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*Synthetic details and characterization data for new compounds*

**1,4-Diisopropyl-7-(2-pyridylmethyl)-1,4,7-triazacyclononane** ($L^{Py}$): To a solution of 1,4-diisopropyl-1,4,7-triazacyclononane [a] (0.718 g, 3.36 mmol) in dry $CH_3CN$ (10 mL) was added 2-picolyl chloride hydrochloride (0.553 g, 3.37 mmol), $Na_2CO_3$ (1.45 g), and *n*-$Bu_4NBr$ (30 mg). The resultant mixture was heated at reflux under an $N_2$ atmosphere for 8h, after which time it was cooled to room temperature and then poured into 1M NaOH (40 mL). The heterogenous mixture was extracted with $CHCl_3$ (3 x 30 mL) and the organic extracts dried ($MgSO_4$) and filtered. Removal of solvent under reduced pressure yielded an orange oil which was purified by vacuum distillation (118-122 °C, 0.05 torr) to afford the pure product as a light yellow oil (0.927 g, 90%). GC/MS: $t_R$ = 12.96 min; $^1H$ NMR (300 MHz, $CDCl_3$) δ 8.50-8.47 (m, 1H), 7.63-7.54 (m, 2H), 7.12-7.07 (m, 1H), 3.82 (s, 2H), 2.94-2.91 (m, 4H), 2.86 (septet, $J$ = 6.6 Hz, 2H), 2.63-2.60 (m, 4H), 2.56 (s, 4H), 0.93 (d, $J$ = 6.6 Hz, 12H) ppm; $^{13}C\{^1H\}$ NMR (75 MHz, $CDCl_3$) δ 161.0, 148.8, 136.2, 123.0, 121.6, 63.9, 55.6, 54.8, 52.8, 52.3, 18.3 ppm; HREIMS Calcd for $C_{18}H_{32}N_4$, 304.2627; Found 304.2628. Anal. Calcd for $C_{18}H_{32}N_4$: C, 71.01; H, 10.59; N, 18.40. Found: C, 70.14; H, 10.64; N, 17.74.

**$d_{14}$-1,4-Diisopropyl-7-(2-pyridylmethyl)-1,4,7-triazacyclononane** ($d_{14}$-$L^{Py}$): This ligand was prepared and isolated in an analogous fashion to $L^{Py}$ by substituting $d_{14}$-1,4-diisopropyl-1,4,7-triazacyclononane. Deuterium content (by $^1H$ NMR): > 98%. $^1H$ NMR (300 MHz, $CDCl_3$) δ 8.48 (d, $J$ = 4.8 Hz, 1H), 7.64-7.53 (m, 2H), 7.10 (m, 1H), 3.81 (s, 2H), 2.92 (t, $J$ = 4.5 Hz, 4H), 2.60 (t, J = 4.5 Hz, 4H), 2.56 (s; 4H) ppm; HREIMS calcd for $C_{18}H_{18}D_{14}N_4$ 332.3298; Found 332.3301.

**$d_2$-1,4-Diisopropyl-7-(2-pyridylmethyl)-1,4,7-triazacyclononane** ($d_2$-$L^{Py}$): To a stirred suspension of $LiAlD_4$ (0.108 g, 2.57 mmol) in THF (5 mL) was added a solution of $L^{PyO}$ (0.407 g, 1.28 mmol) in THF (10 mL). The suspension was stired at room temperature for 12 h, and then warmed on an oil bath to 55 °C and heated for 24 h. During this time small amounts of $LiAlD_4$ (50 mg) were added (3 x 50 mg). The mixture was then cooled in an ice bath and $D_2O$ (3 mL) added, followed by aqueous NaOH (3 M, 10 mL) and $CHCl_3$ (20 mL). The organic phase was separated, dried ($Na_2SO_4$) and evaporated. After heating at 100 °C under vacuum for 1 h to remove 1,4-diisopropyl-1,4,7-triazacyclononane, the product was isolated as a yellow oil by kugelrohr distillation as above for $L^{Py}$ (0.128 g, 33%). Deuterium content (by $^1H$ NMR): > 98%. $^1H$ NMR (300 MHz, $CDCl_3$) δ 8.50-8.47 (m, 1H), 7.63-7.54 (m, 2H), 7.12-7.07 (m, 1H), 2.94-2.91 (m, 4H), 2.86 (septet, $J$ = 6.6 Hz, 2H), 2.63-2.60 (m, 4H), 2.56 (s, 4H), 0.93 (d, $J$ = 6.6 Hz, 12H) ppm; LREIMS *m/z* 306 ($M^+$, 2%).

**Independent synthesis of 1,4-diisopropyl-7-(2-picolylamido)-1,4,7-triazacyclononane** ($L^{PyO}$): To a stirred suspension of 2-picolinoyl chloride hydrochloride

(0.347 g, 1.96 mmol) in $CH_2Cl_2$ (5 mL) was added a solution of 1,4-diisopropyl-1,4,7-triazacyclononane (0.220 g, 1.03 mmol) in $CH_2Cl_2$ (2 mL). After stirring for 30 min, aqueous NaOH (3M, 10 mL) was added and stirring continued for 1 h. The organic phase was then removed, the aqueous phase extracted with $CHCl_3$ (2 x 20 mL) and the combined organic phases dried ($Na_2SO_4$) and evaporated. Kugelrohr distillation (bp 172-178 °C, 0.05 torr) of the crude residue afforded the pure product as a yellow oil, 0.381 g (97%). GC/MS: $t_R$ = 14.98 min; $^1$H NMR (300 MHz, $CDCl_3$) δ 8.56-8.54 (m, 1H), 7.76-7.70 (m, 1H), 7.58-7.54 (m, 1H), 7.29-7.24 (m, 1H), 3.80-3.76 (m, 2H), 3.43-3.40 (m, 2H), 2.95-2.93 (m, 2H), 2.90-2.87 (m, 2H), 2.85 (heptet, $J$ = 6.6 Hz, 1H), 2.64 (heptet, $J$ = 6.6 Hz, 1H), 2.63-2.60 (m, 2H), 2.39-2.36 (m, 2H), 0.97 (d, $J$ = 6.6 Hz, 6H), 0.83 (d, $J$ = 6.6 Hz, 6H) ppm; $^{13}$C{$^1$H} NMR (75 MHz, $CDCl_3$) δ 169.2, 155.9, 148.3, 136.6, 123.7, 123.5, 55.3, 53.8, 53.4, 52.8, 52.4, 51.2, 50.4, 48.8, 18.3, 18.2 ppm; FTIR (neat) 1633 (amide I) cm$^{-1}$; HREIMS calcd for $C_{18}H_{30}N_4O$, 318.2419; found 318.2410.

**[L$^{Py}$Cu]CF$_3$SO$_3$:** To a solution of L$^{Py}$ (0.304 g, 1.0 mmol) in THF/$CH_3CN$ (1:4, 5 mL) was added [Cu($CH_3CN$)$_4$]$CF_3SO_3$ (0.353 g, 0.94 mmol) in $CH_3CN$ (3 mL), generating a deep orange solution. After stirring for 1 h, the solution was concentrated under vacuum and $Et_2O$ (15 mL) was added to induce deposition of the product as a yellow microcrystalline solid, which was filtered, washed with $Et_2O$ and dried under vacuum (0.461 g, 95%). UV-Vis (THF) [$\lambda_{max}$, nm (ε, M$^{-1}$cm$^{-1}$)] 360 (4100); FTIR (KBr, cm$^{-1}$) 3069, 2972, 1603, 1468, 1372, 1270 ($CF_3SO_3^-$), 1146 ($CF_3SO_3^-$), 1030 ($CF_3SO_3^-$), 953, 788, 639 ($CF_3SO_3^-$), 571, 516; FAB-MS (MNBA) *m/e* (relative intensity) 367 ([M-$CF_3SO_3$]$^+$, 100%); Anal. Calcd for $C_{19}H_{32}CuF_3NO_3S$: C, 44.13; H, 6.24; N, 10.83. Found: 43.77; H, 5.99; N, 10.71. Crystals suitable for X-ray diffraction were obtained from a solution of the complex in $CH_3CN/Et_2O$ which was maintained at -20 °C for several days. An analogous procedure was used to prepare [L$^{Py}$Cu]$ClO_4$ substituting [Cu($CH_3CN$)$_4$]$ClO_4$ as the Cu(I) source, which was isolated as a microcrystalline yellow solid in 82% yield. Anal. Calcd for $C_{19}H_{32}O_4ClCu$: C, 46.25; H, 6.90; N, 11.98. Found: C, 46.31; H, 6.88; N, 12.01.

**[L$^{iPr3}$Cu(pyridine)]ClO$_4$:** A solution of [L$^{iPr3}$Cu($CH_3CN$)]$ClO_4$ (0.075 g, 0.16 mol) in THF (3 mL) was treated with pyridine (0.5 mL), causing the solution to develop a yellow color. Addition of $Et_2O$ (10 mL) caused the deposition of the product as a yellow microcrystalline solid (0.062 g, 78%). $^1$H NMR (300 MHz, acetone-$d_6$) δ 8.73 (m, 2H), 8.06 (m, 1H), 7.62 (m, 2H), 3.34 (heptet, $J$ = 6.6 Hz, 3H), 3.05 (m, 6H), 2.82 (m, 6H), 1.21 (d, $J$ = 6.6 Hz, 18H) ppm; $^{13}$C{$^1$H} NMR (75 MHz, acetone-$d_6$) δ 150.2, 137.8, 126.1, 57.4, 50.3, 18.6 ppm.

