Table 1. Crystal data, data collection, and solution and refinement for 97011.

Crystal Data	
Empirical formula	$C_{18}H_{38}CuF_6N_4Sb$
Crystal Habit, color	block, colorless
Crystal size	0.35 x 0.25 x 0.22 mm
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
	a = 9.9638(4) Å α = 90°
	b = 13.3049(6) Å β = 90°
	c = 19.5424(9) Å γ = 90°
Volume	2590.7(2) $Å^3$
Z	4
Formula weight	609.81
Density (calculated)	1.563 Mg/m^3
Absorption coefficient	1.917 mm^{-1}
F(000)	1232
Data Collection	
Diffractometer	Siemens SMART Platform CCD
Wavelength	0.71073 Å
Temperature	293(2) K
θ range for data collection	1.85 to 25.03°
Index ranges	$-11 \le h \le 11$, $0 \le k \le 15$, $0 \le \ell \le 23$
Reflections collected	13008
Independent reflections	4549 (R_{int} = 0.0254)
C.3403 **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 = 0.0682, and B = 0.3403
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.0000 and 0.6856
Absolute structure parameter	0.03(4)
Data / restraints / parameters	4548 / 24 / 271
R indices (I>2σ(I) = 3603)	R1 = 0.0461, wR2 = 0.1094
R indices (all data)	R1 = 0.0612, wR2 = 0.1190
Goodness-of-fit on F^2	1.040
Largest diff. peak and hole	0.437 and -0.377 $eÅ^{-3}$

Table 2. Atomic coordinates [$x_3 10^4$] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 97011. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
Cu(1)	2414(1)	72(1)	6215(1)	73(1)	1
N(1)	4474(6)	156(6)	6440(3)	97(2)	1
N(2)	2777(5)	1137(4)	5417(3)	75(1)	1
N(3)	1630(8)	1289(5)	6813(4)	106(2)	1
N(4)	1321(7)	-1051(5)	6004(3)	87(2)	1
C(1)	2495(17)	1538(11)	7384(8)	180(6)	1
C(2)	3629(17)	1018(18)	7461(8)	222(8)	1
C(3)	4730(12)	851(15)	7014(6)	199(7)	1
C(4)	5041(8)	588(7)	5804(5)	118(3)	1
C(5)	4212(8)	1395(6)	5448(5)	99(2)	1
C(6)	1856(13)	1958(6)	5632(4)	121(3)	1
C(7)	1782(15)	2151(7)	6351(6)	149(5)	1
C(8)	4940(14)	-1054(14)	6442(7)	205(8)	1
C(9)	6521(15)	-930(18)	6570(9)	277(11)	1
C(10)	4232(19)	-1581(12)	7009(10)	240(10)	1
C(11)	196(14)	1111(11)	7016(7)	162(5)	1
C(12)	-651(18)	1830(15)	7280(9)	272(13)	1
C(13)	197(13)	145(11)	7471(6)	170(5)	1
C(14)	2309(7)	781(6)	4714(3)	90(2)	1
C(15)	3021(8)	-201(6)	4531(4)	104(2)	1
C(16)	2477(12)	1573(9)	4133(5)	148(4)	1
C(17)	659(7)	-1680(5)	5852(4)	86(2)	1
C(18)	-230(9)	-2489(5)	5635(5)	110(3)	1
Sb(1)	-1825(1)	-226(1)	4167(1)	84(1)	1
F(1)	-1102(9)	744(6)	3631(6)	221(5)	1
F(2)	-2267(8)	-1001(7)	3435(4)	211(4)	1
F(3)	-2575(8)	-1188(6)	4726(5)	215(4)	1
F(4)	-1355(8)	523(8)	4919(4)	226(4)	1
F(5)	-190(5)	-869(4)	4195(4)	148(2)	1
F(6)	-3479(5)	404(5)	4147(3)	145(2)	1

Table 3. Bond lengths [Å] and angles [°] for 97011.

Cu(1)-N(4)	1.895(6)	Cu(1)-N(1)	2.102(6)
Cu(1)-N(2)	2.137(5)	Cu(1)-N(3)	2.144(7)
N(1)-C(3)	1.476(14)	N(1)-C(4)	1.480(10)
N(1)-C(8)	1.68(2)	N(2)-C(5)	1.471(10)
N(2)-C(6)	1.487(10)	N(2)-C(14)	1.527(9)
N(3)-C(1)	1.45(2)	N(3)-C(7)	1.468(12)
N(3)-C(11)	1.502(14)	N(4)-C(17)	1.107(9)
C(1)-C(2)	1.33(2)	C(2)-C(3)	1.42(2)
C(4)-C(5)	1.523(11)	C(6)-C(7)	1.430(14)
C(8)-C(10)	1.49(2)	C(8)-C(9)	1.60(2)
C(11)-C(12)	1.38(2)	C(11)-C(13)	1.56(2)
C(14)-C(15)	1.529(11)	C(14)-C(16)	1.557(12)
C(17)-C(18)	1.456(10)	Sb(1)-F(1)	1.812(6)
Sb(1)-F(2)	1.818(6)	Sb(1)-F(4)	1.836(7)
Sb(1)-F(5)	1.841(5)	Sb(1)-F(3)	1.841(7)
Sb(1)-F(6)	1.850(5)		

N(4)-Cu(1)-N(1)	130.4(3)	N(4)-Cu(1)-N(2)	117.5(2)
N(1)-Cu(1)-N(2)	87.3(2)	N(4)-Cu(1)-N(3)	120.3(3)
N(1)-Cu(1)-N(3)	101.6(3)	N(2)-Cu(1)-N(3)	87.7(2)
C(3)-N(1)-C(4)	109.2(9)	C(3)-N(1)-C(8)	123.5(10)
C(4)-N(1)-C(8)	105.6(7)	C(3)-N(1)-Cu(1)	111.1(6)
C(4)-N(1)-Cu(1)	102.6(4)	C(8)-N(1)-Cu(1)	102.7(7)
C(5)-N(2)-C(6)	114.7(7)	C(5)-N(2)-C(14)	114.0(6)
C(6)-N(2)-C(14)	107.1(6)	C(5)-N(2)-Cu(1)	106.8(4)
C(6)-N(2)-Cu(1)	100.2(5)	C(14)-N(2)-Cu(1)	113.5(4)
C(1)-N(3)-C(7)	103.5(10)	C(1)-N(3)-C(11)	113.5(10)
C(7)-N(3)-C(11)	112.5(10)	C(1)-N(3)-Cu(1)	112.1(7)
C(7)-N(3)-Cu(1)	102.5(5)	C(11)-N(3)-Cu(1)	111.8(7)
C(17)-N(4)-Cu(1)	176.3(7)	C(2)-C(1)-N(3)	118.1(11)
C(1)-C(2)-C(3)	132(2)	C(2)-C(3)-N(1)	115.6(10)
N(1)-C(4)-C(5)	116.7(7)	N(2)-C(5)-C(4)	112.5(6)
C(7)-C(6)-N(2)	116.1(8)	C(6)-C(7)-N(3)	118.1(8)
C(10)-C(8)-C(9)	113.4(13)	C(10)-C(8)-N(1)	108.9(11)
C(9)-C(8)-N(1)	100(2)	C(12)-C(11)-N(3)	125.0(14)
C(12)-C(11)-C(13)	111.1(10)	N(3)-C(11)-C(13)	106.2(10)
N(2)-C(14)-C(15)	109.5(6)	N(2)-C(14)-C(16)	114.4(7)
C(15)-C(14)-C(16)	111.0(6)	N(4)-C(17)-C(18)	178.3(9)
F(1)-Sb(1)-F(2)	92.6(6)	F(1)-Sb(1)-F(4)	88.5(5)
F(2)-Sb(1)-F(4)	178.3(5)	F(1)-Sb(1)-F(5)	89.8(3)
F(2)-Sb(1)-F(5)	88.5(3)	F(4)-Sb(1)-F(5)	90.2(3)
F(1)-Sb(1)-F(3)	178.6(4)	F(2)-Sb(1)-F(3)	88.5(5)
F(4)-Sb(1)-F(3)	90.4(5)	F(5)-Sb(1)-F(3)	91.1(3)
F(1)-Sb(1)-F(6)	91.1(4)	F(2)-Sb(1)-F(6)	91.4(3)
F(4)-Sb(1)-F(6)	89.9(3)	F(5)-Sb(1)-F(6)	179.1(3)
F(3)-Sb(1)-F(6)	88.1(3)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for 97011. 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)	79(1)	63(1)	77(1)	-2(1)	-3(1)	-8(1)
N(1)	83(4)	124(5)	84(4)	15(4)	-5(3)	12(4)
N(2)	79(4)	69(3)	77(3)	3(2)	-3(3)	3(2)
N(3)	126(6)	95(4)	97(5)	4(4)	20(4)	6(4)
N(4)	106(5)	73(3)	81(4)	1(3)	-2(3)	-16(3)
C(1)	218(13)	174(11)	148(11)	-97(9)	2(11)	-2(10)
C(2)	175(12)	349(22)	141(10)	-124(13)	-44(8)	24(12)
C(3)	108(7)	371(21)	118(9)	-64(11)	-33(6)	-75(10)
C(4)	70(4)	152(8)	132(7)	38(7)	-7(5)	-2(4)
C(5)	85(5)	88(5)	122(6)	23(5)	-13(5)	-18(4)
C(6)	179(10)	84(5)	98(6)	3(4)	-10(6)	41(6)
C(7)	184(11)	97(6)	167(11)	2(6)	63(10)	37(7)
C(8)	158(10)	299(19)	157(11)	111(12)	36(8)	128(11)
C(9)	161(10)	450(30)	221(15)	151(19)	57(11)	159(14)
C(10)	229(15)	197(15)	295(22)	94(16)	69(16)	78(14)
C(11)	165(11)	176(10)	144(9)	4(7)	99(8)	18(8)
C(12)	248(20)	340(22)	229(19)	93(17)	134(17)	168(20)
C(13)	173(11)	195(11)	141(8)	12(9)	67(8)	-24(10)
C(14)	74(4)	133(5)	63(4)	-5(4)	-1(3)	2(4)
C(15)	96(5)	131(5)	84(4)	-27(4)	6(4)	-1(5)
C(16)	155(9)	200(9)	88(6)	36(7)	8(6)	57(8)
C(17)	82(4)	76(4)	99(5)	10(4)	5(4)	-16(4)
C(18)	116(7)	82(5)	132(7)	-10(5)	-24(5)	-27(4)
Sb(1)	69(1)	91(1)	92(1)	-3(1)	1(1)	-3(1)
F(1)	202(8)	146(5)	314(11)	92(7)	138(8)	35(5)
F(2)	172(7)	266(9)	194(7)	-123(7)	-69(6)	59(7)
F(3)	159(7)	203(7)	284(10)	120(7)	39(7)	-11(6)
F(4)	149(6)	315(11)	213(7)	-151(8)	-28(5)	5(7)
F(5)	85(3)	134(4)	226(6)	-9(5)	-24(4)	21(3)
F(6)	80(3)	179(5)	176(5)	7(5)	14(3)	29(3)

Table 5. Torsion angles [°] for 97011.

