Electronic Supplementary Material (ESI) for Dalton Transactions

Copper(II) chemistry of the functionalized macrocycle cyclam tetraproprionic acid.

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Details of the potentiometric titration

For reasons of comparison the determination of the complex stability of H_4TETP with Cu^{II} was done by potentiometric titrations according to Martell et al.[1]

In a typical competition experiment for the determination of the formation constants of the complex $[Cu(H_nTETP)]^{m+}$ a ternary aqueous solution of 0.025 mmol H_4EDTA, 0.025 mmol H_4TETP and 0.025 mmol $CuSO_4$ was prepared in a total volume of 50.25 ml. Titrations were performed from acidic to basic milieu and *vice versa* (with the same solution) in order to assure that the equilibrium was reached after each addition of base or acid, respectively (Figure S1). Starting in acidic milieu 4 ml of a 0.1 M KOH was added in 0.05 ml increments (80 points of measurements) allowing a minimum stirring time of 10 minutes.[2]

As can be seen in Figure S1, the titration curves are almost congruent indicating that the system was in the equilibrium all over the titration.

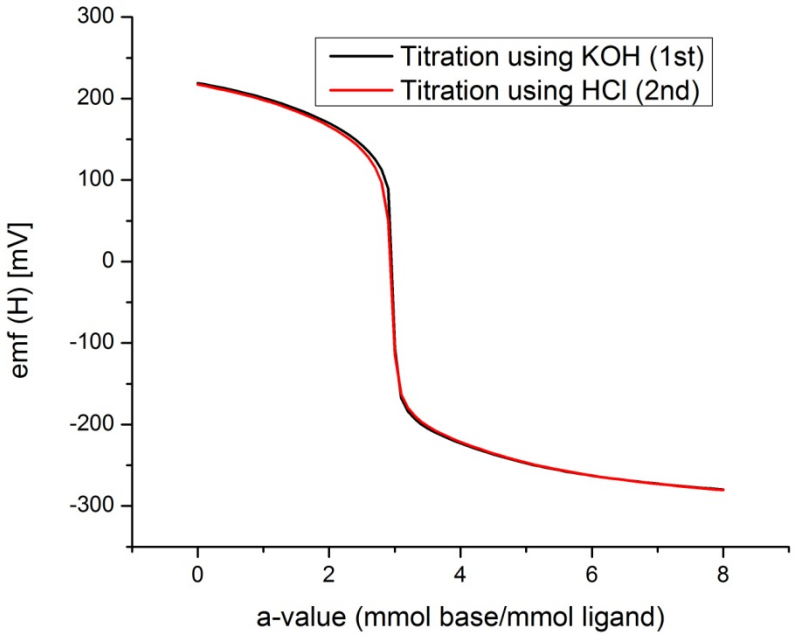

Figure S1: Titration of the ternary solution of EDTA, TETP, Cu(II) (each 0.025 mmol in 50.25 ml H_2O). The titration from acidic to basic milieu (black) *et vice versa* (red) is shown.

For the analysis, literature-known values for the dissociation constants of EDTA and complex formation constants of EDTA-Cu were used:

EDTA (μ = 0.1 M, 25 °C); log K 10.17, 6.11, 2.68, 2.0; Cu(II)-EDTA: log K_{ML} = 18.78, log K_{MHL} = 3.10, log K_{MH2L} = 2.0.[1] The dissociation constants, although literature-known[3] were re-determined under the conditions mentioned above and are in good agreement:

TETP (μ = 0.1 M, 25 °C); log K 10.23 [10.47], 10.49 [10.65], 4.12 [4.33], 3.76 [3.60], 2.89 [3.06], 2.75 [2.28] (literature-known values in brackets).

Thus, the following tableau (see Table S1) was used to determine the complex formation constant of TETP-Cu. The program Hyperquad 2008 Version 5.2.19 [4] was used to fit the parameter values (Figure S2).