**Low Temperature Reaction of [L$^{Py}$Cu]CF$_3$SO$_3$ with O$_2$. Characterization of [(L$^{Py}$Cu)$_2$(O$_2$)](CF$_3$SO$_3$)$_2$:** Solutions of [L$^{Py}$Cu]$CF_3SO_3$ in THF/$CH_3CN$ (10:1, 0.5-20 mM in [L$^{Py}$Cu]$^+$) were cooled to -78 °C and exposed to dry $O_2$, causing an immediate color change from light yellow to intense purple. Samples for EPR (0.5-2.0 mM) and resonance Raman (20 mM)

spectroscopies were prepared by removing aliquots of the purple solution with a frozen pipette. Samples for UV-visible spectroscopy were prepared by direct oxygenation of cold (-78 °C) solutions of $[L^{Py}Cu]CF_3SO_3$ in THF/$CH_3CN$ (10:1, ca. 0.2 mM) in a quartz UV-visible cuvette modified for use at low temperature. EPR (9.46 GHz, 77K, 1:0.1:1 THF/$CH_3CN$/Toluene) *silent*; UV-Vis (THF/$CH_3CN$, 10:1) [$\lambda_{max}$, nm ($\varepsilon$, $M^{-1}cm^{-1}$)] 550 (10200), 600 (9700); resonance Raman ($\lambda_{ex}$ 572 nm, 77K, 10:1 THF/$CH_3CN$) 822 ($^{18}O_2$ 771), 530 ($^{18}O_2$ 506) $cm^{-1}$.

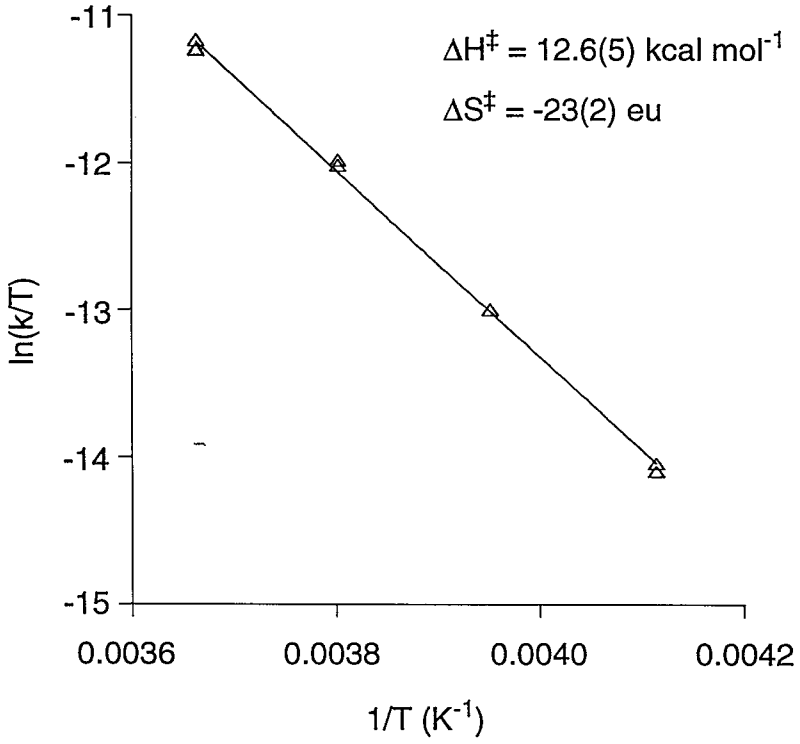
**Ligand recovery and characterization after the decomposition of $[(L^{Py}Cu)_2(O_2)](ClO_4)_2$** A solution of $[(L^{Py}Cu)_2(O_2)](ClO_4)_2$, prepared by oxygenation of a solution of $[L^{Py}Cu]ClO_4$ (0.110 g, 0.23 mmol) in THF/$CH_3CN$ (2:1, 5 mL) at -75 °C for 35 min, was purged with Ar for 10 minutes and then allowed to warm to room temperature under an Ar atmosphere. After warming, the turquoise solution was treated with aq $NH_4OH$ (10 mL) and $CHCl_3$ (10 mL) with vigorous stirring. The organic phase was removed, and the aqueous phase extracted with $CHCl_3$ (3 x 10 mL). The combined organic extracts were dried ($Na_2SO_4$) and evaporated to yield a tan residue (47 mg, 65% mass recovery; a replicate experiment resulted in a 77% mass recovery). The residue was then subjected to analysis by GC/MS and $^1H$ NMR, revealing the presence of $L^{Py}$ and $L^{PyO}$ in a 4:1 ratio. GC/MS: $t_R$ = 12.96 ($L^{Py}$; *m/z* 304 [$M^+$]), 14.98 ($L^{PyO}$; *m/z* = 318 [$M^+$]).

[a] Houser, R.P.; Halfen, J.A.; Young, V.G., Jr.; Blackburn, N.J.; Tolman, W.B. *J. Am. Chem. Soc.* **1995**, *117*, 10745-10746; Halfen, J.A.; Tolman, W.B. *Inorg. Synth.* in press; Mahapatra, S.; Halfen, J.A.; Wilkinson, E.C.; Pan, G.; Wang, X.; Young, V.G., Jr.; Cramer, C.J.; Que, L., Jr.; Tolman, W.B. submitted for publication.

Tabulation of observed rate constants ($k_{obs}$) for the decomposition of $[(L^{Py}Cu)_2(O_2)](ClO_4)_2$ in $CH_3CN$

| T (K) | $k_{obs}^{H}$, $sec^{-1}$ |
|---|---|
| 273 | $3.85(2) \times 10^{-3}$ |
| | $3.59(2) \times 10^{-3}$ |
| | $3.62(3) \times 10^{-3}$ |
| 263 | $1.65(4) \times 10^{-3}$ |
| | $1.59(2) \times 10^{-3}$ |
| 253 | $5.74(3) \times 10^{-4}$ |
| | $5.74(4) \times 10^{-4}$ |
| 243 | $1.92(4) \times 10^{-4}$ |
| | $1.82(1) \times 10^{-4}$ |

Eyring plot for the decomposition of $[(L^{Py}Cu)_2(O_2)](ClO_4)_2$ in $CH_3CN$



**REFERENCE NUMBER: 96061**

CRYSTAL STRUCTURE REPORT

$C_{19}H_{32}CuF_3N_4O_3S$

Report prepared for:

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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 30 frames. These initial sets of frames are oriented such that orthogonal wedges of reciprocal space were surveyed. This produces orientation matrices determined from 171 reflections. Final cell constants are calculated from a set of 7588 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 $0.30^{\circ}$ steps in $\omega$. This collection stratagy provides a high degree of redundancy. The redundant data provide good $\psi$ input in the event an empirical absorption correction is applied (see Table 1).

**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. 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 is the same as previously solved by JH. The lower temperature of the data collection allowed disorder modelling of one triflate anion and one triple-rotational disordered isopropyl group. The ordered triflate anion was used as a paradigm for the disordered anion. The $C_3$ symmetry was exploited with a series of SHELXL SAME and DELU restraints. The two postions are found in a 0.63:0.37 ratio. The disordered isopropyl group was apparently rotationally disordered in exact $120^{\circ}$ fragments. The SHELXL contraints EXYZ and EADP were used to maintain the same positions and anisotropic displacement parameters for related partial atoms. The SUMP restraint was used to maintain full occupancy for the sum of the three fragments. These are found in a 0.144:0.404:0.452 ratio. 258 restaints/constraints were used in total.

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:

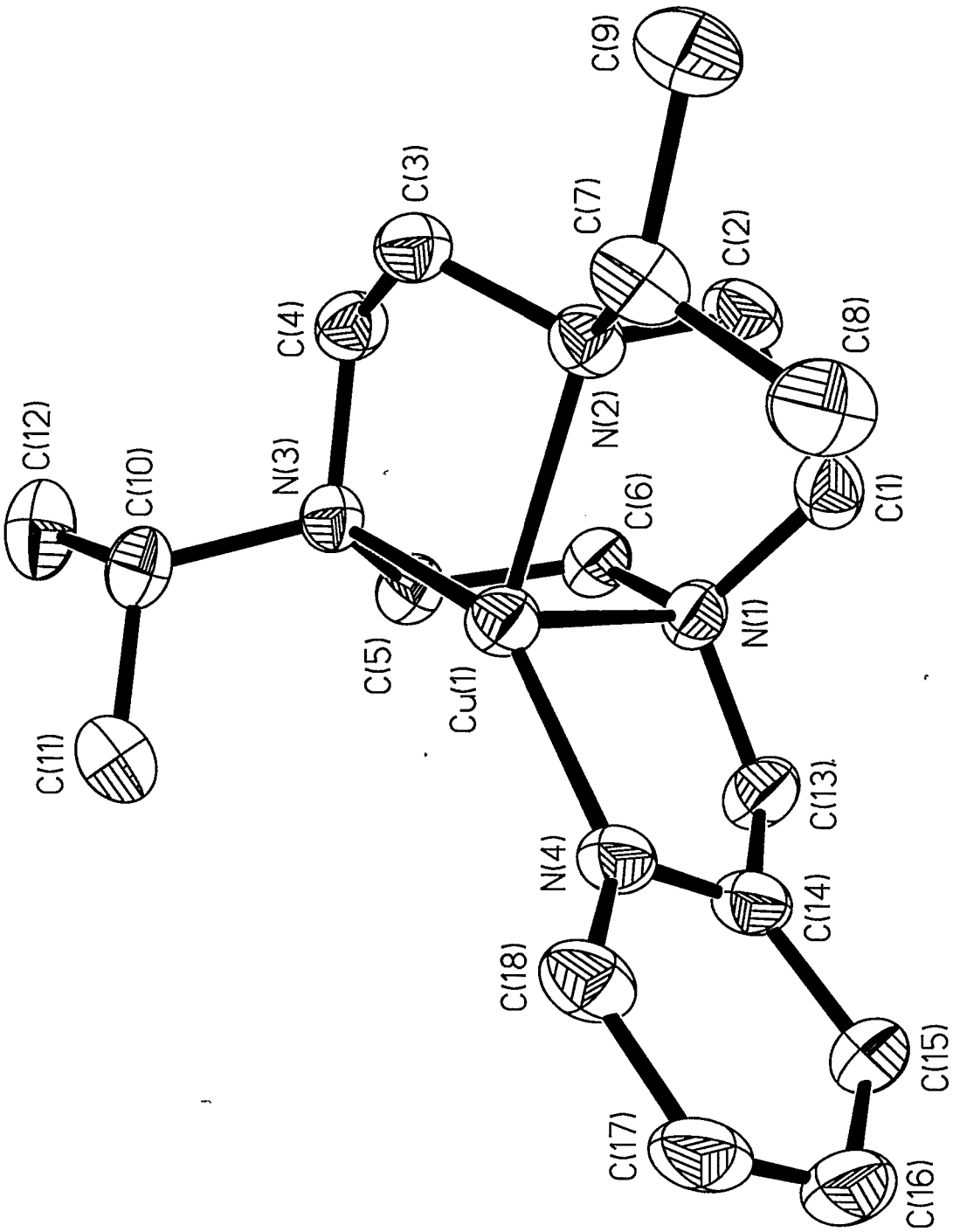
$$R_{int}=\sum|F_o^2-\langle F_o^2\rangle|/\sum|F_o^2|$$

