JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
J. Am. Chem. Soc., 1996, 118(8), 2101-2102, DOI:10.1021/ja953776m

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Synthetic Procedures and Characterization Data for New Compounds

1,5-bis(*p*-toluenesulfonyl)-1,5-diazacyclooctane.[1] To a freshly prepared solution of sodium methoxide (7 g Na in 1 L methanol) was added *N,N'*-bis(*p*-toluenesulfonyl)propane-1,3-diamine[2] (52.3 g, 0.14 mol). The solution was heated to reflux with stirring for 2 h. The solvent was removed under reduced pressure, and the solid residue was dissolved in 800 mL of fresh, dry DMF (Aldrich Sure-Seal). The DMF solution was heated under N_2 to 100 °C, and to this solution was added a 150 mL DMF solution of 1,3-bis(*p*-toluenesulfonyloxy)propane[2] (52.7 g, 0.14 mol) with stirring over a 1 h period. The reaction was allowed to proceed at 100-110 °C for an additional 2 h, followed by stirring at room temperature overnight. The product was precipitated by addition of 1 L of water added drop-wise, collected by filtration, and washed with 1 x 100 mL water, 1 x 100 mL absolute ethanol, and 2 x 100 mL diethyl ether (37.9 g, 65%). Mp 218° C (dec); ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.03 (quint, J = 6 Hz, 4H), 2.42 (s, 6H), 3.26 (t, J = 6 Hz, 8H), 7.30 (d, J = 8 Hz, 4H), 7.67 (d, J = 8 Hz, 4H) ppm; ^{13}C{^{1}H} NMR (50 MHz, $CDCl_3$) δ 21.5, 30.2, 47.1, 127.1, 129.8, 135.7, 143.5 ppm; FTIR (KBr, cm^{-1}) 2933, 2860, 1599, 1463, 1328, 1152, 1082, 919, 813, 710, 687, 642, 564, 548; HRCIMS $[M + H]^+$ = 423.1445, $[M + NH_4]^+$ = 440.1696; Anal. calcd for $C_{20}H_{26}N_2O_4S_2$: H, 6.20; C, 56.85; N, 6.63. Found: H, 6.21; C, 56.92; N, 6.60. Diazacyclooctane has in the past been prepared by the low yield reaction of 1,3-dibromopropane with hydrazine.[3] Besides the desired product, this reaction produces numerous side products, requiring multi-step isolation and purification procedures. We believe the ring closure procedure outlined above to be superior based upon its higher yield and the functional utility introduced by the *p*-toluenesulfonyl protecting groups.

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[1] 1,5-bis(*p*-toluenesulfonyl)-1,5-diazacyclooctane has been prepared by treating diazacyclooctane (prepared according to reference 3) with *p*-toluenesulfonyl chloride: Stetter, H.; Spangenberger, H. *Chem. Ber.* **1958**, *91*, 1982-1988.

[2] Searle, G. H.; Geue, R. J. *Aust. J. Chem.* **1984**, *37*, 959-970.

[3] Buhle, E. L.; Moore, A. M.; Wiselogle, F. Y. *J. Am. Chem. Soc.* **1943**, *65*, 29-32.

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1-*p*-toluenesulfonyl-1,5-diazacyclooctane. 1,5-bis(*p*-toluenesulfonyl)-1,5-diazacyclooctane (20.2 g, 0.048 mol) was treated with 33% HBr/glacial acetic acid at 50 °C for 4 h. The reaction mixture was cooled to 0 °C, and 800 mL of diethyl ether was added, forming a white precipitate. The solid was collected by filtration and was dissolved in 200 mL of water, followed by treatment with NaOH until the pH was ~13-14. The aqueous solution was extracted with 3 x 200 mL of diethyl ether. The ether fractions were combined, dried over $MgSO_4$, and the solvent was removed under reduced pressure. The colorless oil which remained was dried under vacuum, leaving the product as a white solid (4.3 g, 34%). ^{1}H NMR (300 MHz, CD_3CN) δ 1.67 (quint, J = 6 Hz, 4H), 2.39 (s, 3H), 2.81 (t, J = 6 Hz, 4H), 3.16 (t, J = 6 Hz, 4H), 7.36 (d, J = 8 Hz, 2H), 7.65 (d, J = 8 Hz, 2H) ppm; ^{13}C{^{1}H} NMR (75 MHz, CD_3CN) δ 21.5, 31.4, 48.1, 48.3, 127.9, 130.6, 136.7, 144.3 ppm; FTIR (KBr, cm^{-1}) 2926, 2854, 1597, 1493, 1457, 1372, 1332, 1305, 1158, 1090, 991, 848, 818, 714, 692, 645, 548; HREIMS M^+ = 268.1246; Anal. calcd for $C_{13}H_{20}N_2O_2S$: H, 7.51; C, 58.18; N, 10.44. Found: H, 7.49; C, 58.17; N, 10.47.

1-isopropyl-5-*p*-toluenesulfonyl-1,5-diazacyclooctane. To a 30 mL acetonitrile solution of 1-*p*-toluenesulfonyl -1,5-diazacyclooctane (2.64 g, 9.84 mmol) was added 2-bromopropane (2.50 g, 20 mmol), Na_2CO_3 (3 g, 30 mmol) and 5 mg of Bu_4NBr as a phase transfer catalyst. The reaction mixture was heated under nitrogen to reflux for 18 h. The mixture was cooled to room temperature, filtered, and dried over $MgSO_4$. The solution was filtered again, and the solvent was removed under vacuum, leaving the product as a pale yellow solid (2.20 g, 72%). Mp 45° C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.94 (d, J = 7 Hz, 6H), 1.75 (quint, J = 6 Hz, 4H), 2.38 (s, 3H), 2.57 (t, J = 6 Hz, 4H), 2.79 (sept, J = 7 Hz, 1H), 3.28 (t, J = 6 Hz, 4H), 7.25 (d, J = 8 Hz, 2H), 7.66 (d, J = 8 Hz, 2H) ppm; ^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$) δ 18.5, 21.3, 30.3, 45.3, 48.7, 53.1, 126.6, 129.4, 137.2, 142.5 ppm; FTIR (KBr, cm^{-1}) 2955, 2790, 1599, 1453, 1335, 1266, 1225, 1157, 1091, 1070, 1050, 1001, 953, 924, 865, 820, 804, 765, 691, 645, 582; HREIMS M^+ = 310.1716; Anal. calcd for $C_{16}H_{26}N_2O_2S$: H, 8.44; C, 61.90; N, 9.02. Found: H, 8.53; C, 60.68; N, 8.98.