The determination of the stability constants of CuTETP was performed on a qualitative level only. A fit of several different datasets (different titrations of the same ternary solution) did not afford consistent values of the formation constant by using the same model (i.e. inclusion of protonated complex species of TETP). That means, a fit using Hyperquad 2008 was not successful when assuming the appearance of species like TETPCuH or $TETPCuH_2$. When assuming a stability constant of CuTETP higher than 17.54 the measured and simulated titration curves are not congruent, thus the present data available allow for the before mentioned qualitative statement.

Table S1: Tableau of the ternary mixture of EDTA, TETP and Cu^{II}.

Formula	log *beta*	EDTA	TETP	Cu	H
EDTAH	10.17	1	0	0	1
$EDTAH_2$	16.28	1	0	0	2
$EDTAH_3$	18.96	1	0	0	3
$EDTAH_4$	20.96	1	0	0	4
EDTACu	18.78	1	0	1	0
EDTACuH	21.88	1	0	1	1
$EDTACuH_2$	23.88	1	0	1	2
TETPH	10.23	0	1	0	1
$TETPH_2$	20.72	0	1	0	2
$TETPH_3$	24.84	0	1	0	3
$TETPH_4$	28.60	0	1	0	4
$TETPH_5$	31.49	0	1	0	5
$TETPH_6$	34.24	0	1	0	6
TETPCu	**17.54**	0	1	1	0
H_{-1}	-13.78	0	0	0	-1

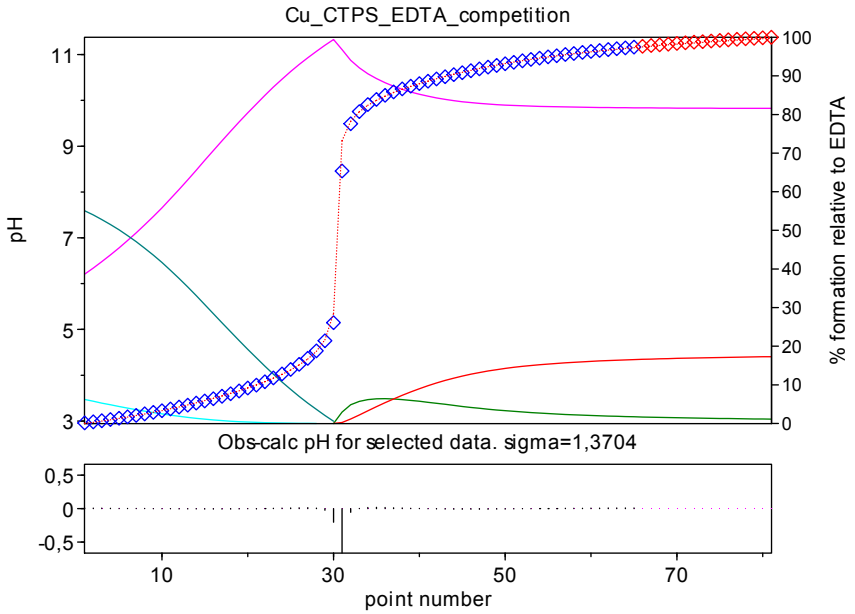

Figure S2: Obtained fit using the tableau of Table S1 and Hyperquad 2008.

General Procedure

The potentiometric titration were performed on a Titrando 905 equipped with an automated burette Dosino 800 that is controlled by the software package Tiamo Version 2.3 (Metrohm AG, Herisau, Switzerland), using a "blue line 17 pH"electrode from Schott Instruments (Schott AG / SI Analytics, Mainz, Germany). Aqueous volumetric solutions (0.1 M KOH, 0.1 M HCl, 0.1 M $CuSO_4$) as well as potassium chloride (trace metal basis) as electrolyte were purchased from Sigma-Aldrich GmbH (Taufkirchen, Germany). For the titrations doubly distilled water was used. Therefore, a MilliQ-UV-Plus-apparatus from Millipore (APS Water Services Inc., Van Nuys, CA, USA) was charged with deionized water. The resistance of the water taken for the titrations was 18.2 MΩ. The titration was done in a sealed and temperature controlled (25 °C) vessel, applying a slight stream of Argon over the solution.