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

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

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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C(7)
C(2)
C(4)
C(8)
N(2)
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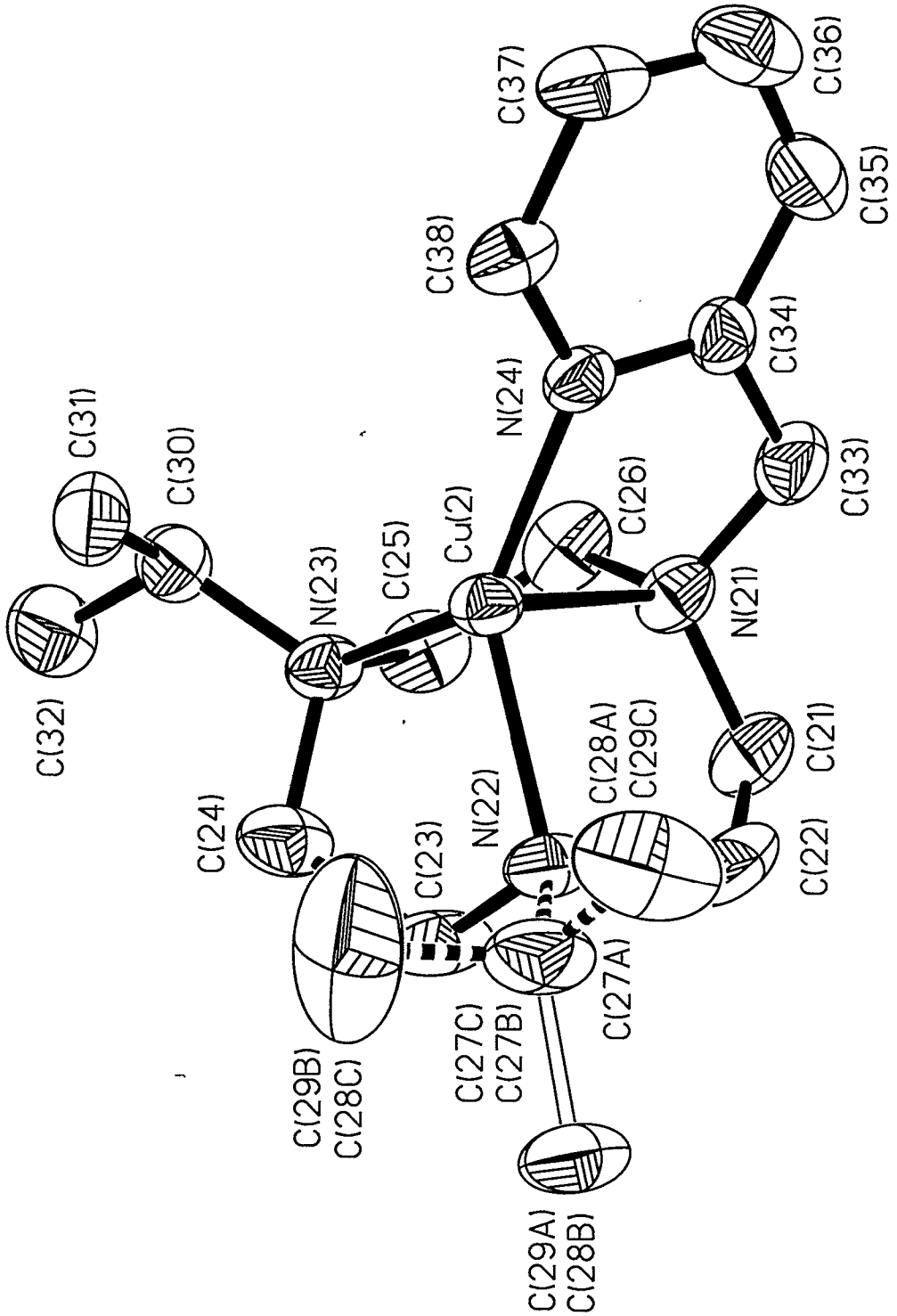
C(6)
C(1)
C(5)
Cu(1)
N(1)
C(11)
C(13)
N(4)
C(14)
C(18)
C(15)
C(17)
C(16)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C(26)
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Cu(2)
C(25)
N(23)
N(21)
C(32)
C(28A)
C(29C)
C(21)
C(24)
N(22)
C(23)
C(22)
C(29B)
C(28C)
C(27C)
C(27B)
C(27A)
C(29A)
C(28B)

**Table 1. Crystal data, data collection, and solution and refinement for 96061.**

| | |
|---|---|
| **Crystal Data** | |
| Empirical formula | $C_{19}H_{32}CuF_3N_4O_3S$ |
| Crystal Habit, color | Plate, Yellow |
| Crystal size | 0.50 x 0.28 x 0.09 mm |
| Crystal system | Triclinic |
| Space group | $P\bar{1}$ |
| | $a$ = 11.8518(1) Å $\alpha$ = 89.264(1)° |
| | $b$ = 12.2046(2) Å $\beta$ = 88.864(1)° |
| | $c$ = 16.4397(1) Å $\gamma$ = 77.512(1)° |
| Volume | 2321.14(5) $Å^3$ |
| Z | 4 |
| Formula weight | 517.09 |
| Density (calculated) | 1.480 $Mg/m^3$ |
| Absorption coefficient | 1.082 $mm^{-1}$ |
| F(000) | 1080 |
| **Data Collection** | |
| Diffractometer | Siemens SMART Platform CCD |
| Wavelength | 0.71073 Å |
| Temperature | 173(2) K |
| $\theta$ range for data collection | 1.24 to 25.01° |
| Index ranges | $-14 \le h \le 8$, $-14 \le k \le 14$, $-19 \le \ell \le 19$ |
| Reflections collected | 12057 |
| Independent reflections | 7847 ($R_{int}$ = 0.0346) |
| **Solution and Refinement** | |
| System used | SHELXTL-V5.0 |
| Solution | Direct methods |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Weighting scheme | $w = [\sigma^2(F_o^2)+(AP)^2+(BP)]^{-1}$, where $P = (Fo^2+2Fc^2))/3$, A = 0.0123, and B = 5.4248 |
| Absorption correction | Semi-empirical |
| Max. and min. transmission | 0.86106 and 0.62089 |
| Data / restraints / parameters | 7845 / 258 / 701 |
| Final R indices [$I>2\sigma(I)$] | R1 = 0.0490, wR2 = 0.1000 |
| R indices (all data) | R1 = 0.0616, wR2 = 0.1104 |
| Goodness-of-fit on $F^2$ | 1.115 |
| Largest diff. peak and hole | 0.521 and -0.510 $eÅ^{-3}$ |

