JA010789T-28-86

Enantioselective Fluorination Mediated by Cinchona Alkaloid Derivatives/Selectfluor Combinations: Reaction Scope and Structural Information of *N*-Fluorocinchona Alkaloids

Norio Shibata,*,† Emiko Suzuki,† Toru Asahi‡ and Motoo Shiro#

Experimental Section

General Information. Melting points were determined on a Yanagimoto micro-melting-point apparatus and uncollected. IR spectra (cm^{-1}) were recorded on a Perkin-Elmer 1600 spectrometer. ^1H NMR spectra were measured as solutions in $CDCl_3$, and chemical shifts are expressed in ppm relative to internal Me_4Si (0.00 ppm) and were recorded on a JEOL GX-270 (270 MHz) or a Varian Gemini 300 (300 MHz) spectrometer. ^{19}F NMR spectra were measured with $CFCl_3$ as an internal standard and were taken with a JEOL GX-270 (254 MHz) spectrometer. Upfield shifts are quoted as negative δ values. EI mass spectra were taken with a JEOL JMS-D300 spectrometer. Column chromatography and preparative TLC were performed on BW-200 (Fuji Silysia) and Kieselgel 60 (Merck, art. 7748), respectively. All reactions involving oxygen- or moisture-sensitive compounds were carried out under a dry N_2 atmosphere. Unless otherwise noted, reagents were added by syringe. THF was distilled from sodium/benzophenone immediately prior to use.

A typical experimental procedure for the fluorination of 1; 2-Benzyl-2-fluoro-1-indanone (2a). A solution of **1a** (40.0 mg, 0.136 mmol) in MeCN (3 mL) was added to DHQB/Selectfluor combination [prepared *in situ* from DHQB (98%, 78.0 mg, 0.163 mmol) and Selectfluor (95%, 60.0 mg, 0.163 mmol) in MeCN (3 mL) stirred in the presence of 3Å MS at room temperature for 1h] at –20 °C. After stirring overnight, water was added to the reaction mixture and it was extracted with AcOEt. The organic phase was washed with 5% HCl, sat. $NaHCO_3$, brine and dried over Na_2SO_4. The solvent was removed under reduced pressure to give a crude oil, which was purified by preparative TLC on silica-gel (benzene) to

give **2a** (32.2 mg, 99%) as a colorless oil. Ee was determined to be 89% by HPLC analysis using a Chiralcel OB column (10% isopropanol in hexane). $[\alpha]_D^{30}$ +110.2° (c= 1.53, $CHCl_3$); Spectral data for **2a** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS) corresponded to literature values.[7a]

2-Fluoro-2-methyl-1-indanone (2b). Using the same procedure, reaction of 16.8 mg (0.0769 mmol) of **1b** with reagent generated *in situ* from 102 mg of DHQB (0.215 mmol) and 78 mg of Selectfluor (0.215 mmol) gave 11.7 mg (93 %) of **2b** as a colorless oil. Ee = 54% (HPLC, Chiralcel OB, 10% isopropanol in hexane); $[\alpha]_D^{31}$ +24.2° (c= 0.295, $CHCl_3$); Spectral data for **2b** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS) corresponded to literature values.[7a]

2-Ethyl-2-fluoro-1-indanone (2c). Using the same procedure, reaction of 40.0 mg (0.172 mmol) of **1c** with reagent generated *in situ* from 96.0 mg of DHQB (0.200 mmol) and 73.0 mg of Selectfluor (0.200 mmol) gave 37.5 mg (99 %) of **2c** as a colorless oil. Ee = 73% (HPLC, Chiralcel OB, 1% isopropanol in hexane); $[\alpha]_D^{26}$ +65.1° (c= 1.01, $CHCl_3$); Spectral data for **2c** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS) corresponded to literature values.[7a]

2-Fluoro-2-methyl-1-tetralone (2d). Using the same procedure, reaction of 40.0 mg (0.172 mmol) of **1d** with reagent generated *in situ* from 96.0 mg of DHQB (0.200 mmol) and 73.0 mg of Selectfluor (0.200 mmol) gave 29.0 mg (94 %) of **2d** as a colorless oil. Ee = 40% (HPLC, Chiralcel OB, 10% isopropanol in hexane); $[\alpha]_D^{26}$ +65.1°(c= 1.60, $CHCl_3$); Spectral data for **2d** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS) corresponded to literature values.[7a]

2-Ethyl-2-fluoro-1-tetralone (2e). Using the same procedure, except reaction temperature (–50 °C) and solvent ($MeCN/CH_2Cl_2$ =3/4), reaction of 30.0 mg (0.122 mmol) of **1e** with reagent generated *in situ* from 86.9 mg of DHQB (0.183 mmol) and 67.0 mg of Selectfluor (0.183 mmol) gave 16.7 mg (71 %) of **2e** as a colorless oil. Ee = 67% (HPLC, Chiralcel OB, 1% isopropanol in hexane); $[\alpha]_D^{26}$ +28.1° (c= 1.27, $CHCl_3$); Spectral data for **2e** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS, optical rotation) corresponded to literature values.[7a]

2-Benzyl-2-fluoro-1-tetralone (2f). Using the same procedure, reaction of 40.0 mg (0.129 mmol) of **1f** with reagent generated *in situ* from 73.0 mg of DHQB (0.151 mmol) and 55.0 mg of Selectfluor (0.151 mmol) gave 31.3 mg (95 %) of **2f** as a colorless oil. Ee = 71% (HPLC, Chiralcel OJ, 10% isopropanol in hexane); $[\alpha]_D^{26}$ +26.2° (c= 0.863, $CHCl_3$); Spectral data for **2f** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS, optical rotation) corresponded to literature values.[7a]

A typical experimental procedure for the fluorination of 4; Ethyl α-cyano-α-fluoro-α-tolylacetate (3a). A solution of **4a** (50.0 mg, 0.246 mmol) in CH_2Cl_2 (4 mL) was added to DHQDA/Selectfluor combination [prepared *in situ* from DHQDA (181 mg, 0.493 mmol) and Selectfluor (95%, 138 mg, 0.370 mmol) in MeCN (3 mL) in the presence of 3Å MS at room temperature for 1h] at –80 °C. After stirring for 2 h, water was added, and the reaction mixture was extracted with AcOEt. Work-up similar to that used in the fluorination of **1a** gave **3a** (43.5 mg, 80%) as a colorless oil. Ee = 87% (HPLC, Chiralcel OJ, 1% isopropanol in hexane); $[\alpha]_D^{25}$ –20.7° (c= 2.69, $CHCl_3$); Spectral data for **3a** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS, optical rotation) corresponded to literature value.[13b]

Methyl α-cyano-α-fluoro-α-naphthylacetate (3b). Using the same procedure, reaction of 20.0 mg (0.0888 mmol) of **4b** with reagent generated *in situ* from 66.0 mg of DHQDA (0.177 mmol) and 49.0 mg of Selectfluor (0.134 mmol) gave 18.7 mg (87 %) of **3b** as colorless crystals. Ee = 76% (HPLC, Chiralpak AD, 2% ethanol in hexane); $[\alpha]_D^{24}$ –40.6° (c= 2.19, $CHCl_3$); Spectral data for **3b** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS, optical rotation) corresponded to literature values.[14]

Ethyl α-cyano-α-fluoro-α-phenylacetate (3c). Using the same procedure, reaction of 50.0 mg (0.265 mmol) of **4c** with reagent generated *in situ* from 195 mg of DHQDA (0.529 mmol) and 145 mg of Selectfluor (0.397 mmol) gave 44.2 mg (81 %) of **3c** as a colorless oil. Ee = 83% (HPLC, Chiralcel OJ, 1% isopropanol in hexane); $[\alpha]_D^{26}$ –18.7° (c= 2.26, $CHCl_3$); Spectral data for **4c** (^{1}H NMR, ^{19}F NMR, IR, Mass, HRMS, optical rotation) corresponded to literature values.[15]

Methyl α-cyano-α-fluoro-α-isopropylphenylacetate (3d). Using the same procedure, reaction of 50.0 mg (0.230 mmol) of **4d** with reagent generated *in situ* from 170 mg of DHQDA (0.463 mmol) and 126 mg of Selectfluor (0.345 mmol) gave 44.6 mg (82 %) of **3d** as a colorless oil. Ee = 76% (HPLC, Chiralpak AS, 1% isopropanol in hexane); $[\alpha]_D^{26}$ –33.1° (c = 2.07, $CHCl_3$); ^1H NMR δ 7.36—7.57 (m, 2H, ArH), 7.32—7.35 (m, 2H, ArH), 3.90 (s, 3H, OMe), 2.95 (m, 1H, CHMe$_2$), 2.52 (d, *J* = 6.83 Hz, 6H, Me$_2$CH). ^{19}F NMR δ –144.3 (s); IR (neat) ν_{max} 2964, 1778 cm^{-1}; MS *m/z* 235 (M^+); HRMS calcd for $C_{13}H_{14}FNO_2$ 235.1009, found 235.1009.

Methyl α-chlorophenyl-α-cyano-α-fluoroacetate (3e). Using the same procedure, reaction of 30.0 mg (0.143 mmol) of **4e** with reagent generated *in situ* from 106 mg of DHQDA (0.286 mmol) and 80.0 mg of Selectfluor (0.215 mmol) gave 18.1 mg (56 %) of **3e** as a colorless oil. Ee = 68% (HPLC, Chiralcel OJ, 5% ethanol in hexane); $[\alpha]_D^{26}$ –23.5° (c = 1.12, $CHCl_3$); ^1H NMR (270 MHz, $CDCl_3$) δ 7.58—7.61 (m, 2H, ArH), 7.26—7.49 (m, 2H, ArH), 3.91 (s, 3H, CH_3); ^{19}F NMR (254 MHz, $CDCl_3$) δ –146.9 (s); IR (neat) ν_{max} 1777 cm^{-1}; MS *m/z* 227 (M^+); HRMS calcd for $C_{10}H_{17}Cl_{35}FNO_2$ 227.0149, found 227.0143, HRMS calcd for $C_{10}H_{17}Cl_{37}FNO_2$ 229.0119, found 229.0096.