1-isopropyl-1,5-diazacyclooctane. To neat 1-isopropyl-5-*p*-toluenesulfonyl-1,5-diazacyclooctane (3.9 g, 12.6 mmol) was added 10 mL of conc. H_2SO_4. The reaction mixture was heated with stirring to 110 °C for 18 h. The reaction mixture was cooled in an ice bath to 0 °C, and

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cold 10 M NaOH was *carefully* added until the pH was ~12-13. The resulting slurry was extracted with 3 x 100 mL of diethyl ether, the ether extracts were combined and dried over $MgSO_4$, and the ether was removed under reduced pressure, leaving the product as a tan oil (1.1 g, 57%). ^{1}H NMR (300 MHz, benzene-d_6) δ 0.80 (d, J = 7 Hz, 6H), 1.35 (quint, J = 6 Hz, 4H), 2.34 (t, J = 6 Hz, 4H), 2.57 (sept, J = 7 Hz, 1H), 2.77 (t, J = 6 Hz, 4H), 3.24 (br s, 1H) ppm; ^{13}C{^{1}H} NMR (75 MHz, benzene-d_6) δ 18.8, 30.7, 48.5, 48.6, 55.0 ppm.

Sodium(1-isopropyl-5-ethylthiolato-1,5-diazacyclooctane), NaL^{iPrdacoS}. To a benzene solution of 1-isopropyl-1,5-diazacyclooctane (1.0 g, 6.4 mmol) was added thiirane (0.39 g, 6.4 mmol) under an atmosphere of N_2. The reaction mixture was stirred at 45 °C for 6 h, at which time the reaction was deemed complete by GC/MS analysis. The mixture was cooled to room temperature, and the solvent removed under vacuum, leaving the protonated ligand, L^{iPrdacoS}, as a yellowish oil. This oil was dissolved under N_2 in 20 mL THF, and to this stirred solution excess NaH was added until bubbling ceased. The solution was filtered, and the volume reduced to about 1 mL. Excess diethyl ether was added, forming a white precipitate which was collected by filtration, washed with diethyl ether, and dried under vacuum (0.75 g, 50%). ^{1}H NMR (500 MHz, CD_3OD) δ 1.07 (d, J = 6.5 Hz, 6H), 1.78 (quint, J = 5.5 Hz, 4H), 2.64 (t, J = 5.0 Hz, 2H), 2.6 - 2.8 (m, 7H), 2.94 (t, J = 5.5 Hz, 4H) ppm; ^{13}C{^{1}H} NMR (125 MHz, CD_3OD) δ 18.7, 22.9, 27.2, 50.5, 53.2, 56.9, 64.6 ppm.

[CuL^{iPrdacoS}]$_2$(CF_3SO_3) (1). Two equivalents of cupric triflate dissolved in methanol (0.122 g, 0.34 mmol) were treated with three equivalents of NaL^{iPrdacoS} in methanol (0.120 g, 0.50 mmol), immediately producing a deep purple-blue solution. This solution was added to excess diethyl ether, and cooled to -20° C, whereupon blue and white solids co-precipitated. The blue solid was extracted with dichloromethane, and this solution was filtered to remove the sodium triflate. The dichloromethane was removed under reduced pressure, and pure, crystalline **1** was obtained by recrystallization from methanol/diethyl ether at -20° C (0.071 g, 60%). FTIR (KBr, cm^{-1}) 2954, 2881, 1463, 1393, 1265 [ν(OTf)], 1224, 1147 [ν(OTf)], 1098, 1029 [ν(OTf)], 997, 974, 706, 636 [ν(OTf)]; UV/vis/NIR [CH_2Cl_2, nm ($M^{-1}cm^{-1}$)] 298 (8500), 358 (2700), 602 (800), 786 (sh, 550), 1466 (br, 1200); EPR (50:50 methanol/toluene, 4.2K, 9.2132 MHz) g_1 = 2.010, g_2 = 2.046, g_3 = 2.204, A_2^{Cu} = 36.3 G, A_3^{Cu} = 49.9 G; Ion Spray MS (methanol) M^+ = 556.2,

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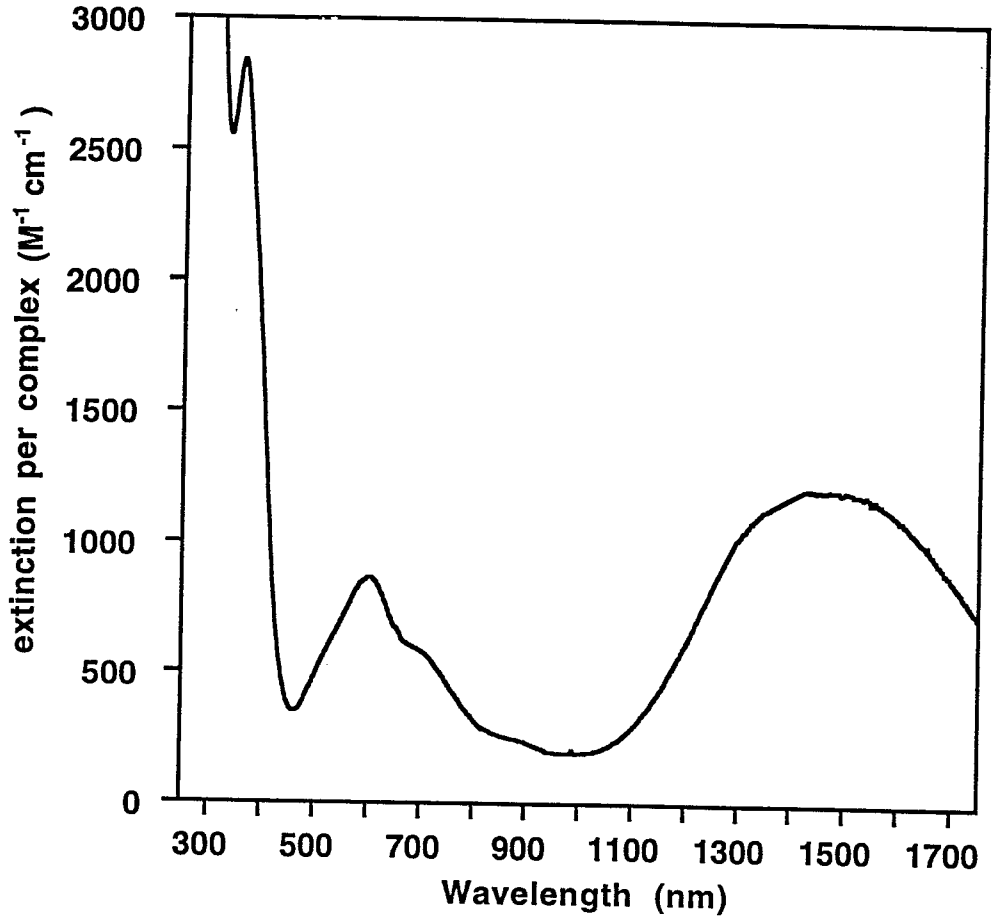
100%; Molar conductance (methanol) $\Lambda_M = 81$ $cm^{-1}mol^{-1}$ Ω^{-1}; Anal. calcd for $C_{23}H_{46}Cu_2F_3N_4O_3S_3$: H, 6.56; C, 39.08; N, 7.93. Found: H, 6.36; C, 38.37; N, 7.48.