Table 2. Atomic coordinates [ $x_3 10^4$] and equivalent isotropic displacement parameters [$Å^2$ x $10^3$] for 96061. U(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | x | y | z | U(eq) | SOF |
|---|---|---|---|---|---|
| Cu(1) | 7153(1) | 7338(1) | 2195(1) | 30(1) | 1 |
| N(1) | 8340(2) | 5828(2) | 1788(2) | 30(1) | 1 |
| C(1) | 8638(3) | 6171(3) | 963(2) | 34(1) | 1 |
| C(2) | 7563(3) | 6715(3) | 482(2) | 35(1) | 1 |
| N(2) | 6748(3) | 7629(2) | 941(2) | 29(1) | 1 |
| C(3) | 5533(3) | 7456(3) | 960(2) | 32(1) | 1 |
| C(4) | 5422(3) | 6387(3) | 1412(2) | 32(1) | 1 |
| N(3) | 5925(2) | 6320(2) | 2239(2) | 28(1) | 1 |
| C(5) | 6657(3) | 5191(3) | 2403(2) | 32(1) | 1 |
| C(6) | 7711(3) | 4911(3) | 1831(2) | 31(1) | 1 |
| C(7) | 6772(4) | 8788(3) | 633(2) | 37(1) | 1 |
| C(8) | 7966(4) | 9046(4) | 726(3) | 45(1) | 1 |
| C(9) | 6362(4) | 9023(4) | -249(2) | 45(1) | 1 |
| C(10) | 4979(3) | 6660(3) | 2875(2) | 35(1) | 1 |
| C(11) | 5461(4) | 6999(4) | 3661(3) | 52(1) | 1 |
| C(12) | 4238(4) | 5800(4) | 3032(3) | 48(1) | 1 |
| C(13) | 9300(3) | 5704(3) | 2361(2) | 35(1) | 1 |
| C(14) | 9391(3) | 6809(3) | 2748(2) | 33(1) | 1 |
| C(15) | 10439(4) | 6928(4) | 3066(2) | 40(1) | 1 |
| C(16) | 10482(4) | 7881(4) | 3504(3) | 47(1) | 1 |
| C(17) | 9490(4) | 8697(4) | 3603(2) | 44(1) | 1 |
| C(18) | 8491(4) | 8557(3) | 3242(2) | 37(1) | 1 |
| N(4) | 8428(3) | 7625(3) | 2821(2) | 31(1) | 1 |
| Cu(2) | 2803(1) | 2240(1) | 2861(1) | 30(1) | 1 |
| N(21) | 3971(3) | 652(3) | 3143(2) | 40(1) | 1 |
| C(21) | 3894(5) | 482(4) | 4023(3) | 56(1) | 1 |
| C(22) | 3396(5) | 1558(4) | 4467(3) | 56(1) | 1 |
| N(22) | 2333(3) | 2262(3) | 4094(2) | 40(1) | 1 |
| C(23) | 1349(5) | 1682(5) | 4170(3) | 61(2) | 1 |
| C(24) | 732(4) | 1612(5) | 3378(3) | 52(1) | 1 |
| N(23) | 1540(3) | 1263(3) | 2678(2) | 36(1) | 1 |
| C(25) | 2167(4) | 72(3) | 2761(3) | 49(1) | 1 |
| C(26) | 3463(4) | -87(3) | 2628(3) | 52(1) | 1 |
| C(27A) | 2059(4) | 3421(4) | 4449(3) | 56(1) | 0.144(12) |
| C(28A) | 2945(9) | 4006(8) | 4304(6) | 74(3) | 0.144(12) |
| C(29A) | 1661(8) | 3654(7) | 5303(5) | 50(3) | 0.144(12) |
| C(27B) | 2059(4) | 3421(4) | 4449(3) | 56(1) | 0.404(12) |
| C(28B) | 1661(8) | 3654(7) | 5303(5) | 50(3) | 0.404(12) |
| C(29B) | 1077(8) | 4103(7) | 4042(6) | 137(5) | 0.404(12) |
| C(27C) | 2059(4) | 3421(4) | 4449(3) | 56(1) | 0.452(11) |
| C(28C) | 1077(8) | 4103(7) | 4042(6) | 137(5) | 0.452(11) |
| C(29C) | 2945(9) | 4006(8) | 4304(6) | 74(3) | 0.452(11) |
| C(30) | 950(4) | 1473(4) | 1869(3) | 43(1) | 1 |
| C(31) | 733(4) | 2704(4) | 1634(3) | 53(1) | 1 |
| C(32) | -151(4) | 1002(5) | 1814(3) | 67(2) | 1 |
| C(33) | 5103(4) | 790(4) | 2845(3) | 48(1) | 1 |
| C(34) | 5047(3) | 1725(3) | 2223(2) | 35(1) | 1 |
| C(35) | 6025(4) | 1784(4) | 1757(3) | 47(1) | 1 |
| C(36) | 5993(4) | 2660(4) | 1221(3) | 55(1) | 1 |
| C(37) | 4977(4) | 3457(4) | 1149(2) | 45(1) | 1 |
| C(38) | 4031(4) | 3367(3) | 1627(2) | 35(1) | 1 |
| N(24) | 4054(3) | 2509(3) | 2163(2) | 31(1) | 1 |
| S(101) | 2036(1) | 6238(1) | 593(1) | 35(1) | 1 |
| O(101) | 1197(3) | 6439(3) | -39(2) | 76(1) | 1 |

| | | | | | |
|---|---|---|---|---|---|
| O(102) | 1739(3) | 5629(3) | 1282(2) | 65(1) | 1 |
| O(103) | 3220(2) | 5906(2) | 314(2) | 45(1) | 1 |
| C(101) | 1915(4) | 7648(4) | 994(3) | 49(1) | 1 |
| F(101) | 2263(4) | 8315(3) | 469(3) | 118(2) | 1 |
| F(102) | 2583(3) | 7627(3) | 1646(2) | 96(1) | 1 |
| F(103) | 852(2) | 8108(2) | 1232(2) | 57(1) | 1 |
| S(201) | 7564(3) | 1561(3) | 4685(2) | 35(1) | 0.633(5) |
| O(201) | 6948(5) | 823(5) | 4296(3) | 59(2) | 0.633(5) |
| O(202) | 7118(4) | 1935(4) | 5479(3) | 50(1) | 0.633(5) |
| O(203) | 8796(5) | 1233(8) | 4648(4) | 51(2) | 0.633(5) |
| C(201) | 7233(8) | 2825(6) | 4056(5) | 53(2) | 0.633(5) |
| F(201) | 6134(8) | 3239(8) | 4011(8) | 111(4) | 0.633(5) |
| F(202) | 7606(5) | 2619(5) | 3290(3) | 91(2) | 0.633(5) |
| F(203) | 7760(10) | 3594(7) | 4306(6) | 115(4) | 0.633(5) |
| S(202) | 7423(6) | 1562(6) | 4381(4) | 43(1) | 0.367(5) |
| O(204) | 6307(8) | 1427(9) | 4656(6) | 61(3) | 0.367(5) |
| O(205) | 7682(7) | 1275(8) | 3538(5) | 62(3) | 0.367(5) |
| O(206) | 8395(12) | 1144(14) | 4900(8) | 79(5) | 0.367(5) |
| C(202) | 7297(13) | 3074(10) | 4394(8) | 61(4) | 0.367(5) |
| F(204) | 6354(14) | 3612(15) | 4033(14) | 127(7) | 0.367(5) |
| F(205) | 8168(14) | 3374(13) | 4046(12) | 114(7) | 0.367(5) |
| F(206) | 7164(12) | 3468(9) | 5141(7) | 129(5) | 0.367(5) |

**Table 3. Bond lengths [Å] and angles [°] for 96061.**

| | | | |
|---|---|---|---|
| Cu(1)-N(4) | 1.941(3) | Cu(1)-N(3) | 2.108(3) |
| Cu(1)-N(2) | 2.133(3) | Cu(1)-N(1) | 2.167(3) |
| N(1)-C(6) | 1.473(4) | N(1)-C(1) | 1.473(5) |
| N(1)-C(13) | 1.474(5) | C(1)-C(2) | 1.533(5) |
| C(2)-N(2) | 1.508(5) | N(2)-C(3) | 1.499(4) |
| N(2)-C(7) | 1.503(5) | C(3)-C(4) | 1.523(5) |
| C(4)-N(3) | 1.490(4) | N(3)-C(5) | 1.485(5) |
| N(3)-C(10) | 1.511(5) | C(5)-C(6) | 1.529(5) |
| C(7)-C(8) | 1.525(6) | C(7)-C(9) | 1.542(5) |
| C(10)-C(11) | 1.520(6) | C(10)-C(12) | 1.523(5) |
| C(13)-C(14) | 1.524(5) | C(14)-N(4) | 1.347(5) |
| C(14)-C(15) | 1.394(5) | C(15)-C(16) | 1.387(6) |
| C(16)-C(17) | 1.375(6) | C(17)-C(18) | 1.380(6) |
| C(18)-N(4) | 1.356(5) | Cu(2)-N(24) | 1.939(3) |
| Cu(2)-N(22) | 2.090(3) | Cu(2)-N(23) | 2.133(3) |
| Cu(2)-N(21) | 2.175(3) | N(21)-C(21) | 1.462(5) |
| N(21)-C(33) | 1.462(6) | N(21)-C(26) | 1.472(5) |
| C(21)-C(22) | 1.510(7) | C(22)-N(22) | 1.501(6) |
| N(22)-C(23) | 1.492(5) | N(22)-C(27C) | 1.504(6) |
| N(22)-C(27B) | 1.504(6) | N(22)-C(27A) | 1.504(6) |
| C(23)-C(24) | 1.519(6) | C(24)-N(23) | 1.489(5) |
| N(23)-C(25) | 1.488(5) | N(23)-C(30) | 1.507(5) |
| C(25)-C(26) | 1.517(7) | C(27A)-C(28A) | 1.406(9) |
| C(27A)-C(29A) | 1.485(8) | C(27B)-C(29B) | 1.446(9) |
| C(27B)-C(28B) | 1.485(8) | C(27C)-C(29C) | 1.406(9) |
| C(27C)-C(28C) | 1.446(9) | C(30)-C(31) | 1.515(6) |
| C(30)-C(32) | 1.540(6) | C(33)-C(34) | 1.515(6) |
| C(34)-N(24) | 1.351(5) | C(34)-C(35) | 1.390(6) |
| C(35)-C(36) | 1.372(7) | C(36)-C(37) | 1.380(7) |
| C(37)-C(38) | 1.380(6) | C(38)-N(24) | 1.355(5) |
| S(101)-O(102) | 1.426(3) | S(101)-O(101) | 1.434(3) |
| S(101)-O(103) | 1.441(3) | S(101)-C(101) | 1.826(4) |
| C(101)-F(101) | 1.300(5) | C(101)-F(103) | 1.317(5) |
| C(101)-F(102) | 1.341(5) | S(201)-O(203) | 1.429(6) |
| S(201)-O(202) | 1.439(5) | S(201)-O(201) | 1.440(6) |
| S(201)-C(201) | 1.821(8) | C(201)-F(201) | 1.294(11) |
| C(201)-F(203) | 1.310(10) | C(201)-F(202) | 1.335(9) |
| S(202)-O(204) | 1.432(10) | S(202)-O(205) | 1.445(9) |
| S(202)-O(206) | 1.446(11) | S(202)-C(202) | 1.819(13) |
| C(202)-F(205) | 1.29(2) | C(202)-F(204) | 1.32(2) |
| C(202)-F(206) | 1.318(14) | | |

