydrazones constitute a class of readily available compounds, which are quite stable and, at the same time, poorly electrophilic substrates.[10] Recently, we introduced an approach for the activation of C=N bond by means of intramolecular complexation with Lewis acidic difluoroboryl group.[11] Concerning the hydrazone functionality, two modes of chelating activation can be proposed (Scheme 1). In path A, the acidic activator is provided from the side of hydrazine moiety, and the validity of this mode was demonstrated for the trifluoromethylation of *N*-benzoylhydrazones.[12] Here, we report the trifluoromethylation of hydrazones activated according to path B, where the delivery of Lewis acidic group occurs from the side of the azomethine fragment.

To implement this approach, the starting hydrazone should contain a functional group capable of generating the chelating Lewis acid. Thus, *N*,*N*-dialkylhydrazones bearing adjacent hydroxyl substituent were considered, and compounds of this type (hydrazones **1**) were readily prepared from salicyl aldehydes and *N*-aminopiperidine and *N*-aminomorpholine.†



Scheme 1



Scheme 2

Table 1 Trifluoromethylation of hydrazones.

Compound	R^1	R^2	Y	Isolated yield of **3** (%)
1a	H	H	O	91%
1b	MeO	H	O	82%
1c	H	H	CH_2	82%
1d	MeO	H	CH_2	82%
1e	benzo		O	97%

Treatment of hydrazones **1** with boron trifluoride etherate in the presence of allyltrimethylsilane in dichloroethane[12] results

† All reactions were performed under argon. Dichloroethane was distilled from CaH_2 before use. DMF was distilled in a vacuum from P_2O_5 and stored over MS 4 Å. Starting compounds were obtained from commercially available aldehydes and hydrazines in methanol. Compounds **1a**,**e**[13] and **1c**[14] have been described.

*2-Methoxy-6-[(*E*)-(morpholin-4-ylimino)methyl]phenol* **1b**: mp 110–112 °C. ^{1}H NMR (200 MHz, $CDCl_3$) δ: 2.98–3.20 (m, 4H, $2CH_2N$), 3.73–3.95 (m, 7H, MeO + $2CH_2O$), 6.67–6.93 (m, 3H, $2CH_{Ar}$), 7.66 (s, 1H, CH=N), 11.72 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 51.4, 55.8, 65.8, 111.8, 118.4, 118.7, 121.3, 140.4, 147.0, 147.9. Found (%): C, 60.87; H, 6.77; N, 11.74. Calc. for $C_{12}H_{16}N_2O_3$ (%): C, 61.00; H, 6.83; N, 11.86.

*2-Methoxy-6-[(*E*)-(piperidin-1-ylimino)methyl]phenol* **1d**: mp 60–61 °C. ^{1}H NMR (200 MHz, $CDCl_3$) δ: 1.37–1.89 [m, 6H, $(CH_2)_3$], 2.90–3.26 (m, 4H, $2CH_2N$), 3.86 (s, 3H, MeO), 6.61–6.93 (m, 3H, $2CH_{Ar}$), 7.61 (s, 1H, CH=N), 12.06 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 23.6, 24.5, 51.8, 55.8, 111.3, 118.3, 119.3, 121.1, 138.9, 147.0, 147.9. Found (%): C, 66.60; H, 7.88; N, 11.88. Calc. for $C_{13}H_{18}N_2O_2$ (%): C, 66.64; H, 7.74; N, 11.96.

in the formation of chelate difluoroboron complexes **2** (Scheme 2). Then, the solvent was exchanged from dichloroethane to dimethylformamide followed by addition of Me_3SiCF_3 and sodium acetate. The trifluoromethylation reaction was effected by heating at 50–52 °C for 2 h with subsequent work-up with aqueous sodium carbonate. Under these conditions, substrates **1a–e** were trifluoromethylated affording CF_3-substituted hydrazines **3a–e** in high yields (Table 1).‡