Figure S1. Absorption spectrum of **1** in CH_3OH (250 nm - 820 nm) or CD_3OD (> 820 nm).



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CRYSTAL STRUCTURE REPORT

$C_{23}H_{46}Cu_2F_3N_4O_3S_3$

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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 - 30 frames. These initial sets of frames are oriented such that orthogonal wedges of reciprocal space were surveyed. This produces orientation matrices determined from 244 reflections. Final cell constants are calculated from a set of 8192 strong reflections from the actual data collection. Final cell constants reported in this manner usually are about one order of magnetude better in precision than reported from four-circle diffractometers. 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. This collection stratagy provides a high degree of redundancy. The redundant data provide good ψ input in the event an empirical absorption correction is applied (see Table 1).

STRUCTURE SOLUTION AND REFINEMENT

The space group $P2_1/n$ 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. Several full-matrix least squares / difference Fourier cycles were performed which located the remainder of the non-hydrogen atoms. 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 individual (or group if appropriate) isotropic displacement parameters.

The structure consists of two independent half-dimers. Both are numbered in similar fashion, except the second is primed. The thermal ellipsoid drawings are provided at 50% probability.

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 preformed 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.

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

Some equations of interest:

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

$$R1=\sum||F_o|-|F_c||/\sum|F_o|$$

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

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

$$GooF=S=[\sum[w(F_o^2-F_c^2)^2]/(n-p)^2$$

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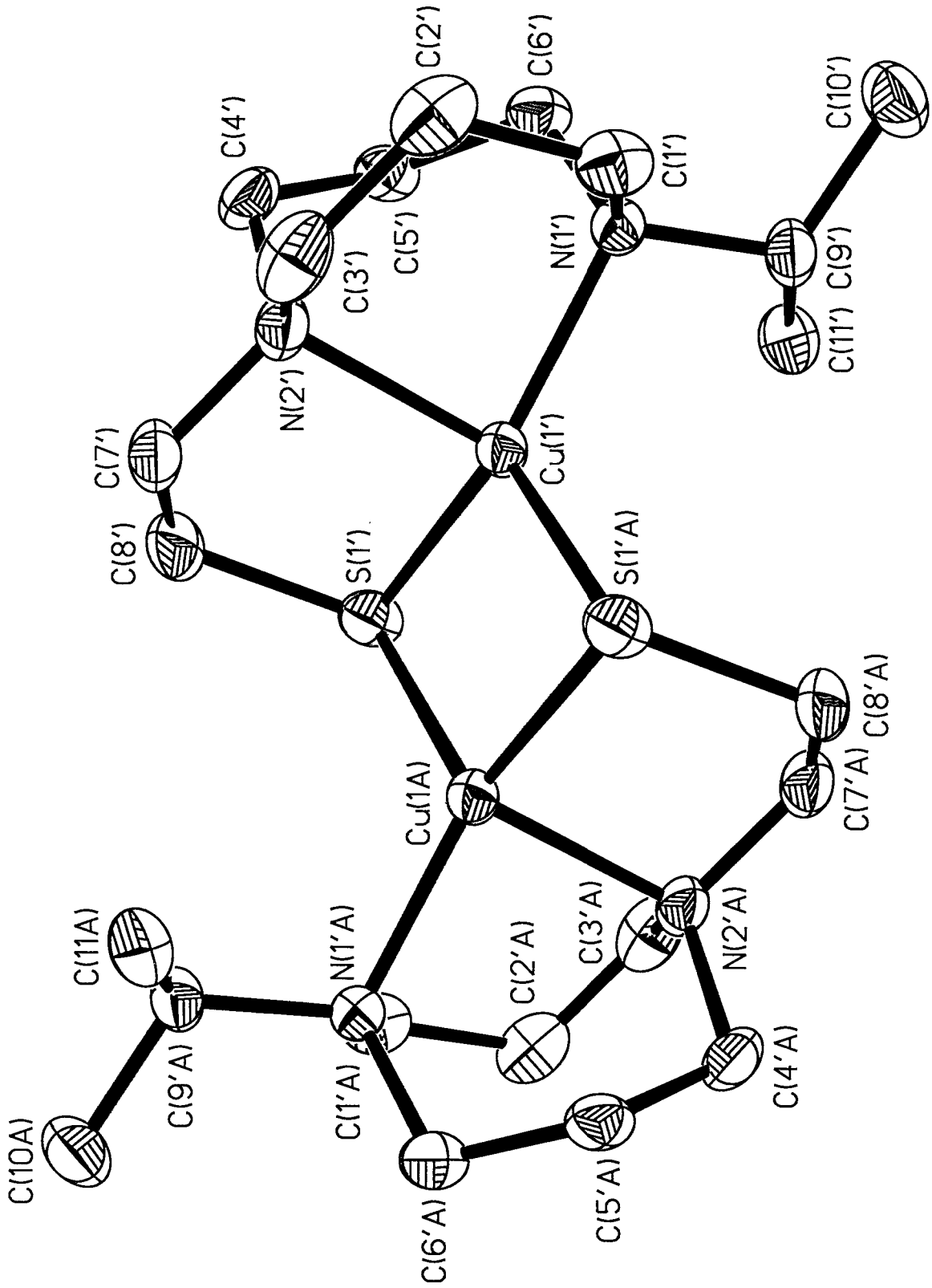
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C(6')
C(10')
C(4')
C(1')
C(5')
C(3')
N(1')
C(9')
C(11')
N(2')
C(7')
Cu(1')
C(8')
S(1')
S(1'A)
C(8'A)
C(7'A)
Cu(1A)
C(11A)
N(1'A)
C(3'A)
N(2'A)
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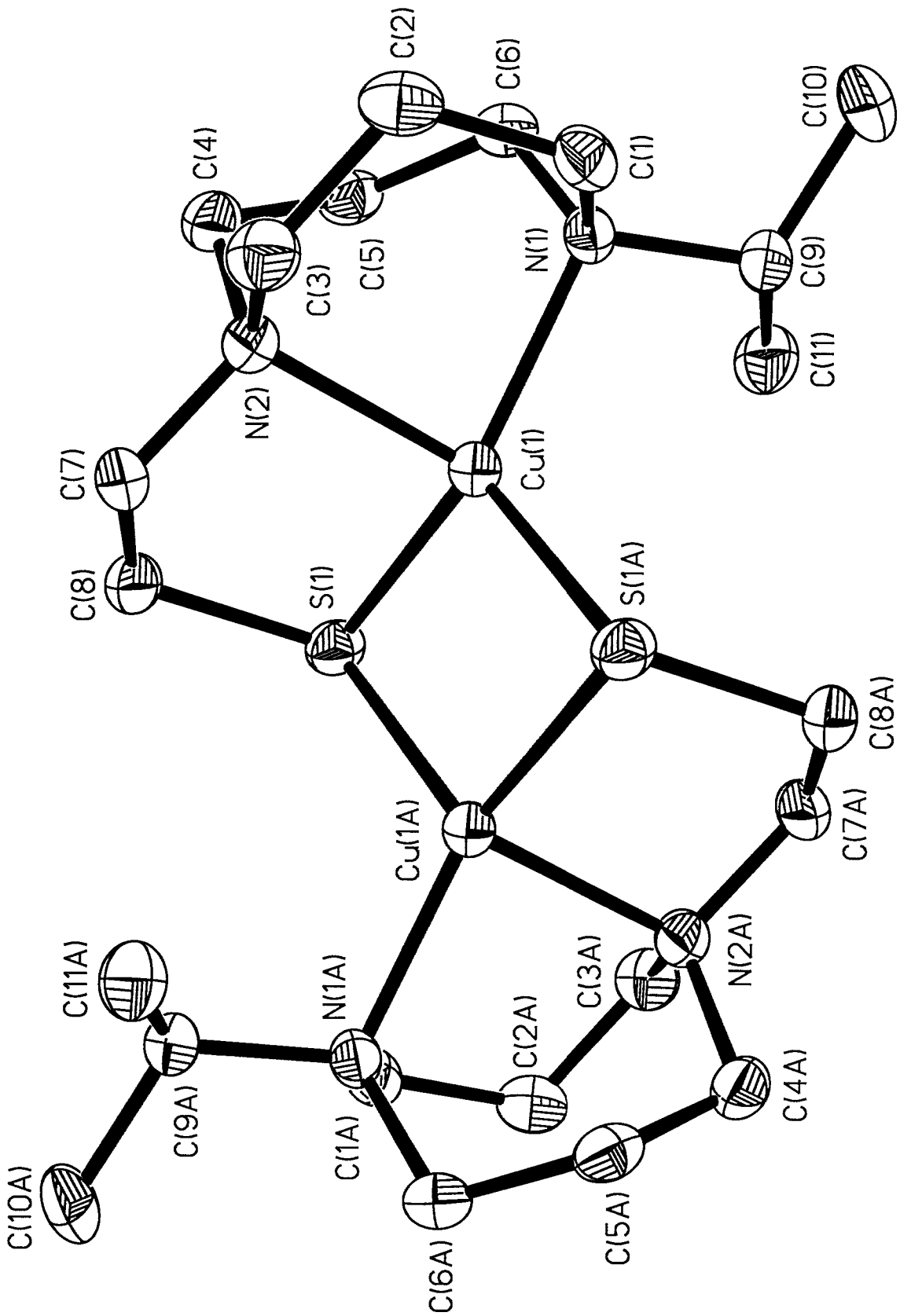
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C(6A)
C(5A)

