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Supporting Information

Journal of Organic Chemistry

General. Unless otherwise stated, all reactions were carried out under strictly anhydrous, air-free conditions. Formation of all ligand-metal complexes was done in a glove box under N₂. All solvents were dried and distilled by standard procedures. Flash chromatography was performed using silica gel (EM Science 60, 230-400 mesh), unless otherwise noted. The ¹H and ¹³C NMR spectra were acquired on a Varian Unity 400 MHz spectrometer. The ¹H (400 MHz) and ¹³C (101 MHz) chemical shifts are given in parts per million (δ) with respect to internal TMS or residual solvent peaks. The ¹³C NMR spectra were acquired with proton decoupling, and ¹⁹F nondecoupling. FTIR spectra were recorded on a Bruker IFS-55 spectrometer. Compounds **1b**,¹ **3b**,² and (*R*) **11**³ were synthesized by known literature methods. All other aziridines were generated by the method outlined below. All starting materials were purchased from Aldrich Chemical Company and used as received.

General procedure for the synthesis of acyl aziridines (1a-d): To a solution of 7-azabicyclo[4.1.0]heptane (1.26 g, 12.9 mmol) in diethyl ether (50 mL) was added triethylamine (2.58 mL, 18.5 mmol). The solution was cooled to -10 °C and the substituted benzoyl chloride (12.9 mmol) in diethyl ether (total volume 5 mL) added dropwise via syringe pump over 30 min. The reaction was warmed to room temperature and allowed to stir for 15 h. To the crude reaction mixture was added MgSO₄ and a minimal amount of decolorizing carbon and stirring was continued for 30 min. The solids were removed by filtration through Celite, and the filtrate was concentrated *in vacuo*. The crude product was purified by crystallization from diethyl ether/hexanes. Analytical samples were generated by a second recrystallization.

7-(4-Anisoyl)-7-azabicyclo[4.1.0]heptane (1a) was obtained as a white crystalline solid in 84% yield. ¹H NMR (CDCl₃) δ 7.96 (d, 2H), 6.93 (d, 2H), 3.85 (s, 3H), 2.73 (m, 2H), 2.04 (m, 2H), 1.90 (m, 2H), 1.55 (m, 2H), 1.35 (m, 2H) ppm; ¹³C NMR (CDCl₃) δ 179.6, 162.9, 131.0, 126.1, 113.4,

¹ Zhang, Z.; Scheffold, R. *Helv. Chim. Acta* **1993**, *76*, 2602-2615.

² Winstein, S.; Boschan, R. *J. Am. Chem. Soc.* **1950**, *72*, 4669-4677.

³ Nabeya, A.; Shigemoto, T.; Iwakura, Y. *J. Org. Chem.* **1975**, *40*, 3536.

55.3, 36.9, 23.9, 19.9 ppm; IR (CH₂Cl₂): 1662 cm⁻¹ (C=O). Anal. Calcd for C₁₄H₁₇NO₂: C, 72.69; H, 7.41; N, 6.06. Found: C, 72.45; H, 7.46; N, 6.02.

7-(4-Fluorobenzoyl)-7-azabicyclo[4.1.0]heptane (1c) was obtained as white needles in 79% yield. ¹H NMR (CDCl₃) δ 7.97 (q, 2H), 7.08 (t, 2H), 2.72 (m, 2H), 2.02(m, 2H), 1.89 (m, 2H), 1.52 (m, 2H), 1.33 (m, 2H) ppm; ¹³C NMR (CDCl₃) δ 179.0, 165.4 (d, *J*_{C-F} = 251.5 Hz), 131.4 (d, *J*_{C-F} = 9.1 Hz), 129.9, 115.4 (d, *J*_{C-F} = 21.9 Hz), 37.2, 23.9, 19.9 ppm; IR (CH₂Cl₂): 1666 cm⁻¹ (C=O.) Anal. Calcd for C₁₃H₁₄FNO: C, 71.20; H, 6.44; N, 6.39. Found; C, 71.24; H, 6.46; N, 6.41.