Prior to each titration the E_0- and slope-values of the electrode were determined according to the procedure of Gans et al., using the program GLEE Version 3.0.21.[5]

Comparison of the Solution and Solid State Electronic Spectra

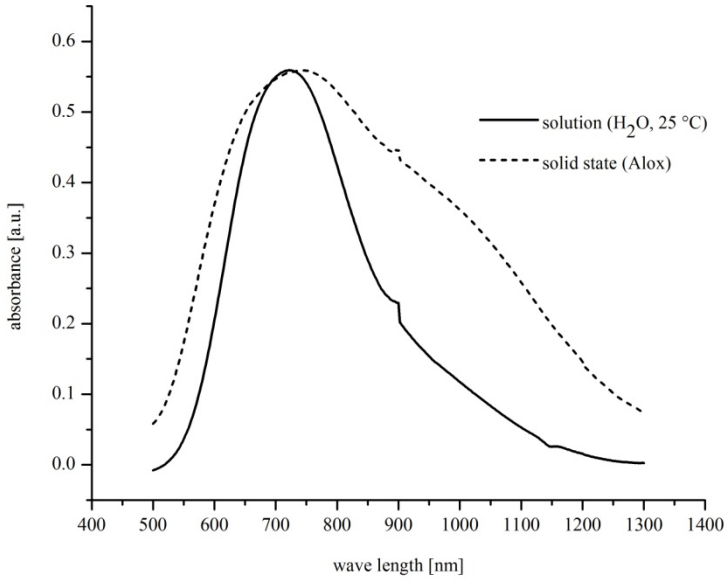

Figure S3: Overlay of the solid state - and solution-UV-vis-spectra of [Cu(H_3TETP)]NO_3.

CSD Structures used for the Structural Analysis

Table S2: CSD-Reference Codes and copper-donor bond lengths of similar cyclam structures used for comparison.

CCSD-Ref-Code		N1-Cu	N2-Cu	N3-Cu	N4-Cu	O5-Cu		axial donor
$[Cu(H_3TETP)]^+$		2.058	2.058	2.112	2.092	2.110		
DESQEX		2.010	2.067	2.012	2.000	2.252		acetate
TOKTAO		2.038	1.980	2.106	2.053	2.223		acetate
SALKUL		2.068	2.073	2.071	2.084	2.223		propionic
RAQCIW		2.112	2.094	2.119	2.080	2.252		propylate
OCOSOO		2.043	2.093	2.022	2.099	2.276		propylate
QUSFAL		2.078	2.074	2.070	2.074	2.181		ethylen amide
QUSFAL		2.076	2.060	2.100	2.061	2.147		
LOTJUZ		2.122	2.070	2.108	2.046	2.146		ethylen amide
FECBEV		2.097	2.076	2.077	2.067	2.165		acetamide
FEBZUI		2.066	2.106	2.046	2.143	2.143		acetamide
FEBZOC		2.063	2.064	2.078	2.033	2.174		acetamide
GUTKAH		2.017	2.067	2.005	2.077	2.218		phosphate
GUTKAH		2.074	2.005	2.069	2.021	2.234		
GUTKEL		2.101	2.038	2.108	2.060	2.226		phosphate
SALKOF		2.080	2.073	2.069	2.094	2.160		ethylene phosphate
KIWMEI		2.045	2.022	2.039	2.053	2.275		ether axial
CUCJOY		1.992	2.000	1.991	2.000	2.434		H_2O
ACUXOL		2.170	2.094	2.100	2.158	2.147		H_2O
AXIQAZ		2.000	1.992	2.022	1.989	2.296		H_2O
BAPFAA		2.034	2.034	2.034	2.034	2.330		H_2O
DUKPOO		2.079	2.040	2.087	2.095	2.398		H_2O
TEKZIS		2.039	2.030	2.033	2.031	2.354		H_2O
JUZZUZ		2.144	2.084	2.144	2.085	2.225		H_2O
NUVJOD		2.033	2.092	1.940	2.044	2.235		H_2O
VIDYIQ		1.960	2.003	1.971	1.999	2.257		NO_3^-
VIFBIV		2.000	1.997	2.000	1.986	2.222		ClO_4^-
VIFBIV		2.004	1.988	2.001	1.997	2.250		ClO_4^-
YEWVAX		2.034	2.061	2.083	2.005	2.515		ClO_4^-
YEWVEB		2.052	2.075	2.085	2.030	2.280		ClO_4^-
FACFIZ		2.006	1.983	2.010	2.003	2.594		ClO_4^-