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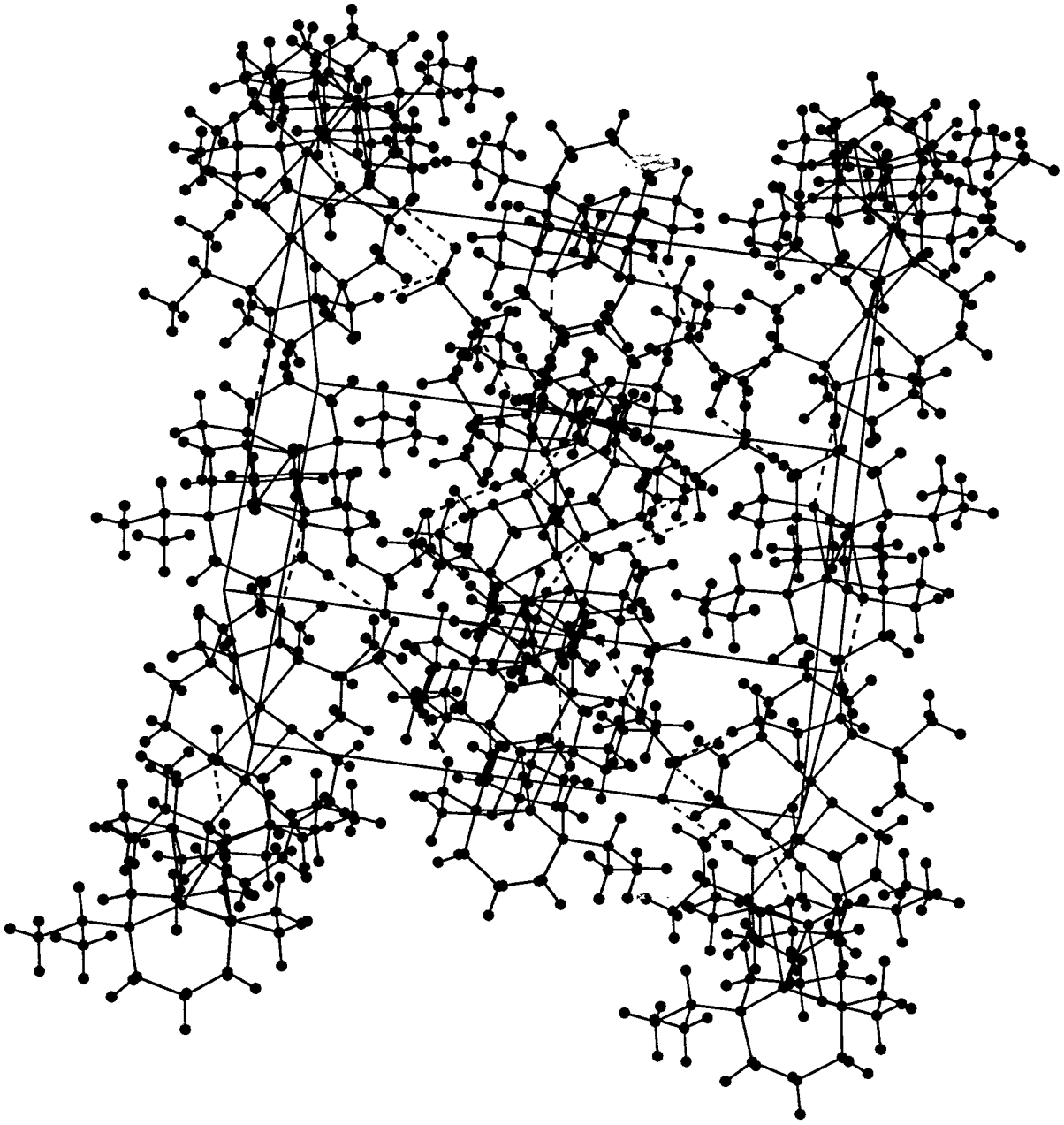

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Table 1. Crystal data and structure refinement for 95148.

