Supporting Information

Chlorodifluoromethyl phenyl sulfone: a novel non-ODSbased difluorocarbene reagent for *O*- and *N*difluoromethylations

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General.

Unless otherwise mentioned, reagents were purchased from commercial sources and used as received. The solvent THF was distilled from sodium. High-resolution mass data were recorded on a high-resolution mass spectrometer in the EI , ESI or MALDI mode. ¹H NMR spectra were recorded at 300 MHz in CDCl₃ and were referenced to residual CHCl₃ or to tetramethylsilane (TMS). ¹⁹F NMR spectra were recorded at 282 MHz using CFCl₃ as external standard. ¹³C NMR spectra were recorded at 75, 100 or 125 MHz in CDCl₃. All chemical shifts are reported in δ parts per million and coupling constants are in hertz. PhSO₂CF₂H, PhSO₂CF₂Br, PhSO₂CF₂I, PhSO₂CF₃ were prepared by using the known procedures.¹

Preparation of chlorodifluoromethyl phenyl sulfone (2). <u>Method A</u>: Under N₂ atmosphere, into a 500 mL three-neck flask containing *N*-chlorosuccinimide (16.0 g, 120 mmol), PhSO₂CF₂H (**3**) (6.7 g, 35 mmol) and THF (140 mL) at -78° C, was added LHMDS (90 mmol, dissolved in 60 mL of THF) dropwise via a needle over a period of 18 min, followed by stirring at -78° C for additional 2 h. Then the reaction was quenched by adding excess amount of saturated NH₄Cl (aqueous solution) at -78° C, followed by extraction with diethyl ether. The organic phase was washed with brine and then dried over anhydrous MgSO₄. After the solution was filtered and the solvent was evaporated under vacuum, the residue was subjected to silica gel column chromatography using a mixture of ethyl acetate and petroleum ether (1:30, v/v) as eluent to give product **2** (6.3 g; yield: 79%) as a colorless liquid. IR (film): 3073, 1585, 1451, 1365, 1190, 1120, 1079, 930, 757, 719, 684, 657, 606, 582, 551, 428 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz): δ 7.67 (t,

J = 16.2 Hz, 2H), 7.84 (t, J = 15.3 Hz, 1H), 8.05 (d , J = 8.1 Hz, 2H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -63.0 (s, 2F). ¹³C NMR(CDCl₃, 125 MHz): δ 136.3, 131.2, 130.8, 129.7, 126.4 (t, J = 333.6 Hz). MS (EI, m/z, %): 226 (M⁺, 0.57), 77 (100.00). HRMS (EI): calcd. for C₇H₃F₂O₂ClS: 225.9667; Found: 225.9670.

<u>Method B</u>: (a) Under N₂ atmosphere, thioanisole (4) (15.86 g, 114 mmol), tetrachloromethane (10 mL), and PCl₅ (0.53 g, 2.5 mmol) were added into a 100-L threeneck flask equipped with a magnetic stirrer, a condensor with a gas outlet on the top, and a septum. With vigorous stirring of the solution at rt, chlorine gas was bubbled slowly into the mixture via a teflon needle. Immediately, an exothermic reaction occurred and HCl gas evolved, which was collected and neutralized by NaOH solution through the gas outlet. After the mixture was stirred for 1 h, the reaction mixture was further stirred at 80°C for 10 h (with chlorine gas was keeping passing through the reaction mixture). The progress of the chlorination process was monitored by ¹H NMR until all of the thioanisole was consumed. Then CH₂Cl₂ (20 mL) was added, and the mixture was washed with H₂O and saturated NaHCO₃ aqueous solution. The separated organic phase was further washed with brine and dried over anhydrous MgSO₄. After filtration and removal of the solvent under vacuum, trichloromethyl phenyl sulfide (**5**) was obtained through fractional vaccum distillation (bp: 86-87°C/1mmHg): 22.56 g (99.18 mmol, 87% yield).