| | | | |
|---|---|---|---|
| N(4)-Cu(1)-N(3) | 139.67(12) | N(4)-Cu(1)-N(2) | 131.05(12) |
| N(3)-Cu(1)-N(2) | 87.27(11) | N(4)-Cu(1)-N(1) | 86.21(12) |
| N(3)-Cu(1)-N(1) | 84.64(11) | N(2)-Cu(1)-N(1) | 85.75(11) |
| C(6)-N(1)-C(1) | 115.7(3) | C(6)-N(1)-C(13) | 114.2(3) |
| C(1)-N(1)-C(13) | 113.5(3) | C(6)-N(1)-Cu(1) | 107.2(2) |
| C(1)-N(1)-Cu(1) | 100.9(2) | C(13)-N(1)-Cu(1) | 103.4(2) |
| N(1)-C(1)-C(2) | 112.1(3) | N(2)-C(2)-C(1) | 113.5(3) |
| C(3)-N(2)-C(7) | 110.3(3) | C(3)-N(2)-C(2) | 112.2(3) |
| C(7)-N(2)-C(2) | 113.3(3) | C(3)-N(2)-Cu(1) | 99.5(2) |
| C(7)-N(2)-Cu(1) | 114.9(2) | C(2)-N(2)-Cu(1) | 105.9(2) |
| N(2)-C(3)-C(4) | 112.6(3) | N(3)-C(4)-C(3) | 112.2(3) |
| C(5)-N(3)-C(4) | 111.4(3) | C(5)-N(3)-C(10) | 112.4(3) |
| C(4)-N(3)-C(10) | 110.3(3) | C(5)-N(3)-Cu(1) | 102.1(2) |
| C(4)-N(3)-Cu(1) | 105.9(2) | C(10)-N(3)-Cu(1) | 114.4(2) |
| N(3)-C(5)-C(6) | 112.6(3) | N(1)-C(6)-C(5) | 111.7(3) |
| N(2)-C(7)-C(8) | 111.2(3) | N(2)-C(7)-C(9) | 114.3(3) |
| C(8)-C(7)-C(9) | 110.3(3) | N(3)-C(10)-C(11) | 111.2(3) |

| | | | |
|---|---|---|---|
| N(3)-C(10)-C(12) | 114.5(3) | C(11)-C(10)-C(12) | 111.5(4) |
| N(1)-C(13)-C(14) | 113.0(3) | N(4)-C(14)-C(15) | 121.5(3) |
| N(4)-C(14)-C(13) | 118.6(3) | C(15)-C(14)-C(13) | 119.7(4) |
| C(16)-C(15)-C(14) | 119.4(4) | C(17)-C(16)-C(15) | 119.1(4) |
| C(16)-C(17)-C(18) | 118.9(4) | N(4)-C(18)-C(17) | 122.9(4) |
| C(14)-N(4)-C(18) | 118.1(3) | C(14)-N(4)-Cu(1) | 113.0(2) |
| C(18)-N(4)-Cu(1) | 128.6(3) | N(24)-Cu(2)-N(22) | 139.76(13) |
| N(24)-Cu(2)-N(23) | 130.97(12) | N(22)-Cu(2)-N(23) | 86.98(12) |
| N(24)-Cu(2)-N(21) | 84.90(13) | N(22)-Cu(2)-N(21) | 85.48(13) |
| N(23)-Cu(2)-N(21) | 85.69(13) | C(21)-N(21)-C(33) | 114.5(3) |
| C(21)-N(21)-C(26) | 116.8(4) | C(33)-N(21)-C(26) | 113.0(4) |
| C(21)-N(21)-Cu(2) | 106.9(3) | C(33)-N(21)-Cu(2) | 104.6(2) |
| C(26)-N(21)-Cu(2) | 98.8(2) | N(21)-C(21)-C(22) | 112.4(4) |
| N(22)-C(22)-C(21) | 114.6(4) | C(23)-N(22)-C(22) | 110.3(4) |
| C(23)-N(22)-C(27C) | 113.0(4) | C(22)-N(22)-C(27C) | 110.9(3) |
| C(23)-N(22)-C(27B) | 113.0(4) | C(22)-N(22)-C(27B) | 110.9(3) |
| C(23)-N(22)-C(27A) | 113.0(4) | C(22)-N(22)-C(27A) | 110.9(3) |
| C(23)-N(22)-Cu(2) | 106.7(2) | C(22)-N(22)-Cu(2) | 102.0(2) |
| C(27C)-N(22)-Cu(2) | 113.3(3) | C(27B)-N(22)-Cu(2) | 113.3(3) |
| C(27A)-N(22)-Cu(2) | 113.3(3) | N(22)-C(23)-C(24) | 114.1(4) |
| N(23)-C(24)-C(23) | 113.1(4) | C(25)-N(23)-C(24) | 111.4(3) |
| C(25)-N(23)-C(30) | 111.1(3) | C(24)-N(23)-C(30) | 112.6(3) |
| C(25)-N(23)-Cu(2) | 105.6(2) | C(24)-N(23)-Cu(2) | 101.6(2) |
| C(30)-N(23)-Cu(2) | 113.9(2) | N(23)-C(25)-C(26) | 113.1(3) |
| N(21)-C(26)-C(25) | 112.5(4) | C(28A)-C(27A)-C(29A) | 106.5(6) |
| C(28A)-C(27A)-N(22) | 112.7(5) | C(29A)-C(27A)-N(22) | 123.2(5) |
| C(29B)-C(27B)-C(28B) | 98.6(6) | C(29B)-C(27B)-N(22) | 109.8(4) |
| C(28B)-C(27B)-N(22) | 123.2(5) | C(29C)-C(27C)-C(28C) | 103.6(7) |
| C(29C)-C(27C)-N(22) | 112.7(5) | C(28C)-C(27C)-N(22) | 109.8(4) |
| N(23)-C(30)-C(31) | 111.1(3) | N(23)-C(30)-C(32) | 114.0(4) |
| C(31)-C(30)-C(32) | 112.0(4) | N(21)-C(33)-C(34) | 113.9(3) |
| N(24)-C(34)-C(35) | 121.9(4) | N(24)-C(34)-C(33) | 118.5(3) |
| C(35)-C(34)-C(33) | 119.5(4) | C(36)-C(35)-C(34) | 119.7(4) |
| C(35)-C(36)-C(37) | 118.8(4) | C(38)-C(37)-C(36) | 119.4(4) |
| N(24)-C(38)-C(37) | 122.3(4) | C(34)-N(24)-C(38) | 117.9(3) |
| C(34)-N(24)-Cu(2) | 114.5(2) | C(38)-N(24)-Cu(2) | 127.6(3) |
| O(102)-S(101)-O(101) | 115.2(2) | O(102)-S(101)-O(103) | 115.2(2) |
| O(101)-S(101)-O(103) | 115.0(2) | O(102)-S(101)-C(101) | 102.8(2) |
| O(101)-S(101)-C(101) | 101.6(2) | O(103)-S(101)-C(101) | 104.5(2) |
| F(101)-C(101)-F(103) | 108.8(4) | F(101)-C(101)-F(102) | 105.8(4) |
| F(103)-C(101)-F(102) | 106.5(4) | F(101)-C(101)-S(101) | 112.2(3) |
| F(103)-C(101)-S(101) | 112.3(3) | F(102)-C(101)-S(101) | 110.9(3) |
| O(203)-S(201)-O(202) | 113.3(4) | O(203)-S(201)-O(201) | 116.0(5) |
| O(202)-S(201)-O(201) | 114.6(4) | O(203)-S(201)-C(201) | 104.1(5) |
| O(202)-S(201)-C(201) | 104.0(4) | O(201)-S(201)-C(201) | 103.0(4) |
| F(201)-C(201)-F(203) | 109.8(9) | F(201)-C(201)-F(202) | 105.8(9) |
| F(203)-C(201)-F(202) | 104.9(8) | F(201)-C(201)-S(201) | 112.7(7) |
| F(203)-C(201)-S(201) | 111.8(7) | F(202)-C(201)-S(201) | 111.5(6) |
| O(204)-S(202)-O(205) | 114.2(7) | O(204)-S(202)-O(206) | 117.8(9) |
| O(205)-S(202)-O(206) | 112.3(8) | O(204)-S(202)-C(202) | 103.6(7) |
| O(205)-S(202)-C(202) | 103.6(7) | O(206)-S(202)-C(202) | 103.2(8) |
| F(205)-C(202)-F(204) | 107.9(14) | F(205)-C(202)-F(206) | 109.4(13) |
| F(204)-C(202)-F(206) | 103.2(14) | F(205)-C(202)-S(202) | 112.5(11) |
| F(204)-C(202)-S(202) | 111.7(12) | F(206)-C(202)-S(202) | 111.7(9) |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 96061. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [ (ha^*)^2 U_{11} + \ldots + 2hka^* b^* U_{12} ]$