Empirical formula	$C_{23}H_{46}Cu_2F_3N_4O_3S_3$
Formula weight	706.90
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 12.4726(3) Å α = 90° b = 17.9093(5) Å β = 91.7860(10)° c = 13.0768(3) Å γ = 90°
Volume	2919.62(13) $Å^3$
Z	4
Density (calculated)	1.608 Mg/m^3
Absorption coefficient	1.723 mm^{-1}
F(000)	1476
Crystal size	0.40 x 0.15 x 0.13 mm
θ range for data collection	1.93 to 25.07°
Index ranges	$-12 \le h \le 14$, $-17 \le k \le 21$, $-15 \le \ell \le 15$
Reflections collected	14799
Independent reflections	5149 (R_{int} = 0.0509)
Absorption correction	Semi-empirical
Max. and min. transmission	0.59570 and 0.51790
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5149 / 0 / 393
Goodness-of-fit on F^2	1.130
Final R indices [$I>2\sigma(I)$]	R1 = 0.0444, wR2 = 0.0867
R indices (all data)	R1 = 0.0630, wR2 = 0.0944
Largest diff. peak and hole	0.379 and -0.540 $eÅ^{-3}$

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Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 95148. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
Cu(1)	9080(1)	94(1)	658(1)	23(1)	1
N(1)	8111(2)	704(2)	1661(2)	22(1)	1
C(1)	8611(3)	594(2)	2700(3)	27(1)	1
C(2)	8525(3)	-188(2)	3135(3)	30(1)	1
C(3)	9054(3)	-814(2)	2545(3)	30(1)	1
N(2)	8638(3)	-880(2)	1472(2)	25(1)	1
C(4)	7453(3)	-987(2)	1429(3)	29(1)	1
C(5)	6852(3)	-306(2)	1016(3)	28(1)	1
C(6)	6980(3)	427(2)	1594(3)	26(1)	1
C(7)	9211(4)	-1497(2)	972(3)	33(1)	1
C(8)	9034(4)	-1504(2)	-179(3)	34(1)	1
S(1)	9224(1)	-585(1)	-780(1)	26(1)	1
C(9)	8190(3)	1522(2)	1395(3)	30(1)	1
C(10)	7503(4)	2041(2)	2041(4)	40(1)	1
C(11)	7921(4)	1640(2)	263(3)	37(1)	1
Cu(1')	5591(1)	176(1)	5943(1)	23(1)	1
N(1')	6576(2)	846(2)	6885(2)	24(1)	1
C(1')	7666(3)	788(2)	6457(3)	31(1)	1
C(2')	8187(3)	24(2)	6556(3)	37(1)	1
C(3')	7619(3)	-640(2)	6073(3)	38(1)	1
N(2')	6529(3)	-756(2)	6446(2)	29(1)	1
C(4')	6519(3)	-843(2)	7583(3)	31(1)	1
C(5')	6022(3)	-176(2)	8104(3)	29(1)	1
C(6')	6565(3)	579(2)	7976(3)	28(1)	1
C(7')	6019(4)	-1406(2)	5932(3)	39(1)	1
C(8')	4821(4)	-1423(2)	6053(3)	43(1)	1
S(1')	4143(1)	-544(1)	5714(1)	29(1)	1
C(9')	6205(3)	1648(2)	6794(3)	30(1)	1
C(10')	6849(4)	2208(2)	7458(3)	40(1)	1
C(11')	5019(4)	1700(2)	7007(3)	38(1)	1
S(101)	4381(1)	2345(1)	539(1)	40(1)	1
O(101)	4871(3)	1675(2)	159(3)	60(1)	1
O(102)	4625(3)	2511(2)	1595(2)	53(1)	1
O(103)	3290(3)	2465(2)	209(3)	56(1)	1
C(101)	5095(3)	3074(2)	-120(3)	32(1)	1
F(101)	6138(2)	3060(2)	120(2)	49(1)	1
F(102)	4974(2)	3016(2)	-1132(2)	53(1)	1
F(103)	4743(2)	3759(1)	124(2)	46(1)	1

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Table 3. Selected bond lengths [Å], angles [°], and torsion angles [°] for 95148.

Cu(1)-N(1)	2.115(3)	Cu(1)-N(2)	2.125(3)
Cu(1)-S(1)	2.2500(10)	Cu(1)-S(1)#1	2.2919(11)
Cu(1)-Cu(1)#1	2.9306(9)	N(1)-C(1)	1.490(5)
N(1)-C(6)	1.494(5)	N(1)-C(9)	1.511(5)
C(1)-C(2)	1.517(5)	C(2)-C(3)	1.522(5)
C(3)-N(2)	1.485(5)	N(2)-C(7)	1.479(5)
N(2)-C(4)	1.490(5)	C(4)-C(5)	1.522(5)
C(5)-C(6)	1.522(5)	C(7)-C(8)	1.514(6)
C(8)-S(1)	1.842(4)	S(1)-Cu(1)#1	2.2919(11)
C(9)-C(11)	1.522(6)	C(9)-C(10)	1.534(5)
Cu(1')-N(1')	2.091(3)	Cu(1')-N(2')	2.132(3)
Cu(1')-S(1')	2.2320(11)	Cu(1')-S(1')#2	2.2993(10)
Cu(1')-Cu(1')#2	2.9035(9)	N(1')-C(1')	1.489(5)
N(1')-C(6')	1.506(5)	N(1')-C(9')	1.512(5)
C(1')-C(2')	1.520(6)	C(2')-C(3')	1.512(6)
C(3')-N(2')	1.474(5)	N(2')-C(7')	1.478(5)
N(2')-C(4')	1.495(5)	C(4')-C(5')	1.517(5)
C(5')-C(6')	1.524(5)	C(7')-C(8')	1.507(6)
C(8')-S(1')	1.835(4)	S(1')-Cu(1')#2	2.2994(10)
C(9')-C(11')	1.517(6)	C(9')-C(10')	1.538(5)
S(101)-O(103)	1.430(3)	S(101)-O(102)	1.435(3)
S(101)-O(101)	1.443(4)	S(101)-C(101)	1.813(4)
C(101)-F(101)	1.329(5)	C(101)-F(102)	1.332(5)
C(101)-F(103)	1.345(5)		