(*b*) Into a 250-mL round-bottom teflon flask, was added a magnetic stirrer and 50 g of Olah's reagent (molar ratio: HF/pyridine = 10:1). Thereafter, trichloromethyl phenyl sulfide **5** (10 g, 44.15 mmol) was slowly added to the HF-pyridine solution at 20°C, and a gas-outlet was placed on the top of the reaction flask for the purpose of HCl gas collection. The reaction mixture was stirred for 4 h at rt, and the progress of fluorination was monitored by ¹⁹F NMR. After the reaction was finished, the reaction mixture was poured into iced water, and extracted with cold CH₂Cl₂ (200 mL x 3). The combined organic phase was washed with cold saturated NaHCO₃ aqueous solution and brine, successively. After drying over MgSO₄ and solvent removal, crude chlorodifluoromethyl phenyl sulfide (**6**) (¹⁹F NMR: δ -27 ppm, (s)) was obtained (purity > 94%, determined by NMR). The impurities were mainly PhSCF₃ and PhSCClF₂.

(c) To a 250-mL round-bottom flask with a magnetic stirrer containing the aboveobtained crude product (6), were added CH₃CN (75 mL), CCl₄ (75 mL), H₂O (150 mL), NaIO₄ (40 g) and ruthenium trichloride hydrate (150 mg). The resulting mixture was stirred at rt for 10 h. The completion of the reaction was monitored by ¹⁹F NMR. Thereafter, 150 mL of water was added, and the resulting reaction mixture was extracted with ether (150 mL x 2). The combined organic phase was washed successively with saturated NaHCO₃ and NaCl solutions. After drying over anhydrous MgSO₄ and solvent removal, the crude product **2** was further purified by silica gel column chromatography (pet ether/ethyl acetate = 50:1 v/v) to give 8.153 g chlorodifluoromethyl phenyl sulfone as a colorless liquid (96% purity, determined by ¹H and ¹⁹F NMR; the main impurity was PhSO₂CF₃), yield for 2 steps: 78%. The ¹H and ¹⁹F NMR data showed that the product was identical to compound **2** prepared via preparative method A (see above).

We applied this chlorodifluoromethyl phenyl sulfone sample **2** (with 4% $PhSO_2CF_3$ impurity) in the *O*- and *N*-difluoromethylation reactions, and found that it worked equally well as pure compound **2**.

Typical procedure for difluoromethylation using chlorodifluoromethyl phenyl sulfone (2). Into a pressure tube containing a mixture of 1-naphthol (7n) (144 mg, 1.0 mmol), chlorodifluoromethyl phenyl sulfone 2 (521 mg, 2.3 mmol), CH₃CN (7 mL) and a magnetic stirrer, was added aqueous KOH (25 wt.%, 2 mL, ca. 11 mmol). The pressure tube was then sealed with a cap. The reaction mixture was stirred at 50 °C for 5 h. After cooling to RT, the mixture was extracted with Et₂O (25 mL x 3), and the combined organic layers were dried over anhydrous MgSO₄. After the removal of solvents under vacuum, the crude product was further purified by silica gel column chromatography to give product **8n** as a colorless liquid, yield: 79% (153 mg). Colorless liquid. ¹H NMR(CDCl₃, 300 MHz): δ 6.67 (t, *J* = 73.8 Hz, 1H), 7.19 (d, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.56 (t, *J* = 8.1 Hz, 1H), 7.71 (d, *J* = 8.4 Hz, 1H), 7.86 (t, *J* = 5.1 Hz, 1H), 8.19 (t, *J* = 5.4 Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -79.83 (d, *J* = 74.8 Hz, 2F). The characterization data was consistent with reference.¹⁰

Characterization date for the isolated compounds:

1-(Difluoromethoxy)benzene (8a): ¹⁹F NMR (CDCl₃, 282 MHz): δ -82.71 (d, J = 75.3 Hz, 2F). The characterization data were consistent with those reported in reference.⁵

1-(Difluoromethoxy)-2-iodobenzene (**8b**): Colorless liquid. ¹H NMR (CDCl₃, 300 MHz): δ 6.52 (t, *J* = 73.2 Hz, 1H), 6.96 (t, *J* = 7.8 Hz, 1H), 7.16 (d, *J* = 9.0Hz, 1H), 7.35 (t, *J* = 8.1 Hz, 1H), 7.85(d, *J* = 8.1Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -81.36 (d, *J* = 72.8 Hz, 2F). The characterization data was consistent with reference.¹⁰

l-(Difluoromethoxy)-4-iodobenzene (**8c**): Colorless liquid. ¹H NMR (CDCl₃, 300 MHz): δ 6.48 (t, J = 73.5 Hz, 1H), 6.89 (d, J = 9.0 Hz, 2H), 7.68 (d, J = 9.0Hz, 2H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -81.18 (d, J = 73.6 Hz, 2F). The characterization data was consistent with reference.²