7-(4-trifluoromethyl)benzoyl-7-azabicyclo[4.1.0]heptane (1d) was obtained as white needles in 78% yield. ¹H NMR (CDCl₃) δ 8.04 (d, 2H), 7.66 (d, 2H), 2.74 (m, 2H), 2.01 (m, 2H), 1.88 (m, 2H), 1.51 (m, 2H), 1.33 (m, 2H) ppm; ¹³C NMR (CDCl₃) δ 178.6, 136.7, 133.7 (q, *J*_{C-F} = 32.5 Hz), 129.3, 125.3 (d, *J*_{C-F} = 7.5 Hz), 123.6 (d, *J*_{C-F} = 217 Hz), 37.4, 23.8, 19.8 ppm; IR (CH₂Cl₂): 1669 cm⁻¹ (C=O). Anal. Calcd for C₁₄H₁₄F₃NO: C, 58.31; H, 4.90; N, 4.86. Found: C, 58.26; H, 4.91; N, 4.89.

General procedure for TMSN₃ ring opening of acyl aziridines. To a solution of the preformed metal ligand complex (0.05 mmol) in either THF or CH₂Cl₂ (1 mL) was added TMSN₃ (133 μL, 1.0 mmol). The solution was stirred momentarily and the appropriate acyl aziridine was added (0.5 mmol). The reactions were monitored by TLC (25% EtOAc/hexanes) until all starting material was consumed or no further reaction progress was noted after 6 h. The crude reaction mixture was absorbed onto silica and chromatographed directly (5% EtOAc/hexanes) giving the compounds **2a-d** as white crystalline solids. Analytical samples were generated by recrystallization from Et₂O/hexanes.

Preparation of the Yb(biphenol)OTf. To a 1 mL solution of 2,2'-biphenol (9.3 mg, 0.05 mmol) in THF at 0 °C was added a 1.6 M solution of n-BuLi in hexanes (62.5 μL, 0.10 mmol). The solution was stirred 35 min with Yb(OTf)₃ (31.0 mg, 0.05 mmol), and allowed to settle. The mixture was then filtered through a plug of cotton to remove solid LiOTf. The filtrate was used without further purification.

Preparation of Zr(Cp)₂(SbF₆)₂. To a solution of bis(cyclopentadienyl)zirconium dichloride (14.6 mg, 0.05 mmol) in THF (1 mL) was added AgSbF₆ (34.3 mg, 0.10 mmol). The solution was

stirred 30 min at room temperature and the fluffy white precipitate of AgCl was removed by filtration. The filtrate was used as the active catalyst with out further purification.

***trans*-2-Azido-*N*-(4-anisoyl)cyclohexyl amine (2a).** ^1H NMR (CDCl_3) δ 7.75 (d, 2H), 6.91 (d, 2H), 6.09 (d, 1H), 3.95 (m, 1H), 3.83 (s, 3H), 3.27 (m, 1H), 2.14 (m, 2H), 1.73 (m, 1H), 1.50 (m, 1H), 1.33 (m, 4H) ppm; ^{13}C NMR (CDCl_3) δ 166.8, 162.2, 128.8, 126.9, 113.7, 63.9, 55.4, 52.9, 32.2, 30.7, 24.3, 24.2 ppm; IR (CH_2Cl_2): 3440 (N-H), 2100 (N_3), 1660 (C=O) cm^{-1} . Anal. Calcd for $\text{C}_{14}\text{H}_{18}\text{N}_4\text{O}_2$: C, 61.28; H, 6.62; N, 20.43. Found: C, 61.03; H, 6.54; N, 20.24.