N(1)-Cu(1)-N(2)	87.16(12)	N(1)-Cu(1)-S(1)	149.23(9)
N(2)-Cu(1)-S(1)	90.25(9)	N(1)-Cu(1)-S(1)#1	107.64(9)
N(2)-Cu(1)-S(1)#1	122.20(9)	S(1)-Cu(1)-S(1)#1	99.64(3)
N(1)-Cu(1)-Cu(1)#1	153.71(9)	N(2)-Cu(1)-Cu(1)#1	114.84(9)
S(1)-Cu(1)-Cu(1)#1	50.45(3)	S(1)#1-Cu(1)-Cu(1)#1	49.19(3)
C(1)-N(1)-C(6)	112.1(3)	C(1)-N(1)-C(9)	108.1(3)
C(6)-N(1)-C(9)	112.1(3)	C(1)-N(1)-Cu(1)	105.4(2)
C(6)-N(1)-Cu(1)	110.4(2)	C(9)-N(1)-Cu(1)	108.4(2)
N(1)-C(1)-C(2)	115.6(3)	C(1)-C(2)-C(3)	116.9(3)
N(2)-C(3)-C(2)	113.1(3)	C(7)-N(2)-C(3)	108.5(3)
C(7)-N(2)-C(4)	112.2(3)	C(3)-N(2)-C(4)	111.3(3)
C(7)-N(2)-Cu(1)	104.8(2)	C(3)-N(2)-Cu(1)	108.7(2)
C(4)-N(2)-Cu(1)	111.1(2)	N(2)-C(4)-C(5)	112.7(3)
C(6)-C(5)-C(4)	118.2(3)	N(1)-C(6)-C(5)	113.6(3)
N(2)-C(7)-C(8)	112.8(3)	C(7)-C(8)-S(1)	113.6(3)
C(8)-S(1)-Cu(1)	96.40(13)	C(8)-S(1)-Cu(1)#1	116.3(2)
Cu(1)-S(1)-Cu(1)#1	80.36(3)	N(1)-C(9)-C(11)	110.2(3)
N(1)-C(9)-C(10)	114.7(3)	C(11)-C(9)-C(10)	110.0(4)
N(1')-Cu(1')-N(2')	87.77(12)	N(1')-Cu(1')-S(1')	150.35(9)
N(2')-Cu(1')-S(1')	91.30(10)	N(1')-Cu(1')-S(1')#2	106.91(9)
N(2')-Cu(1')-S(1')#2	114.92(9)	S(1')-Cu(1')-S(1')#2	100.32(4)
N(1')-Cu(1')-Cu(1')#2	153.97(9)	N(2')-Cu(1')-Cu(1')#2	110.56(9)
S(1')-Cu(1')-Cu(1')#2	51.18(3)	S(1')#2-Cu(1')-Cu(1')#2	49.14(3)
C(1')-N(1')-C(6')	111.7(3)	C(1')-N(1')-C(9')	108.5(3)
C(6')-N(1')-C(9')	111.4(3)	C(1')-N(1')-Cu(1')	105.4(2)
C(6')-N(1')-Cu(1')	110.7(2)	C(9')-N(1')-Cu(1')	109.0(2)
N(1')-C(1')-C(2')	115.1(3)	C(3')-C(2')-C(1')	118.6(4)
N(2')-C(3')-C(2')	113.4(3)	C(3')-N(2')-C(7')	110.4(3)
C(3')-N(2')-C(4')	112.4(3)	C(7')-N(2')-C(4')	110.7(3)
C(3')-N(2')-Cu(1')	106.9(2)	C(7')-N(2')-Cu(1')	104.6(2)
C(4')-N(2')-Cu(1')	111.5(2)	N(2')-C(4')-C(5')	112.4(3)
C(4')-C(5')-C(6')	117.4(3)	N(1')-C(6')-C(5')	113.8(3)
N(2')-C(7')-C(8')	112.5(3)	C(7')-C(8')-S(1')	114.1(3)
C(8')-S(1')-Cu(1')	95.7(2)	C(8')-S(1')-Cu(1')#2	113.2(2)

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Cu(1')-S(1')-Cu(1')#2	79.68(4)	N(1')-C(9')-C(11')	110.0(3)
N(1')-C(9')-C(10')	114.9(3)	C(11')-C(9')-C(10')	110.6(3)
O(103)-S(101)-O(102)	115.5(2)	O(103)-S(101)-O(101)	115.4(2)
O(102)-S(101)-O(101)	115.1(2)	O(103)-S(101)-C(101)	103.1(2)
O(102)-S(101)-C(101)	102.5(2)	O(101)-S(101)-C(101)	102.4(2)
F(101)-C(101)-F(102)	108.2(3)	F(101)-C(101)-F(103)	106.6(3)
F(102)-C(101)-F(103)	106.2(3)	F(101)-C(101)-S(101)	111.5(3)
F(102)-C(101)-S(101)	112.0(3)	F(103)-C(101)-S(101)	112.0(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y,-z #2 -x+1,-y,-z+1

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Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for 95148. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$(ha^*)^2 U_{11} + ... + 2hka^*b^* U_{12}$]

	U11	U22	U33	U23	U13	U12
Cu(1)	25(1)	20(1)	24(1)	-1(1)	4(1)	-2(1)
N(1)	26(2)	18(2)	23(2)	0(1)	1(1)	1(1)
C(1)	29(2)	26(2)	25(2)	-6(2)	0(2)	0(2)
C(2)	29(2)	42(2)	19(2)	1(2)	-1(2)	3(2)
C(3)	31(2)	31(2)	28(2)	6(2)	0(2)	4(2)
N(2)	30(2)	21(2)	25(2)	2(1)	6(1)	0(1)
C(4)	31(2)	26(2)	31(2)	-4(2)	6(2)	-8(2)
C(5)	22(2)	34(2)	28(2)	-1(2)	4(2)	-6(2)
C(6)	22(2)	31(2)	24(2)	1(2)	3(2)	1(2)
C(7)	41(3)	18(2)	40(2)	2(2)	15(2)	3(2)
C(8)	40(3)	23(2)	40(2)	-8(2)	14(2)	-9(2)
S(1)	25(1)	28(1)	24(1)	-3(1)	2(1)	-5(1)
C(9)	30(2)	20(2)	40(2)	-2(2)	8(2)	-3(2)
C(10)	52(3)	20(2)	48(3)	-7(2)	9(2)	4(2)
C(11)	41(3)	28(2)	41(3)	10(2)	5(2)	4(2)
Cu(1')	27(1)	20(1)	21(1)	1(1)	0(1)	-1(1)
N(1')	28(2)	22(2)	22(2)	1(1)	-1(1)	0(1)
C(1')	23(2)	41(2)	30(2)	3(2)	3(2)	-4(2)
C(2')	24(2)	49(3)	37(3)	10(2)	8(2)	6(2)
C(3')	39(3)	39(3)	35(2)	6(2)	12(2)	16(2)
N(2')	42(2)	21(2)	23(2)	0(1)	0(2)	8(2)
C(4')	35(2)	29(2)	28(2)	10(2)	-4(2)	3(2)
C(5')	35(2)	34(2)	18(2)	1(2)	1(2)	-5(2)
C(6')	29(2)	33(2)	23(2)	0(2)	-1(2)	-3(2)
C(7')	70(3)	20(2)	27(2)	-1(2)	-5(2)	7(2)
C(8')	70(4)	22(2)	35(3)	8(2)	-9(2)	-13(2)
S(1')	34(1)	32(1)	21(1)	2(1)	2(1)	-10(1)
C(9')	45(3)	19(2)	27(2)	1(2)	0(2)	-1(2)
C(10')	58(3)	28(2)	35(3)	-6(2)	1(2)	-8(2)
C(11')	47(3)	26(2)	42(3)	-7(2)	-2(2)	6(2)
S(101)	42(1)	34(1)	45(1)	0(1)	4(1)	-13(1)
O(101)	69(3)	29(2)	81(3)	1(2)	5(2)	-3(2)
O(102)	62(2)	60(2)	35(2)	10(2)	6(2)	-23(2)
O(103)	37(2)	65(2)	67(2)	-19(2)	2(2)	-17(2)
C(101)	37(3)	31(2)	29(2)	-1(2)	6(2)	3(2)
F(101)	33(2)	49(2)	64(2)	16(1)	7(1)	-5(1)
F(102)	75(2)	54(2)	30(1)	1(1)	6(1)	10(2)
F(103)	59(2)	27(1)	53(2)	-3(1)	8(1)	3(1)

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Table 5. Torsion angles [°] for 95148.