1-Bromo-4-(difluoromethoxy)benzene (8d): Colorless liquid. ¹H NMR (CDCl₃, 300 MHz): δ 6.49 (t, J = 73.5 Hz, 1H), 7.02 (d, J = 12.0 Hz, 2H), 7.48 (d, J = 15.0 Hz, 2H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -81.60 (d, J = 73.1 Hz, 2F). The characterization data were consistent with those reported previously.²

1-Chloro-4-(difluoromethoxy)benzene (8e): Colorless liquid. ¹H NMR (CDCl₃, 300 MHz): δ 6.48 (t, J = 73.2 Hz, 1H), 7.08 (d, J = 12.0 Hz, 2H), 7.33 (d, J = 11.7 Hz, 2H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -81.58 (d, J = 73.1 Hz, 2F). The characterization data was consistent with previous report.³

1-(Difluoromethoxy)-2-nitrobenzene (**8f**): Yellow liquid. ¹H NMR (CDCl₃, 300MHz): δ 6.63 (t, J = 147.0 Hz, 1H), 7.39 (t, J = 6.0 Hz, 2H), 7.63 (t, J = 9.1 Hz, 1H), 7.93 (d, J = 7.8 Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -82.01 (d, J = 73.4 Hz, 2F). The characterization data was consistent with previous report.⁴

1-(Difluoromethoxy)-4-nitrobenzene (**8g**): Yellow liquid. ¹H NMR (CDCl₃, 300 MHz): δ 6.63 (t, J = 72.0 Hz, 1H), 7.26 (d, J = 9.0 Hz, 2H), 8.26 (d, J = 15.3 Hz, 2H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -82.49 (d, J = 71.7 Hz, 2F). The characterization data was consistent with reference.⁵

1-tert-Butyl-4-(difluoromethoxy)benzene (**8h**): Colorless liquid. IR (film): 2967, 1608, 1513, 1382, 1231, 1137 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz): δ 6.48 (t, *J* = 74.8 Hz, 1H), 7.04 (d, *J* = 8.7 Hz, 2H), 7.37 (d, *J* = 9.3 Hz, 2H), 1.31 (s, 9H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -80.37 (d, *J* = 73.4, 2F). ¹³C NMR (CDCl₃, 125 MHz): δ 149.0, 148.4, 126.7, 119.1, 116.2 (t, *J* = 257.3 Hz), 34.4, 31.4. MS (EI, m/z, %): 200 (M⁺, 13.11), 185 (100.00). Anal. calcd. for C₁₁H₁₄F₂O: C, 65.98; H, 7.05; Found: C, 65.84; H, 7.35.

1-(Difluoromethoxy)-3-phenylbenzene (**8i**): White solid, mp. 26-28 °C. ¹H NMR (CDCl₃, 300 MHz): δ 7.56 (t, *J* = 6.9 Hz, 4H), 7.44 (t, *J* = 7.8 Hz, 2H), 7.35 (t, *J* = 7.2 Hz, 1H), 7.19 (d, *J* = 8.4 Hz, 2H), 6.55 (t, *J* = 73.8 Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -81.07 (d, *J* = 71.7 Hz, 2F). The characterization data was consistent with reference.¹⁰

1-(difluoromethoxy)-2-methoxybenzene (**8j**): ¹⁹F NMR (CDCl₃, 282MHz): δ -82.69 (d, J = 72.7, 2F).

2,4-Dichloro-1-(difluoromethoxy)benzene (**8k**): Colorless liquid. ¹H NMR (CDCl₃, 300 MHz): δ 6.51(t, J = 73.1 Hz, 1H), 7.17-7.26 (m, J = 29.2 Hz, 2H), 7.46 (d , J = 2.5 Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -81.68 (d, J = 71.1 Hz, 2F). The characterization data was consistent with previous report.⁶

1,3,5-Tribromo-2-(difluoromethoxy)benzene (81): White solid. Mp. 65-66°C; ¹H NMR (CDCl₃, 300 MHz): δ 7.74 (s, 2H), 6.59 (t, J = 73.8 Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -81.31 (d, J = 71.7 Hz, 2F). The characterization data was consistent with reference.¹⁰