2-Ethoxycarbonyl-2-fluoro-1-indanone (6a). Using the same procedure, reaction of 40.0 mg (0.196 mmol) of **5a** with reagent generated *in situ* from 144 mg of DHQDA (0.392 mmol) and 107 mg of Selectfluor (0.294 mmol) gave 38.7 mg (89 %) of **6a** as a colorless oil. Ee = 78% (HPLC, Chiralcel OJ, 10% isopropanol in hexane); $[\alpha]_D^{26}$ +15.1° (c = 2.13, $CHCl_3$); ^1H NMR δ 7.82 (m, 1H, ArH), 7.71 (m, 1H, ArH), 7.48 (m, 2H, ArH), 4.28 (q, *J* = 7.1 Hz, 2H, CH_2Me), 3.80 (dd, *J* = 11.6, 17.7Hz, 1H, CHHAr), 3.43 (dd, *J* = 23.3, 17.8 Hz, 1H, CHHAr), 1.26 (t, *J* = 7.2 Hz, 3H, Me). ^{19}F NMR δ –164.9 (dd, *J* = 12.0, 23.5 Hz); IR (KBr) ν_{max} 2984, 1726, 1607 cm^{-1}; MS *m/z* 222 (M^+); HRMS calcd for $C_{12}H_{11}FO_3$ 222.0692, found 222.0682.

2-Ethoxycarbonyl-2-fluorocoumaranone (6b). Using the same procedure, reaction of 30.0 mg (0.146 mmol) of **5b** with reagent generated *in situ* from 107 mg of DHQDA (0.292 mmol) and 81.0 mg of Selectfluor (0.218 mmol) gave 30.1 mg (92 %) of **6b** as a colorless oil. Ee = 80% (HPLC, Chiralcel

OJ, 10% isopropanol in hexane): $[\alpha]_D^{25}$ –27.7° (c = 1.70, $CHCl_3$); ^1H NMR δ 7.74 (m, 2H, ArH), 7.25 (m, 2H, ArH), 4.35 (qd, *J* = 7.3, 1.2 Hz, 2H, CH_2), 1.31 (t, *J* = 7.2 Hz, 3H, Me). ^{19}F NMR δ –127.43 (s); IR (KBr) ν_{max} 1745 cm^{-1}; MS *m/z* 224 (M^+); HRMS calcd for $C_{11}H_9FO_4$ 224.0485, found 224.0466.

2-Fluoro-2-methoxycarbonyl-1-tetralone (6c). Using the same procedure, reaction of 40.0 mg (0.196 mmol) of **5c** with reagent generated *in situ* from 128 mg of DHQD (0.392 mmol) and 107 mg of Selectfluor (0.294 mmol) gave 34.4 mg (79 %) of **6c** as a white solid. Ee = 59% (HPLC, Chiralcel OJ, 1% isopropanol in hexane); Mp 60—63 °C (hexane/CH_2Cl_2); ^1H NMR (270 MHz, $CDCl_3$) δ 8.07 (m, 1H, ArH), 7.56 (m, 1H, ArH), 7.37 (m, 1H, ArH), 7.29 (m, 1H, ArH), 3.83 (s, 3H, CH_3), 3.77—3.89 (m, 2H, CH_2), 2.48—3.23 (m, 2H, $ArCH_2$); ^{19}F NMR (254 MHz, $CDCl_3$) δ –164.6 (dd, *J*= 23.1, 11.1 Hz); IR (KBr) ν_{max} 1765, 1697 cm^{-1}; MS *m/z* 222 (M^+); HRMS calcd for $C_{11}H_9FO_4$ 222.0692, found 222.0685.

2-Benzoxycarbonyl-2-fluorocyclopentanone (6d). Using the same procedure, reaction of 40.0 mg (0.184 mmol) of **5d** with reagent generated *in situ* from 120 mg of DHQ (0.367 mmol) and 101 mg of Selectfluor (0.275 mmol) gave 24.0 mg (55 %) of **6d** as a colorless oil. Ee = 43% (HPLC, Chiralcel OJ, 50% ethanol in hexane); ^1H NMR (270 MHz, $CDCl_3$) δ 7.26—7.41 (m, 5H, ArH), 5.25 (s, 2H, CH_2Ph), 2.42—2.63 (m, 2H, CH_2), 2.32 (ddd, J= 28.5, 14.5, 6.8 Hz, 2H, $COCH_2$), 2.11 (pentet, 2H, *J*=7.3 Hz, $CH_3CH_2CH_2$); ^{19}F NMR (254 MHz, $CDCl_3$) δ –164.4 (t, *J*= 21.3 Hz); IR (neat) ν_{max} 2965, 1767 cm^{-1}; MS *m/z* 236 (M^+); HRMS calcd for $C_{13}H_{13}FO_3$ 236.0848, found 236.0821.

A typical experimental procedure for the fluorination of 8; 3-Benzyl-3-fluorooxindole (7a). A solution of **8a** (20 mg, 0.0896 mmol) in MeCN (4 mL) was added to $(DHQ)_2AQN$/Selectfluor combination [prepared *in situ* from $(DHQ)_2AQN$ (115 mg, 0.134 mmol) and Selectfluor (97%, 49 mg, 0.134 mmol) in MeCN (3 mL) at room temperature for 1h] at 0 °C. (The reaction was performed in the absence of 3Å MS since all the commercially available bis-cinchona alkaloids are anhydrates.) After stirring at 0 °C for 1—2 days, water was added, and the reaction mixture was extracted with AcOEt.

Work-up similar to that used in the fluorination of **1a** gave 7a (19.7 mg, 91%) as white solid, Ee = 78%

(HPLC, Chiralcel OB, 3% isopropanol in hexane); Mp 122—124 °C (hexane/CH_2Cl_2); $[\alpha]_D^{25}$ –18.8° (c = 1.17, MeOH); ^{1}H NMR (270 MHz, $CDCl_3$) δ 8.24 (brs, 1H, NH), 7.19—7.28 (m, 4H, ArH), 7.08—7.09 (m, 2H, ArH), 6.98—7.00 (m, 2H, ArH), 6.79 (d, *J*= 7.6 Hz, 1H, ArH), 3.57 (dd, 1H, *J*= 13.2, 10.6 Hz, CHH), 3.23 (dd, 1H, *J*= 22.4, 13.5 Hz, CHH); ^{19}F NMR (254 MHz, $CDCl_3$) δ –155.77 (dd, *J*= 22.2, 10.2 Hz); IR (KBr) ν_{max} 3162, 1718 cm^{-1}; MS *m/z* 241 (M^+); HRMS calcd for $C_{15}H_{12}NFO$ 241.0903, found 241.0904.

3-Fluoro-3-(*p*-methoxybenzyl)oxindole (7b). Using the same procedure, reaction of 20.0 mg (0.0790 mmol) of **8b** with reagent generated *in situ* from 104 mg of $(DHQD)_2PYR$ (0.118 mmol) and 42.0 mg of Selectfluor (0.118 mmol) gave 16.9 mg (79 %) of **7b** as a light yellow oil. Ee = 82% (HPLC, Chiralpak AD, 5% ethanol in hexane); $[\alpha]_D^{27}$ –2.23° (c = 1.13, $CHCl_3$); ^{1}H NMR (270 MHz, $CDCl_3$) δ 8.30 (brs, 1H, NH), 7.26 (m, 1H, ArH), 6.99 (m, 4H, ArH), 6.80 (d, 1H, *J*= 7.9 Hz, ArH), 6.73 (d, 2H, *J*= 8.6 Hz, ArH), 3.75 (s, 3H, OMe), 3.51 (dd, *J*= 13.5, 10.5 Hz, 1H, CHH), 3.17 (dd, 1H, *J*= 22.4, 13.5 Hz, CHH); ^{19}F NMR (254 MHz, $CDCl_3$) δ –155.93 (dd, *J*= 22.2, 11.1 Hz); IR (neat) ν_{max} 3260, 1737 cm^{-1}; MS *m/z* 271 (M^+); HRMS calcd for $C_{16}H_{14}NFO_2$ 271.1000, found 271.1026.

3-Fluoro-3-methyloxindole (7c). Using the same procedure, reaction of 20.0 mg (0.136 mmol) of **8c** with reagent generated *in situ* from 180 mg of $(DHQD)_2PYR$ (0.204 mmol) and 75.0 mg of Selectfluor (0.204 mmol) gave 21.1 mg (94 %) of **7c** as light yellow solid. Ee = HPLC, Chiralpak AD, 5% ethanol in hexane); Mp 87—89 °C (hexane/CH_2Cl_2); $[\alpha]_D^{25}$ +10.7° (c = 1.20, $CHCl_3$); ^{1}H NMR (270 MHz, $CDCl_3$) δ 8.33 (br.s, 1H, NH), 7.42 (d, 1H, *J*= 7.6 Hz, ArH), 7.30—7.36 (m, 1H, ArH), 7.10 (t, *J*= 7.6 Hz, 1H, ArH), 6.92 (d, 1H, *J*= 7.5 Hz, ArH), 1.78 (d, 3H, *J*= 22.1, 13.5 Hz, CH_3); ^{19}F NMR (254 MHz, $CDCl_3$) δ –152.2 (dd, *J*= 43.5, 22.2 Hz); IR (KBr) ν_{max} 3208, 1734 cm^{-1}; MS *m/z* 165 (M^+); HRMS calcd for C_9H_8NFO 165.0590, found 165.0582.

3-Ethyl-3-fluorooxindole (7d). Using the same procedure, reaction of 20.0 mg (0.124 mmol) of **8d** with reagent generated *in situ* from 164 mg of $(DHQD)_2PYR$ (0.186 mmol) and 68.0 mg of Selectfluor

(0.186 mmol) gave 17.6 mg (79 %) of **7d** as white solid. Ee = 76% (HPLC, Chiralpak AD, 5% isopropanol in hexane); Mp 63—66 °C (hexane/CH_2Cl_2); $[\alpha]_D^{25}$ +44.7° (c = 0.94, $CHCl_3$); ^1H NMR (270 MHz, $CDCl_3$) δ 8.18 (br.s, 1H, NH), 7.30—7.41 (m, 2H, ArH), 7.07—7.13 (m, 1H, ArH), 6.91 (d, 1H, *J*= 7.9 Hz, ArH), 2.20 (td, 2H, *J*= 21.8, 7.4 Hz, ArH), 0.89 (t, 3H, *J*= 7.6 Hz, CH_3); ^{19}F NMR (254 MHz, $CDCl_3$) δ –157.4 (t, *J*= 13.9 Hz); IR (KBr) ν_{max} 3242, 1740, 1701 cm^{-1}; MS *m/z* 179 (M^+); HRMS calcd for $C_{10}H_{10}NFO$ 179.0746, found 179.0722.