N(4)-Cu(1)-N(1)-C(3)	-137.7(9)	N(2)-Cu(1)-N(1)-C(3)	97.8(9)
N(3)-Cu(1)-N(1)-C(3)	10.8(9)	N(4)-Cu(1)-N(1)-C(4)	105.7(6)
N(2)-Cu(1)-N(1)-C(4)	-18.8(6)	N(3)-Cu(1)-N(1)-C(4)	-105.8(6)
N(4)-Cu(1)-N(1)-C(8)	-3.7(7)	N(2)-Cu(1)-N(1)-C(8)	-128.2(6)
N(3)-Cu(1)-N(1)-C(8)	144.8(6)	N(4)-Cu(1)-N(2)-C(5)	-136.7(5)
N(1)-Cu(1)-N(2)-C(5)	-1.7(5)	N(3)-Cu(1)-N(2)-C(5)	100.1(5)
N(4)-Cu(1)-N(2)-C(6)	103.5(6)	N(1)-Cu(1)-N(2)-C(6)	-121.5(5)
N(3)-Cu(1)-N(2)-C(6)	-19.7(6)	N(4)-Cu(1)-N(2)-C(14)	-10.2(5)
N(1)-Cu(1)-N(2)-C(14)	124.7(5)	N(3)-Cu(1)-N(2)-C(14)	-133.5(5)
N(4)-Cu(1)-N(3)-C(1)	128.7(9)	N(1)-Cu(1)-N(3)-C(1)	-23.8(9)
N(2)-Cu(1)-N(3)-C(1)	-110.5(9)	N(4)-Cu(1)-N(3)-C(7)	-120.8(8)
N(1)-Cu(1)-N(3)-C(7)	86.6(8)	N(2)-Cu(1)-N(3)-C(7)	-0.1(8)
N(4)-Cu(1)-N(3)-C(11)	-0.1(8)	N(1)-Cu(1)-N(3)-C(11)	-152.7(7)
N(2)-Cu(1)-N(3)-C(11)	120.6(7)	N(1)-Cu(1)-N(4)-C(17)	-129(11)
N(2)-Cu(1)-N(4)-C(17)	-18(11)	N(3)-Cu(1)-N(4)-C(17)	87(11)
C(7)-N(3)-C(1)-C(2)	-109(2)	C(11)-N(3)-C(1)-C(2)	129(2)
Cu(1)-N(3)-C(1)-C(2)	1(2)	N(3)-C(1)-C(2)-C(3)	56(3)
C(1)-C(2)-C(3)-N(1)	-73(3)	C(4)-N(1)-C(3)-C(2)	136(2)
C(8)-N(1)-C(3)-C(2)	-100(2)	Cu(1)-N(1)-C(3)-C(2)	23(2)
C(3)-N(1)-C(4)-C(5)	-79.9(10)	C(8)-N(1)-C(4)-C(5)	145.4(9)
Cu(1)-N(1)-C(4)-C(5)	38.1(9)	C(6)-N(2)-C(5)-C(4)	132.3(8)
C(14)-N(2)-C(5)-C(4)	-103.9(8)	Cu(1)-N(2)-C(5)-C(4)	22.3(9)
N(1)-C(4)-C(5)-N(2)	-43.6(11)	C(5)-N(2)-C(6)-C(7)	-74.7(12)
C(14)-N(2)-C(6)-C(7)	157.8(10)	Cu(1)-N(2)-C(6)-C(7)	39.2(11)
N(2)-C(6)-C(7)-N(3)	-47(2)	C(1)-N(3)-C(7)-C(6)	140.0(13)
C(11)-N(3)-C(7)-C(6)	-97.0(14)	Cu(1)-N(3)-C(7)-C(6)	23(2)
C(3)-N(1)-C(8)-C(10)	62(2)	C(4)-N(1)-C(8)-C(10)	-171.3(13)
Cu(1)-N(1)-C(8)-C(10)	-64(2)	C(3)-N(1)-C(8)-C(9)	-56.8(13)
C(4)-N(1)-C(8)-C(9)	69.6(11)	Cu(1)-N(1)-C(8)-C(9)	176.8(8)
C(1)-N(3)-C(11)-C(12)	66(2)	C(7)-N(3)-C(11)-C(12)	-52(2)
Cu(1)-N(3)-C(11)-C(12)	-166.5(14)	C(1)-N(3)-C(11)-C(13)	-65.8(14)
C(7)-N(3)-C(11)-C(13)	177.0(10)	Cu(1)-N(3)-C(11)-C(13)	62.3(11)
C(5)-N(2)-C(14)-C(15)	64.0(7)	C(6)-N(2)-C(14)-C(15)	-168.1(7)
Cu(1)-N(2)-C(14)-C(15)	-58.6(6)	C(5)-N(2)-C(14)-C(16)	-61.3(8)
C(6)-N(2)-C(14)-C(16)	66.6(9)	Cu(1)-N(2)-C(14)-C(16)	176.2(6)
Cu(1)-N(4)-C(17)-C(18)	-8(38)		

Symmetry transformations used to generate equivalent atoms:

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 97011.

	x	y	z	U(eq)	SOF
H(1A)	2731(17)	2243(11)	7345(8)	217	1
H(1B)	1975(17)	1463(11)	7801(8)	217	1
H(2A)	3333(17)	348(18)	7586(8)	266	1
H(2B)	4034(17)	1296(18)	7871(8)	266	1
H(3A)	5473(12)	589(15)	7280(6)	239	1
H(3B)	5009(12)	1493(15)	6827(6)	239	1
H(4A)	5186(8)	43(7)	5482(5)	142	1
H(4B)	5913(8)	871(7)	5912(5)	142	1
H(5A)	4318(8)	2025(6)	5692(5)	118	1
H(5B)	4548(8)	1488(6)	4987(5)	118	1
H(6A)	2134(13)	2572(6)	5405(4)	145	1
H(6B)	961(13)	1798(6)	5469(4)	145	1
H(7A)	1031(15)	2600(7)	6430(6)	179	1
H(7B)	2589(15)	2509(7)	6483(6)	179	1
H(8A)	4759(14)	-1374(14)	6000(7)	245	1
H(9A)	6919(15)	-586(18)	6189(9)	333	1
H(9B)	6924(15)	-1581(18)	6619(9)	333	1
H(9C)	6666(15)	-547(18)	6980(9)	333	1
H(10A)	3294(19)	-1633(12)	6902(10)	288	1
H(10B)	4344(19)	-1209(12)	7426(10)	288	1
H(10C)	4603(19)	-2242(12)	7064(10)	288	1
H(11A)	-242(14)	899(11)	6591(7)	194	1
H(12A)	-634(18)	2415(15)	6992(9)	327	1
H(12B)	-361(18)	2009(15)	7732(9)	327	1
H(12C)	-1548(18)	1568(15)	7299(9)	327	1
H(13A)	794(13)	-344(11)	7276(6)	203	1
H(13B)	-694(13)	-128(11)	7491(6)	203	1
H(13C)	492(13)	313(11)	7924(6)	203	1
H(14A)	1348(7)	631(6)	4750(3)	108	1
H(15A)	2903(8)	-676(6)	4896(4)	124	1
H(15B)	3960(8)	-74(6)	4466(4)	124	1
H(15C)	2645(8)	-469(6)	4117(4)	124	1
H(16A)	2023(12)	2182(9)	4259(5)	177	1
H(16B)	2098(12)	1315(9)	3717(5)	177	1
H(16C)	3414(12)	1710(9)	4066(5)	177	1
H(18A)	-1072(9)	-2213(5)	5489(5)	165	1
H(18B)	-377(9)	-2940(5)	6012(5)	165	1
H(18C)	175(9)	-2849(5)	5263(5)	165	1

REFERENCE NUMBER: 97328aaa

$[(Me_3TACD)Cu(CH_3CN)]ClO_4$

CRYSTAL STRUCTURE REPORT

$C_{12}H_{26}ClCuN_4O_4$

Report prepared for:

B. Lam / Prof. W. Tolman

18 December 1997

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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. The specimen initially indexed with a *c* three times greater than the current cell. This could be transformed, although poorly, to a C-centered orthorhombic cell. The monoclinic cell had poor fits for most reflections in *I*. This is an indication of twinning. DIRAX[1] was used to index a pair of major and minor twins with 50 reflections. SAINT was used to integrate these twin components iteratively. The structure is reported for the major twin only. The model from the major was used in the refinement of the minor. It was determined that the twin components were related as reflection twins based on Flack x. Final cell constants are calculated from a set of 3903 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 $P2_1$ was determined based on systematic absences and intensity statistics.[3] 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 relative isotropic displacement parameters.

The structure was found as expected. A good residual was determined despite the presence of the twins found in a 0.87:0.13 ratio.

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:

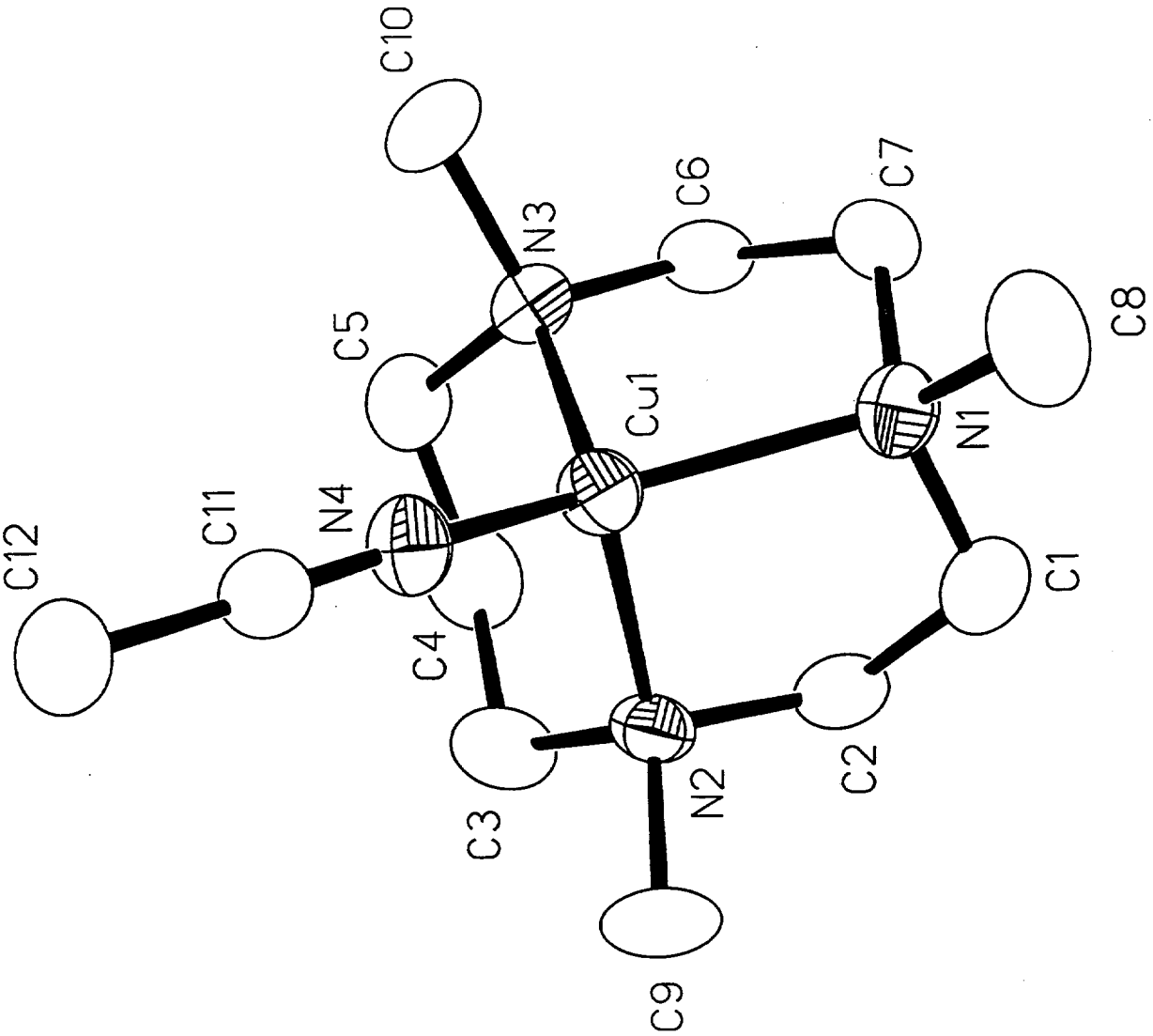
$$R_{int}=\sum|F_o^2-\langle F_o^2\rangle|/\sum|F_o^2|$$