To provide the support for the formation of chelate boron complexes as key reaction intermediates complex **2e** derived from naphthalene substrate **1e** ($R^1 + R^2$ = benzo) was isolated in individual form.§ This substance was purified by recrystallization from dichloroethane, and its structure was proved by ^1H, ^{19}F and ^{11}B NMR spectroscopy and X-ray diffraction analysis (Figure 1).¶

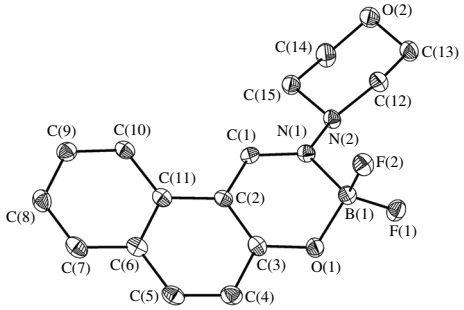


Figure 1 Molecular structure of **2e** presented by thermal ellipsoids with 50% probability. The hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): N(1)–C(1) 1.303(2), N(1)–B(1) 1.600(3), N(1)–N(2) 1.403(2), O(1)–B(1) 1.448(3); C(1)–N(1)–N(2) 122.45(16), N(1)–C(1)–C(2) 121.52(17), C(1)–N(1)–B(1) 119.28(16), O(1)–B(1)–N(1) 108.14(15).

In summary, we demonstrated that *N*,*N*-dialkylhydrazones bearing adjacent hydroxyl group can be smoothly trifluoromethylated with the Ruppert–Prakash reagent. The Lewis acidic activation of the hydrazone C=N double bond is achieved through the intramolecular complexation with difluoroboryl group.

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‡ *General procedure for the synthesis of* **3a–e**. To the flask containing hydrazone **1** (1.0 mmol) were successively added dichloroethane (2 ml), allyltrimethylsilane (238 μl, 1.5 mmol) and $BF_3{\cdot}OEt_2$ (190 μl, 1.5 mmol), and the mixture was heated under gentle reflux for 10 min. The solvent was evaporated under vacuum, DMF (2 ml) was added, and the mixture was stirred for 2 h at 50–52 °C. The reaction mixture was cooled to room temperature, quenched by the addition of saturated aqueous Na_2CO_3 (0.5 ml), the mixture was stirred for 2 min, diluted with water (7 ml) and extracted with diethyl ether (3×7 ml). The combined organic phase was filtered through Na_2SO_4, concentrated under vacuum, and the crude product was chromatographed on silica gel.

2-[2,2,2-Trifluoro-1-(morpholin-4-ylamino)ethyl]phenol **3a**: R_f 0.36 (hexanes–EtOAc, 1:1), mp 92–94 °C. ^1H NMR (200 MHz, $CDCl_3$) δ: 2.65–2.95 (m, 4H, $2CH_2N$), 3.22 (br. s, 1H, NH, $\Delta\nu_{1/2}$ 11.9 Hz), 3.62–3.78 (m, 4H, $2CH_2O$), 4.59 (q, 1H, CHN, *J* 8.2 Hz), 6.82–6.96 (m, 2H, $2CH_{Ar}$), 7.05–7.15 (m, 1H, CH_{Ar}), 7.22–7.34 (m, 1H, CH_{Ar}), 9.58 (br. s, 1H, OH, $\Delta\nu_{1/2}$ 12.6 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 56.0, 63.0 (q, *J* 28.4 Hz), 66.5, 116.8, 117.4, 119.6, 124.6 (q, *J* 282.0 Hz), 130.5, 130.6, 157.2. ^{19}F NMR (282 MHz, $CDCl_3$) δ: –73.13 (d, *J* 8.2 Hz). Found (%): C, 52.24; H, 5.44; N, 10.07. Calc. for $C_{12}H_{15}F_3N_2O_2$ (%): C, 52.17; H, 5.47; N, 10.14.