3-Fluoro-3-isopropylphenyloxindole (7e). Using the same procedure, reaction of 20.0 mg (0.114 mmol) of **8e** with reagent generated *in situ* from 134 mg of $(DHQ)_2PHAL$ (0.171 mmol) and 63.0 mg of Selectfluor (0.171 mmol) gave 2.6 mg (12 %) of **7e** as a colorless oil. Ee = 40% (HPLC, Chiralpak AD, 5% isopropanol in hexane); ^1H NMR (270 MHz, $CDCl_3$) δ 7.69 (br.s, 1H, NH), 7.29—7.54 (m, 2H, ArH), 7.08 (t, 1H, *J*= 7.6 Hz, ArH), 6.86 (d, 1H, *J*= 7.6 Hz, ArH), 2.48—2.59 (m, 1H, CH), 1.13 (d, 3H, *J*= 6.9 Hz, CH_3), 0.89 (d, 3H, *J*= 6.6 Hz, CH_3); ^{19}F NMR (254 MHz, $CDCl_3$) δ –161.4 (d, *J*= 9.2 Hz); MS *m/z* 193 (M^+); HRMS calcd for $C_{11}H_{12}NFO$ 193.0902, found 193.0901.

3-Ethoxycarbonyl-3-fluoroxindole (7f). Using the same procedure, reaction of 20.0 mg (0.0975 mmol) of **8f** with reagent generated *in situ* from 129 mg of $(DHQD)_2PYR$ (0.146 mmol) and 53.0 mg of Selectfluor (0.146 mmol) gave 20.1 mg (93 %) of **7f** as white solid. Ee = 37% (HPLC, Chiralpak AD, 5% isopropanol in hexane); Mp 109—110 °C (hexane/CH_2Cl_2); ^1H NMR (270 MHz, $CDCl_3$) δ 7.97 (br.s, 1H, NH), 7.37—7.42 (m, 2H, ArH), 7.11 (t, 1H, *J*= 7.6 Hz, ArH), 6.94 (d, 1H, *J*= 7.9 Hz, ArH), 4.22—4.36 (m, 2H, CH_2), 1.25 (t, 3H, *J*= 7.1 Hz, CH_3); ^{19}F NMR (254 MHz, $CDCl_3$) δ –164.7 (s); IR (KBr) ν_{max} 3182, 1748 cm^{-1}; MS *m/z* 223 (M^+); HRMS calcd for $C_{11}H_{10}NFO_3$ 223.0645, found 223.0658.

***N*-Fluoroquininine tetrafluoroborate (*N*F-Q·BF_4).** Selectfluor (97%, 2.23 g, 6.16 mmol) was added to a suspension of quinine (2.00 g, 6.16 mmol) in MeCN (12 mL) at room temperature. After the mixture was stirred for several minutes, a homogeneous solution was formed. After an additional several minutes, the pale yellow clear solution became cloudy, and a powder began to precipitate. The white

precipitate that was formed filtered, quickly washed with cold MeCN and dried under ... The crystalline *N*F-Q·BF_4 was recrystallized by slow evaporation of its solution in MeCN/AcOEt to yield colorless crystals of excellent quality. Mp 138—140 °C (AcOEt/MeCN); $[\alpha]_D^{26}$ –118° (c = 0.95, MeCN); ^{1}H NMR (270 MHz, $CDCl_3$) δ 8.84 (br.s, 1H), 8.06 (d, 1H, *J*= 8.9 Hz), 7.71 (br.s, 1H), 7.46 (d, 1H, *J*= 6.9 Hz), 7.15 (s, 1H), 6.35 (d, 1H, *J*= 2.9 Hz), 5.70—5.83 (m, 1H), 5.12—5.21 (m, 2H), 4.82—4.94 (m, 1H), 4.70 (d, 1H, *J*= 2.5 Hz), 4.35—4.48 (m, 2H), 4.11—4.15 (m, 1H), 3.99—4.05 (m, 1H), 3.96 (s, 3H), 3.32 (m, 1H), 2.34—2.67 (m, 3H), 2.17 (m, 1H), 1.94—1.96 (m, 1H); ^{19}F NMR (254 MHz, $CDCl_3$) δ –43 (s); IR (KBr) ν_{max} 3507, 3400—3150, 3150—2800, 2361, 1626 cm^{-1}; FAB-MS *m/z* 343 (–BF_4); *Anal.* Calcd. for $C_{20}H_{25}BF_5N_2O_{2.5}$ (+1/2H_2O): C, 54.69; H, 5.74; N, 6.38; Found: C, 54.98; H, 5.86; N, 6.41.

***N*-Fluoroquininine 4-chlorobenzoate tetrafluoroborate (*N*F-DHQB·BF_4).** ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.72 (br.s, 1H), 8.21 (d, 2H, *J*= 8.5 Hz), 8.04 (d, 1H, *J*= 9.4 Hz), 7.62 (d, 2H, *J*= 8.5 Hz), 7.55 (d, 1H, *J*= 4.3 Hz), 7.48 (dd, 1H, *J*= 9.4, 2.6 Hz), 7.3 (s, 1H), 7.24 (d, 1H, *J*= 2.6 Hz), 4.66 (m, 1H), 4.24—4.51 (m, 2H), 4.17—4.22 (m, 1H), 3.84—3.88 (m, 1H), 2.96 (m, 1H), 2.40—2.53 (m, 3H), 2.30—2.40 (m, 2H), 1.41—1.53 (m, 2H), 0.85 (t, 3H, *J*= 7.3 Hz); FAB-MS *m/z* 483 (–BF_4).

Supporting Information 2

JA010789T-28-86

Enantioselective Fluorination Mediated by Cinchona Alkaloid Derivatives/Selectfluor Combinations: Reaction Scope and Structural Information of *N*-Fluorocinchona Alkaloids

Norio Shibata, Emiko Suzuki, Toru Asahi and Motoo Shiro*

Contribution from the Faculty of Pharmaceutical Sciences, Toyama Medical and Pharmaceutical University, Sugitani 2630, Toyama 930-0194, Japan, and Kagami Memorial Laboratory for Materials Science and Technology, Waseda University, 2-8-26, Nishi-Waseda, Shinjuku-ku, Tokyo 169-0051, Japan, and Rigaku Corporation, 3-9-12 Matsubara-cho, Akishima, Tokyo 196-8666, Japan.

Crystallographic Data of *N*F-Q·BF_4

Experimental

Data Collection

A colorless needle crystal of $C_{20}H_{25}BF_5N_2O_{2.50}$ having approximate dimensions of 0.30 x 0.10 x 0.10 mm was mounted in a loop. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations which were exposed for 1.0 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 7.8475(2) $\AA$

b = 15.5819(4) $\AA$ β = 95.9737(4)°

c = 16.2805(3) $\AA$

V = 1979.95(8) $\AA^3$

For Z = 4 and F.W. = 439.23, the calculated density is 1.47 g/cm^3. Based on the systematic absences of:

0k0: k $\neq$ 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P2_1$ (#4)

The data were collected at a temperature of -160 $\pm$ 1°C to a maximum 2θ value of 70.0°. A total of 75 images, corresponding to 225.0 ° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 4.00 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 34449 reflections which were collected, 8582 were unique (R_{int} = 0.019); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.3 cm^{-1}. A symmetry-related absorption correction using the program ABSCOR[1] was applied which resulted in transmission factors ranging from 0.88 to 0.99. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[4] was based on 8567 all reflections ($2\theta < 69.99$) and 550 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma(Fo^2 - Fc^2)/\Sigma Fo^2 = 0.057$$

$$R_w = \sqrt{\Sigma w(Fo^2 - Fc^2)^2/\Sigma w(Fo^2)^2} = 0.131$$

$$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.040 \quad for \quad I > 2.0\sigma(I) \quad data$$

The standard deviation of an observation of unit weight[5] was 1.09. The weighting scheme was based on counting statistics and included a factor (p = 0.095) to downweight the intense reflections. Plots of $\Sigma w(Fo^2 - Fc^2)^2$ versus Fo^2, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.34 and -0.19 $e^-/\mathring{A}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6] .The values for the mass attenuation coefficients are those of Creagh and Hubbel[7]. All calculations were performed using the teXsan[8] crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(2) SIR97: Altomare, A., Burla, M.C., Camalli, M., Cascarano, G.L., Giacovazzo, C., Guagliardi, A., Moliterni, A.G.G., Polidori, G. & Spagna, R. (1999). J. Appl. Cryst. 32, 115-119.