(*E*)-1-Difluoromethoxy-4-styrylbenzene (**8m**): White solid. Mp. 95-96 °C. ¹H NMR (CDCl₃, 300 MHz): δ 7.51 (d, J = 8.4 Hz, 4H), 7.37 (d, J = 7.8 Hz, 2H), 7.27 (t, J = 6.9 Hz, 1H), 7.10 (d, J = 9.9 Hz, 4H), 6.52 (t, J = 73.5 Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -82.53 (d, J = 74.7 Hz, 2F). The characterization data was consistent with reference.¹⁰

1-(Difluoromethoxy)naphthalene (8n): Colorless liquid. ¹H NMR(CDCl₃, 300 MHz): δ 6.67 (t, J = 73.8 Hz, 1H), 7.19 (d, J = 7.5 Hz, 1H), 7.44 (t, J = 7.8 Hz, 1H), 7.56 (t, J = 8.1 Hz, 2H), 7.71 (d, J = 8.4 Hz, 1H), 7.86 (t, J = 5.1 Hz, 1H), 8.19 (t, J = 5.4 Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -79.83 (d, J = 74.8 Hz, 2F). The characterization data was consistent with reference.¹⁰

2-(difluoromethoxy)naphthalene (**80**): Colorless liquid. ¹H NMR (CDCl₃, 300 MHz): δ 6.63 (t, J = 74.1 Hz, 1H), 7.25-7.30 (m, J = 15.0 Hz, 1H), 7.44-7.54 (m, J = 30.0 Hz, 3H), 7.79-7.86 (m, J = 23.6 Hz, 3H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -79.83 (d, J = 74.8 Hz, 2F). The characterization data was consistent with previous reports. ^{5,6}

8-(Difluoromethoxy)quinoline (**8p**): White solid, m.p. 70-73°C. IR (film): 3097, 1584, 1452, 1362, 1189, 1120, 1077, 931, 760, 719, 685, 655, 582, 551, 429 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz): δ 7.10 (t, J = 75.3 Hz, 1H), 7.46-7.53 (m, 3H), 7.69-7.72 (m, 1H), 8.21 (d, J = 10.2 Hz, 1H), 8.98 (d, J = 6.0 Hz, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ - 81.85 (d, J = 73.9 Hz, 2F). ¹³C NMR(CDCl₃, 125MHz): δ 150.5, 147.0, 136.2, 129.7, 126.4, 125.3, 122.0, 119.8, 119.0, 116.5 (t, J = 258 Hz). MS (EI, m/z, %): 195 (M⁺, 7.00), 129 (100.00). Anal. calcd. for C₁₀H₇F₂ON: C, 61.25; H, 3.68; N, 9.92; Found: C, 61.54; H, 3.62; N, 7.18.

1-(Difluoromethyl)-1H-benzo[d]imidazole (**13a**): White solid, m.p. 40-41 °C. ¹H NMR (CDCl₃, 300MHz): δ 7.34 (t, *J* = 60.3 Hz, 1H), 7.36-7.43 (m, 2H), 7.60-7.63 (m, 1H), 7.84-7.87 (m, 1H), 8.14 (m, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -93.7 (d, *J* = 61.1, 2F). The characterization data was consistent with previous report.⁹

1-(Difluoromethyl)-2-phenyl-1H-imidazole (**13b**): White solid, m.p. 67-68 °C . ¹H NMR (CDCl₃, 300 MHz): δ 7.07 (t, *J* = 59.9 Hz, 1H), 7.23 (s, 1H), 7.40 (s, 1H), 7.50-7.53 (m, 3H), 7.58-7.62 (m, 2H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -90.44 (d, *J* = 59.7, 2F). The characterization data was consistent with previous report.⁸

1-Difluoromethyl-1H-benzotriazole (**13c**): White solid, mp. 38–40°C. ¹H NMR (CDCl₃, 300 MHz): δ 7.48–7.53 (m, 1H), 7.62–7.67 (m, 1H), 7.83 (d, *J* = 8.5Hz, 1H), 7.86 (t, *J* = 58.3 Hz, 1H, CHF₂), 8.14–8.16 (m, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -103.1 (d, *J* = 58.3 Hz, 2F). The characterization data was consistent with previous report.⁷