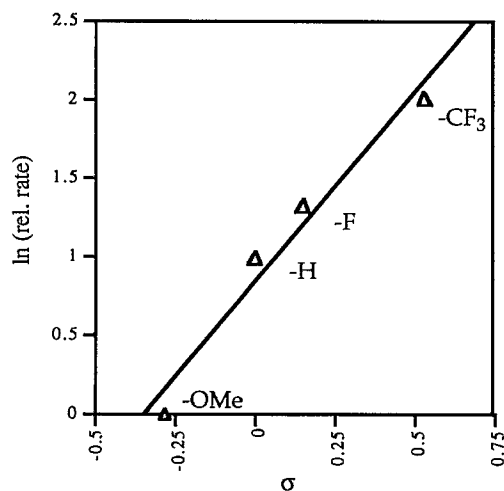
***trans*-2-Azido-*N*-benzoylcyclohexyl amine (2b).** ^1H NMR (CDCl_3) δ 7.79 (d, 2H), 7.48 (t, 1H), 7.39 (t, 2H), 6.50 (d, 1H), 3.96 (m, 1H), 3.30 (m, 1H), 2.10 (m, 2H), 1.80 (m, 1H), 1.71 (m, 1H), 1.44 (m, 1H), 1.32 (m, 3H) ppm; ^{13}C NMR (CDCl_3) δ 167.4, 134.8, 131.4, 128.4, 126.9, 63.8, 52.9, 32.0, 30.7, 24.2, 24.1 ppm; IR (CH_2Cl_2): 3440 (N-H), 2098 (N_3), 1664 (C=O) cm^{-1} . Anal. Calcd for $\text{C}_{13}\text{H}_{16}\text{N}_4\text{O}$: C, 63.90; H, 6.61; N, 22.94. Found: C, 63.83; H, 6.55; N, 22.90.

***trans*-2-Azido-*N*-(4-fluorobenzoyl)-cyclohexyl amine (2c).** ^1H NMR (CDCl_3) δ 7.79 (m, 2H), 7.07 (t, 2H), 6.42 (d, 1H), 3.94 (m, 1H), 3.29 (m, 1H), 2.12 (m, 2H), 1.82 (m, 1H), 1.73 (m, 1H), 1.47 (m, 1H), 1.34 (m, 3H) ppm; ^{13}C NMR (CDCl_3) δ 166.4, 164.5 (d, $J_{\text{C-F}} = 250.6$ Hz), 130.7 (d, $J_{\text{C-F}} = 3.2$ Hz), 129.3 (d, $J_{\text{C-F}} = 9.1$ Hz), 115.4 (d, $J_{\text{C-F}} = 21.8$ Hz), 63.8, 53.2, 32.1, 30.8, 24.3, 24.2 ppm; IR (CH_2Cl_2): 3440 (N-H), 2100 (N_3), 1669 (C=O) cm^{-1} . Anal. Calcd for $\text{C}_{13}\text{H}_{15}\text{FN}_4\text{O}$: C, 59.51; H, 5.77; N, 21.37. Found: C, 59.54; H, 5.73; N, 21.36.

***trans*-2-Azido-*N*-(4-(trifluoromethyl)benzoyl)cyclohexyl amine (2d).** ^1H NMR (CDCl_3) δ 7.88 (d, 2H), 7.70 (d, 2H), 6.10 (d, 1H), 3.97 (m, 1H), 3.28 (m, 1H), 2.18 (m, 2H), 1.75 (m, 1H), 1.67 (m, 1H), 1.52 (m, 1H), 1.33 (m, 3H) ppm; ^{13}C NMR (CDCl_3) δ 166.2, 137.8, 133.2 (q, $J_{\text{C-F}} = 32.2$ Hz), 127.4, 125.6 (q, $J_{\text{C-F}} = 3.6$ Hz), 123.6 (q, $J_{\text{C-F}} = 272.4$ Hz), 63.8, 53.3, 32.0, 30.7, 29.7, 24.2 ppm; IR(CH_2Cl_2): 3435 (N-H), 2100 (N_3), 1671 (C=O) cm^{-1} . Anal. Calcd for $\text{C}_{14}\text{H}_{15}\text{F}_3\text{N}_4\text{O}$: C, 53.83; H, 4.84; N, 17.95. Found: C, 53.72; H, 4.91; N, 17.83.