(3) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least-Squares:

Function minimized: $\Sigma w(Fo^2 - Fc^2)^2$

where w = $\frac{1}{\sigma^2(Fo^2)} = [\sigma_c^2(Fo^2) + (p(Max(Fo^2, 0) + 2Fc^2)/3)^2]^{-1}$

$\sigma_c(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

(5) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(8) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{20}H_{25}BF_5N_2O_{2.50}$
Formula Weight	439.23
Crystal Color, Habit	colorless, needle
Crystal Dimensions	0.30 X 0.10 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	32623 (3.6 - 80.4°)
Indexing Images	3 oscillations at 1.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 7.8475(2) Å b = 15.5819(4) Å c = 16.2805(3) Å β = 95.9737(4)° V = 1979.95(8) Å^3
Space Group	$P2_1$ (#4)
Z value	4
D_{calc}	1.473 g/cm^3
F_{000}	916.00
μ(MoKα)	1.27 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated

Temperature	-160.0 °C
Voltage, Current	60 kV, 90 mA
Collimator Size	0.5 mm
Detector Aperture	450.0 mm x 256.0 mm
Data Images	75 exposures at 4.0 minutes per degree
Oscillation Range (ϕ=0.0°,χ=50.0°)	ω 130.0 - 190.0° with 3.0° step
Oscillation Range (ϕ=180.0°,χ=50.0°)	ω 0.0 - 165.0° with 3.0° step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	70.0°
No. of Reflections Measured	Total: 34449 Unique: 8582 (R_{int} = 0.019)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.8813 - 0.9874)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo^2)}$
p-factor	0.0950
No. of Reflections (All, 2θ<69.99°)	8567
No. Variables	550
Reflection/Parameter Ratio	15.58
Residuals: R; Rw	0.057 ; 0.131
Residuals: R1	0.040
No. of Reflections to calc R1	7637

Goodness of Fit Indicator	1.09
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.34 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.19 $e^-/\AA^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
F(1)	-0.0068(2)	0.5947(3)	0.09522(9)	2.47(2)
F(2)	0.6639(1)	0.6909(3)	0.51909(6)	1.81(2)
F(3)	-0.2235(3)	0.8896(3)	-0.0647(1)	3.66(3)
F(4)	-0.4427(2)	0.7970(3)	-0.09709(8)	2.83(3)
F(5)	-0.1775(2)	0.7470(3)	-0.05265(9)	2.62(2)
F(6)	-0.3378(2)	0.8125(3)	0.03697(8)	3.10(3)
F(7)	0.1465(2)	0.6029(3)	0.42203(7)	1.92(2)
F(8)	0.4320(2)	0.5828(3)	0.4160(1)	4.23(4)
F(9)	0.2623(2)	0.4698(3)	0.43933(9)	2.96(3)
F(10)	0.2374(2)	0.5328(3)	0.31249(9)	3.62(3)
O(1)	-0.1336(2)	0.6062(3)	0.30239(8)	1.80(2)
O(2)	0.1784(2)	0.8990(3)	0.05920(8)	1.61(2)
O(3)	0.3896(1)	0.6565(3)	0.67988(8)	1.49(2)
O(4)	0.8008(2)	0.3777(3)	0.46603(8)	1.59(2)
O(5)	0.1060(2)	0.5865(3)	0.59356(9)	1.80(2)
N(1)	-0.1759(2)	0.5787(3)	0.11495(10)	1.66(2)
N(2)	-0.1415(2)	0.9238(3)	0.34419(9)	1.43(2)
N(3)	0.6968(2)	0.7263(3)	0.59934(8)	1.25(2)
N(4)	0.5477(2)	0.3571(3)	0.77085(9)	1.55(2)
C(1)	-0.2866(3)	0.5740(3)	0.0336(1)	1.99(3)
C(2)	-0.4686(3)	0.5495(3)	0.0506(1)	1.87(3)
C(3)	-0.4752(2)	0.5505(3)	0.1456(1)	1.75(3)
C(4)	-0.3552(3)	0.4802(3)	0.1830(1)	2.04(3)
C(5)	-0.1731(3)	0.4935(3)	0.1577(1)	2.06(3)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(6)	-0.4152(2)	0.6371(3)	0.1822(1)	1.63(3)
C(7)	-0.2277(2)	0.6547(3)	0.16546(10)	1.37(2)
C(8)	-0.1012(2)	0.6688(3)	0.2431(1)	1.42(2)
C(9)	-0.5975(3)	0.6085(3)	0.0037(1)	2.22(3)
C(10)	-0.7432(3)	0.5812(3)	-0.0359(2)	2.88(4)
C(11)	-0.2043(2)	0.8557(3)	0.37842(10)	1.56(3)
C(12)	-0.1972(2)	0.7728(3)	0.34603(10)	1.51(2)
C(13)	-0.1197(2)	0.7593(3)	0.27520(9)	1.25(2)
C(14)	0.0316(2)	0.8251(3)	0.16194(9)	1.29(2)
C(15)	0.0923(2)	0.8981(3)	0.12722(9)	1.29(2)
C(16)	0.0696(2)	0.9792(3)	0.1627(1)	1.44(2)
C(17)	-0.0078(2)	0.9867(3)	0.23396(10)	1.46(2)
C(18)	-0.0687(2)	0.9129(3)	0.27256(9)	1.20(2)
C(19)	-0.0528(2)	0.8312(3)	0.23532(9)	1.12(2)
C(20)	0.2296(2)	0.8177(3)	0.0291(1)	1.81(3)
C(21)	0.8748(2)	0.7623(3)	0.60438(10)	1.41(2)
C(22)	0.9090(2)	0.8099(3)	0.68855(9)	1.24(2)
C(23)	0.7604(2)	0.7897(3)	0.74002(9)	1.25(2)
C(24)	0.5950(2)	0.8285(3)	0.6959(1)	1.54(2)
C(25)	0.5669(2)	0.7958(3)	0.6063(1)	1.53(2)
C(26)	0.7351(2)	0.6928(3)	0.74721(9)	1.21(2)
C(27)	0.6893(2)	0.6528(3)	0.66050(9)	1.13(2)
C(28)	0.5167(2)	0.6055(3)	0.64828(9)	1.20(2)
C(29)	1.0867(2)	0.7877(3)	0.7275(1)	1.49(2)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(30)	1.1315(2)	0.7701(3)	0.8065(1)	1.96(3)
C(31)	0.4857(2)	0.4255(3)	0.8051(1)	1.60(3)
C(32)	0.4766(2)	0.5076(3)	0.76828(10)	1.42(2)
C(33)	0.5358(2)	0.5192(3)	0.69202(9)	1.25(2)
C(34)	0.6721(2)	0.4524(3)	0.57512(9)	1.18(2)
C(35)	0.7358(2)	0.3801(3)	0.54099(10)	1.31(2)
C(36)	0.7355(2)	0.3003(3)	0.5823(1)	1.50(2)
C(37)	0.6726(2)	0.2942(3)	0.6573(1)	1.55(2)
C(38)	0.6091(2)	0.3678(3)	0.69574(9)	1.26(2)
C(39)	0.6072(2)	0.4476(3)	0.65348(9)	1.14(2)
C(40)	0.8039(2)	0.4573(3)	0.4226(1)	1.87(3)
B(1)	-0.2950(3)	0.8128(3)	-0.0443(1)	1.85(3)
B(2)	0.2729(3)	0.5464(3)	0.3962(1)	1.85(3)
H(1O)	-0.0319	0.6031	0.3414	2.2
H(1a)	-0.2880	0.6279	0.0068	2.4
H(1b)	-0.2425	0.5315	-0.0007	2.4
H(2)	-0.4906	0.4923	0.0315	2.2
H(3)	-0.5888	0.5390	0.1579	2.1
H(3O)	0.2821	0.6298	0.6526	1.8
H(4a)	-0.3514	0.4818	0.2415	2.4
H(4b)	-0.3970	0.4255	0.1637	2.4
H(5O)	0.1140	0.5821	0.5456	2.2
H(5O')	0.1113	0.5478	0.6049	2.2
H(5a)	-0.1453	0.4489	0.1213	2.5

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(5b)	-0.0916	0.4936	0.2051	2.5
H(6a)	-0.4869	0.6809	0.1576	2.0
H(6b)	-0.4221	0.6361	0.2401	2.0
H(7)	-0.2267	0.7044	0.1321	1.6
H(8)	0.0122	0.6611	0.2289	1.7
H(9)	-0.5725	0.6678	0.0025	2.7
H(10a)	-0.8198	0.6205	-0.0645	3.5
H(10b)	-0.7711	0.5216	-0.0356	3.5
H(11)	-0.2573	0.8631	0.4278	1.9
H(12)	-0.2456	0.7257	0.3729	1.8
H(15)	0.0458	0.7705	0.1371	1.5
H(16)	0.1084	1.0289	0.1369	1.7
H(17)	-0.0206	1.0414	0.2577	1.8
H(20a)	0.2881	0.8259	-0.0185	2.2
H(20b)	0.3037	0.7896	0.0707	2.2
H(20c)	0.1312	0.7829	0.0152	2.2
H(21a)	0.9553	0.7168	0.6015	1.7
H(21b)	0.8840	0.8009	0.5602	1.7
H(22)	0.9067	0.8695	0.6775	1.5
H(23)	0.7829	0.8138	0.7935	1.5
H(24a)	0.5003	0.8119	0.7243	1.8
H(24b)	0.6044	0.8890	0.6957	1.8
H(25a)	0.5838	0.8408	0.5688	1.8
H(25b)	0.4543	0.7732	0.5948	1.8

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(26a)	0.8378	0.6673	0.7722	1.5
H(26b)	0.6447	0.6816	0.7804	1.5
H(27)	0.7768	0.6126	0.6510	1.4
H(28)	0.4863	0.5960	0.5910	1.4
H(29)	1.1751	0.7859	0.6919	1.8
H(30a)	1.0477	0.7704	0.8446	2.4
H(30b)	1.2473	0.7565	0.8249	2.4
H(31)	0.4447	0.4188	0.8576	1.9
H(32)	0.4302	0.5542	0.7958	1.7
H(34)	0.6714	0.5052	0.5465	1.4
H(36)	0.7790	0.2503	0.5578	1.8
H(37)	0.6714	0.2398	0.6841	1.9
H(40a)	0.6900	0.4767	0.4088	2.2
H(40b)	0.8664	0.4982	0.4565	2.2
H(40c)	0.8575	0.4488	0.3734	2.2