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Cu(1) | 29(1) | 29(1) | 30(1) | -8(1) | -2(1) | -6(1) |
| N(1) | 25(2) | 32(2) | 33(2) | -6(1) | 0(1) | -7(1) |
| C(1) | 33(2) | 38(2) | 34(2) | -12(2) | 6(2) | -12(2) |
| C(2) | 41(2) | 40(2) | 25(2) | -7(2) | 2(2) | -14(2) |
| N(2) | 33(2) | 29(2) | 27(2) | -2(1) | 0(1) | -9(1) |
| C(3) | 31(2) | 35(2) | 29(2) | -2(2) | -4(2) | -7(2) |
| C(4) | 28(2) | 34(2) | 34(2) | -4(2) | -3(2) | -9(2) |
| N(3) | 25(2) | 29(2) | 29(2) | -2(1) | 1(1) | -3(1) |
| C(5) | 29(2) | 30(2) | 37(2) | -2(2) | -1(2) | -6(2) |
| C(6) | 28(2) | 27(2) | 38(2) | -6(2) | -2(2) | -4(2) |
| C(7) | 49(2) | 29(2) | 32(2) | 0(2) | 1(2) | -11(2) |
| C(8) | 55(3) | 43(3) | 43(2) | 3(2) | -5(2) | -21(2) |
| C(9) | 55(3) | 46(3) | 35(2) | 9(2) | -4(2) | -10(2) |
| C(10) | 26(2) | 33(2) | 43(2) | -2(2) | 3(2) | 0(2) |
| C(11) | 47(3) | 68(3) | 38(2) | -12(2) | 7(2) | -7(2) |
| C(12) | 38(2) | 45(3) | 61(3) | 4(2) | 14(2) | -7(2) |
| C(13) | 27(2) | 36(2) | 42(2) | -6(2) | -5(2) | -4(2) |
| C(14) | 37(2) | 39(2) | 25(2) | -4(2) | -3(2) | -13(2) |
| C(15) | 32(2) | 55(3) | 39(2) | -4(2) | -2(2) | -19(2) |
| C(16) | 45(3) | 67(3) | 39(2) | 0(2) | -8(2) | -32(2) |
| C(17) | 58(3) | 53(3) | 29(2) | -6(2) | -3(2) | -32(2) |
| C(18) | 51(3) | 34(2) | 30(2) | -6(2) | 1(2) | -17(2) |
| N(4) | 33(2) | 35(2) | 26(2) | -4(1) | -1(1) | -12(1) |
| Cu(2) | 34(1) | 31(1) | 26(1) | 3(1) | 3(1) | -6(1) |
| N(21) | 51(2) | 31(2) | 34(2) | 4(1) | -6(2) | -4(2) |
| C(21) | 63(3) | 63(3) | 39(2) | 22(2) | -12(2) | -8(3) |
| C(22) | 74(3) | 69(3) | 28(2) | 13(2) | -11(2) | -25(3) |
| N(22) | 50(2) | 48(2) | 28(2) | -5(2) | 7(2) | -22(2) |
| C(23) | 72(3) | 93(4) | 36(2) | -11(3) | 18(2) | -55(3) |
| C(24) | 47(3) | 73(3) | 45(2) | -17(2) | 11(2) | -35(3) |
| N(23) | 41(2) | 39(2) | 30(2) | -5(1) | 1(1) | -15(2) |
| C(25) | 67(3) | 36(2) | 50(3) | -2(2) | -1(2) | -24(2) |
| C(26) | 71(3) | 26(2) | 55(3) | 2(2) | -10(2) | -1(2) |
| C(27A) | 61(3) | 70(3) | 40(2) | -27(2) | 13(2) | -22(3) |
| C(28A) | 89(7) | 64(6) | 83(7) | -33(5) | 25(5) | -46(5) |
| C(29A) | 62(6) | 63(6) | 30(4) | -11(4) | 11(4) | -22(4) |
| C(27B) | 61(3) | 70(3) | 40(2) | -27(2) | 13(2) | -22(3) |
| C(28B) | 62(6) | 63(6) | 30(4) | -11(4) | 11(4) | -22(4) |
| C(29B) | 117(7) | 99(7) | 161(9) | -85(6) | -69(7) | 60(5) |
| C(27C) | 61(3) | 70(3) | 40(2) | -27(2) | 13(2) | -22(3) |
| C(28C) | 117(7) | 99(7) | 161(9) | -85(6) | -69(7) | 60(5) |
| C(29C) | 89(7) | 64(6) | 83(7) | -33(5) | 25(5) | -46(5) |
| C(30) | 39(2) | 51(3) | 41(2) | -11(2) | -3(2) | -12(2) |
| C(31) | 44(3) | 62(3) | 49(3) | -3(2) | -10(2) | -1(2) |
| C(32) | 50(3) | 94(5) | 61(3) | -23(3) | -5(3) | -26(3) |
| C(33) | 43(3) | 55(3) | 39(2) | 4(2) | -9(2) | 8(2) |
| C(34) | 32(2) | 41(2) | 32(2) | -7(2) | -6(2) | -6(2) |
| C(35) | 33(2) | 61(3) | 47(3) | -18(2) | 3(2) | -12(2) |
| C(36) | 56(3) | 71(3) | 47(3) | -18(2) | 15(2) | -35(3) |
| C(37) | 69(3) | 45(2) | 32(2) | -6(2) | 7(2) | -38(2) |
| C(38) | 52(3) | 33(2) | 27(2) | -2(2) | -3(2) | -21(2) |
| N(24) | 35(2) | 36(2) | 23(2) | -2(1) | -3(1) | -11(1) |
| S(101) | 31(1) | 39(1) | 37(1) | -10(1) | 4(1) | -10(1) |
| O(101) | 56(2) | 102(3) | 61(2) | -34(2) | -25(2) | 5(2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O(102) | 68(2) | 49(2) | 73(2) | 10(2) | 31(2) | -4(2) |
| O(103) | 34(2) | 49(2) | 49(2) | -13(1) | 12(1) | -8(1) |
| C(101) | 36(2) | 44(3) | 65(3) | -13(2) | 7(2) | -6(2) |
| F(101) | 145(4) | 46(2) | 165(4) | -2(2) | 83(3) | -32(2) |
| F(102) | 56(2) | 96(2) | 128(3) | -74(2) | -30(2) | 7(2) |
| F(103) | 40(1) | 52(2) | 71(2) | -15(1) | 1(1) | 6(1) |
| S(201) | 36(2) | 36(1) | 33(2) | 3(1) | -3(1) | -11(1) |
| O(201) | 76(4) | 49(3) | 61(4) | 2(3) | -20(3) | -32(3) |
| O(202) | 43(3) | 75(4) | 36(2) | -5(2) | 11(2) | -20(2) |
| O(203) | 36(3) | 59(4) | 53(4) | -6(3) | 10(2) | 1(3) |
| C(201) | 60(5) | 41(4) | 57(5) | 8(4) | -4(4) | -10(4) |
| F(201) | 79(5) | 79(6) | 154(7) | 46(5) | -19(5) | 28(4) |
| F(202) | 123(5) | 102(4) | 47(3) | 33(3) | -1(3) | -22(4) |
| F(203) | 173(10) | 50(5) | 138(8) | 6(4) | -19(7) | -60(7) |
| S(202) | 41(2) | 43(2) | 46(3) | 7(3) | -11(3) | -9(2) |
| O(204) | 62(6) | 62(6) | 61(6) | 0(5) | 9(5) | -21(5) |
| O(205) | 53(5) | 82(7) | 56(5) | -28(5) | 2(4) | -24(5) |
| O(206) | 80(9) | 75(8) | 82(10) | 15(9) | -49(9) | -14(9) |
| C(202) | 79(9) | 48(7) | 60(9) | -3(6) | 16(8) | -21(7) |
| F(204) | 88(8) | 83(11) | 184(12) | 69(11) | 37(8) | 29(8) |
| F(205) | 91(8) | 52(7) | 198(16) | 10(9) | 70(9) | -22(6) |
| F(206) | 194(13) | 78(7) | 115(8) | -56(6) | 63(8) | -31(7) |