Competition Experiments for TMSN_3 addition. An equimolar amount (0.25 mmol each) of two of the aziridines (**1a-d**) were dissolved in CH_2Cl_2 followed by the addition of TMSN_3 and Yb(biphenol)OTf. The reactions were monitored by TLC or ^1H NMR (25% EtOAc/hexanes) until approximately 10% of the product appeared. The reactions were then flushed through a

plug of silica gel to remove the metal salts. The filtrate was analyzed by HPLC (5% EtOH/hexane, Chiralcel OD column) to determine the product ratios and percent conversion. All standards for the products were developed from the synthesis of compounds **2a-d** above. A plot of the $\ln(\text{relative rate})$ vs. σ_p is shown below and the ρ value found was 2.4.



General procedure for the isomerization of acyl aziridines. To a solution of the metal triflate [Cu(II), Zn(II) or Sn(II)] (0.05 mmol), and 200 μL DME in 1 mL of either THF or CH_2Cl_2 was added the appropriate acyl aziridine (0.5 mmol). The reactions were monitored by TLC (25% EtOAc/hexanes) until all starting material was consumed or no further reaction progress was noted after 6 hr. The crude reaction mixture was absorbed onto silica and chromatographed directly (5% EtOAc/hexanes) giving the compounds **3a-d** as low melting solids or oils.

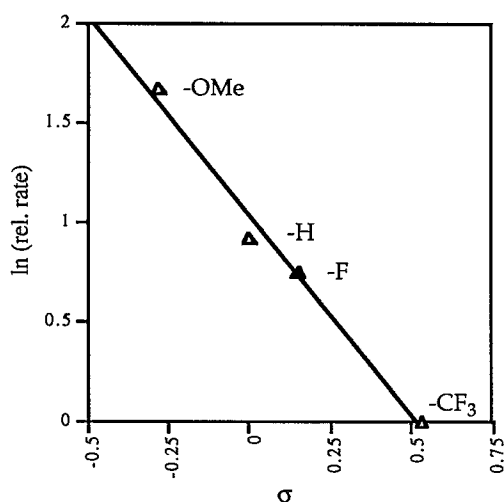
4-Anisoyl-*cis*-oxazoline (3a). ^1H NMR (CDCl_3) δ 7.84 (d, 2H), 6.87 (d, 2H), 4.61 (m, 1H), 4.09 (q, 1H), 3.81 (s, 3H), 1.92 (m, 3H), 1.63 (m, 1H), 1.56 (m, 2H), 1.38 (m, 2H) ppm; ^{13}C NMR (CDCl_3) δ 164.0, 161.9, 129.8, 120.9, 113.6, 78.7, 63.4, 55.3, 27.8, 26.3, 19.9, 19.2 ppm; IR (CH_2Cl_2): 1636 (C=N) cm^{-1} . Anal. Calcd for $\text{C}_{14}\text{H}_{17}\text{NO}_2$: C, 72.69; H, 7.41; N, 6.06. Found: C, 72.61; H, 7.45; N, 6.03.

4-Fluoro-*cis*-oxazoline (3c). ^1H NMR (CDCl_3) δ 7.96 (q, 2H), 7.08 (t, 2H), 4.68 (m, 1H), 4.11 (q, 1H), 1.88 (m, 3H), 1.62 (m, 1H), 1.55 (m, 2H), 1.41 (m, 2H) ppm; ^{13}C NMR (CDCl_3) δ 165.8,

163.3, 130.3 (d, $J_{C-F} = 9.1$ Hz), 124.5 (d, $J_{C-F} = 3.2$ Hz), 115.3 (d, $J_{C-F} = 21.9$ Hz), 78.9, 63.5, 27.7, 26.2, 19.8, 19.1 ppm; IR (CH₂Cl₂): 1642 (C=N) cm⁻¹. Anal. Calcd for C₁₃H₁₄FNO: C, 71.20; H, 6.44; N, 6.39. Found; C, 71.34; H, 6.46; N, 6.42.