$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(1)	0.0248(5)	0.0318(6)	0.0403(6)	0.0000(5)	0.0174(5)	-0.0036(5)
F(2)	0.0278(5)	0.0280(5)	0.0128(4)	-0.0034(4)	0.0016(3)	-0.0011(4)
F(3)	0.066(1)	0.0228(6)	0.0510(9)	-0.0067(7)	0.0122(8)	0.0025(6)
F(4)	0.0244(5)	0.0591(9)	0.0240(5)	0.0060(6)	0.0015(4)	0.0015(6)
F(5)	0.0271(6)	0.0300(6)	0.0442(7)	0.0094(5)	0.0120(5)	0.0106(5)
F(6)	0.0398(7)	0.0604(10)	0.0180(5)	0.0087(7)	0.0044(5)	0.0005(6)
F(7)	0.0285(5)	0.0177(4)	0.0263(5)	0.0038(4)	0.0003(4)	-0.0018(4)
F(8)	0.0246(6)	0.0425(9)	0.092(1)	-0.0016(6)	-0.0044(7)	0.0000(9)
F(9)	0.0559(9)	0.0177(5)	0.0389(7)	0.0096(5)	0.0058(6)	0.0040(5)
F(10)	0.064(1)	0.0476(9)	0.0262(6)	0.0233(8)	0.0067(6)	-0.0021(6)
O(1)	0.0240(5)	0.0186(5)	0.0253(6)	-0.0033(4)	-0.0008(4)	0.0093(4)
O(2)	0.0241(6)	0.0187(5)	0.0197(5)	-0.0007(4)	0.0085(4)	0.0020(4)
O(3)	0.0139(4)	0.0161(5)	0.0271(5)	0.0022(4)	0.0049(4)	-0.0028(4)
O(4)	0.0247(6)	0.0148(5)	0.0215(5)	0.0022(4)	0.0057(4)	-0.0025(4)
O(5)	0.0236(5)	0.0187(5)	0.0261(6)	0.0017(4)	0.0020(4)	0.0007(4)
N(1)	0.0221(6)	0.0158(5)	0.0265(6)	0.0001(5)	0.0090(5)	-0.0007(5)
N(2)	0.0163(5)	0.0211(6)	0.0166(5)	0.0014(4)	0.0005(4)	-0.0003(4)
N(3)	0.0175(5)	0.0169(5)	0.0133(5)	-0.0009(4)	0.0021(4)	0.0009(4)
N(4)	0.0181(6)	0.0201(6)	0.0204(6)	-0.0029(5)	0.0006(4)	0.0045(5)
C(1)	0.0359(9)	0.0200(7)	0.0210(7)	-0.0013(6)	0.0084(6)	-0.0029(6)
C(2)	0.0310(8)	0.0160(6)	0.0237(7)	-0.0016(6)	0.0013(6)	-0.0024(5)
C(3)	0.0224(7)	0.0223(7)	0.0220(6)	-0.0043(6)	0.0024(5)	0.0006(6)
C(4)	0.0325(8)	0.0187(7)	0.0256(8)	-0.0058(6)	0.0007(6)	0.0043(6)
C(5)	0.0298(8)	0.0146(6)	0.0342(9)	0.0040(6)	0.0051(7)	0.0034(6)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	0.0176(6)	0.0218(7)	0.0228(7)	0.0005(5)	0.0028(5)	-0.0035(5)
C(7)	0.0203(6)	0.0117(5)	0.0202(6)	-0.0005(5)	0.0034(5)	-0.0005(5)
C(8)	0.0181(6)	0.0139(6)	0.0221(6)	-0.0001(5)	0.0028(5)	0.0041(5)
C(9)	0.0362(9)	0.0217(7)	0.0257(8)	0.0017(7)	-0.0007(6)	-0.0013(6)
C(10)	0.041(1)	0.033(1)	0.0336(10)	0.0002(9)	-0.0082(8)	0.0039(8)
C(11)	0.0185(6)	0.0241(7)	0.0174(6)	0.0005(5)	0.0048(5)	-0.0001(5)
C(12)	0.0182(6)	0.0208(6)	0.0187(6)	-0.0012(5)	0.0029(5)	0.0022(5)
C(13)	0.0149(5)	0.0152(6)	0.0174(6)	-0.0005(5)	0.0020(4)	0.0024(5)
C(14)	0.0163(6)	0.0140(6)	0.0191(6)	-0.0011(5)	0.0033(5)	0.0000(5)
C(15)	0.0164(6)	0.0157(6)	0.0173(6)	0.0001(5)	0.0034(4)	0.0012(5)
C(16)	0.0195(6)	0.0148(6)	0.0208(6)	-0.0016(5)	0.0033(5)	0.0014(5)
C(17)	0.0195(6)	0.0157(6)	0.0202(6)	-0.0008(5)	0.0012(5)	-0.0009(5)
C(18)	0.0148(6)	0.0156(6)	0.0149(5)	0.0003(4)	0.0003(4)	0.0000(4)
C(19)	0.0138(5)	0.0142(5)	0.0143(5)	-0.0007(4)	0.0005(4)	0.0021(4)
C(20)	0.0267(7)	0.0204(7)	0.0233(7)	-0.0004(6)	0.0094(6)	-0.0025(6)
C(21)	0.0159(6)	0.0194(6)	0.0188(6)	-0.0016(5)	0.0052(5)	0.0013(5)
C(22)	0.0141(5)	0.0148(6)	0.0183(6)	-0.0006(4)	0.0024(4)	0.0007(5)
C(23)	0.0159(5)	0.0139(5)	0.0179(6)	0.0001(4)	0.0037(4)	-0.0005(5)
C(24)	0.0159(6)	0.0167(6)	0.0266(7)	0.0036(5)	0.0052(5)	0.0007(5)
C(25)	0.0166(6)	0.0173(6)	0.0241(7)	0.0024(5)	0.0012(5)	0.0029(5)
C(26)	0.0159(5)	0.0151(6)	0.0150(5)	-0.0013(5)	0.0013(4)	0.0001(4)
C(27)	0.0138(5)	0.0131(5)	0.0163(5)	0.0008(4)	0.0019(4)	0.0017(4)
C(28)	0.0135(5)	0.0142(5)	0.0183(6)	0.0005(4)	0.0034(4)	-0.0011(5)
C(29)	0.0136(5)	0.0198(6)	0.0235(7)	-0.0005(5)	0.0026(5)	-0.0019(5)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(30)	0.0183(6)	0.0312(9)	0.0244(7)	-0.0008(6)	-0.0011(5)	0.0009(6)
C(31)	0.0176(6)	0.0245(7)	0.0188(6)	-0.0026(5)	0.0025(5)	0.0040(5)
C(32)	0.0170(6)	0.0194(6)	0.0180(6)	-0.0012(5)	0.0033(5)	0.0018(5)
C(33)	0.0135(5)	0.0143(6)	0.0196(6)	-0.0003(4)	0.0021(4)	0.0000(5)
C(34)	0.0157(6)	0.0120(5)	0.0171(6)	0.0003(4)	0.0015(4)	-0.0005(4)
C(35)	0.0169(6)	0.0144(6)	0.0181(6)	0.0006(4)	0.0001(5)	-0.0018(5)
C(36)	0.0214(6)	0.0118(6)	0.0228(6)	0.0031(5)	-0.0019(5)	-0.0005(5)
C(37)	0.0229(7)	0.0128(6)	0.0223(6)	-0.0001(5)	-0.0012(5)	0.0008(5)
C(38)	0.0157(6)	0.0137(6)	0.0178(6)	-0.0014(4)	-0.0008(5)	0.0022(4)
C(39)	0.0135(5)	0.0122(5)	0.0177(6)	0.0001(4)	0.0015(4)	0.0003(4)
C(40)	0.0288(8)	0.0197(7)	0.0239(7)	0.0008(6)	0.0087(6)	-0.0004(6)
B(1)	0.0263(8)	0.0242(8)	0.0204(7)	0.0037(6)	0.0053(6)	0.0022(6)
B(2)	0.0279(8)	0.0157(7)	0.0260(8)	0.0047(6)	-0.0001(7)	0.0016(6)

The general temperature factor expression:

$$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Table 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
F(1)	N(1)	1.419(2)	F(2)	N(3)	1.417(2)
F(3)	B(1)	1.377(3)	F(4)	B(1)	1.391(3)
F(5)	B(1)	1.396(3)	F(6)	B(1)	1.398(2)
F(7)	B(2)	1.422(2)	F(8)	B(2)	1.379(3)
F(9)	B(2)$^{-}$	1.391(3)	F(10)	B(2)	1.379(3)
O(1)	C(8)	1.414(2)	O(2)	C(15)	1.356(2)
O(2)	C(20)	1.429(2)	O(3)	C(28)	1.414(2)
O(4)	C(35)	1.372(2)	O(4)	C(40)	1.429(2)
N(1)	C(1)	1.509(3)	N(1)	C(5)	1.498(2)
N(1)	C(7)	1.521(2)	N(2)	C(11)	1.317(2)
N(2)	C(18)	1.362(2)	N(3)	C(21)	1.499(2)
N(3)	C(25)	1.499(2)	N(3)	C(27)	1.523(2)
N(4)	C(31)	1.318(2)	N(4)	C(38)	1.370(2)
C(1)	C(2)	1.532(3)	C(2)	C(3)	1.552(3)
C(2)	C(9)	1.514(3)	C(3)	C(4)	1.530(3)
C(3)	C(6)	1.529(3)	C(4)	C(5)	1.541(3)
C(6)	C(7)	1.549(2)	C(7)	C(8)	1.539(2)
C(8)	C(13)	1.516(2)	C(9)	C(10)	1.322(3)
C(11)	C(12)	1.400(3)	C(12)	C(13)	1.374(2)
C(13)	C(19)	1.423(2)	C(14)	C(15)	1.377(2)
C(14)	C(19)	1.428(2)	C(15)	C(16)	1.408(2)
C(16)	C(17)	1.370(2)	C(17)	C(18)	1.417(2)
C(18)	C(19)	1.421(2)	C(21)	C(22)	1.557(2)
C(22)	C(23)	1.538(2)	C(22)	C(29)	1.511(2)

Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance
C(23)	C(24)	1.541(2)
C(24)	C(25)	1.539(3)
C(27)	C(28)	1.536(2)
C(29)	C(30)	1.326(3)
C(32)	C(33)	1.382(2)
C(34)	C(35)	1.373(2)
C(35)	C(36)	1.414(2)
C(37)	C(38)	1.421(2)
C(23)	C(26)	1.529(2)
C(26)	C(27)	1.551(2)
C(28)	C(33)	1.522(2)
C(31)	C(32)	1.411(3)
C(33)	C(39)	1.423(2)
C(34)	C(39)	1.424(2)
C(36)	C(37)	1.367(3)
C(38)	C(39)	1.421(2)