2-Methoxy-6-[2,2,2-trifluoro-1-(morpholin-4-ylamino)ethyl]phenol **3b**: R_f 0.53 (hexanes–EtOAc, 1:1), mp 100–101 °C. ^1H NMR (200 MHz, $CDCl_3$) δ: 2.64–2.77 (m, 4H, $2CH_2N$), 3.04 (br. s, 1H, NH, $\Delta\nu_{1/2}$ 60.0 Hz), 3.64–3.73 (m, 4H, $2CH_2O$), 3.88 (s, 3H, OMe), 4.89 (q, 1H, CHN, *J* 7.6 Hz), 6.82–7.00 (m, 4H, $3CH_{Ar}$ + OH). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 55.9, 57.0, 58.7 (q, *J* 28.8 Hz), 66.8, 111.1, 119.5, 119.6, 120.9 (q, *J* 1.2 Hz), 125.2 (q, *J* 282.1 Hz), 144.6, 146.9. ^{19}F NMR (282 MHz, $CDCl_3$) δ: –73.79 (d, *J* 7.6 Hz). Found (%): C, 51.11; H, 5.61; N, 9.14. Calc. for $C_{13}H_{17}F_3N_2O_3$ (%): C, 50.98; H, 5.59; N, 9.15.

2-[2,2,2-Trifluoro-1-(piperidin-1-ylamino)ethyl]phenol **3c**: R_f 0.15 (hexanes–EtOAc, 10:1), mp 50–52 °C. ^1H NMR (200 MHz, $CDCl_3$) δ: 1.33–1.50 and 1.55–1.75 [m, 2H and 4H, $(CH_2)_3$], 2.56–2.96 (m, 4H, $2CH_2N$), 3.15 (br. s, 1H, NH, $\Delta\nu_{1/2}$ 11.3 Hz), 4.55 (q, 1H, CHN, *J* 8.3 Hz), 6.80–7.01 (m, 2H, $2CH_{Ar}$), 7.07–7.18 (m, 1H, CH_{Ar}), 7.21–7.33 (m, 1H, CH_{Ar}), 10.59 (br. s, 1H, OH, $\Delta\nu_{1/2}$ 15.1 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 23.2, 25.5, 57.0, 63.3 (q, *J* 28.2 Hz), 117.7, 117.9 (q, *J* 1.1 Hz), 119.3, 124.8 (q, *J* 282.1 Hz), 130.2, 130.3, 157.6. ^{19}F NMR (282 MHz, $CDCl_3$) δ: –72.57 (d, *J* 8.3 Hz). Found (%): C, 57.09; H, 6.34; N, 10.47. Calc. for $C_{13}H_{17}F_3N_2O$ (%): C, 56.93; H, 6.25; N, 10.21.

2-Methoxy-6-[2,2,2-trifluoro-1-(piperidin-1-ylamino)ethyl]phenol **3d**: R_f 0.36 (hexanes–EtOAc, 3:1), mp 97–100 °C. ^1H NMR (200 MHz, $CDCl_3$) δ: 1.27–1.43 and 1.52–1.69 [m, 2H and 4H, $(CH_2)_3$], 2.57–2.77 (m, 4H, $2CH_2N$), 2.94 (br. s, 1H, NH, $\Delta\nu_{1/2}$ 11.3 Hz), 3.87 (s, 3H, OMe), 4.76 (q, 1H, CHN, *J* 8.2 Hz), 6.76–6.93 (m, 3H, $3CH_{Ar}$), 8.48 (br. s, 1H, OH, $\Delta\nu_{1/2}$ 18.7 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 23.3, 25.6, 55.8, 57.3, 60.3 (q, *J* 28.4 Hz), 111.3, 119.2, 119.5, 121.1 (q, *J* 1.1 Hz), 125.1 (q, *J* 282.0 Hz), 145.7, 147.7. ^{19}F NMR (282 MHz, $CDCl_3$) δ: –73.29 (d, *J* 8.2 Hz). Found (%): C, 55.05; H, 6.36; N, 9.02. Calc. for $C_{14}H_{19}F_3N_2O_2$ (%): C, 55.26; H, 6.29; N, 9.21.