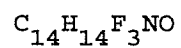
4-Trifluoromethyl-*cis*-oxazoline (3d). ¹H NMR (CDCl₃) δ 8.06 (d, 2H), 7.62 (d, 2H), 4.68 (m, 1H), 4.16 (m, 1H), 1.90 (m, 3H), 1.61 (m, 1H), 1.54 (m, 2H), 1.41 (m, 2H) ppm; ¹³C NMR (CDCl₃) δ 163.0, 132.7 (q, $J_{C-F} = 30.4$ Hz), 131.6, 125.2 (q, $J_{C-F} = 3.7$ Hz), 123.8 (q, $J_{C-F} = 275.9$ Hz), 79.3, 63.7, 27.5, 26.1, 19.7, 19.0 ppm; IR (CH₂Cl₂): 1644 (C=N) cm⁻¹. Anal. Calcd for C₁₄H₁₄F₄NO: C, 58.31; H, 4.90; N, 4.86. Found; C, 58.24; H, 4.93; N, 4.88.

General procedure for isomerization competition experiments between aziridines 1a-d. Equimolar amounts (0.25 mmol each) of two of the aziridines (1a-d) were dissolved in 1 mL THF/DME (20/1) followed by the addition Cu(OTf)₂ (0.05 mmol). The reactions were monitored by TLC (25% EtOAc/hexanes) until approximately 10% of the product oxazolines appeared. The reactions were then flushed through a plug of silica gel to remove the metal salts. The filtrate was analyzed by HPLC (5 % EtOH/hexane, Chiralcel OD column) to determine the product ratios and percent conversion. All standards for the products were developed from the synthesis of compounds 3a-d above. A plot of the ln(relative rate) vs. σ_p is shown below and the ρ value found was -2.0.



REFERENCE NUMBER: 98029

CRYSTAL STRUCTURE REPORT



Report prepared for:
W. Drury / Prof. T. Lectka

28 January 1998

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DATA COLLECTION

A crystal of the compound was attached to a glass fiber and mounted on the Siemens SMART system for a data collection at 173(2) K. An initial set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames are oriented such that orthogonal wedges of reciprocal space were surveyed. This produces orientation matrices determined from 12 reflections. Final cell constants are calculated from a set of 1643 strong reflections from the actual data collection. Please refer to Table 1 for additional crystal and refinement information.

The data collection technique used for this specimen is generally known as a hemisphere collection. Here a randomly oriented region of reciprocal space is surveyed to the extent of 1.3 hemispheres to a resolution of 0.84 Å. Three major swaths of frames are collected with 0.30° steps in ω . In the event the lattice is triclinic some additional sets of frames are collected to better model the absorption correction.

STRUCTURE SOLUTION AND REFINEMENT

The space group $P\bar{1}$ was determined based on systematic absences and intensity statistics.¹ A successful direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. All non-hydrogen atoms were refined with anisotropic displacement parameters unless stated otherwise. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters.

The material was found as expected. It was difficult to trim a sample suitable for data collection from the large crystals grown in NMR tubes. The soft crystals would acquire crack when cut. A small specimen was used that had a minimum of agitation.

Data collection and structure solution were conducted at the X-Ray Crystallographic Laboratory, 160 Kolthoff Hall, Chemistry Department, The University of Minnesota. All calculations were performed using SGI INDY R4400-SC or Pentium computers using the SHELXTL V5.0 suite of programs. All publications arising from this report MUST either 1) include Victor G. Young, Jr. as a coauthor or 2) acknowledge both Victor G. Young, Jr. and the X-Ray Crystallographic Laboratory.

¹. SHELXTL-Plus V5.0, Siemens Industrial Automation, Inc., Madison, WI.

Some equations of interest:

$$R_{int} = \frac{\sum |F_o^2 - \langle F_o^2 \rangle|}{\sum |F_o^2|}$$

$$R1 = \frac{\sum ||F_o^2| - |F_c^2||}{\sum |F_o^2|}$$

$$wR2 = \left[\frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right]^{1/2},$$

where $w = q/\sigma^2(F_o^2) + (a \cdot P)^2 + b \cdot P$

$$Goof = S = \left[\frac{\sum [w(F_o^2 - F_c^2)^2]}{(n-p)} \right]^{1/2}$$

Table 1. Crystal data, data collection, and solution and refinement for 98029.