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

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

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

$$GooF=S=[\sum[w(F_o^2-F_c^2)^2]/(n-p)^{1/2}$$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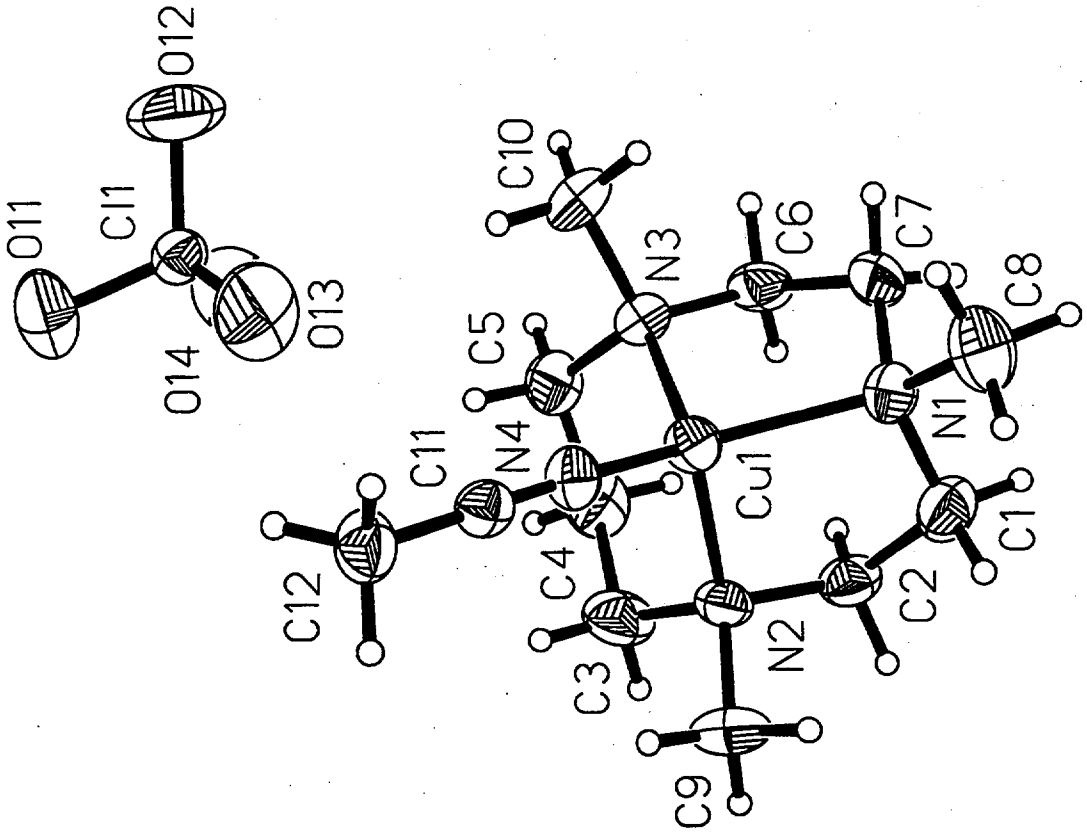
C7
C8
C5
Cu1
N1
C12
C11
N4
C1
C4
C2
C3
N2
C9


O12
O11
Cl1
O13
O14
C10
N3
C6
C7
C8
C5
N1
C11
N4
Cu1
C1
C12
C4
C2
C3
N2
C9

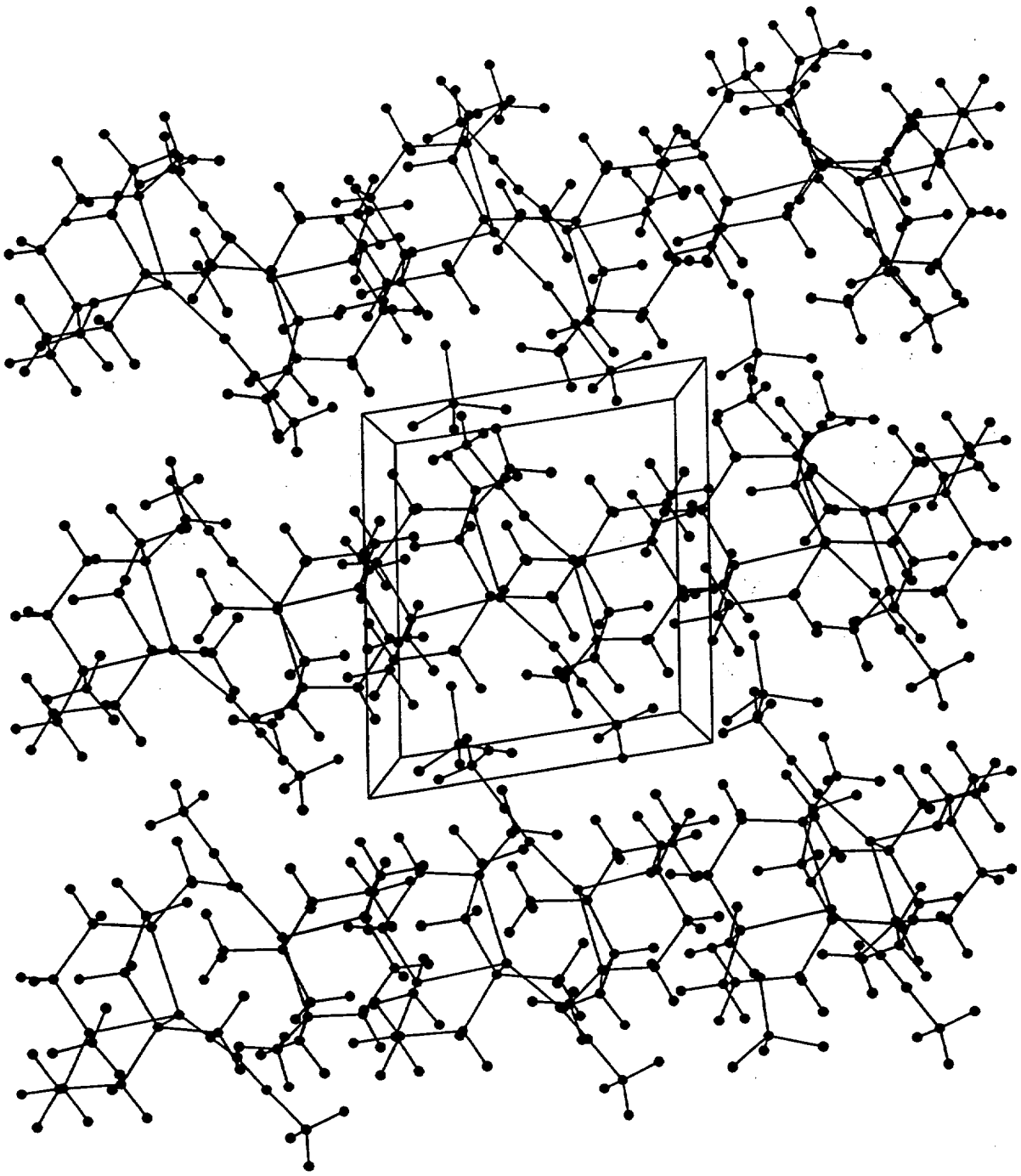


Table 1. Crystal data, data collection, and solution and refinement for 97328aaa.

Crystal Data	
Empirical formula	$C_{12}H_{26}ClCuN_4O_4$
Crystal Habit, color	Plate, Colorless
Crystal size	0.26 x 0.21 x 0.06 mm
Crystal system	Monoclinic
Space group	$P2_1$
	a = 7.5134(1) Å α = 90°
	b = 14.2737(3) Å β = 98.2640(10)°
	c = 8.2653(2) Å γ = 90°
Volume	877.20(3) Å^3
Z	2
Formula weight	389.36
Density (calculated)	1.474 Mg/m^3
Absorption coefficient	1.418 mm^{-1}
F(000)	408
Data Collection	
Diffractometer	Siemens SMART Platform CCD
Wavelength	0.71073 Å
Temperature	173(2) K
θ range for data collection	2.49 to 25.11°
Index ranges	$-8 \le h \le 8$, $-16 \le k \le 16$, $-9 \le \ell \le 9$
Reflections collected	4498
Independent reflections	2845 (R_{int} = 0.0327)
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 = 0.117300, and B = 0.0
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.000 and 0.569
Absolute structure parameter	-0.03(3)
Data / restraints / parameters	2845 / 1 / 204
R indices (I>2σ(I) = 2645)	R1 = 0.0562, wR2 = 0.1561
R indices (all data)	R1 = 0.0596, wR2 = 0.1611
Goodness-of-fit on F^2	1.075
Largest diff. peak and hole	1.193 and -0.444 eÅ^{-3}

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 97328aaa. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
Cu(1)	3844(1)	7481(1)	5375(1)	33(1)	1
N(1)	3059(7)	7511(5)	2847(6)	35(1)	1
C(1)	1209(12)	7141(5)	2571(9)	45(2)	1
C(2)	81(9)	7422(7)	3886(8)	41(2)	1
N(2)	1075(8)	7292(4)	5556(7)	35(1)	1
C(3)	452(12)	7884(6)	6820(10)	51(2)	1
C(4)	684(13)	8944(7)	6553(11)	55(2)	1
N(3)	3585(8)	8971(4)	5294(7)	36(1)	1
C(5)	2568(11)	9303(6)	6582(9)	44(2)	1
C(6)	2707(10)	9206(5)	3658(9)	39(2)	1
C(7)	3183(11)	8520(5)	2380(8)	40(2)	1
C(8)	4173(15)	6983(7)	1837(10)	60(2)	1
C(9)	863(12)	6283(6)	6078(11)	50(2)	1
C(10)	5425(12)	9372(7)	5605(11)	54(2)	1
N(4)	5450(8)	6883(4)	6971(7)	33(1)	1
C(11)	6285(10)	6566(4)	8086(8)	31(1)	1
C(12)	7405(10)	6156(6)	9504(9)	41(2)	1
Cl(1)	7666(2)	9361(1)	10525(2)	33(1)	1
O(11)	7908(10)	9277(5)	12269(7)	65(2)	1
O(12)	8945(12)	9959(7)	9979(11)	89(3)	1
O(13)	7708(16)	8468(5)	9840(9)	86(3)	1
O(14)	5950(10)	9768(7)	10068(10)	82(2)	1

Table 3. Bond lengths [Å] and angles [°] for 97328aaa.