Table 4. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
O(1)	H(1O)	0.97	O(3)	H(3O)	1.00
O(5)	H(5O)	0.79	O(5)	H(5O')	0.63
C(1)	H(1a)	0.94	C(1)	H(1b)	0.95
C(2)	H(2)	0.95	C(3)	H(3)	0.95
C(4)	H(4a)	0.95	C(4)	H(4b)	0.95
C(5)	H(5a)	0.95	C(5)	H(5b)	0.95
C(6)	H(6a)	0.95	C(6)	H(6b)	0.95
C(7)	H(7)	0.94	C(8)	H(8)	0.95
C(9)	H(9)	0.94	C(10)	H(10a)	0.94
C(10)	H(10b)	0.96	C(11)	H(11)	0.95
C(12)	H(12)	0.95	C(14)	H(15)	0.95
C(16)	H(16)	0.94	C(17)	H(17)	0.94
C(20)	H(20a)	0.95	C(20)	H(20b)	0.95
C(20)	H(20c)	0.95	C(21)	H(21a)	0.95
C(21)	H(21b)	0.95	C(22)	H(22)	0.94
C(23)	H(23)	0.95	C(24)	H(24a)	0.95
C(24)	H(24b)	0.95	C(25)	H(25a)	0.95
C(25)	H(25b)	0.95	C(26)	H(26a)	0.95
C(26)	H(26b)	0.95	C(27)	H(27)	0.95
C(28)	H(28)	0.95	C(29)	H(29)	0.95
C(30)	H(30a)	0.95	C(30)	H(30b)	0.95
C(31)	H(31)	0.95	C(32)	H(32)	0.95
C(34)	H(34)	0.94	C(36)	H(36)	0.95
C(37)	H(37)	0.95	C(40)	H(40a)	0.95

Table 4. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C(40)	H(40b)	0.95	C(40)	H(40c)	0.95

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(15)	O(2)	C(20)	116.9(1)	C(35)	O(4)	C(40)	116.3(1)
F(1)	N(1)	C(1)	106.0(1)	F(1)	N(1)	C(5)	107.0(1)
F(1)	N(1)	C(7)	107.1(1)	C(1)	N(1)	C(5)	110.1(1)
C(1)	N(1)	C(7)	110.6(1)	C(5)	N(1)	C(7)	115.5(1)
C(11)	N(2)	C(18)	118.1(1)	F(2)	N(3)	C(21)	106.0(1)
F(2)	N(3)	C(25)	106.9(1)	F(2)	N(3)	C(27)	107.1(1)
C(21)	N(3)	C(25)	111.3(1)	C(21)	N(3)	C(27)	110.1(1)
C(25)	N(3)	C(27)	114.9(1)	C(31)	N(4)	C(38)	117.3(1)
N(1)	C(1)	C(2)	108.4(1)	C(1)	C(2)	C(3)	107.8(2)
C(1)	C(2)	C(9)	110.1(2)	C(3)	C(2)	C(9)	114.0(2)
C(2)	C(3)	C(4)	107.8(2)	C(2)	C(3)	C(6)	110.9(1)
C(4)	C(3)	C(6)	109.0(1)	C(3)	C(4)	C(5)	110.3(1)
N(1)	C(5)	C(4)	105.9(1)	C(3)	C(6)	C(7)	110.3(1)
N(1)	C(7)	C(6)	105.6(1)	N(1)	C(7)	C(8)	111.7(1)
C(6)	C(7)	C(8)	115.1(1)	O(1)	C(8)	C(7)	108.4(1)
O(1)	C(8)	C(13)	112.1(1)	C(7)	C(8)	C(13)	109.8(1)
C(2)	C(9)	C(10)	123.2(2)	N(2)	C(11)	C(12)	123.6(1)
C(11)	C(12)	C(13)	119.7(2)	C(8)	C(13)	C(12)	119.8(1)
C(8)	C(13)	C(19)	121.5(1)	C(12)	C(13)	C(19)	118.6(1)
C(15)	C(14)	C(19)	120.0(1)	O(2)	C(15)	C(14)	124.5(1)
O(2)	C(15)	C(16)	114.9(1)	C(14)	C(15)	C(16)	120.6(1)
C(15)	C(16)	C(17)	120.7(1)	C(16)	C(17)	C(18)	120.4(1)
N(2)	C(18)	C(17)	118.0(1)	N(2)	C(18)	C(19)	122.7(1)
C(17)	C(18)	C(19)	119.3(1)	C(13)	C(19)	C(14)	123.7(1)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle
C(13)	C(19)	C(18)	117.2(1)
N(3)	C(21)	C(22)	107.5(1)
C(21)	C(22)	C(29)	109.1(1)
C(22)	C(23)	C(24)	108.4(1)
C(24)	C(23)	C(26)	108.3(1)
N(3)	C(25)	C(24)	106.2(1)
N(3)	C(27)	C(26)	105.8(1)
C(26)	C(27)	C(28)	115.3(1)
O(3)	C(28)	C(33)	111.4(1)
C(22)	C(29)	C(30)	126.5(2)
C(31)	C(32)	C(33)	119.6(2)
C(28)	C(33)	C(39)	120.7(1)
C(35)	C(34)	C(39)	119.9(1)
O(4)	C(35)	C(36)	114.7(1)
C(35)	C(36)	C(37)	120.2(1)
N(4)	C(38)	C(37)	117.9(1)
C(37)	C(38)	C(39)	118.6(1)
C(33)	C(39)	C(38)	117.2(1)
F(3)	B(1)	F(4)	109.8(2)
F(3)	B(1)	F(6)	112.0(2)
F(4)	B(1)	F(6)	108.5(2)
F(7)	B(2)	F(8)	108.6(2)
F(7)	B(2)	F(10)	108.2(2)
F(8)	B(2)	F(10)	112.2(2)
C(14)	C(19)	C(18)	119.0(1)
C(21)	C(22)	C(23)	108.1(1)
C(23)	C(22)	C(29)	116.3(1)
C(22)	C(23)	C(26)	110.9(1)
C(23)	C(24)	C(25)	110.0(1)
C(23)	C(26)	C(27)	110.3(1)
N(3)	C(27)	C(28)	111.7(1)
O(3)	C(28)	C(27)	109.2(1)
C(27)	C(28)	C(33)	108.7(1)
N(4)	C(31)	C(32)	123.9(2)
C(28)	C(33)	C(32)	120.7(1)
C(32)	C(33)	C(39)	118.6(1)
O(4)	C(35)	C(34)	124.6(1)
C(34)	C(35)	C(36)	120.7(1)
C(36)	C(37)	C(38)	120.9(1)
N(4)	C(38)	C(39)	123.4(1)
C(33)	C(39)	C(34)	123.3(1)
C(34)	C(39)	C(38)	119.5(1)
F(3)	B(1)	F(5)	108.9(2)
F(4)	B(1)	F(5)	109.0(2)
F(5)	B(1)	F(6)	108.4(2)
F(7)	B(2)	F(9)	107.6(2)
F(8)	B(2)	F(9)	109.6(2)
F(9)	B(2)	F(10)	110.5(2)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(8)	O(1)	H(1O)	106.5	C(28)	O(3)	H(3O)	101.6
H(5O)	O(5)	H(5O')	101.8	N(1)	C(1)	H(1a)	109.8
N(1)	C(1)	H(1b)	109.6	C(2)	C(1)	H(1a)	109.8
C(2)	C(1)	H(1b)	109.6	H(1a)	C(1)	H(1b)	109.6
C(1)	C(2)	H(2)	108.3	C(3)	C(2)	H(2)	108.1
C(9)	C(2)	H(2)	108.3	C(2)	C(3)	H(3)	109.7
C(4)	C(3)	H(3)	109.4	C(6)	C(3)	H(3)	110.0
C(3)	C(4)	H(4a)	109.5	C(3)	C(4)	H(4b)	109.5
C(5)	C(4)	H(4a)	109.3	C(5)	C(4)	H(4b)	109.2
H(4a)	C(4)	H(4b)	109.1	N(1)	C(5)	H(5a)	110.5
N(1)	C(5)	H(5b)	110.6	C(4)	C(5)	H(5a)	110.2
C(4)	C(5)	H(5b)	110.3	H(5a)	C(5)	H(5b)	109.1
C(3)	C(6)	H(6a)	109.0	C(3)	C(6)	H(6b)	108.9
C(7)	C(6)	H(6a)	109.4	C(7)	C(6)	H(6b)	109.4
H(6a)	C(6)	H(6b)	109.8	N(1)	C(7)	H(7)	107.9
C(6)	C(7)	H(7)	108.1	C(8)	C(7)	H(7)	108.2
O(1)	C(8)	H(8)	108.6	C(7)	C(8)	H(8)	108.8
C(13)	C(8)	H(8)	109.1	C(2)	C(9)	H(9)	118.2
C(10)	C(9)	H(9)	118.5	C(9)	C(10)	H(10a)	120.2
C(9)	C(10)	H(10b)	119.8	H(10a)	C(10)	H(10b)	120.0
N(2)	C(11)	H(11)	118.6	C(12)	C(11)	H(11)	117.9
C(11)	C(12)	H(12)	120.4	C(13)	C(12)	H(12)	119.9
C(15)	C(14)	H(15)	120.2	C(19)	C(14)	H(15)	119.8
C(15)	C(16)	H(16)	119.4	C(17)	C(16)	H(16)	119.9

Table 6. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(16)	C(17)	H(17)	120.0	C(18)	C(17)	H(17)	119.6
O(2)	C(20)	H(20a)	109.8	O(2)	C(20)	H(20b)	109.7
O(2)	C(20)	H(20c)	109.7	H(20a)	C(20)	H(20b)	109.3
H(20a)	C(20)	H(20c)	109.4	H(20b)	C(20)	H(20c)	109.0
N(3)	C(21)	H(21a)	109.8	N(3)	C(21)	H(21b)	109.9
C(22)	C(21)	H(21a)	110.0	C(22)	C(21)	H(21b)	110.2
H(21a)	C(21)	H(21b)	109.4	C(21)	C(22)	H(22)	107.7
C(23)	C(22)	H(22)	107.6	C(29)	C(22)	H(22)	107.7
C(22)	C(23)	H(23)	109.9	C(24)	C(23)	H(23)	110.0
C(26)	C(23)	H(23)	109.4	C(23)	C(24)	H(24a)	109.2
C(23)	C(24)	H(24b)	109.3	C(25)	C(24)	H(24a)	109.2
C(25)	C(24)	H(24b)	109.3	H(24a)	C(24)	H(24b)	109.8
N(3)	C(25)	H(25a)	110.1	N(3)	C(25)	H(25b)	110.0
C(24)	C(25)	H(25a)	110.5	C(24)	C(25)	H(25b)	110.3
H(25a)	C(25)	H(25b)	109.7	C(23)	C(26)	H(26a)	109.6
C(23)	C(26)	H(26b)	109.6	C(27)	C(26)	H(26a)	109.1
C(27)	C(26)	H(26b)	109.1	H(26a)	C(26)	H(26b)	109.2
N(3)	C(27)	H(27)	108.2	C(26)	C(27)	H(27)	108.0
C(28)	C(27)	H(27)	107.7	O(3)	C(28)	H(28)	109.4
C(27)	C(28)	H(28)	109.3	C(33)	C(28)	H(28)	108.8
C(22)	C(29)	H(29)	116.8	C(30)	C(29)	H(29)	116.7
C(29)	C(30)	H(30a)	120.1	C(29)	C(30)	H(30b)	120.0
H(30a)	C(30)	H(30b)	119.9	N(4)	C(31)	H(31)	117.7
C(32)	C(31)	H(31)	118.4	C(31)	C(32)	H(32)	120.0