N(2)-Cu(1)-N(1)-C(1)	62.5(2)	S(1)-Cu(1)-N(1)-C(1)	148.2(2)
S(1)#1-Cu(1)-N(1)-C(1)	-60.5(2)	Cu(1)#1-Cu(1)-N(1)-C(1)	-85.7(3)
N(2)-Cu(1)-N(1)-C(6)	-58.8(2)	S(1)-Cu(1)-N(1)-C(6)	26.9(3)
S(1)#1-Cu(1)-N(1)-C(6)	178.2(2)	Cu(1)#1-Cu(1)-N(1)-C(6)	153.0(2)
N(2)-Cu(1)-N(1)-C(9)	178.0(2)	S(1)-Cu(1)-N(1)-C(9)	-96.3(3)
S(1)#1-Cu(1)-N(1)-C(9)	55.0(2)	Cu(1)#1-Cu(1)-N(1)-C(9)	29.8(4)
C(6)-N(1)-C(1)-C(2)	51.1(4)	C(9)-N(1)-C(1)-C(2)	175.2(3)
Cu(1)-N(1)-C(1)-C(2)	-69.1(3)	N(1)-C(1)-C(2)-C(3)	61.5(5)
C(1)-C(2)-C(3)-N(2)	-58.2(5)	C(2)-C(3)-N(2)-C(7)	179.6(3)
C(2)-C(3)-N(2)-C(4)	-56.4(4)	C(2)-C(3)-N(2)-Cu(1)	66.2(4)
N(1)-Cu(1)-N(2)-C(7)	-179.4(3)	S(1)-Cu(1)-N(2)-C(7)	31.3(2)
S(1)#1-Cu(1)-N(2)-C(7)	-70.3(3)	Cu(1)#1-Cu(1)-N(2)-C(7)	-14.3(3)
N(1)-Cu(1)-N(2)-C(3)	-63.6(2)	S(1)-Cu(1)-N(2)-C(3)	147.1(2)
S(1)#1-Cu(1)-N(2)-C(3)	45.5(3)	Cu(1)#1-Cu(1)-N(2)-C(3)	101.5(2)
N(1)-Cu(1)-N(2)-C(4)	59.2(2)	S(1)-Cu(1)-N(2)-C(4)	-90.1(2)
S(1)#1-Cu(1)-N(2)-C(4)	168.3(2)	Cu(1)#1-Cu(1)-N(2)-C(4)	-135.7(2)
C(7)-N(2)-C(4)-C(5)	-128.8(3)	C(3)-N(2)-C(4)-C(5)	109.4(4)
Cu(1)-N(2)-C(4)-C(5)	-11.9(4)	N(2)-C(4)-C(5)-C(6)	-60.3(4)
C(1)-N(1)-C(6)-C(5)	-105.2(4)	C(9)-N(1)-C(6)-C(5)	133.0(3)
Cu(1)-N(1)-C(6)-C(5)	12.0(4)	C(4)-C(5)-C(6)-N(1)	60.6(4)
C(3)-N(2)-C(7)-C(8)	-167.1(3)	C(4)-N(2)-C(7)-C(8)	69.5(4)
Cu(1)-N(2)-C(7)-C(8)	-51.2(4)	N(2)-C(7)-C(8)-S(1)	48.1(4)
C(7)-C(8)-S(1)-Cu(1)	-18.0(3)	C(7)-C(8)-S(1)-Cu(1)#1	64.4(3)
N(1)-Cu(1)-S(1)-C(8)	-91.9(2)	N(2)-Cu(1)-S(1)-C(8)	-7.1(2)
S(1)#1-Cu(1)-S(1)-C(8)	115.7(2)	Cu(1)#1-Cu(1)-S(1)-C(8)	115.7(2)
N(1)-Cu(1)-S(1)-Cu(1)#1	152.4(2)	N(2)-Cu(1)-S(1)-Cu(1)#1	-122.77(9)
S(1)#1-Cu(1)-S(1)-Cu(1)#1	0.0	Cu(1)#1-Cu(1)-S(1)-Cu(1)#1	0.0
C(1)-N(1)-C(9)-C(11)	167.5(3)	C(6)-N(1)-C(9)-C(11)	-68.4(4)
Cu(1)-N(1)-C(9)-C(11)	53.7(4)	C(1)-N(1)-C(9)-C(10)	-67.7(4)
C(6)-N(1)-C(9)-C(10)	56.3(4)	Cu(1)-N(1)-C(9)-C(10)	178.5(3)
N(2')-Cu(1')-N(1')-C(1')	63.7(2)	S(1')-Cu(1')-N(1')-C(1')	152.4(2)
S(1')#2-Cu(1')-N(1')-C(1')	-51.7(2)	Cu(1')#2-Cu(1')-N(1')-C(1')	-72.4(3)
N(2')-Cu(1')-N(1')-C(6')	-57.2(2)	S(1')-Cu(1')-N(1')-C(6')	31.5(3)
S(1')#2-Cu(1')-N(1')-C(6')	-172.6(2)	Cu(1')#2-Cu(1')-N(1')-C(6')	166.7(2)
N(2')-Cu(1')-N(1')-C(9')	180.0(2)	S(1')-Cu(1')-N(1')-C(9')	-91.3(3)
S(1')#2-Cu(1')-N(1')-C(9')	64.6(2)	Cu(1')#2-Cu(1')-N(1')-C(9')	43.9(4)
C(6')-N(1')-C(1')-C(2')	52.9(4)	C(9')-N(1')-C(1')-C(2')	176.1(3)
Cu(1')-N(1')-C(1')-C(2')	-67.3(4)	N(1')-C(1')-C(2')-C(3')	58.7(5)
C(1')-C(2')-C(3')-N(2')	-57.6(5)	C(2')-C(3')-N(2')-C(7')	179.2(3)
C(2')-C(3')-N(2')-C(4')	-56.6(4)	C(2')-C(3')-N(2')-Cu(1')	66.0(4)
N(1')-Cu(1')-N(2')-C(3')	-64.9(3)	S(1')-Cu(1')-N(2')-C(3')	144.8(2)
S(1')#2-Cu(1')-N(2')-C(3')	42.8(3)	Cu(1')#2-Cu(1')-N(2')-C(3')	96.2(2)
N(1')-Cu(1')-N(2')-C(7')	178.0(3)	S(1')-Cu(1')-N(2')-C(7')	27.6(2)
S(1')#2-Cu(1')-N(2')-C(7')	-74.4(3)	Cu(1')#2-Cu(1')-N(2')-C(7')	-21.0(3)
N(1')-Cu(1')-N(2')-C(4')	58.3(3)	S(1')-Cu(1')-N(2')-C(4')	-92.0(3)
S(1')#2-Cu(1')-N(2')-C(4')	165.9(2)	Cu(1')#2-Cu(1')-N(2')-C(4')	-140.6(2)
C(3')-N(2')-C(4')-C(5')	109.6(4)	C(7')-N(2')-C(4')-C(5')	-126.4(4)
Cu(1')-N(2')-C(4')-C(5')	-10.4(4)	N(2')-C(4')-C(5')-C(6')	-61.2(5)
C(1')-N(1')-C(6')-C(5')	-106.7(4)	C(9')-N(1')-C(6')-C(5')	131.8(4)
Cu(1')-N(1')-C(6')-C(5')	10.4(4)	C(4')-C(5')-C(6')-N(1')	62.3(5)
C(3')-N(2')-C(7')-C(8')	-163.8(3)	C(4')-N(2')-C(7')-C(8')	71.0(4)
Cu(1')-N(2')-C(7')-C(8')	-49.2(4)	N(2')-C(7')-C(8')-S(1')	50.8(4)
C(7')-C(8')-S(1')-Cu(1')	-22.7(3)	C(7')-C(8')-S(1')-Cu(1')#2	58.5(3)
N(1')-Cu(1')-S(1')-C(8')	-90.8(2)	N(2')-Cu(1')-S(1')-C(8')	-3.0(2)
S(1')#2-Cu(1')-S(1')-C(8')	112.6(2)	Cu(1')#2-Cu(1')-S(1')-C(8')	112.6(2)
N(1')-Cu(1')-S(1')-Cu(1')#2	156.6(2)	N(2')-Cu(1')-S(1')-Cu(1')#2	-115.63(9)
S(1')#2-Cu(1')-S(1')-Cu(1')#2	0.0	Cu(1')#2-Cu(1')-S(1')-Cu(1')#2	0.0
C(1')-N(1')-C(9')-C(11')	167.9(3)	C(6')-N(1')-C(9')-C(11')	-68.7(4)
Cu(1')-N(1')-C(9')-C(11')	53.6(4)	C(1')-N(1')-C(9')-C(10')	-66.5(4)