Cu(1)-N(4)	1.862(5)	Cu(1)-N(1)	2.088(5)
Cu(1)-N(2)	2.124(6)	Cu(1)-N(3)	2.136(6)
N(1)-C(8)	1.472(10)	N(1)-C(1)	1.474(10)
N(1)-C(7)	1.497(10)	C(1)-C(2)	1.525(10)
C(2)-N(2)	1.484(9)	N(2)-C(3)	1.472(10)
N(2)-C(9)	1.518(10)	C(3)-C(4)	1.542(13)
C(4)-C(5)	1.503(13)	N(3)-C(6)	1.456(9)
N(3)-C(5)	1.474(9)	N(3)-C(10)	1.484(10)
C(6)-C(7)	1.520(10)	N(4)-C(11)	1.132(9)
C(11)-C(12)	1.464(10)	Cl(1)-O(13)	1.397(7)
Cl(1)-O(12)	1.408(7)	Cl(1)-O(14)	1.415(7)
Cl(1)-O(11)	1.432(6)		

N(4)-Cu(1)-N(1)	141.2(2)	N(4)-Cu(1)-N(2)	115.7(2)
N(1)-Cu(1)-N(2)	86.3(2)	N(4)-Cu(1)-N(3)	121.7(2)
N(1)-Cu(1)-N(3)	86.4(2)	N(2)-Cu(1)-N(3)	92.5(2)
C(8)-N(1)-C(1)	109.2(7)	C(8)-N(1)-C(7)	106.6(6)
C(1)-N(1)-C(7)	113.4(6)	C(8)-N(1)-Cu(1)	117.1(5)
C(1)-N(1)-Cu(1)	105.8(4)	C(7)-N(1)-Cu(1)	105.0(4)
N(1)-C(1)-C(2)	113.8(6)	N(2)-C(2)-C(1)	111.9(6)
C(3)-N(2)-C(2)	114.8(6)	C(3)-N(2)-C(9)	106.6(6)
C(2)-N(2)-C(9)	108.9(6)	C(3)-N(2)-Cu(1)	113.1(5)
C(2)-N(2)-Cu(1)	106.6(4)	C(9)-N(2)-Cu(1)	106.5(5)
N(2)-C(3)-C(4)	114.1(7)	C(5)-C(4)-C(3)	117.3(8)
C(6)-N(3)-C(5)	112.5(6)	C(6)-N(3)-C(10)	111.0(6)
C(5)-N(3)-C(10)	108.5(6)	C(6)-N(3)-Cu(1)	106.6(4)
C(5)-N(3)-Cu(1)	110.6(5)	C(10)-N(3)-Cu(1)	107.5(5)
N(3)-C(5)-C(4)	117.6(6)	N(3)-C(6)-C(7)	112.1(6)
N(1)-C(7)-C(6)	114.3(5)	C(11)-N(4)-Cu(1)	170.7(6)
N(4)-C(11)-C(12)	178.5(8)	O(13)-Cl(1)-O(12)	111.5(6)
O(13)-Cl(1)-O(14)	109.9(7)	O(12)-Cl(1)-O(14)	107.6(6)
O(13)-Cl(1)-O(11)	108.9(5)	O(12)-Cl(1)-O(11)	112.3(5)
O(14)-Cl(1)-O(11)	106.4(5)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for 97328aaa. 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)	32(1)	31(1)	33(1)	4(1)	1(1)	2(1)
N(1)	35(3)	35(3)	34(2)	-4(3)	3(2)	3(3)
C(1)	52(5)	38(4)	46(4)	-8(3)	3(3)	-6(3)
C(2)	30(3)	45(4)	48(3)	2(4)	6(3)	-11(4)
N(2)	40(3)	33(4)	35(3)	2(2)	14(2)	-7(2)
C(3)	49(5)	54(5)	55(4)	1(4)	26(4)	-6(4)
C(4)	66(6)	49(5)	54(5)	-10(4)	22(4)	13(4)
N(3)	35(3)	35(3)	38(3)	-2(2)	4(3)	-7(2)
C(5)	53(4)	37(4)	43(4)	-10(3)	12(3)	3(4)
C(6)	46(4)	26(3)	47(4)	11(3)	7(3)	-5(3)
C(7)	52(5)	36(4)	32(3)	7(3)	7(3)	-6(3)
C(8)	70(6)	65(5)	47(4)	-12(4)	16(4)	10(5)
C(9)	47(5)	37(4)	69(5)	13(4)	14(4)	-12(4)
C(10)	52(5)	50(4)	58(5)	-10(4)	1(4)	-24(4)
N(4)	34(3)	33(3)	31(3)	0(2)	1(2)	6(2)
C(11)	36(3)	23(3)	35(3)	-3(3)	8(3)	0(3)
C(12)	37(4)	50(5)	35(4)	0(3)	0(3)	1(3)
Cl(1)	33(1)	30(1)	34(1)	-2(1)	4(1)	-2(1)
O(11)	84(5)	71(4)	38(3)	-1(3)	2(3)	5(4)
O(12)	86(6)	94(6)	96(5)	-21(5)	48(5)	-54(5)
O(13)	148(8)	39(4)	72(4)	-18(3)	16(5)	-9(5)
O(14)	52(4)	111(7)	81(4)	28(5)	2(4)	26(4)

Table 5. Torsion angles [°] for 97328aaa.

N(4)-Cu(1)-N(1)-C(8)	-9.0(9)	N(2)-Cu(1)-N(1)-C(8)	-136.7(7)
N(3)-Cu(1)-N(1)-C(8)	130.5(7)	N(4)-Cu(1)-N(1)-C(1)	112.9(5)
N(2)-Cu(1)-N(1)-C(1)	-14.8(5)	N(3)-Cu(1)-N(1)-C(1)	-107.6(5)
N(4)-Cu(1)-N(1)-C(7)	-126.9(5)	N(2)-Cu(1)-N(1)-C(7)	105.3(5)
N(3)-Cu(1)-N(1)-C(7)	12.6(4)	C(8)-N(1)-C(1)-C(2)	162.9(7)
C(7)-N(1)-C(1)-C(2)	-78.4(8)	Cu(1)-N(1)-C(1)-C(2)	36.1(8)
N(1)-C(1)-C(2)-N(2)	-46.2(9)	C(1)-C(2)-N(2)-C(3)	155.5(7)
C(1)-C(2)-N(2)-C(9)	-85.1(8)	C(1)-C(2)-N(2)-Cu(1)	29.5(7)
N(4)-Cu(1)-N(2)-C(3)	78.2(5)	N(1)-Cu(1)-N(2)-C(3)	-135.2(5)
N(3)-Cu(1)-N(2)-C(3)	-49.0(5)	N(4)-Cu(1)-N(2)-C(2)	-154.7(5)
N(1)-Cu(1)-N(2)-C(2)	-8.1(5)	N(3)-Cu(1)-N(2)-C(2)	78.1(5)
N(4)-Cu(1)-N(2)-C(9)	-38.5(5)	N(1)-Cu(1)-N(2)-C(9)	108.1(5)
N(3)-Cu(1)-N(2)-C(9)	-165.7(5)	C(2)-N(2)-C(3)-C(4)	-63.1(9)
C(9)-N(2)-C(3)-C(4)	176.2(7)	Cu(1)-N(2)-C(3)-C(4)	59.5(8)
N(2)-C(3)-C(4)-C(5)	-62.5(10)	N(4)-Cu(1)-N(3)-C(6)	161.8(4)
N(1)-Cu(1)-N(3)-C(6)	10.3(4)	N(2)-Cu(1)-N(3)-C(6)	-75.8(5)
N(4)-Cu(1)-N(3)-C(5)	-75.6(5)	N(1)-Cu(1)-N(3)-C(5)	133.0(5)
N(2)-Cu(1)-N(3)-C(5)	46.9(5)	N(4)-Cu(1)-N(3)-C(10)	42.7(6)
N(1)-Cu(1)-N(3)-C(10)	-108.7(5)	N(2)-Cu(1)-N(3)-C(10)	165.2(5)
C(6)-N(3)-C(5)-C(4)	59.6(9)	C(10)-N(3)-C(5)-C(4)	-177.1(8)
Cu(1)-N(3)-C(5)-C(4)	-59.5(8)	C(3)-C(4)-C(5)-N(3)	64.2(10)
C(5)-N(3)-C(6)-C(7)	-153.0(6)	C(10)-N(3)-C(6)-C(7)	85.2(8)
Cu(1)-N(3)-C(6)-C(7)	-31.5(7)	C(8)-N(1)-C(7)-C(6)	-159.1(7)
C(1)-N(1)-C(7)-C(6)	80.7(7)	Cu(1)-N(1)-C(7)-C(6)	-34.3(7)
N(3)-C(6)-C(7)-N(1)	46.7(9)	N(1)-Cu(1)-N(4)-C(11)	-164(4)
N(2)-Cu(1)-N(4)-C(11)	-45(4)	N(3)-Cu(1)-N(4)-C(11)	65(4)
Cu(1)-N(4)-C(11)-C(12)	-156(25)		

Symmetry transformations used to generate equivalent atoms:

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 97328aaa.

	x	y	z	U(eq)	SOF
H(1A)	1261(12)	6449(5)	2520(9)	55	1
H(1B)	605(12)	7367(5)	1499(9)	55	1
H(2A)	-276(9)	8087(7)	3733(8)	49	1
H(2B)	-1029(9)	7039(7)	3765(8)	49	1
H(3A)	1124(12)	7710(6)	7898(10)	61	1
H(3B)	-836(12)	7753(6)	6852(10)	61	1
H(4A)	-14(13)	9111(7)	5484(11)	66	1
H(4B)	137(13)	9281(7)	7405(11)	66	1
H(5A)	2513(11)	9995(6)	6522(9)	53	1
H(5B)	3263(11)	9137(6)	7653(9)	53	1
H(6A)	3070(10)	9846(5)	3375(9)	47	1
H(6B)	1387(10)	9207(5)	3647(9)	47	1
H(7A)	2367(11)	8629(5)	1346(8)	48	1
H(7B)	4424(11)	8651(5)	2173(8)	48	1
H(8A)	3786(69)	7127(42)	680(12)	90	1
H(8B)	4038(79)	6310(7)	2021(70)	90	1
H(8C)	5437(20)	7161(39)	2136(65)	90	1
H(9A)	-388(24)	6172(14)	6234(80)	75	1
H(9B)	1656(69)	6166(15)	7107(44)	75	1
H(9C)	1185(85)	5861(6)	5231(39)	75	1
H(10A)	5354(13)	10057(7)	5540(83)	81	1
H(10B)	6133(31)	9137(39)	4783(53)	81	1
H(10C)	6003(36)	9187(41)	6697(36)	81	1
H(12A)	8588(31)	5998(41)	9214(24)	62	1
H(12B)	6828(43)	5587(25)	9842(47)	62	1
H(12C)	7548(70)	6608(17)	10406(27)	62	1

REFERENCE NUMBER: 97301a

$[(iPr_3TACDD)Cu]ClO_4$

CRYSTAL STRUCTURE REPORT

$C_{18}H_{39}ClCuN_3O_4$

Report prepared for:

B. Lam / Prof. W. Tolman

11 November 1997

Victor G. Young, Jr.
X-Ray Crystallographic Laboratory
160 Kolthoff Hall
Chemistry Department
207 Pleasant St. S.E.
The University of Minnesota
Minneapolis, MN 55455

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 116 reflections. Final cell constants are calculated from a set of 7785 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 $Pca2_1$ 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 relative isotropic displacement parameters.

The complex was found as expected. The solid material appears to be stable in air for at least 18 hours.

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:

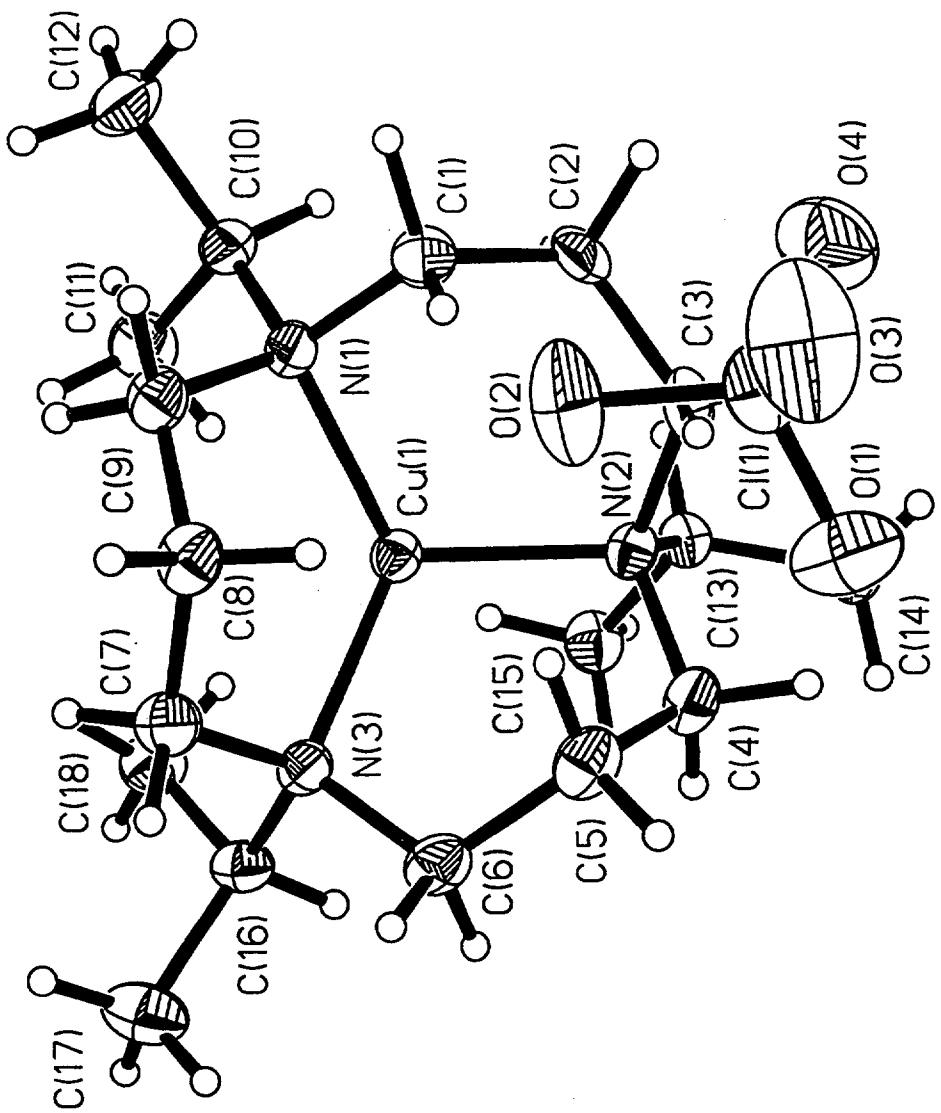
$$R_{int}=\sum|F_o^2-\langle F_o^2\rangle|/\sum|F_o^2|$$

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

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

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

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



isopropyl grp blocking entry in adjacent molecule.