1-[2,2,2-Trifluoro-1-(morpholin-4-ylamino)ethyl]-2-naphthol **3e**: R_f 0.24 (hexanes–EtOAc, 2:1), mp 101–103 °C. ^1H NMR (200 MHz, $CDCl_3$) δ: 2.67–3.02 (m, 4H, $2CH_2N$), 3.25 (br. s, 1H, NH, $\Delta\nu_{1/2}$ 130 Hz), 3.57–3.76 (m, 4H, $2CH_2O$), 5.56 (q, 1H, CHN, *J* 8.0 Hz), 7.18 (d, 1H, CH_{Ar}, *J* 8.9 Hz), 7.31–7.44 (m, 1H, CH_{Ar}), 7.53 (t, 1H, CH_{Ar}, *J* 7.5 Hz), 7.75–7.90 (m, 3H, $3CH_{Ar}$), 10.65 (br. s, 1H, OH, $\Delta\nu_{1/2}$ 130 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 55.7, 57.9 (q, *J* 28.5 Hz), 66.4, 106.0, 119.9, 120.8, 122.9, 125.0 (q, *J* 283.8 Hz), 127.1, 128.7, 129.0, 131.5, 133.5, 157.3. ^{19}F NMR (282 MHz, $CDCl_3$) δ: –72.16 (d, *J* 8.0 Hz). Found (%): C, 58.94; H, 5.18; N, 8.62. Calc. for $C_{16}H_{17}F_3N_2O_2$ (%): C, 58.89; H, 5.25; N, 8.58.

§ *Complex* **2e**. Allyltrimethylsilane (83 μl, 0.52 mmol) and $BF_3{\cdot}OEt_2$ (66 μl, 0.52 mmol) were successively added to a solution of hydrazone **1e** (89 mg, 0.35 mmol) in dichloroethane (1.5 ml), and the mixture was heated under gentle reflux for 10 min. The solution was allowed to cool slowly and kept overnight at 5 °C. The solvent was decanted, the crystals were washed with cold dichloroethane and hexane, and dried in a vacuum to give 62 mg of **2e** as pale yellow crystals; mp 187–188 °C. ^1H NMR (300 MHz, CD_2Cl_2) δ: 3.33–3.41 (m, 4H, $2CH_2O$), 3.86–3.93 (m, 4H, $2CH_2N$), 7.26 (d, 1H, CH_{Ar}, *J* 9.2 Hz), 7.48–7.55 (m, 1H, CH_{Ar}), 7.65–7.73 (m, 1H, CH_{Ar}), 7.88 (d, 1H, CH_{Ar}, *J* 8.1 Hz), 8.04–8.12 (m, 2H, $2CH_{Ar}$), 9.18 (br. s, 1H, CH=N, $\Delta\nu_{1/2}$ 11 Hz). ^{19}F NMR (282 MHz, CD_2Cl_2) δ: –134.97 (q, J_{F-B} 15.5 Hz). ^{11}B NMR (160 MHz, CD_2Cl_2) δ: –0.09 (t, J_{F-B} 15.5 Hz).

¶ *Crystallographic data for* **2e**: crystals of $C_{15}H_{15}BF_2N_2O_2$ are orthorhombic, space group *Pbca*, a = 13.425(2), b = 8.4912(15) and c = = 24.387(4) Å, V = 2780.0(8) Å^3, Z = 8, M = 304.10, d_{calc} = 1.453 g cm^{-3}, μ(MoKα) = 1.14 mm^{-1}, F(000) = 1264. Intensities of 17786 reflections were measured with a Smart APEX II diffractometer at 100 K [λ(MoKα) = = 0.71072 Å, ω-scans, $2\theta < 55.74°$] and 3316 independent reflections (R_{int} = 0.0913) were used in further refinement. The structure was solved by direct method and refined by the full-matrix least-squares technique against F^2 in the anisotropic–isotropic approximation. Hydrogen atoms were calculated and refined in the rigid body approximation with the U(H) = 1.2U_{eq}(C). The refinement converged to wR_2 = 0.1098 and GOF = = 0.993 for all independent reflections [R_1 = 0.0456 was calculated against F for 2042 observed reflections with $I > 2\sigma(I)$].

CCDC 714416 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif. For details, see 'Notice to Authors', *Mendeleev Commun.*, Issue 1, 2009.

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