Table 6. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(33)	C(32)	H(32)	120.4	C(35)	C(34)	H(34)	119.9
C(39)	C(34)	H(34)	120.2	C(35)	C(36)	H(36)	120.1
C(37)	C(36)	H(36)	119.7	C(36)	C(37)	H(37)	119.4
C(38)	C(37)	H(37)	119.6	O(4)	C(40)	H(40a)	109.2
O(4)	C(40)	H(40b)	109.3	O(4)	C(40)	H(40c)	109.2
H(40a)	C(40)	H(40b)	109.9	H(40a)	C(40)	H(40c)	109.5
H(40b)	C(40)	H(40c)	109.7				

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle
F(1)	N(1)	C(1)	C(2)	175.5(1)
F(1)	N(1)	C(7)	C(6)	177.4(1)
F(2)	N(3)	C(21)	C(22)	174.4(1)
F(2)	N(3)	C(27)	C(26)	177.1(1)
O(1)	C(8)	C(7)	N(1)	-76.4(2)
O(1)	C(8)	C(13)	C(12)	-13.6(2)
O(2)	C(15)	C(14)	C(19)	178.0(1)
O(3)	C(28)	C(27)	N(3)	-74.8(2)
O(3)	C(28)	C(33)	C(32)	-19.4(2)
O(4)	C(35)	C(34)	C(39)	179.8(1)
N(1)	C(1)	C(2)	C(3)	7.2(2)
N(1)	C(5)	C(4)	C(3)	7.8(2)
N(1)	C(7)	C(8)	C(13)	160.8(1)
N(2)	C(18)	C(17)	C(16)	178.7(2)
N(2)	C(18)	C(19)	C(14)	-177.5(1)
N(3)	C(21)	C(22)	C(29)	136.4(1)
N(3)	C(27)	C(26)	C(23)	3.1(2)
N(4)	C(31)	C(32)	C(33)	-0.2(3)
N(4)	C(38)	C(39)	C(33)	-0.6(2)
C(1)	N(1)	C(5)	C(4)	-68.5(2)
C(1)	N(1)	C(7)	C(8)	-171.9(1)
C(1)	C(2)	C(3)	C(6)	54.8(2)
C(2)	C(1)	N(1)	C(5)	60.1(2)
C(2)	C(3)	C(4)	C(5)	56.1(2)

atom	atom	atom	atom	angle
F(1)	N(1)	C(5)	C(4)	176.7(1)
F(1)	N(1)	C(7)	C(8)	-56.8(2)
F(2)	N(3)	C(25)	C(24)	175.5(1)
F(2)	N(3)	C(27)	C(28)	-56.6(2)
O(1)	C(8)	C(7)	C(6)	44.0(2)
O(1)	C(8)	C(13)	C(19)	164.1(1)
O(2)	C(15)	C(16)	C(17)	-176.7(2)
O(3)	C(28)	C(27)	C(26)	46.0(2)
O(3)	C(28)	C(33)	C(39)	157.6(1)
O(4)	C(35)	C(36)	C(37)	-179.5(2)
N(1)	C(1)	C(2)	C(9)	132.2(2)
N(1)	C(7)	C(6)	C(3)	2.1(2)
N(2)	C(11)	C(12)	C(13)	0.7(3)
N(2)	C(18)	C(19)	C(13)	1.2(2)
N(3)	C(21)	C(22)	C(23)	9.1(2)
N(3)	C(25)	C(24)	C(23)	9.2(2)
N(3)	C(27)	C(28)	C(33)	163.4(1)
N(4)	C(38)	C(37)	C(36)	179.8(2)
N(4)	C(38)	C(39)	C(34)	-179.3(1)
C(1)	N(1)	C(7)	C(6)	62.2(2)
C(1)	C(2)	C(3)	C(4)	-64.5(2)
C(1)	C(2)	C(9)	C(10)	137.5(2)
C(2)	C(1)	N(1)	C(7)	-68.7(2)
C(2)	C(3)	C(6)	C(7)	-60.8(2)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle
C(3)	C(2)	C(9)	C(10)	-101.2(3)
C(4)	C(3)	C(2)	C(9)	172.9(2)
C(4)	C(5)	N(1)	C(7)	57.6(2)
C(5)	N(1)	C(7)	C(8)	62.2(2)
C(6)	C(3)	C(2)	C(9)	-67.8(2)
C(7)	C(8)	C(13)	C(12)	107.0(2)
C(8)	C(13)	C(12)	C(11)	175.9(1)
C(8)	C(13)	C(19)	C(18)	-176.8(1)
C(11)	N(2)	C(18)	C(19)	-2.2(2)
C(12)	C(11)	N(2)	C(18)	1.3(3)
C(12)	C(13)	C(19)	C(18)	0.9(2)
C(13)	C(19)	C(18)	C(17)	-178.5(1)
C(14)	C(15)	C(16)	C(17)	2.3(3)
C(15)	C(14)	C(19)	C(18)	-1.6(2)
C(16)	C(15)	O(2)	C(20)	169.8(1)
C(16)	C(17)	C(18)	C(19)	-1.6(2)
C(21)	N(3)	C(27)	C(26)	62.3(1)
C(21)	C(22)	C(23)	C(24)	-65.0(2)
C(21)	C(22)	C(29)	C(30)	-138.0(2)
C(22)	C(21)	N(3)	C(27)	-70.1(2)
C(22)	C(23)	C(26)	C(27)	-61.3(2)
C(23)	C(26)	C(27)	C(28)	-120.9(1)
C(24)	C(23)	C(26)	C(27)	57.4(2)
C(25)	N(3)	C(27)	C(26)	-64.3(2)
C(3)	C(6)	C(7)	C(8)	-121.6(2)
C(4)	C(3)	C(6)	C(7)	57.8(2)
C(5)	N(1)	C(7)	C(6)	-63.6(2)
C(5)	C(4)	C(3)	C(6)	-64.4(2)
C(6)	C(7)	C(8)	C(13)	-78.7(2)
C(7)	C(8)	C(13)	C(19)	-75.4(2)
C(8)	C(13)	C(19)	C(14)	1.7(2)
C(11)	N(2)	C(18)	C(17)	177.4(2)
C(11)	C(12)	C(13)	C(19)	-1.8(2)
C(12)	C(13)	C(19)	C(14)	179.4(1)
C(13)	C(19)	C(14)	C(15)	179.9(1)
C(14)	C(15)	O(2)	C(20)	-9.2(2)
C(14)	C(19)	C(18)	C(17)	2.9(2)
C(15)	C(16)	C(17)	C(18)	-1.0(3)
C(16)	C(15)	C(14)	C(19)	-1.0(2)
C(21)	N(3)	C(25)	C(24)	-69.2(2)
C(21)	N(3)	C(27)	C(28)	-171.4(1)
C(21)	C(22)	C(23)	C(26)	53.7(2)
C(22)	C(21)	N(3)	C(25)	58.6(2)
C(22)	C(23)	C(24)	C(25)	54.7(2)
C(23)	C(22)	C(29)	C(30)	-15.4(3)
C(24)	C(23)	C(22)	C(29)	171.9(1)
C(24)	C(25)	N(3)	C(27)	56.8(2)
C(25)	N(3)	C(27)	C(28)	61.9(2)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle
C(25)	C(24)	C(23)	C(26)	-65.6(2)
C(26)	C(27)	C(28)	C(33)	-75.8(2)
C(27)	C(28)	C(33)	C(39)	-82.0(2)
C(28)	C(33)	C(39)	C(34)	3.1(2)
C(31)	N(4)	C(38)	C(37)	-178.6(2)
C(31)	C(32)	C(33)	C(39)	-1.1(2)
C(32)	C(33)	C(39)	C(34)	-179.9(1)
C(33)	C(39)	C(34)	C(35)	-178.6(1)
C(34)	C(35)	O(4)	C(40)	1.3(2)
C(34)	C(39)	C(38)	C(37)	-1.5(2)
C(35)	C(36)	C(37)	C(38)	-0.9(3)
C(36)	C(35)	C(34)	C(39)	1.0(2)
C(26)	C(23)	C(22)	C(29)	-69.4(2)
C(27)	C(28)	C(33)	C(32)	101.1(2)
C(28)	C(33)	C(32)	C(31)	175.9(1)
C(28)	C(33)	C(39)	C(38)	-175.5(1)
C(31)	N(4)	C(38)	C(39)	-0.7(2)
C(32)	C(31)	N(4)	C(38)	1.2(3)
C(32)	C(33)	C(39)	C(38)	1.5(2)
C(33)	C(39)	C(38)	C(37)	177.3(1)
C(34)	C(35)	C(36)	C(37)	-0.5(2)
C(35)	C(34)	C(39)	C(38)	0.0(2)
C(36)	C(35)	O(4)	C(40)	-179.8(2)
C(36)	C(37)	C(38)	C(39)	1.9(2)