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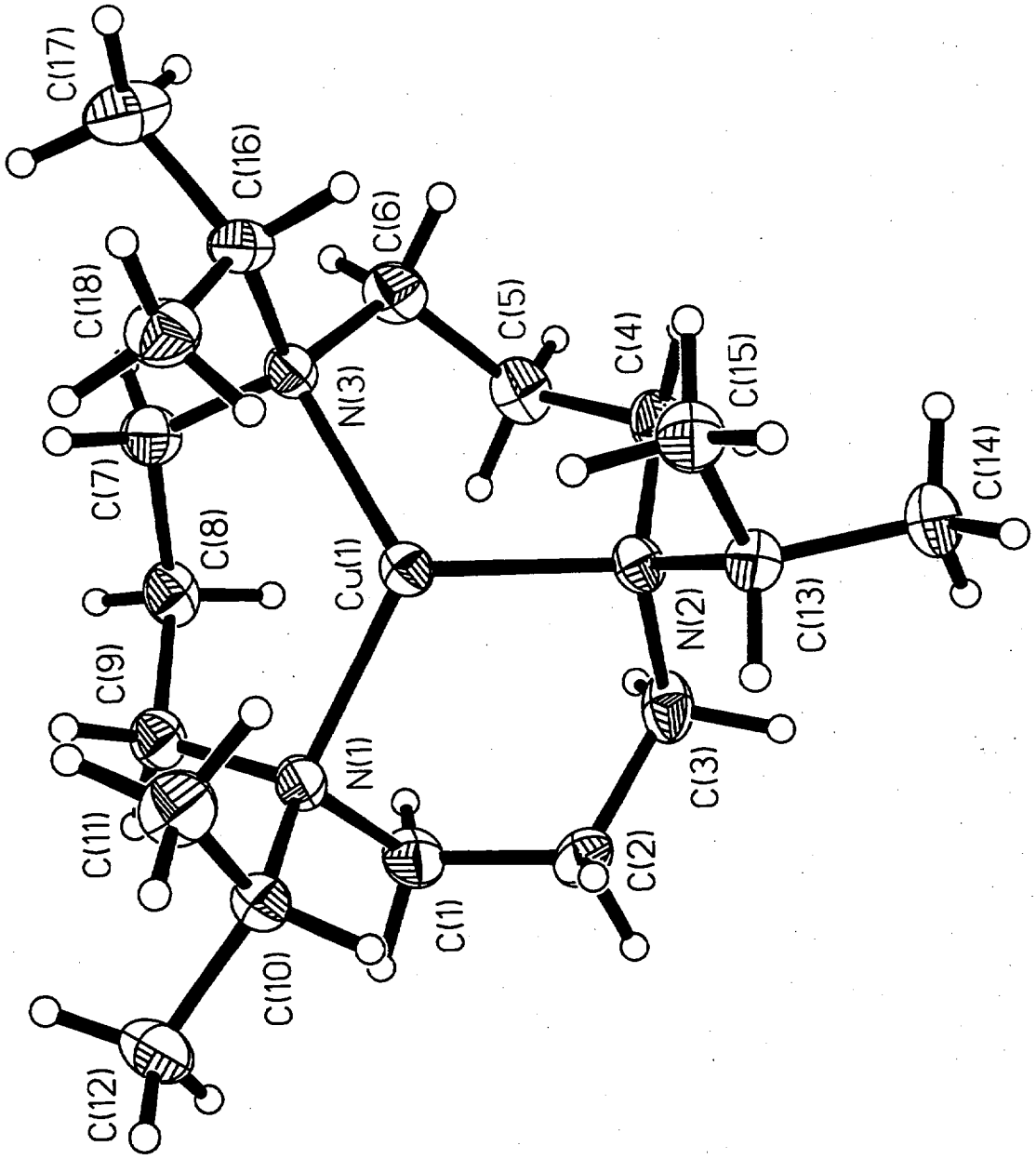

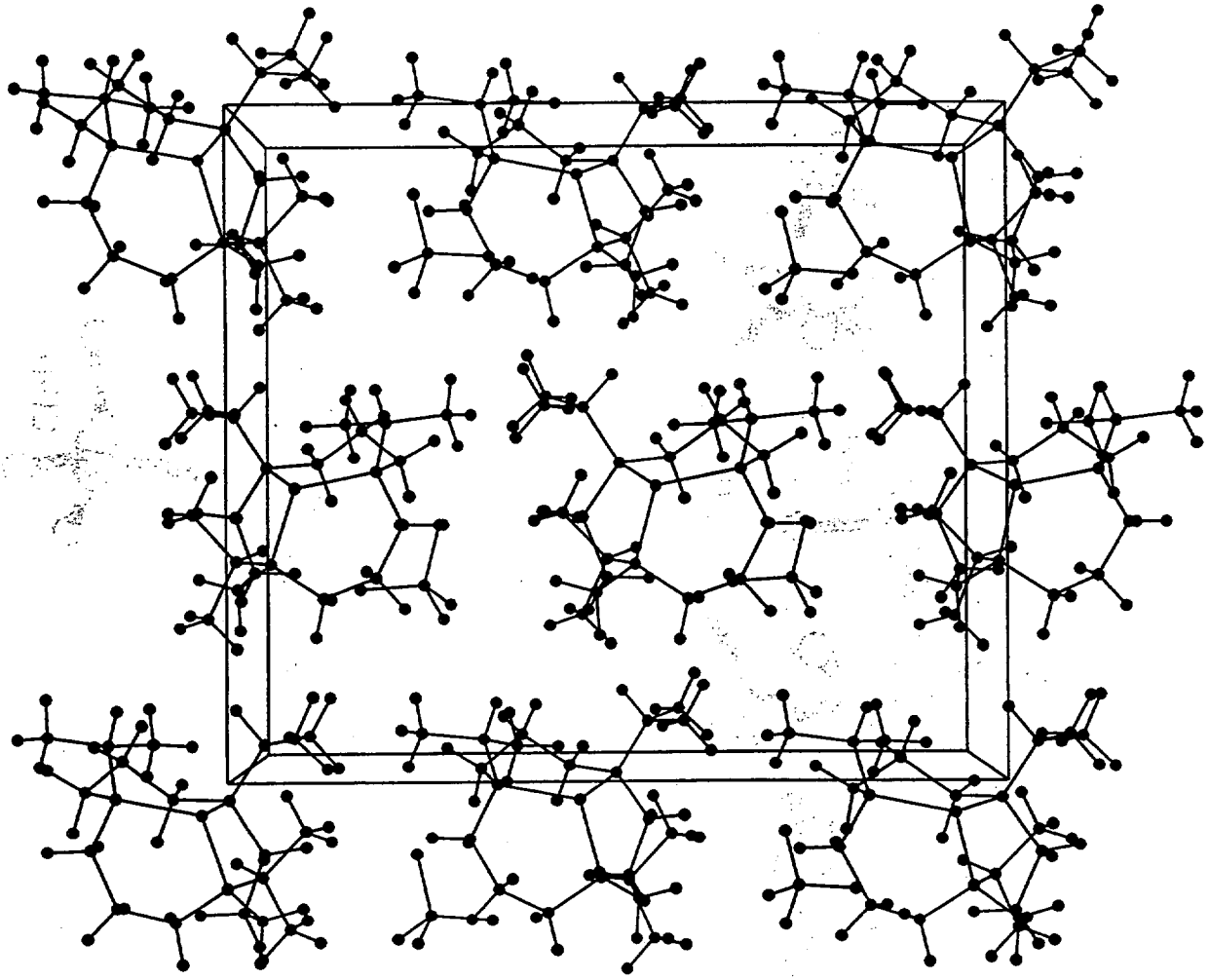

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Least-squares plane number 1 (XO = orthogonal, x = Crystal coordinates)

-0.3900 XO + 0.6495 YO + 0.6527 ZO = 6.5474
-6.807 x + 5.427 y + 9.807 z = 6.5474

	Deviation	Weight	
	0.4656	1.0000	CU1
+	0.0000	1.0000	N1
	-0.9942	1.0000	C1
	-0.5731	1.0000	C2
	-0.9056	1.0000	C3
+	0.0000	1.0000	N2
	-0.7519	1.0000	C4
	-1.6401	1.0000	C5
	-0.9293	1.0000	C6
+	0.0000	1.0000	N3
	-0.7034	1.0000	C7
	-1.5062	1.0000	C8
	-0.7027	1.0000	C9
	1.1364	1.0000	C10
	2.3360	1.0000	C11
	0.7734	1.0000	C12
	1.2308	1.0000	C13
	1.0054	1.0000	C14
	2.3440	1.0000	C15
	1.2043	1.0000	C16
	0.9017	1.0000	C17
	2.3308	1.0000	C18

Mean deviation from plane = 0.0000 Angstroms

Table 1. Crystal data, data collection, and solution and refinement for 97301a.