**Table 5. Torsion angles [°] for 96061.**

| | | | |
|---|---|---|---|
| N(4)-Cu(1)-N(1)-C(6) | 135.7(2) | N(3)-Cu(1)-N(1)-C(6) | -5.0(2) |
| N(2)-Cu(1)-N(1)-C(6) | -92.6(2) | N(4)-Cu(1)-N(1)-C(1) | -102.8(2) |
| N(3)-Cu(1)-N(1)-C(1) | 116.5(2) | N(2)-Cu(1)-N(1)-C(1) | 28.8(2) |
| N(4)-Cu(1)-N(1)-C(13) | 14.8(2) | N(3)-Cu(1)-N(1)-C(13) | -125.9(2) |
| N(2)-Cu(1)-N(1)-C(13) | 146.4(2) | C(6)-N(1)-C(1)-C(2) | 66.9(4) |
| C(13)-N(1)-C(1)-C(2) | -158.3(3) | Cu(1)-N(1)-C(1)-C(2) | -48.4(3) |
| N(1)-C(1)-C(2)-N(2) | 49.8(4) | C(1)-C(2)-N(2)-C(3) | -127.9(3) |
| C(1)-C(2)-N(2)-C(7) | 106.5(3) | C(1)-C(2)-N(2)-Cu(1) | -20.4(3) |
| N(4)-Cu(1)-N(2)-C(3) | -167.4(2) | N(3)-Cu(1)-N(2)-C(3) | 26.6(2) |
| N(1)-Cu(1)-N(2)-C(3) | 111.4(2) | N(4)-Cu(1)-N(2)-C(7) | -49.6(3) |
| N(3)-Cu(1)-N(2)-C(7) | 144.3(3) | N(1)-Cu(1)-N(2)-C(7) | -130.8(3) |
| N(4)-Cu(1)-N(2)-C(2) | 76.2(3) | N(3)-Cu(1)-N(2)-C(2) | -89.8(2) |
| N(1)-Cu(1)-N(2)-C(2) | -5.0(2) | C(7)-N(2)-C(3)-C(4) | -169.3(3) |
| C(2)-N(2)-C(3)-C(4) | 63.5(4) | Cu(1)-N(2)-C(3)-C(4) | -48.1(3) |
| N(2)-C(3)-C(4)-N(3) | 52.4(4) | C(3)-C(4)-N(3)-C(5) | -134.3(3) |
| C(3)-C(4)-N(3)-C(10) | 100.2(3) | C(3)-C(4)-N(3)-Cu(1) | -24.1(3) |
| N(4)-Cu(1)-N(3)-C(5) | -49.2(3) | N(2)-Cu(1)-N(3)-C(5) | 114.5(2) |
| N(1)-Cu(1)-N(3)-C(5) | 28.5(2) | N(4)-Cu(1)-N(3)-C(4) | -165.8(2) |
| N(2)-Cu(1)-N(3)-C(4) | -2.1(2) | N(1)-Cu(1)-N(3)-C(4) | -88.1(2) |
| N(4)-Cu(1)-N(3)-C(10) | 72.5(3) | N(2)-Cu(1)-N(3)-C(10) | -123.8(2) |
| N(1)-Cu(1)-N(3)-C(10) | 150.2(2) | C(4)-N(3)-C(5)-C(6) | 63.5(4) |
| C(10)-N(3)-C(5)-C(6) | -172.1(3) | Cu(1)-N(3)-C(5)-C(6) | -49.1(3) |
| C(1)-N(1)-C(6)-C(5) | -131.9(3) | C(13)-N(1)-C(6)-C(5) | 93.6(4) |
| Cu(1)-N(1)-C(6)-C(5) | -20.3(3) | N(3)-C(5)-C(6)-N(1) | 49.1(4) |
| C(3)-N(2)-C(7)-C(8) | 171.6(3) | C(2)-N(2)-C(7)-C(8) | -61.8(4) |
| Cu(1)-N(2)-C(7)-C(8) | 60.1(4) | C(3)-N(2)-C(7)-C(9) | -62.8(4) |
| C(2)-N(2)-C(7)-C(9) | 63.9(4) | Cu(1)-N(2)-C(7)-C(9) | -174.2(3) |
| C(5)-N(3)-C(10)-C(11) | 75.3(4) | C(4)-N(3)-C(10)-C(11) | -159.8(3) |
| Cu(1)-N(3)-C(10)-C(11) | -40.6(4) | C(5)-N(3)-C(10)-C(12) | -52.2(4) |
| C(4)-N(3)-C(10)-C(12) | 72.8(4) | Cu(1)-N(3)-C(10)-C(12) | -168.0(3) |
| C(6)-N(1)-C(13)-C(14) | -140.1(3) | C(1)-N(1)-C(13)-C(14) | 84.4(4) |
| Cu(1)-N(1)-C(13)-C(14) | -24.0(3) | N(1)-C(13)-C(14)-N(4) | 26.7(5) |
| N(1)-C(13)-C(14)-C(15) | -157.5(3) | N(4)-C(14)-C(15)-C(16) | 3.5(6) |
| C(13)-C(14)-C(15)-C(16) | -172.2(4) | C(14)-C(15)-C(16)-C(17) | -1.2(6) |
| C(15)-C(16)-C(17)-C(18) | -1.9(6) | C(16)-C(17)-C(18)-N(4) | 2.9(6) |
| C(15)-C(14)-N(4)-C(18) | -2.6(5) | C(13)-C(14)-N(4)-C(18) | 173.1(3) |
| C(15)-C(14)-N(4)-Cu(1) | 171.7(3) | C(13)-C(14)-N(4)-Cu(1) | -12.6(4) |
| C(17)-C(18)-N(4)-C(14) | -0.7(5) | C(17)-C(18)-N(4)-Cu(1) | -173.9(3) |
| N(3)-Cu(1)-N(4)-C(14) | 75.4(3) | N(2)-Cu(1)-N(4)-C(14) | -82.7(3) |
| N(1)-Cu(1)-N(4)-C(14) | -1.7(3) | N(3)-Cu(1)-N(4)-C(18) | -111.1(3) |
| N(2)-Cu(1)-N(4)-C(18) | 90.8(3) | N(1)-Cu(1)-N(4)-C(18) | 171.8(3) |
| N(24)-Cu(2)-N(21)-C(21) | 136.4(3) | N(22)-Cu(2)-N(21)-C(21) | -4.5(3) |
| N(23)-Cu(2)-N(21)-C(21) | -91.8(3) | N(24)-Cu(2)-N(21)-C(33) | 14.6(2) |
| N(22)-Cu(2)-N(21)-C(33) | -126.3(3) | N(23)-Cu(2)-N(21)-C(33) | 146.4(3) |
| N(24)-Cu(2)-N(21)-C(26) | -102.1(3) | N(22)-Cu(2)-N(21)-C(26) | 117.0(3) |
| N(23)-Cu(2)-N(21)-C(26) | 29.7(3) | C(33)-N(21)-C(21)-C(22) | 96.2(5) |
| C(26)-N(21)-C(21)-C(22) | -128.6(4) | Cu(2)-N(21)-C(21)-C(22) | -19.1(5) |
| N(21)-C(21)-C(22)-N(22) | 46.1(6) | C(21)-C(22)-N(22)-C(23) | 67.3(5) |
| C(21)-C(22)-N(22)-C(27C) | -166.7(4) | C(21)-C(22)-N(22)-C(27B) | -166.7(4) |
| C(21)-C(22)-N(22)-C(27A) | -166.7(4) | C(21)-C(22)-N(22)-Cu(2) | -45.7(4) |
| N(24)-Cu(2)-N(22)-C(23) | -166.5(3) | N(23)-Cu(2)-N(22)-C(23) | -4.0(3) |
| N(21)-Cu(2)-N(22)-C(23) | -89.9(3) | N(24)-Cu(2)-N(22)-C(22) | -50.8(3) |
| N(23)-Cu(2)-N(22)-C(22) | 111.8(3) | N(21)-Cu(2)-N(22)-C(22) | 25.9(3) |
| N(24)-Cu(2)-N(22)-C(27C) | 68.5(4) | N(23)-Cu(2)-N(22)-C(27C) | -128.9(3) |
| N(21)-Cu(2)-N(22)-C(27C) | 145.1(3) | N(24)-Cu(2)-N(22)-C(27B) | 68.5(4) |
| N(23)-Cu(2)-N(22)-C(27B) | -128.9(3) | N(21)-Cu(2)-N(22)-C(27B) | 145.1(3) |
| N(24)-Cu(2)-N(22)-C(27A) | 68.5(4) | N(23)-Cu(2)-N(22)-C(27A) | -128.9(3) |
| N(21)-Cu(2)-N(22)-C(27A) | 145.1(3) | C(22)-N(22)-C(23)-C(24) | -129.4(5) |
| C(27C)-N(22)-C(23)-C(24) | 105.8(5) | C(27B)-N(22)-C(23)-C(24) | 105.8(5) |

| | | | |
|---|---|---|---|
| C(27A)-N(22)-C(23)-C(24) | 105.8(5) | Cu(2)-N(22)-C(23)-C(24) | -19.4(5) |
| N(22)-C(23)-C(24)-N(23) | 45.2(6) | C(23)-C(24)-N(23)-C(25) | 68.8(5) |
| C(23)-C(24)-N(23)-C(30) | -165.6(4) | C(23)-C(24)-N(23)-Cu(2) | -43.3(4) |
| N(24)-Cu(2)-N(23)-C(25) | 74.2(3) | N(22)-Cu(2)-N(23)-C(25) | -91.0(3) |
| N(21)-Cu(2)-N(23)-C(25) | -5.3(2) | N(24)-Cu(2)-N(23)-C(24) | -169.5(3) |
| N(22)-Cu(2)-N(23)-C(24) | 25.4(3) | N(21)-Cu(2)-N(23)-C(24) | 111.1(3) |
| N(24)-Cu(2)-N(23)-C(30) | -48.1(3) | N(22)-Cu(2)-N(23)-C(30) | 146.8(3) |
| N(21)-Cu(2)-N(23)-C(30) | -127.5(3) | C(24)-N(23)-C(25)-C(26) | -130.8(4) |
| C(30)-N(23)-C(25)-C(26) | 102.7(4) | Cu(2)-N(23)-C(25)-C(26) | -21.3(4) |
| C(21)-N(21)-C(26)-C(25) | 63.3(5) | C(33)-N(21)-C(26)-C(25) | -160.8(3) |
| Cu(2)-N(21)-C(26)-C(25) | -50.8(4) | N(23)-C(25)-C(26)-N(21) | 53.0(5) |
| C(23)-N(22)-C(27A)-C(28A) | -174.1(6) | C(22)-N(22)-C(27A)-C(28A) | 61.5(7) |
| C(27C)-N(22)-C(27A)-C(28A) | 0(100) | C(27B)-N(22)-C(27A)-C(28A) | 0(100) |
| Cu(2)-N(22)-C(27A)-C(28A) | -52.6(7) | C(23)-N(22)-C(27A)-C(29A) | 56.0(7) |
| C(22)-N(22)-C(27A)-C(29A) | -68.5(6) | C(27C)-N(22)-C(27A)-C(29A) | 0(100) |
| C(27B)-N(22)-C(27A)-C(29A) | 0(100) | Cu(2)-N(22)-C(27A)-C(29A) | 177.5(5) |
| C(23)-N(22)-C(27B)-C(29B) | -59.2(7) | C(22)-N(22)-C(27B)-C(29B) | 176.4(6) |
| C(27C)-N(22)-C(27B)-C(29B) | 0(100) | C(27A)-N(22)-C(27B)-C(29B) | 0(100) |
| Cu(2)-N(22)-C(27B)-C(29B) | 62.3(6) | C(23)-N(22)-C(27B)-C(28B) | 56.0(7) |
| C(22)-N(22)-C(27B)-C(28B) | -68.5(6) | C(27C)-N(22)-C(27B)-C(28B) | 0(100) |
| C(27A)-N(22)-C(27B)-C(28B) | 0(100) | Cu(2)-N(22)-C(27B)-C(28B) | 177.5(5) |
| C(23)-N(22)-C(27C)-C(29C) | -174.1(6) | C(22)-N(22)-C(27C)-C(29C) | 61.5(7) |
| C(27B)-N(22)-C(27C)-C(29C) | 0(100) | C(27A)-N(22)-C(27C)-C(29C) | 0(100) |
| Cu(2)-N(22)-C(27C)-C(29C) | -52.6(7) | C(23)-N(22)-C(27C)-C(28C) | -59.2(7) |
| C(22)-N(22)-C(27C)-C(28C) | 176.4(6) | C(27B)-N(22)-C(27C)-C(28C) | 0(100) |
| C(27A)-N(22)-C(27C)-C(28C) | 0(100) | Cu(2)-N(22)-C(27C)-C(28C) | 62.3(6) |
| C(25)-N(23)-C(30)-C(31) | -157.3(4) | C(24)-N(23)-C(30)-C(31) | 76.9(4) |
| Cu(2)-N(23)-C(30)-C(31) | -38.1(4) | C(25)-N(23)-C(30)-C(32) | 75.1(5) |
| C(24)-N(23)-C(30)-C(32) | -50.8(5) | Cu(2)-N(23)-C(30)-C(32) | -165.8(3) |
| C(21)-N(21)-C(33)-C(34) | -136.4(4) | C(26)-N(21)-C(33)-C(34) | 86.7(4) |
| Cu(2)-N(21)-C(33)-C(34) | -19.7(4) | N(21)-C(33)-C(34)-N(24) | 17.4(5) |
| N(21)-C(33)-C(34)-C(35) | -165.4(4) | N(24)-C(34)-C(35)-C(36) | 0.1(6) |
| C(33)-C(34)-C(35)-C(36) | -176.9(4) | C(34)-C(35)-C(36)-C(37) | -1.0(6) |
| C(35)-C(36)-C(37)-C(38) | 1.4(6) | C(36)-C(37)-C(38)-N(24) | -1.0(6) |
| C(35)-C(34)-N(24)-C(38) | 0.3(5) | C(33)-C(34)-N(24)-C(38) | 177.4(3) |
| C(35)-C(34)-N(24)-Cu(2) | 179.2(3) | C(33)-C(34)-N(24)-Cu(2) | -3.7(4) |
| C(37)-C(38)-N(24)-C(34) | 0.1(5) | C(37)-C(38)-N(24)-Cu(2) | -178.6(3) |
| N(22)-Cu(2)-N(24)-C(34) | 70.5(3) | N(23)-Cu(2)-N(24)-C(34) | -86.1(3) |
| N(21)-Cu(2)-N(24)-C(34) | -6.4(3) | N(22)-Cu(2)-N(24)-C(38) | -110.7(3) |
| N(23)-Cu(2)-N(24)-C(38) | 92.6(3) | N(21)-Cu(2)-N(24)-C(38) | 172.4(3) |
| O(102)-S(101)-C(101)-F(101) | 173.9(4) | O(101)-S(101)-C(101)-F(101) | -66.7(4) |
| O(103)-S(101)-C(101)-F(101) | 53.2(4) | O(102)-S(101)-C(101)-F(103) | -63.2(4) |
| O(101)-S(101)-C(101)-F(103) | 56.2(4) | O(103)-S(101)-C(101)-F(103) | 176.1(3) |
| O(102)-S(101)-C(101)-F(102) | 55.8(4) | O(101)-S(101)-C(101)-F(102) | 175.3(3) |
| O(103)-S(101)-C(101)-F(102) | -64.8(4) | O(203)-S(201)-C(201)-F(201) | 179.3(9) |
| O(202)-S(201)-C(201)-F(201) | -61.9(9) | O(201)-S(201)-C(201)-F(201) | 57.9(9) |
| O(203)-S(201)-C(201)-F(203) | -56.5(9) | O(202)-S(201)-C(201)-F(203) | 62.4(8) |
| O(201)-S(201)-C(201)-F(203) | -177.8(8) | O(203)-S(201)-C(201)-F(202) | 60.6(7) |
| O(202)-S(201)-C(201)-F(202) | 179.4(6) | O(201)-S(201)-C(201)-F(202) | -60.8(7) |
| O(204)-S(202)-C(202)-F(205) | -170.1(14) | O(205)-S(202)-C(202)-F(205) | -51(2) |
| O(206)-S(202)-C(202)-F(205) | 67(2) | O(204)-S(202)-C(202)-F(204) | -48.5(14) |
| O(205)-S(202)-C(202)-F(204) | 70.9(14) | O(206)-S(202)-C(202)-F(204) | -171.9(14) |
| O(204)-S(202)-C(202)-F(206) | 66.4(13) | O(205)-S(202)-C(202)-F(206) | -174.1(11) |
| O(206)-S(202)-C(202)-F(206) | -56.9(14) | | |