Crystal Data

Empirical formula	$C_{14}H_{14}F_3NO$
Crystal Habit, color	Plate, Colorless
Crystal size	0.25 x 0.20 x 0.06 mm
Crystal system	Triclinic
Space group	$P\bar{1}$
	$a = 6.6677(7) \text{ \AA}$ $\alpha = 91.668(2)^\circ$
	$b = 8.2722(9) \text{ \AA}$ $\beta = 97.475(2)^\circ$
	$c = 12.1198(13) \text{ \AA}$ $\gamma = 108.101(2)^\circ$
Volume	$628.29(12) \text{ \AA}^3$
Z	2
Formula weight	269.26
Density (calculated)	1.423 Mg/m^3
Absorption coefficient	0.120 mm^{-1}
F(000)	280

Data Collection

Diffractionmeter	Siemens SMART Platform CCD
Wavelength	0.71073 \AA
Temperature	$173(2) \text{ K}$
θ range for data collection	1.70 to 25.08°
Index ranges	$-7 \leq h \leq 7$, $-9 \leq k \leq 9$, $0 \leq l \leq 14$
Reflections collected	3567
Independent reflections	2133 ($R_{int} = 0.0281$)

Solution and Refinement

System used	SHELXTL-V5.0
Solution	Direct methods
Refinement method	Full-matrix least-squares on F^2
Weighting scheme	$w = [\sigma^2(F^2) + (AP)^2 + (BP)]^{-1}$, where $P = (Fo^2 + 2Fc^2)/3$, $A = 0.12$, and $B = 0.0$
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.000 and 0.568
Data / restraints / parameters	2133 / 0 / 172
R indices ($I > 2\sigma(I)$) = 1416)	$R1 = 0.0705$, $wR2 = 0.1811$
R indices (all data)	$R1 = 0.1043$, $wR2 = 0.2055$
Goodness-of-fit on F^2	1.034
Largest diff. peak and hole	0.725 and -0.387 e\AA^{-3}

Table 2. Atomic coordinates [$\times 10^4$] and equivalent isotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for 98029. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
C(1)	5040(5)	7346(4)	341(2)	28(1)	1
O(1)	3221(3)	7333(3)	417(2)	42(1)	1
N(1)	5732(4)	7218(3)	-680(2)	27(1)	1
C(2)	5070(5)	8082(4)	-1624(2)	30(1)	1
C(3)	4372(5)	7085(5)	-2754(3)	37(1)	1
C(4)	6208(6)	6895(5)	-3307(3)	45(1)	1
C(5)	7976(5)	6603(5)	-2483(3)	38(1)	1
C(6)	8987(5)	8137(4)	-1653(3)	33(1)	1
C(7)	7370(5)	8631(4)	-1083(2)	30(1)	1
C(8)	6629(5)	7403(4)	1340(2)	26(1)	1
C(9)	6237(5)	7908(4)	2378(2)	29(1)	1
C(10)	7670(5)	7998(4)	3332(3)	31(1)	1
C(11)	9517(5)	7587(4)	3249(2)	28(1)	1
C(12)	9923(5)	7080(4)	2220(2)	28(1)	1
C(13)	8475(5)	6981(4)	1269(2)	27(1)	1
C(14)	11113(5)	7686(4)	4258(3)	35(1)	1
F(1)	10496(4)	8042(4)	5197(2)	85(1)	1
F(2)	11686(5)	6344(4)	4366(2)	103(1)	1
F(3)	12928(4)	8921(4)	4238(2)	93(1)	1

Table 3. Bond lengths [Å] and angles [°] for 98029.