Crystal Data	
Empirical formula	$C_{18}H_{39}ClCuN_3O_4$
Crystal Habit, color	Plate, Colorless
Crystal size	0.40 x 0.19 x 0.06 mm
Crystal system	Orthorhombic
Space group	$Pca2_1$
	a = 17.4559(3) Å α = 90°
	b = 8.35520(10) Å β = 90°
	c = 15.0250(3) Å γ = 90°
Volume	2191.36(6) $Å^3$
Z	4
Formula weight	460.51
Density (calculated)	1.396 Mg/m^3
Absorption coefficient	1.146 mm^{-1}
F(000)	984
Data Collection	
Diffractometer	Siemens SMART Platform CCD
Wavelength	0.71073 Å
Temperature	173(2) K
θ range for data collection	2.33 to 25.01°
Index ranges	$0 \le h \le 20$, $0 \le k \le 9$, $-14 \le \ell \le 17$
Reflections collected	10981
Independent reflections	3416 (R_{int} = 0.0347)
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 = 0.0183, and B = 0.3103
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.0000 and 0.823
Absolute structure parameter	0.008(12)
Data / restraints / parameters	3416 / 1 / 250
R indices (I>2σ(I) = 3100)	R1 = 0.0285, wR2 = 0.0560
R indices (all data)	R1 = 0.0352, wR2 = 0.0579
Goodness-of-fit on F^2	1.074
Largest diff. peak and hole	0.213 and -0.238 $eÅ^{-3}$

Table 2. Atomic coordinates [$x\ 10^4$] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 97301a. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
Cu(1)	5533(1)	2941(1)	9364(1)	21(1)	1
N(1)	5032(1)	848(3)	9700(2)	22(1)	1
C(1)	5697(2)	-291(3)	9778(2)	29(1)	1
C(2)	6369(2)	337(3)	10327(2)	30(1)	1
C(3)	6933(2)	1366(3)	9810(2)	29(1)	1
N(2)	6669(1)	3048(3)	9619(1)	21(1)	1
C(4)	7046(2)	3637(4)	8788(2)	28(1)	1
C(5)	6660(2)	3063(4)	7932(2)	30(1)	1
C(6)	5973(2)	4061(4)	7628(2)	29(1)	1
N(3)	5248(1)	3888(3)	8167(2)	22(1)	1
C(7)	4720(2)	2678(3)	7754(2)	29(1)	1
C(8)	4862(2)	924(4)	8004(2)	28(1)	1
C(9)	4560(2)	387(4)	8910(2)	27(1)	1
C(10)	4568(2)	831(4)	10545(2)	26(1)	1
C(11)	4054(2)	2290(4)	10605(2)	35(1)	1
C(12)	4101(2)	-688(4)	10693(2)	40(1)	1
C(13)	6836(2)	4114(3)	10400(2)	24(1)	1
C(14)	7682(2)	4549(4)	10516(2)	30(1)	1
C(15)	6351(2)	5634(3)	10357(2)	29(1)	1
C(16)	4861(2)	5508(3)	8231(2)	26(1)	1
C(17)	4515(2)	6092(4)	7359(2)	41(1)	1
C(18)	4264(2)	5506(4)	8966(2)	32(1)	1
Cl(1)	7464(1)	-1793(1)	7890(1)	34(1)	1
O(1)	7850(2)	-373(4)	7628(2)	73(1)	1
O(2)	6653(1)	-1568(3)	7828(2)	56(1)	1
O(3)	7685(2)	-3123(4)	7353(2)	72(1)	1
O(4)	7659(2)	-2125(3)	8798(2)	54(1)	1

Table 3. Bond lengths [Å] and angles [°] for 97301a.

Cu(1)-N(1)	2.019(2)	Cu(1)-N(2)	2.022(2)
Cu(1)-N(3)	2.026(2)	N(1)-C(9)	1.495(3)
N(1)-C(1)	1.506(3)	N(1)-C(10)	1.507(4)
C(1)-C(2)	1.527(4)	C(2)-C(3)	1.521(4)
C(3)-N(2)	1.506(4)	N(2)-C(4)	1.495(3)
N(2)-C(13)	1.502(3)	C(4)-C(5)	1.529(4)
C(5)-C(6)	1.530(4)	C(6)-N(3)	1.511(3)
N(3)-C(7)	1.502(3)	N(3)-C(16)	1.515(3)
C(7)-C(8)	1.533(4)	C(8)-C(9)	1.528(4)
C(10)-C(11)	1.516(4)	C(10)-C(12)	1.525(4)
C(13)-C(15)	1.527(4)	C(13)-C(14)	1.531(4)
C(16)-C(18)	1.519(4)	C(16)-C(17)	1.523(4)
Cl(1)-O(1)	1.420(3)	Cl(1)-O(3)	1.427(2)
Cl(1)-O(2)	1.430(2)	Cl(1)-O(4)	1.434(2)

N(1)-Cu(1)-N(2)	114.53(9)	N(1)-Cu(1)-N(3)	116.96(9)
N(2)-Cu(1)-N(3)	113.09(8)	C(9)-N(1)-C(1)	108.9(2)
C(9)-N(1)-C(10)	111.7(2)	C(1)-N(1)-C(10)	110.1(2)
C(9)-N(1)-Cu(1)	105.3(2)	C(1)-N(1)-Cu(1)	103.5(2)
C(10)-N(1)-Cu(1)	116.8(2)	N(1)-C(1)-C(2)	114.7(2)
C(3)-C(2)-C(1)	114.5(3)	N(2)-C(3)-C(2)	115.2(2)
C(4)-N(2)-C(13)	111.8(2)	C(4)-N(2)-C(3)	109.3(2)
C(13)-N(2)-C(3)	110.2(2)	C(4)-N(2)-Cu(1)	106.8(2)
C(13)-N(2)-Cu(1)	111.4(2)	C(3)-N(2)-Cu(1)	107.2(2)
N(2)-C(4)-C(5)	113.9(2)	C(4)-C(5)-C(6)	115.2(2)
N(3)-C(6)-C(5)	116.4(2)	C(7)-N(3)-C(6)	110.9(2)
C(7)-N(3)-C(16)	110.8(2)	C(6)-N(3)-C(16)	108.7(2)
C(7)-N(3)-Cu(1)	104.8(2)	C(6)-N(3)-Cu(1)	107.9(2)
C(16)-N(3)-Cu(1)	113.7(2)	N(3)-C(7)-C(8)	116.3(2)
C(9)-C(8)-C(7)	116.3(2)	N(1)-C(9)-C(8)	116.1(2)
N(1)-C(10)-C(11)	111.1(2)	N(1)-C(10)-C(12)	114.7(3)
C(11)-C(10)-C(12)	110.2(2)	N(2)-C(13)-C(15)	110.7(2)
N(2)-C(13)-C(14)	114.7(2)	C(15)-C(13)-C(14)	110.0(2)
N(3)-C(16)-C(18)	110.5(2)	N(3)-C(16)-C(17)	114.1(2)
C(18)-C(16)-C(17)	110.7(2)	O(1)-Cl(1)-O(3)	111.5(2)
O(1)-Cl(1)-O(2)	110.0(2)	O(3)-Cl(1)-O(2)	109.5(2)
O(1)-Cl(1)-O(4)	108.2(2)	O(3)-Cl(1)-O(4)	108.9(2)
O(2)-Cl(1)-O(4)	108.8(2)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for 97301a. 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)	20(1)	19(1)	22(1)	-1(1)	1(1)	-1(1)
N(1)	20(1)	21(1)	26(1)	1(1)	0(1)	-1(1)
C(1)	30(2)	15(2)	41(2)	-1(1)	-2(1)	0(1)
C(2)	27(2)	21(2)	41(2)	7(1)	-9(2)	7(1)
C(3)	19(1)	25(2)	44(2)	-7(1)	-6(1)	5(1)
N(2)	20(1)	20(1)	24(2)	-4(1)	1(1)	-1(1)
C(4)	22(2)	35(2)	28(2)	-2(1)	3(1)	-4(1)
C(5)	30(2)	36(2)	25(2)	-6(1)	7(1)	-2(1)
C(6)	31(2)	34(2)	22(2)	0(1)	7(1)	-2(1)
N(3)	22(1)	23(1)	22(1)	0(1)	1(1)	-3(1)
C(7)	34(2)	27(2)	25(2)	-1(1)	-4(1)	-4(1)
C(8)	31(2)	28(2)	25(2)	-9(1)	-3(1)	-6(1)
C(9)	28(2)	24(2)	29(2)	-3(1)	-4(1)	-4(1)
C(10)	26(2)	24(2)	27(2)	3(1)	-3(1)	-5(1)
C(11)	33(2)	42(2)	30(2)	1(1)	7(1)	6(2)
C(12)	39(2)	42(2)	38(2)	9(2)	2(2)	-15(2)
C(13)	22(1)	25(2)	24(2)	-2(1)	-1(1)	1(1)
C(14)	27(2)	31(2)	32(2)	-7(1)	-4(1)	-5(1)
C(15)	29(2)	24(2)	33(2)	-4(1)	-1(1)	3(1)
C(16)	31(2)	21(2)	26(2)	3(1)	0(1)	-1(1)
C(17)	59(2)	30(2)	36(2)	5(1)	-5(2)	5(2)
C(18)	34(2)	24(2)	39(2)	1(1)	3(1)	5(1)
Cl(1)	31(1)	39(1)	31(1)	-7(1)	-1(1)	5(1)
O(1)	71(2)	65(2)	84(2)	31(2)	12(2)	-9(2)
O(2)	31(1)	78(2)	58(2)	-17(1)	-6(1)	12(1)
O(3)	63(2)	83(2)	70(2)	-51(2)	2(1)	21(2)
O(4)	65(2)	58(2)	40(1)	3(1)	-16(1)	-2(1)

Table 5. Torsion angles [°] for 97301a.

N(2)-Cu(1)-N(1)-C(9)	-132.5(2)	N(3)-Cu(1)-N(1)-C(9)	3.2(2)
N(2)-Cu(1)-N(1)-C(1)	-18.3(2)	N(3)-Cu(1)-N(1)-C(1)	117.4(2)
N(2)-Cu(1)-N(1)-C(10)	102.9(2)	N(3)-Cu(1)-N(1)-C(10)	-121.4(2)
C(9)-N(1)-C(1)-C(2)	159.6(2)	C(10)-N(1)-C(1)-C(2)	-77.7(3)
Cu(1)-N(1)-C(1)-C(2)	47.9(3)	N(1)-C(1)-C(2)-C(3)	-85.0(3)
C(1)-C(2)-C(3)-N(2)	76.3(3)	C(2)-C(3)-N(2)-C(4)	-152.5(2)
C(2)-C(3)-N(2)-C(13)	84.2(3)	C(2)-C(3)-N(2)-Cu(1)	-37.1(3)
N(1)-Cu(1)-N(2)-C(4)	130.5(2)	N(3)-Cu(1)-N(2)-C(4)	-7.0(2)
N(1)-Cu(1)-N(2)-C(13)	-107.3(2)	N(3)-Cu(1)-N(2)-C(13)	115.3(2)
N(1)-Cu(1)-N(2)-C(3)	13.4(2)	N(3)-Cu(1)-N(2)-C(3)	-124.1(2)
C(13)-N(2)-C(4)-C(5)	-156.5(2)	C(3)-N(2)-C(4)-C(5)	81.2(3)
Cu(1)-N(2)-C(4)-C(5)	-34.4(3)	N(2)-C(4)-C(5)-C(6)	83.8(3)
C(4)-C(5)-C(6)-N(3)	-72.7(3)	C(5)-C(6)-N(3)-C(7)	-95.4(3)
C(5)-C(6)-N(3)-C(16)	142.6(2)	C(5)-C(6)-N(3)-Cu(1)	18.8(3)
N(1)-Cu(1)-N(3)-C(7)	-3.1(2)	N(2)-Cu(1)-N(3)-C(7)	133.2(2)
N(1)-Cu(1)-N(3)-C(6)	-121.3(2)	N(2)-Cu(1)-N(3)-C(6)	15.0(2)
N(1)-Cu(1)-N(3)-C(16)	118.0(2)	N(2)-Cu(1)-N(3)-C(16)	-105.7(2)
C(6)-N(3)-C(7)-C(8)	85.2(3)	C(16)-N(3)-C(7)-C(8)	-154.0(2)
Cu(1)-N(3)-C(7)-C(8)	-31.0(3)	N(3)-C(7)-C(8)-C(9)	78.9(3)
C(1)-N(1)-C(9)-C(8)	-79.5(3)	C(10)-N(1)-C(9)-C(8)	158.7(2)
Cu(1)-N(1)-C(9)-C(8)	30.9(3)	C(7)-C(8)-C(9)-N(1)	-78.7(3)
C(9)-N(1)-C(10)-C(11)	-76.3(3)	C(1)-N(1)-C(10)-C(11)	162.6(2)
Cu(1)-N(1)-C(10)-C(11)	45.0(3)	C(9)-N(1)-C(10)-C(12)	49.5(3)
C(1)-N(1)-C(10)-C(12)	-71.6(3)	Cu(1)-N(1)-C(10)-C(12)	170.8(2)
C(4)-N(2)-C(13)-C(15)	77.0(3)	C(3)-N(2)-C(13)-C(15)	-161.2(2)
Cu(1)-N(2)-C(13)-C(15)	-42.4(3)	C(4)-N(2)-C(13)-C(14)	-48.2(3)
C(3)-N(2)-C(13)-C(14)	73.6(3)	Cu(1)-N(2)-C(13)-C(14)	-167.5(2)
C(7)-N(3)-C(16)-C(18)	73.8(3)	C(6)-N(3)-C(16)-C(18)	-164.1(2)
Cu(1)-N(3)-C(16)-C(18)	-43.9(3)	C(7)-N(3)-C(16)-C(17)	-51.7(3)
C(6)-N(3)-C(16)-C(17)	70.3(3)	Cu(1)-N(3)-C(16)-C(17)	-169.4(2)