4-Benzoyl-2-(difluoromethyl)-3-methyl-1-phenyl-1,2-dihydropyrazol-5-one (**13d**): White solid, m.p. 44 °C. IR (KBr): 1641, 1599, 1552, 1505, 1469, 1386, 1309, 1250, 1151, 1088, 1041, 935, 906, 869, 800, 767, 733, 693, 661, 488 cm⁻¹. ¹H NMR (CDCl3, 300 MHz): δ 2.23 (s, 3H), 6.73 (t, J = 73.0 Hz, 1H), 7.39-7.44 (m, 1H), 7.47-7.54 (m, 4H), 7.58-7.68 (m, 3H), 7.80-7.83 (m, 2H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -82.82 (d, J = 74.0 Hz, 2F). ¹³C NMR (CDCl₃, 125 MHz): δ 15.0, 109.8, 112.7, 116.3 (t, J = 269.0 Hz), 123.7, 128.3, 128.6, 129.3, 129.2, 133.0, 137.0, 138.4, 149.7. MS (ESI, m/z): 329.2 (M⁺+H⁺), HRMS (ESI) calcd. For C₁₈H₁₄F₂N₂O₂: (M⁺+H⁺): 328.1023; Found: 328.1096.

2-(Difluoromethyl)-1-phenylpyrazolidin-3-one (**13e**): Colorless liquid. IR (film): 2932, 2270, 1686, 1651, 1601, 1503, 1407, 1350, 1254, 1141, 1084, 751, 693, 658 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz): δ 3.02 (t, J = 9.8 Hz, 2H), 3.83 (t, J = 9.8 Hz, 2H), 6.84-6.89 (m, 1H), 6.92-6.98 (m, 2H), 7.11 (t, J = 72.1 Hz, 1H), 723-7.29 (m, 2H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -87.11 (d, J = 72.2 Hz, 2F). ¹³C NMR (CDCl₃, 125 MHz): δ 30.5, 50.5, 114.2 (t, J = 259.3 Hz), 114.3, 119.7, 129.1, 148.0, 156.6. MS (EI, m/z, %): 212 (M⁺, 31.63), 77 (100.00). HRMS (EI): calcd. for C₁₀H₁₀F₂N₂O: 212.0761; Found: 212.0770.

1-Bromo-3-chloro-5-(difluoromethoxy)benzene (**16**): Colorless liquid. IR (film): 1588, 1574, 1429, 1380, 1222, 1123, 1060, 938, 850, 780, 667, 419, 410 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz): δ 6.42 (t, *J* = 72.7 Hz, 1H), 7.02 (d, *J* = 2.1 Hz, 1H), 7.13 (d, *J* = 1.9 Hz, 1H), 7.29-7.31 (m, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -81.67 (d, *J* = 71.8 Hz, 2F). ¹³C NMR (CDCl₃, 125 MHz): δ 115.3 (t, *J* = 264.6 Hz), 119.2, 121.5, 123.0, 128.7, 135.9, 151.6. MS (EI, m/z, %): 256 (M⁺, 59.27), 208 (100.00). HRMS (EI): calcd. for C₇H₄F₂ClBrO: 255.9102; Found: 255.9103. EA: calcd. for C₇H₄F₂ClBrO: C, 32.66; H, 1.57; Found: C, 33.5; H, 1.76.

2-(3-Chloro-5-(difluoromethoxy)phenyl)-1,3-dioxolane (17): Colorless liquid. IR (film): 3089, 2894, 1590, 1453, 1381, 1308, 1268, 1177, 1121, 1051, 1001, 970, 942, 861, 687, 550, 471 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz): δ 4.31-4.43 (m, 4H), 6.09 (s, 1H), 6.82 (t, *J* = 73.2 Hz, 1H), 7.45 (s, 1H), 7.64 (s, 1H). ¹⁹F NMR (CDCl₃, 282 MHz): δ -80.97 (d, *J* = 73.6 Hz, 2F). ¹³C NMR (CDCl₃, 125 MHz): δ 65.4, 102.2, 115.6 (t, *J* = 261.7 Hz), 115.8, 120.6, 123.8, 135.2, 141.9, 151.6. MS (EI, m/z, %): 250 (M⁺, 16.33), 73 (100.00). Anal. calcd. for C₁₀H₉F₂ClO₃: C, 47.92; H, 3.62; Found: C, 48.04; H, 3.71.

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¹H, ¹³C, and ¹⁹F NMR spectra of all new products















































































¹H and ¹⁹F NMR spectra of all (known) liquid products









