C(1)-O(1)	1.225(4)	C(1)-N(1)	1.388(4)
C(1)-C(8)	1.492(4)	N(1)-C(2)	1.458(4)
N(1)-C(7)	1.475(4)	C(2)-C(7)	1.508(4)
C(2)-C(3)	1.518(4)	C(3)-C(4)	1.516(5)
C(4)-C(5)	1.527(5)	C(5)-C(6)	1.515(5)
C(6)-C(7)	1.502(4)	C(8)-C(13)	1.392(4)
C(8)-C(9)	1.395(4)	C(9)-C(10)	1.385(4)
C(10)-C(11)	1.390(4)	C(11)-C(12)	1.390(4)
C(11)-C(14)	1.496(4)	C(12)-C(13)	1.387(4)
C(14)-F(2)	1.286(4)	C(14)-F(1)	1.316(4)
C(14)-F(3)	1.322(4)		
O(1)-C(1)-N(1)	122.5(3)	O(1)-C(1)-C(8)	122.2(3)
N(1)-C(1)-C(8)	115.2(3)	C(1)-N(1)-C(2)	120.9(3)
C(1)-N(1)-C(7)	122.5(2)	C(2)-N(1)-C(7)	61.9(2)
N(1)-C(2)-C(7)	59.6(2)	N(1)-C(2)-C(3)	117.6(3)
C(7)-C(2)-C(3)	121.1(3)	C(4)-C(3)-C(2)	113.7(3)
C(3)-C(4)-C(5)	113.0(3)	C(6)-C(5)-C(4)	110.4(3)
C(7)-C(6)-C(5)	112.5(3)	N(1)-C(7)-C(6)	115.9(3)
N(1)-C(7)-C(2)	58.5(2)	C(6)-C(7)-C(2)	119.6(3)
C(13)-C(8)-C(9)	119.5(3)	C(13)-C(8)-C(1)	122.3(3)
C(9)-C(8)-C(1)	118.2(3)	C(10)-C(9)-C(8)	120.6(3)
C(9)-C(10)-C(11)	119.4(3)	C(10)-C(11)-C(12)	120.5(3)
C(10)-C(11)-C(14)	121.0(3)	C(12)-C(11)-C(14)	118.5(3)
C(13)-C(12)-C(11)	119.8(3)	C(12)-C(13)-C(8)	120.2(3)
F(2)-C(14)-F(1)	108.1(3)	F(2)-C(14)-F(3)	103.7(3)
F(1)-C(14)-F(3)	103.9(3)	F(2)-C(14)-C(11)	114.4(3)
F(1)-C(14)-C(11)	113.5(3)	F(3)-C(14)-C(11)	112.3(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for 98029. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [(ha)^2 U_{11} + \dots + 2hka b U_{12}]$

	U11	U22	U33	U23	U13	U12
C(1)	22(2)	34(2)	28(2)	-1(1)	6(1)	9(1)
O(1)	22(1)	74(2)	34(1)	-1(1)	2(1)	23(1)
N(1)	21(1)	37(2)	23(1)	1(1)	1(1)	10(1)
C(2)	25(2)	41(2)	27(2)	3(1)	1(1)	15(1)
C(3)	27(2)	53(2)	28(2)	5(2)	-6(1)	12(2)
C(4)	45(2)	58(2)	28(2)	-3(2)	3(2)	11(2)
C(5)	31(2)	51(2)	39(2)	-1(2)	12(2)	19(2)
C(6)	23(2)	45(2)	31(2)	5(1)	7(1)	10(1)
C(7)	24(2)	31(2)	30(2)	-1(1)	-1(1)	5(1)
C(8)	19(2)	34(2)	25(2)	-1(1)	4(1)	8(1)
C(9)	19(2)	38(2)	32(2)	-1(1)	5(1)	11(1)
C(10)	30(2)	37(2)	26(2)	-4(1)	7(1)	9(1)
C(11)	23(2)	31(2)	27(2)	2(1)	2(1)	7(1)
C(12)	22(2)	35(2)	28(2)	3(1)	6(1)	11(1)
C(13)	24(2)	35(2)	22(2)	-1(1)	4(1)	10(1)
C(14)	39(2)	35(2)	30(2)	-3(1)	1(2)	13(2)
F(1)	65(2)	175(3)	29(1)	-12(1)	-7(1)	66(2)
F(2)	156(3)	91(2)	68(2)	-28(1)	-67(2)	84(2)
F(3)	45(2)	130(3)	63(2)	27(2)	-23(1)	-17(2)

Table 5. Torsion angles [$^{\circ}$] for 98029.