Symmetry transformations used to generate equivalent atoms:

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 97301a.

	x	y	z	U(eq)	SOF
H(1A)	5883(2)	-548(3)	9172(2)	34	1
H(1B)	5513(2)	-1299(3)	10050(2)	34	1
H(2A)	6165(2)	973(3)	10829(2)	36	1
H(2B)	6649(2)	-588(3)	10581(2)	36	1
H(3A)	7419(2)	1421(3)	10149(2)	35	1
H(3B)	7045(2)	830(3)	9236(2)	35	1
H(4A)	7587(2)	3277(4)	8786(2)	34	1
H(4B)	7047(2)	4822(4)	8794(2)	34	1
H(5A)	6489(2)	1945(4)	8020(2)	36	1
H(5B)	7046(2)	3059(4)	7449(2)	36	1
H(6A)	6125(2)	5202(4)	7633(2)	34	1
H(6B)	5856(2)	3772(4)	7003(2)	34	1
H(7A)	4760(2)	2777(3)	7099(2)	34	1
H(7B)	4188(2)	2954(3)	7921(2)	34	1
H(8A)	4627(2)	239(4)	7541(2)	34	1
H(8B)	5422(2)	727(4)	7989(2)	34	1
H(9A)	4039(2)	836(4)	8990(2)	32	1
H(9B)	4510(2)	-793(4)	8903(2)	32	1
H(10A)	4940(2)	906(4)	11050(2)	31	1
H(11A)	4366(2)	3262(4)	10558(14)	52	1
H(11B)	3784(9)	2286(14)	11177(6)	52	1
H(11C)	3680(8)	2266(13)	10119(8)	52	1
H(12A)	3873(10)	-665(13)	11288(6)	60	1
H(12B)	4437(3)	-1624(4)	10640(15)	60	1
H(12C)	3694(8)	-753(14)	10245(9)	60	1
H(13A)	6676(2)	3520(3)	10947(2)	28	1
H(14A)	7991(2)	3570(4)	10523(13)	45	1
H(14B)	7751(2)	5125(21)	11079(7)	45	1
H(14C)	7846(3)	5232(20)	10021(7)	45	1
H(15A)	5808(2)	5346(3)	10332(13)	44	1
H(15B)	6489(8)	6247(12)	9824(7)	44	1
H(15C)	6447(8)	6286(11)	10888(6)	44	1
H(16A)	5265(2)	6298(3)	8405(2)	31	1
H(17A)	4903(4)	6044(24)	6888(4)	62	1
H(17B)	4339(11)	7198(10)	7430(5)	62	1
H(17C)	4080(8)	5409(16)	7198(8)	62	1
H(18A)	4514(2)	5297(24)	9538(3)	49	1
H(18B)	3884(6)	4670(17)	8847(7)	49	1
H(18C)	4010(8)	6551(9)	8986(9)	49	1

REFERENCE NUMBER: 98038cc

$[(iPr_3TACD)_2Cu_2(\mu\text{-}O_2)](BPh_4)_2$

CRYSTAL STRUCTURE REPORT

$C_{91}H_{90}D_{42}B_2Cl_4Cu_2N_6O_5$

Report prepared for:

B. Lam / Prof. W. Tolman

3 March 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 148(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 334 reflections. Final cell constants are calculated from a set of 7729 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 Pn 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 relative isotropic displacement parameters.

The structure was found as expected along with three molecules of acetone and two molecules of dichloromethane. All solvent was disordered. PLATON/SQUEEZE was used to remove the effect of the disordered solvent from the data in order to refine the remainder of the structure as accurately as possible. PLATON/SQUEEZE[2] found a potential solvent volume = 1277.8 $Å^3$ out of the unit cell volume of 4557.0 $Å^3$, or 28.0% of the total. The solvent (see packing diagram) appears to form sheets between the dication and anions. The residuals improved about 15% after this was done. PLATON/SQUEEZE estimated 452 electrons of scattering within the void. The empirical formula is correct considering the disordered solvent initially located was all of it. Volume considerations would argue that as much as 32 non-hydrogen atoms could fill the asymmetric unit and this is less than the 18 partially located. The deuterium content was taken into account when calculating the formula, density, and formula weight.

The solution was possible in either Pn or $P2_1/n$. In Pn a pair of ipr groups are ordered, but in $P2_1/n$ they must be disordered. This is the only significant obstacle to this structure being centrosymmetric. As a consequence, the two BPh_4 anions are pseudosymmetrically related as is the pair of copper complex monomers. The residuals for Pn are better than $P2_1/n$ by ~3%. The Hamilton test would select Pn as the better space group at the 99% confidence level. Still many restraints were imposed to keep this fully anisotropic structure positive definite. The parts related by the pseudosymmetry were made to have similar α and β bond distance by using the SAME restraint. Some groups had bizarre thermal ellipsoids so DELU and SIMU restraints were applied where found necessary. The copper and oxygen atoms were not restrained. 771 restraints were applied altogether. The Pn result is racemically twinned in a 0.54:0.46 ratio.

The core does look odd with both oxygens librating above and below the plane. It doesnot seem that oxygen would have two closely spaced positions so these were left as is. The librational correction for bond distances based on the oxygen cannot be computed (indeterminite). The Cu(1)···Cu(2) distance is 3.519(1) Å.

Crystallographers advice: This refinement certainly confirms that the peroxo species is present. Despite the low R1 (due to PLATON correction) and the apparent small esds on bond lengths and angles, I would not push the precision of the results. You should think that these are actually bigger than listed due to the procedure used. Pseudosymmetry is still a problem with these results.

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:

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

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

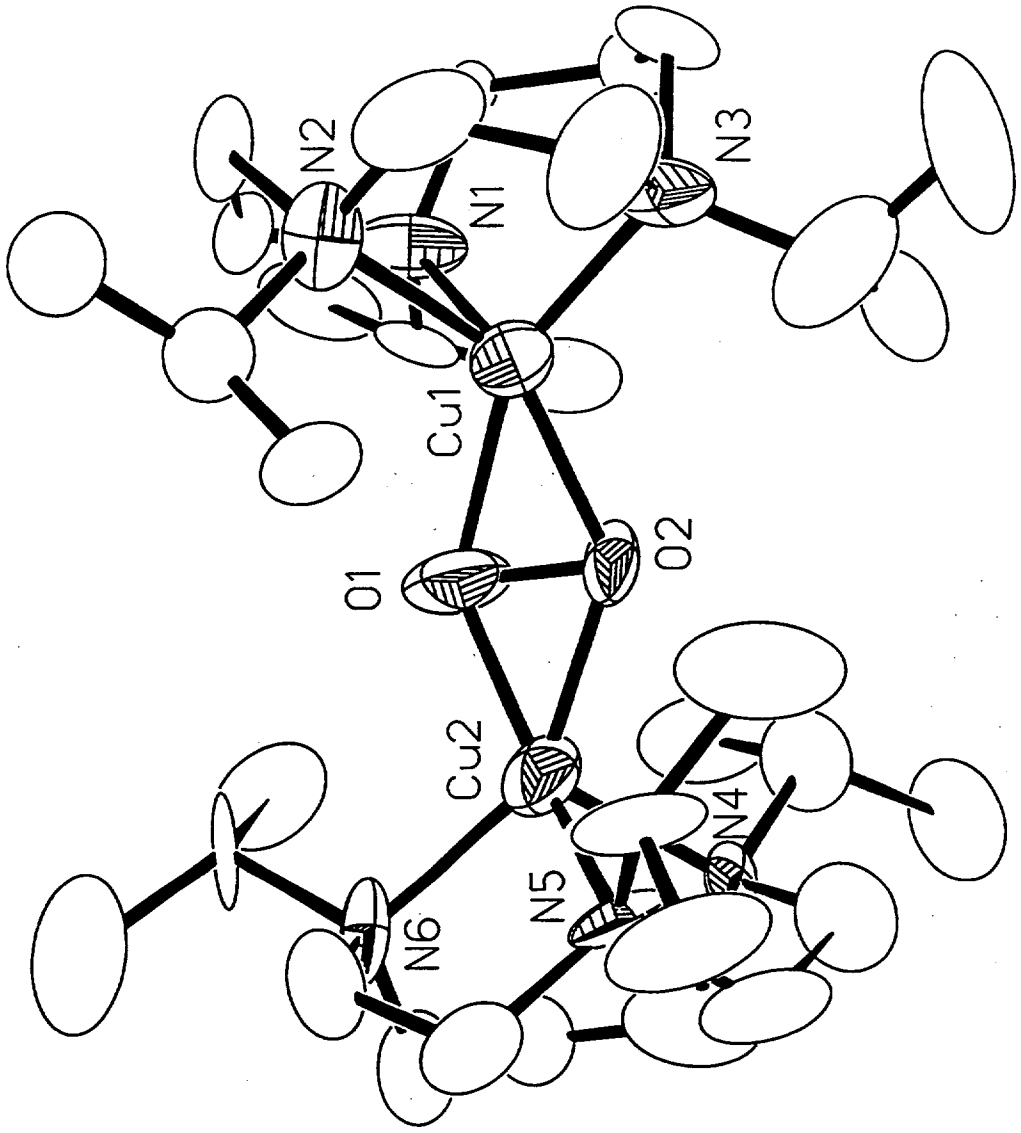
$$wR2=[\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2},$$

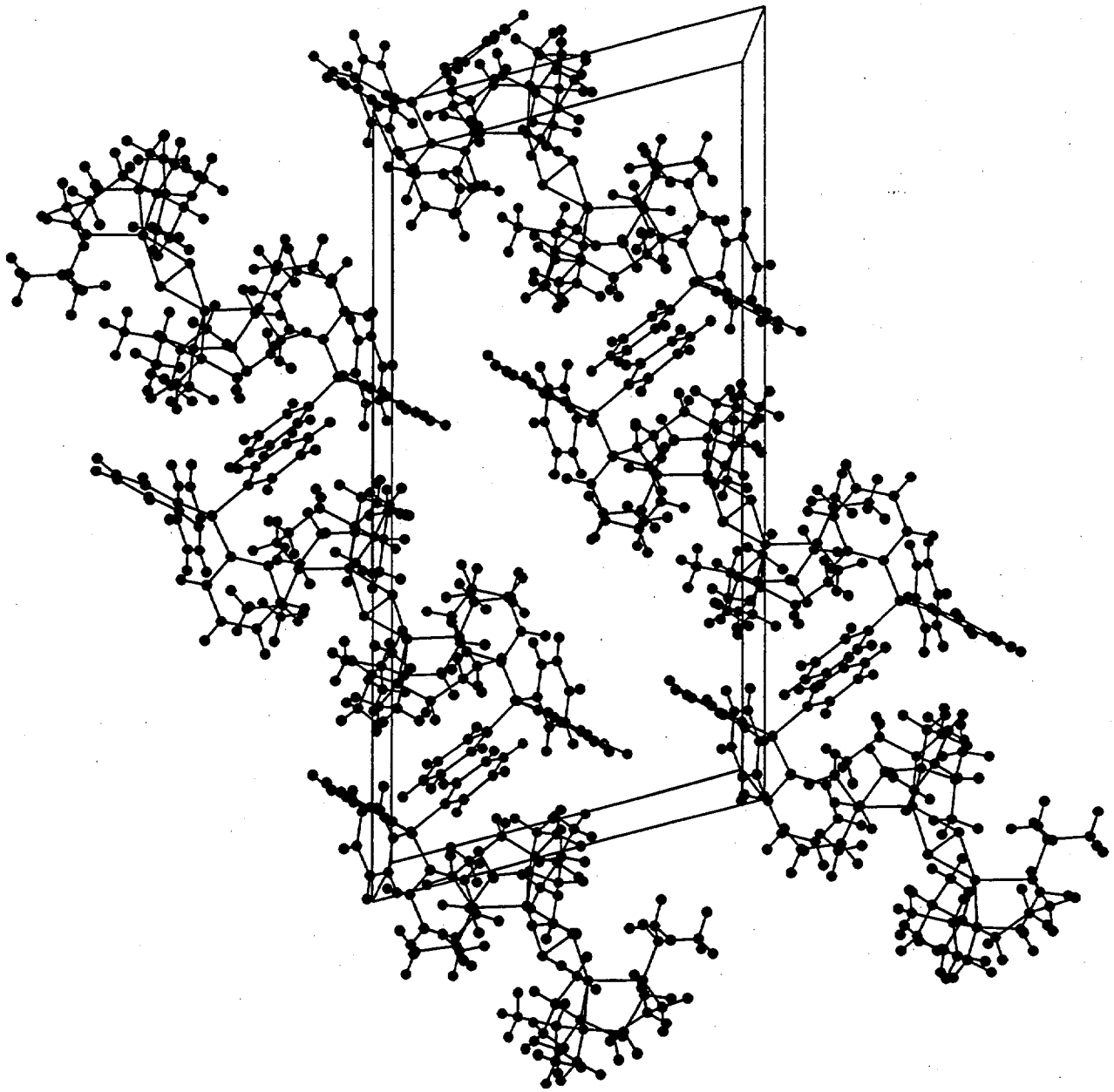
$$\text{where } w=q/\sigma^2(F_o^2)+(a*P)^2+b*P$$

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

Table 1. Crystal data, data collection, and solution and refinement for 98038cc.