Table 8. Non-bonded Contacts out to 3.60 Å

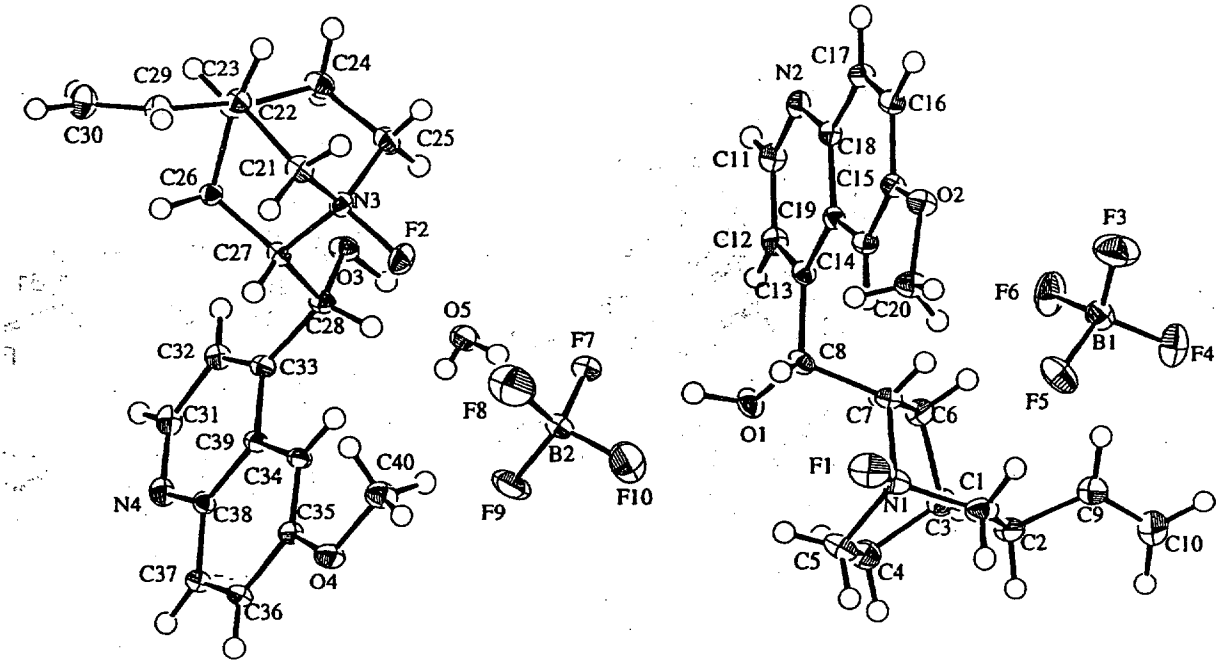
atom	atom	distance	atom	atom	distance
F(1)	C(10)[1)]	3.130(3)	F(1)	F(5)	3.542(2)
F(2)	F(8)	2.887(2)	F(2)	C(12)[1)]	3.376(2)
F(3)	C(10)[2)]	3.428(3)	F(3)	C(2)[2)]	3.494(3)
F(3)	C(23)[3)]	3.530(2)	F(3)	O(2)	3.566(3)
F(4)	C(23)[3)]	3.235(2)	F(4)	C(26)[3)]	3.427(2)
F(4)	C(24)[3)]	3.450(2)	F(4)	C(20)[4)]	3.470(2)
F(4)	C(4)[2)]	3.486(3)	F(4)	C(30)[5)]	3.566(2)
F(5)	C(1)	3.197(3)	F(5)	C(26)[3)]	3.367(2)
F(5)	C(23)[3)]	3.425(2)	F(5)	C(20)	3.509(3)
F(5)	C(30)[3)]	3.525(2)	F(6)	C(7)	3.286(2)
F(6)	C(14)	3.369(2)	F(6)	C(20)[4)]	3.385(3)
F(6)	C(31)[6)]	3.418(2)	F(7)	O(1)	2.782(2)
F(7)	O(5)	2.855(2)	F(7)	C(36)[7)]	3.214(2)
F(7)	C(8)	3.484(2)	F(7)	C(40)[4)]	3.519(2)
F(7)	C(37)[7)]	3.599(2)	F(8)	C(40)	3.505(3)
F(8)	C(37)[7)]	3.571(3)	F(9)	C(25)[8)]	3.146(2)
F(9)	C(24)[8)]	3.384(2)	F(9)	O(5)	3.429(2)
F(9)	C(22)[8)]	3.430(2)	F(9)	C(21)[8)]	3.458(2)
F(9)	C(11)[9)]	3.529(2)	F(9)	C(40)[4)]	3.585(3)
F(10)	O(1)	3.117(2)	F(10)	C(24)[8)]	3.452(3)
F(10)	C(8)	3.494(2)	O(1)	C(40)[4)]	3.107(2)
O(1)	B(2)	3.516(3)	O(2)	C(1)[10)]	3.272(2)
O(2)	C(31)[7)]	3.285(2)	O(2)	N(4)[7)]	3.386(2)
O(3)	O(5)	2.733(2)	O(3)	C(29)[4)]	3.289(2)

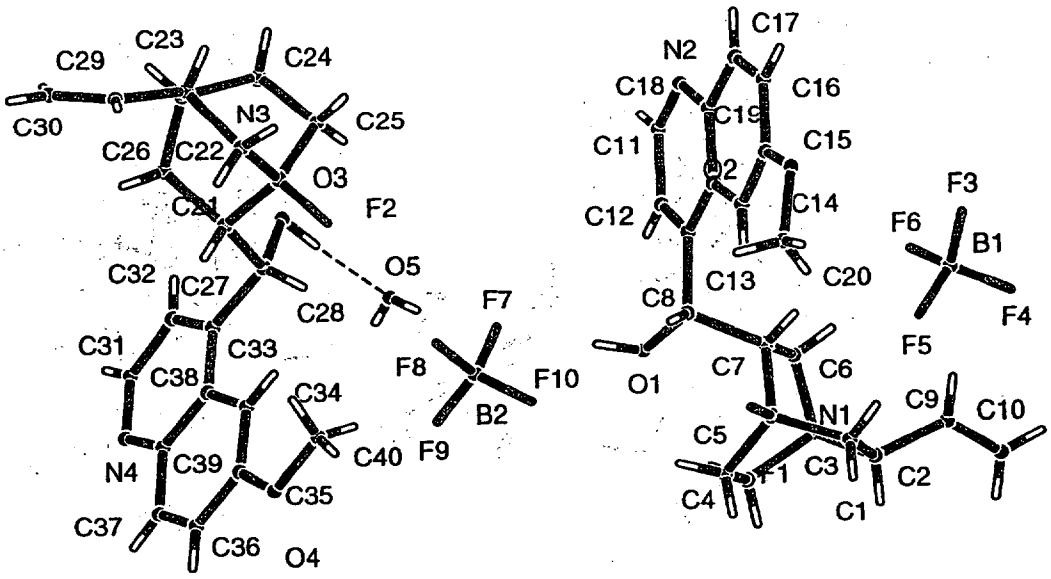
Table 8. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance
O(3)	C(30)$^{4)}$	3.514(2)
O(4)	C(21)$^{11)}$	3.411(2)
O(5)	C(28)	3.267(2)
O(5)	C(17)$^{9)}$	3.368(2)
N(2)	C(32)$^{6)}$	3.312(2)
N(2)	C(31)$^{6)}$	3.440(2)
N(4)	C(15)$^{8)}$	3.189(2)
N(4)	C(11)$^{9)}$	3.436(2)
N(4)	C(20)$^{8)}$	3.585(2)
C(9)	C(20)$^{4)}$	3.572(3)
C(11)	C(38)$^{6)}$	3.284(2)
C(12)	C(38)$^{6)}$	3.557(2)
C(14)	C(37)$^{7)}$	3.586(2)
C(15)	C(38)$^{7)}$	3.552(2)
C(17)	C(39)$^{7)}$	3.522(2)
C(18)	C(32)$^{6)}$	3.525(2)
C(19)	C(38)$^{7)}$	3.591(2)
O(4)	C(25)$^{8)}$	3.261(2)
O(5)	N(2)$^{9)}$	2.734(2)
O(5)	C(21)$^{4)}$	3.300(2)
O(5)	C(18)$^{9)}$	3.505(2)
N(2)	C(33)$^{6)}$	3.428(2)
N(2)	C(35)$^{7)}$	3.585(2)
N(4)	C(14)$^{8)}$	3.405(2)
N(4)	C(12)$^{9)}$	3.439(2)
C(5)	C(30)$^{8)}$	3.540(3)
C(10)	C(16)$^{12)}$	3.500(3)
C(11)	C(39)$^{6)}$	3.461(2)
C(14)	C(38)$^{7)}$	3.521(2)
C(15)	C(31)$^{7)}$	3.407(2)
C(16)	C(31)$^{7)}$	3.574(2)
C(18)	C(31)$^{6)}$	3.388(2)
C(19)	C(37)$^{7)}$	3.347(2)
C(25)	C(35)$^{7)}$	3.454(2)

Symmetry operations

(1)	X+1,Y,Z	(2)	-X-1,Y+1/2,-Z
(3)	X-1,Y,Z-1	(4)	X-1,Y,Z
(5)	X-2,Y,Z-1	(6)	-X,Y+1/2,-Z+1
(7)	-X+1,Y+1/2,-Z+1	(8)	-X+1,Y-1/2,-Z+1
(9)	-X,Y-1/2,-Z+1	(10)	-X,Y+1/2,-Z
(11)	-X+2,Y-1/2,-Z+1	(12)	-X-1,Y-1/2,-Z


C29
C23
C24
C30
C22
C25
C21
C26
N3
F2
C27
O3
C28
C32
C33
C31
C39
C34
C40
N4
C38
C35
C37
O4
C36
O5
F7
F8
B2
F9
F10
C17
N2
C16
C11
C18
C15
O2
C19
C12
C14
C13
C20
C8
O1
C7
C6
F1
N1
C1
C3
C2
C9
C10
C5
C4
F3
F6
B1
F4
F5


C23
C29
C24
C30
N3
C25
C26
C22
O3
C21
F2
O5
C32
C27
F7
C28
C31
C33
F8
F10
C38
B2
C34
F9
C40
N4
C39
C35
C37
C36
O4
C17
N2
C18
C16
C19
C11
C15
O2
F3
C12
C14
F6
B1
C13
C20
C8
F4
F5
C7
C6
O1
C9
C10
N1
C5
C3
C2
C4
C1