Symmetry transformations used to generate equivalent atoms:

Table 6. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$ x $10^3$) for 96061.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 9068(3) | 5504(3) | 666(2) | 48(12) |
| H(1B) | 9150(3) | 6709(3) | 1002(2) | 42(11) |
| H(2A) | 7811(3) | 7036(3) | -30(2) | 32(10) |
| H(2B) | 7144(3) | 6129(3) | 336(2) | 33(10) |
| H(3A) | 5270(3) | 7422(3) | 395(2) | 35(10) |
| H(3B) | 5022(3) | 8107(3) | 1226(2) | 22(9) |
| H(4A) | 4595(3) | 6359(3) | 1461(2) | 22(9) |
| H(4B) | 5821(3) | 5729(3) | 1093(2) | 25(9) |
| H(5A) | 6186(3) | 4619(3) | 2343(2) | 45(12) |
| H(5B) | 6921(3) | 5158(3) | 2972(2) | 38(11) |
| H(6A) | 8237(3) | 4218(3) | 2025(2) | 32(10) |
| H(6B) | 7456(3) | 4769(3) | 1280(2) | 24(9) |
| H(7A) | 6227(4) | 9327(3) | 987(2) | 27(9) |
| H(8A) | 8221(4) | 8884(4) | 1286(3) | 43(11) |
| H(8B) | 7929(4) | 9840(4) | 600(3) | 59(14) |
| H(8C) | 8515(4) | 8578(4) | 351(3) | 50(13) |
| H(9A) | 5571(4) | 8909(4) | -292(2) | 48(12) |
| H(9B) | 6875(4) | 8509(4) | -618(2) | 52(13) |
| H(9C) | 6377(4) | 9799(4) | -399(2) | 46(12) |
| H(10A) | 4450(3) | 7349(3) | 2659(2) | 32(10) |
| H(11A) | 5934(4) | 7550(4) | 3540(3) | 45(12) |
| H(11B) | 5937(4) | 6335(4) | 3921(3) | 66(15) |
| H(11C) | 4821(4) | 7332(4) | 4029(3) | 56(13) |
| H(12A) | 3983(4) | 5563(4) | 2512(3) | 62(15) |
| H(12B) | 3562(4) | 6137(4) | 3366(3) | 65(15) |
| H(12C) | 4693(4) | 5148(4) | 3317(3) | 53(13) |
| H(13A) | 10034(3) | 5387(3) | 2070(2) | 45(12) |
| H(13B) | 9191(3) | 5166(3) | 2796(2) | 53(13) |
| H(15A) | 11118(4) | 6361(4) | 2983(2) | 45(12) |
| H(16A) | 11186(4) | 7968(4) | 3733(3) | 65(15) |
| H(17A) | 9494(4) | 9346(4) | 3915(2) | 38(11) |
| H(18A) | 7817(4) | 9140(3) | 3290(2) | 28(10) |
| H(21A) | 4676(5) | 159(4) | 4230(3) | 53(13) |
| H(21B) | 3403(5) | -65(4) | 4138(3) | 54(13) |
| H(22A) | 3207(5) | 1370(4) | 5034(3) | 57(13) |
| H(22B) | 3996(5) | 2009(4) | 4488(3) | 66(15) |
| H(23A) | 784(5) | 2084(5) | 4574(3) | 86(18) |
| H(23B) | 1644(5) | 912(5) | 4381(3) | 121(27) |
| H(24A) | 227(4) | 1067(5) | 3447(3) | 52(13) |
| H(24B) | 234(4) | 2354(5) | 3255(3) | 69(17) |
| H(25A) | 1862(4) | -386(3) | 2362(3) | 40(11) |
| H(25B) | 2014(4) | -205(3) | 3312(3) | 90(19) |
| H(26A) | 3836(4) | -878(3) | 2747(3) | 68(15) |
| H(26B) | 3624(4) | 67(3) | 2049(3) | 67(15) |
| H(27A) | 1403(4) | 3838(4) | 4117(3) | 67 |
| H(28A) | 2876(9) | 4329(8) | 3754(6) | 111 |
| H(28B) | 3698(9) | 3487(8) | 4355(6) | 111 |
| H(28C) | 2880(9) | 4608(8) | 4702(6) | 111 |
| H(29A) | 1862(8) | 4350(7) | 5481(5) | 76 |
| H(29B) | 2035(8) | 3032(7) | 5654(5) | 76 |
| H(29C) | 820(8) | 3734(7) | 5339(5) | 76 |
| H(27B) | 2741(8) | 3768(7) | 4346(6) | 67 |
| H(28D) | 1333(8) | 4459(7) | 5364(6) | 76 |

| | | | | |
|---|---|---|---|---|
| H(28E) | 2316(8) | 3429(7) | 5668(6) | 76 |
| H(28F) | 1069(8) | 3229(7) | 5442(6) | 76 |
| H(29D) | 1284(8) | 4228(7) | 3474(6) | 205 |
| H(29E) | 844(8) | 4827(7) | 4317(6) | 205 |
| H(29F) | 435(8) | 3715(7) | 4062(6) | 205 |
| H(27C) | 1893(4) | 3381(4) | 5046(3) | 67 |
| H(28G) | 997(4) | 3788(4) | 3507(3) | 205 |
| H(28H) | 1189(4) | 4872(4) | 3976(3) | 205 |
| H(28I) | 376(4) | 4110(4) | 4371(3) | 205 |
| H(29G) | 3116(4) | 4028(4) | 3716(3) | 111 |
| H(29H) | 3647(4) | 3628(4) | 4586(3) | 111 |
| H(29I) | 2702(4) | 4777(4) | 4503(3) | 111 |
| H(30A) | 1509(4) | 1063(4) | 1455(3) | 41(11) |
| H(31A) | 1462(4) | 2959(4) | 1642(3) | 66(15) |
| H(31B) | 181(4) | 3142(4) | 2022(3) | 59(14) |
| H(31C) | 415(4) | 2807(4) | 1086(3) | 55(13) |
| H(32A) | 22(4) | 208(5) | 1976(3) | 81(19) |
| H(32B) | -425(4) | 1075(5) | 1253(3) | 60(14) |
| H(32C) | -751(4) | 1423(5) | 2178(3) | 65(15) |
| H(33A) | 5518(4) | 76(4) | 2599(3) | 62(14) |
| H(33B) | 5557(4) | 943(4) | 3313(3) | 55(13) |
| H(35A) | 6711(4) | 1220(4) | 1811(3) | 69(16) |
| H(36A) | 6658(4) | 2718(4) | 905(3) | 56(13) |
| H(37A) | 4929(4) | 4062(4) | 773(2) | 54(13) |
| H(38A) | 3340(4) | 3925(3) | 1580(2) | 44(12) |