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C(6')-N(1')-C(9')-C(10')	56.8(4)	Cu(1')-N(1')-C(9')-C(10')	179.2(3)
O(103)-S(101)-C(101)-F(101)	178.4(3)	O(102)-S(101)-C(101)-F(101)	58.2(3)
O(101)-S(101)-C(101)-F(101)	-61.4(3)	O(103)-S(101)-C(101)-F(102)	-60.1(3)
O(102)-S(101)-C(101)-F(102)	179.6(3)	O(101)-S(101)-C(101)-F(102)	60.1(3)
O(103)-S(101)-C(101)-F(103)	59.1(3)	O(102)-S(101)-C(101)-F(103)	-61.2(3)
O(101)-S(101)-C(101)-F(103)	179.3(3)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y,-z #2 -x+1,-y,-z+1

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Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 95148.

	x	y	z	U(eq)
H(1A)	8273(3)	946(2)	3176(3)	28(11)
H(1B)	9380(3)	727(2)	2674(3)	32(11)
H(2A)	7754(3)	-311(2)	3187(3)	28(10)
H(2B)	8842(3)	-184(2)	3838(3)	55(14)
H(3A)	9837(3)	-723(2)	2541(3)	18(9)
H(3B)	8937(3)	-1293(2)	2902(3)	31(11)
H(4A)	7207(3)	-1100(2)	2125(3)	51(14)
H(4B)	7276(3)	-1423(2)	988(3)	24(10)
H(5A)	7077(3)	-221(2)	306(3)	24(10)
H(5B)	6078(3)	-431(2)	980(3)	24(10)
H(6A)	6525(3)	811(2)	1251(3)	46(13)
H(6B)	6719(3)	360(2)	2296(3)	19(9)
H(7A)	8964(4)	-1978(2)	1254(3)	22(10)
H(7B)	9988(4)	-1451(2)	1135(3)	32(11)
H(8A)	8296(4)	-1679(2)	-343(3)	37(12)
H(8B)	9538(4)	-1864(2)	-478(3)	40(12)
H(9A)	8955(3)	1674(2)	1515(3)	20(10)
H(10A)	7674(17)	1956(12)	2768(4)	55(15)
H(10B)	6742(4)	1935(11)	1900(17)	53(15)
H(10C)	7655(18)	2562(2)	1867(17)	80(18)
H(11A)	8326(16)	1284(10)	-142(3)	52(14)
H(11B)	8112(19)	2151(5)	69(5)	59(15)
H(11C)	7151(5)	1563(14)	134(5)	24(10)
H(1'A)	8139(3)	1159(2)	6806(3)	76(18)
H(1'B)	7616(3)	924(2)	5723(3)	34(11)
H(2'A)	8907(3)	57(2)	6260(3)	22(10)
H(2'B)	8296(3)	-84(2)	7294(3)	45(13)
H(3'A)	7575(3)	-569(2)	5322(3)	40(12)
H(3'B)	8051(3)	-1095(2)	6214(3)	38(12)
H(4'A)	7264(3)	-910(2)	7851(3)	12(9)
H(4'B)	6110(3)	-1298(2)	7752(3)	32(11)
H(5'A)	6002(3)	-285(2)	8846(3)	33(11)
H(5'B)	5270(3)	-130(2)	7847(3)	17(9)
H(6'A)	6189(3)	954(2)	8390(3)	34(11)
H(6'B)	7314(3)	545(2)	8247(3)	23(10)
H(7'A)	6335(4)	-1871(2)	6221(3)	39(12)
H(7'B)	6174(4)	-1391(2)	5195(3)	35(12)
H(8'A)	4671(4)	-1546(2)	6773(3)	41(12)
H(8'B)	4514(4)	-1827(2)	5618(3)	71(17)
H(9'A)	6291(3)	1800(2)	6065(3)	31(11)
H(10D)	7614(4)	2160(10)	7321(15)	55(15)
H(10E)	6734(16)	2104(9)	8183(3)	30(11)
H(10F)	6610(15)	2716(3)	7295(14)	53(15)
H(11D)	4624(4)	1328(10)	6593(15)	29(11)
H(11E)	4756(6)	2201(5)	6830(18)	61(15)
H(11F)	4909(4)	1604(14)	7734(5)	47(13)