O(1)-C(1)-N(1)-C(2)	39.1(4)	C(8)-C(1)-N(1)-C(2)	-144.2(3)
O(1)-C(1)-N(1)-C(7)	113.4(3)	C(8)-C(1)-N(1)-C(7)	-69.9(4)
C(1)-N(1)-C(2)-C(7)	112.9(3)	C(7)-N(1)-C(2)-C(7)	0.0
C(1)-N(1)-C(2)-C(3)	-135.5(3)	C(7)-N(1)-C(2)-C(3)	111.6(3)
N(1)-C(2)-C(3)-C(4)	-75.9(4)	C(7)-C(2)-C(3)-C(4)	-6.4(4)
C(2)-C(3)-C(4)-C(5)	38.5(4)	C(3)-C(4)-C(5)-C(6)	-63.5(4)
C(4)-C(5)-C(6)-C(7)	53.2(4)	C(1)-N(1)-C(7)-C(6)	139.2(3)
C(2)-N(1)-C(7)-C(6)	-110.3(3)	C(1)-N(1)-C(7)-C(2)	-110.4(3)
C(2)-N(1)-C(7)-C(2)	0.0	C(5)-C(6)-C(7)-N(1)	45.2(4)
C(5)-C(6)-C(7)-C(2)	-21.7(4)	N(1)-C(2)-C(7)-N(1)	0.0
C(3)-C(2)-C(7)-N(1)	-105.9(3)	N(1)-C(2)-C(7)-C(6)	103.9(3)
C(3)-C(2)-C(7)-C(6)	-2.0(4)	O(1)-C(1)-C(8)-C(13)	163.0(3)
N(1)-C(1)-C(8)-C(13)	-13.7(4)	O(1)-C(1)-C(8)-C(9)	-17.3(5)
N(1)-C(1)-C(8)-C(9)	165.9(3)	C(13)-C(8)-C(9)-C(10)	0.2(5)
C(1)-C(8)-C(9)-C(10)	-179.4(3)	C(8)-C(9)-C(10)-C(11)	0.3(5)
C(9)-C(10)-C(11)-C(12)	-0.4(5)	C(9)-C(10)-C(11)-C(14)	179.4(3)
C(10)-C(11)-C(12)-C(13)	-0.1(4)	C(14)-C(11)-C(12)-C(13)	-179.8(3)
C(11)-C(12)-C(13)-C(8)	0.7(4)	C(9)-C(8)-C(13)-C(12)	-0.7(4)
C(1)-C(8)-C(13)-C(12)	178.9(3)	C(10)-C(11)-C(14)-F(2)	130.2(4)
C(12)-C(11)-C(14)-F(2)	-50.1(4)	C(10)-C(11)-C(14)-F(1)	5.5(5)
C(12)-C(11)-C(14)-F(1)	-174.8(3)	C(10)-C(11)-C(14)-F(3)	-112.0(4)
C(12)-C(11)-C(14)-F(3)	67.8(4)		

Symmetry transformations used to generate equivalent atoms:

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 98029.

	x	y	z	U(eq)	SOF
H(2A)	4250(5)	8864(4)	-1457(2)	36	1
H(3A)	3397(5)	5935(5)	-2662(3)	45	1
H(3B)	3562(5)	7666(5)	-3252(3)	45	1
H(4A)	6815(6)	7938(5)	-3692(3)	54	1
H(4B)	5657(6)	5923(5)	-3879(3)	54	1
H(5A)	7376(5)	5579(5)	-2080(3)	46	1
H(5B)	9076(5)	6401(5)	-2894(3)	46	1
H(6A)	10025(5)	7877(4)	-1082(3)	39	1
H(6B)	9777(5)	9114(4)	-2045(3)	39	1
H(7A)	7897(5)	9730(4)	-608(2)	36	1
H(9A)	4977(5)	8193(4)	2430(2)	35	1
H(10A)	7393(5)	8338(4)	4037(3)	37	1
H(12A)	11190(5)	6803(4)	2168(2)	33	1
H(13A)	8743(5)	6623(4)	566(2)	32	1