Crystal Data	
Empirical formula	$C_{91}H_{90}D_{42}B_2Cl_4Cu_2N_6O_5$
Crystal Habit, color	Plate, Purple-brown, translucent
Crystal size	0.45 x 0.21 x 0.04 mm
Crystal system	Monoclinic
Space group	Pn
	a = 14.4665(3) Å α = 90°
	b = 11.7064(2) Å β = 104.728(1)°
	c = 27.8227(6) Å γ = 90°
Volume	4557.0(2) Å^3
Z	2
Formula weight	1722.87
Density (calculated)	1.256 Mg/m^3
Absorption coefficient	0.636 mm^{-1}
F(000)	1792
Data Collection	
Diffractometer	Siemens SMART Platform CCD
Wavelength	0.71073 Å
Temperature	148(2) K
θ range for data collection	1.46 to 25.03°
Index ranges	$-17 \le h \le 13$, $0 \le k \le 13$, $-33 \le \ell \le 32$
Reflections collected	21514
Independent reflections	10337 (R_{int} = 0.0283)
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 = 0.0621, and B = 0.0
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.000 and 0.845
Absolute structure parameter	0.46(3)
Data / restraints / parameters	10335 / 771 / 824
R indices (I>2σ(I) = 6208)	R1 = 0.0524, wR2 = 0.1197
R indices (all data)	R1 = 0.0840, wR2 = 0.1321
Goodness-of-fit on F^2	0.961
Largest diff. peak and hole	0.362 and -0.420 eÅ^{-3}





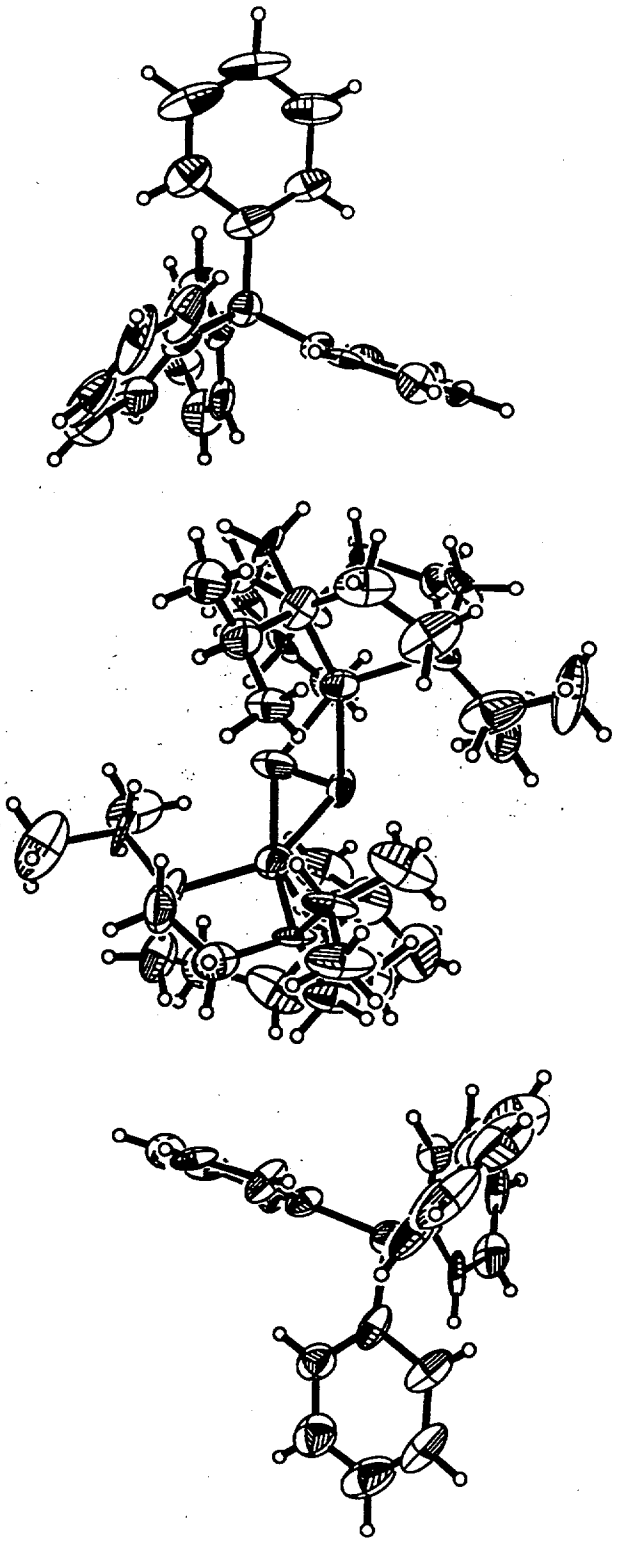


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Table 1. Crystal data, data collection, and solution and refinement for 98038cc.

Crystal Data	
Empirical formula	$C_{91}H_{152}BCl_4Cu_2N_6O_5$
Crystal Habit, color	Plate, Purple-brown, translucent
Crystal size	0.45 x 0.21 x 0.04 mm
Crystal system	Monoclinic
Space group	Pn
	a = 14.4665(3) Å α = 90°
	b = 11.7064(2) Å β = 104.728(1)°
	c = 27.8227(6) Å γ = 90°
Volume	4557.0(2) Å^3
Z	2
Formula weight	1689.88
Density (calculated)	1.232 Mg/m^3
Absorption coefficient	0.636 mm^{-1}
F(000)	1822
Data Collection	
Diffractometer	Siemens SMART Platform CCD
Wavelength	0.71073 Å
Temperature	148(2) K
θ range for data collection	1.46 to 25.03°
Index ranges	$-17 \le h \le 13$, $0 \le k \le 13$, $-33 \le \ell \le 32$
Reflections collected	21514
Independent reflections	10337 (R_{int} = 0.0283)
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 = 0.062100, and B = 0.0
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.000 and 0.845
Absolute structure parameter	0.46(3)
Data / restraints / parameters	10335 / 771 / 824
R indices (I>2σ(I) = 6208)	R1 = 0.0524, wR2 = 0.1197
R indices (all data)	R1 = 0.0840, wR2 = 0.1321
Goodness-of-fit on F^2	0.961
Largest diff. peak and hole	0.362 and -0.420 eÅ^{-3}

Table 2. Atomic coordinates [$x\ 10^4$] and equivalent isotropic displacement parameters [$Å^2\ x\ 10^3$] for 98038cc. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
Cu(1)	1087(1)	2096(1)	4315(1)	58(1)	1
O(1)	-117(7)	2164(10)	3799(3)	106(4)	1
N(1)	865(6)	3184(6)	4878(3)	59(3)	1
C(1)	296(6)	2375(7)	5127(3)	49(3)	1
C(2)	799(7)	1251(7)	5274(3)	55(3)	1
N(2)	1012(5)	663(5)	4826(3)	47(2)	1
C(3)	1969(7)	205(8)	4926(4)	69(4)	1
C(4)	2556(9)	513(6)	4573(4)	80(4)	1
N(3)	2491(6)	1830(6)	4492(3)	54(3)	1
C(5)	3050(7)	2435(8)	4945(3)	56(3)	1
C(6)	2634(6)	3563(8)	5028(4)	50(3)	1
C(7)	1787(4)	3532(7)	5246(2)	27(2)	1
C(8)	273(6)	4193(7)	4666(3)	46(3)	1
C(9)	735(7)	4923(7)	4361(4)	64(4)	1
C(10)	-172(7)	4885(9)	5025(4)	89(5)	1
C(11)	248(7)	-196(9)	4627(4)	56(3)	1
C(12)	397(5)	-707(5)	4162(2)	55(2)	1
C(13)	180(9)	-1141(9)	4992(4)	63(3)	1
C(14)	2931(11)	1958(10)	4055(4)	104(6)	1
C(15)	3982(9)	1570(13)	4141(7)	129(7)	1
C(16)	2894(8)	3215(11)	3902(5)	99(5)	1
Cu(2)	-518(1)	2109(1)	3129(1)	56(1)	1
O(2)	665(6)	2257(8)	3606(3)	74(3)	1
N(4)	-317(4)	3217(6)	2570(3)	40(2)	1
C(17)	238(7)	2424(7)	2300(4)	66(4)	1
C(18)	-215(8)	1268(7)	2186(3)	63(4)	1
N(5)	-379(5)	694(6)	2619(2)	46(2)	1
C(19)	-1409(6)	173(8)	2491(4)	57(3)	1
C(20)	-1950(7)	583(8)	2863(4)	71(4)	1
N(6)	-1940(6)	1816(6)	2965(3)	48(3)	1
C(21)	-2462(8)	2427(8)	2511(4)	64(4)	1
C(22)	-2104(7)	3575(7)	2407(4)	53(3)	1
C(23)	-1219(7)	3561(9)	2213(4)	91(5)	1
C(24)	341(8)	4205(9)	2798(4)	85(5)	1
C(25)	-244(9)	5068(8)	3032(4)	93(5)	1
C(26)	670(9)	4889(9)	2395(5)	107(5)	1
C(27)	318(7)	-242(8)	2823(3)	56(3)	1
C(28)	1316(6)	167(6)	3047(3)	98(3)	1
C(29)	336(10)	-1252(9)	2466(4)	81(5)	1
C(30)	-2356(6)	2045(7)	3411(4)	53(3)	1
C(31)	-3384(8)	1618(10)	3337(6)	103(6)	1
C(32)	-2301(9)	3338(8)	3539(4)	78(4)	1
B(1)	1358(9)	2576(9)	6996(5)	40(1)	1
C(101)	523(8)	1616(8)	6756(4)	54(3)	1
C(102)	-330(9)	1823(9)	6407(4)	82(4)	1
C(103)	-933(9)	885(11)	6193(5)	102(4)	1
C(104)	-659(10)	-222(11)	6297(5)	110(4)	1
C(105)	183(9)	-437(9)	6645(6)	93(4)	1
C(106)	780(8)	465(8)	6851(5)	72(3)	1
C(107)	1791(8)	2343(8)	7573(3)	51(3)	1
C(108)	1246(8)	1913(10)	7895(4)	61(3)	1
C(109)	1599(10)	1749(12)	8395(4)	90(4)	1
C(110)	2542(10)	2012(10)	8620(4)	86(4)	1
C(111)	3084(9)	2470(10)	8331(4)	70(3)	1