Crystallography

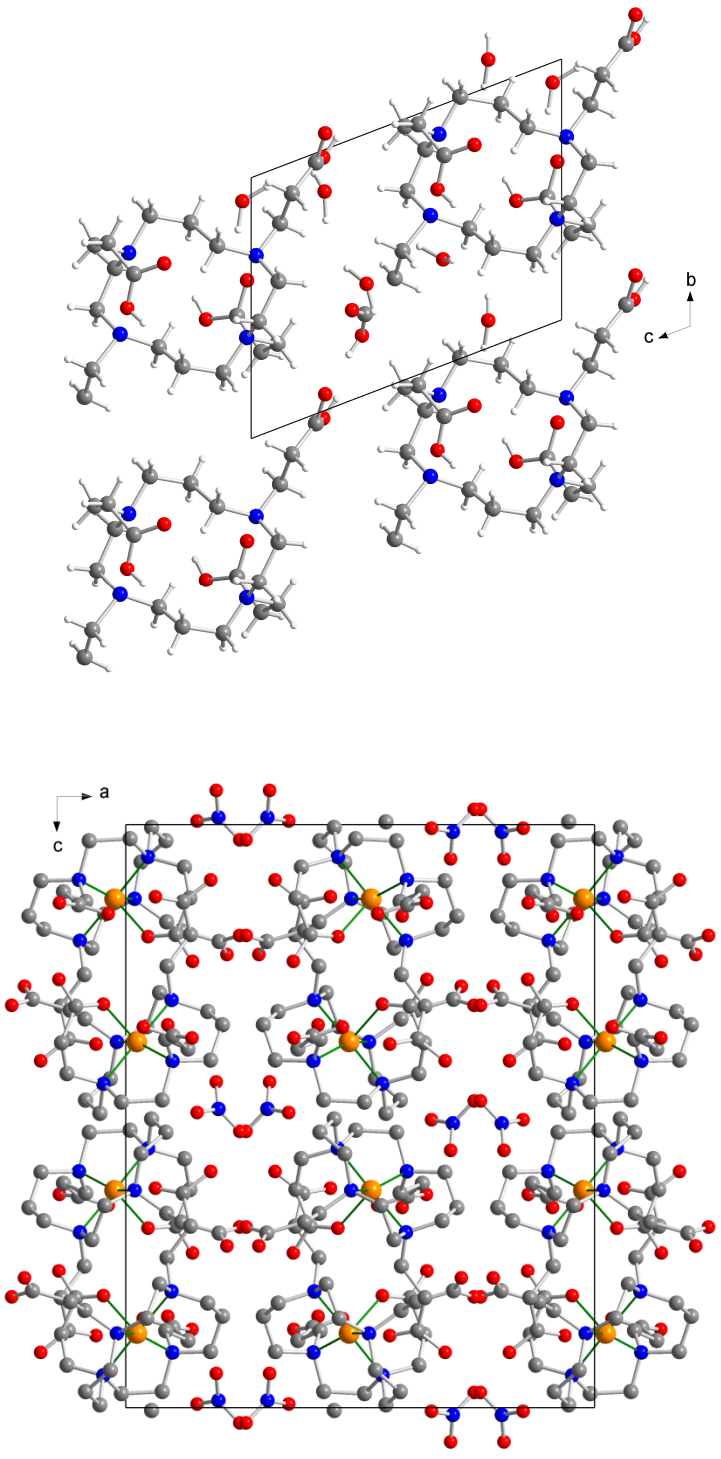

Figure S4. Crystal structure of TETP drawn along the a-axis (top) and Cu^{II}TETP along the b-axis (bottom). The coordination environment around the Cu atoms is indicated by green lines. Protons are omitted for clarity.

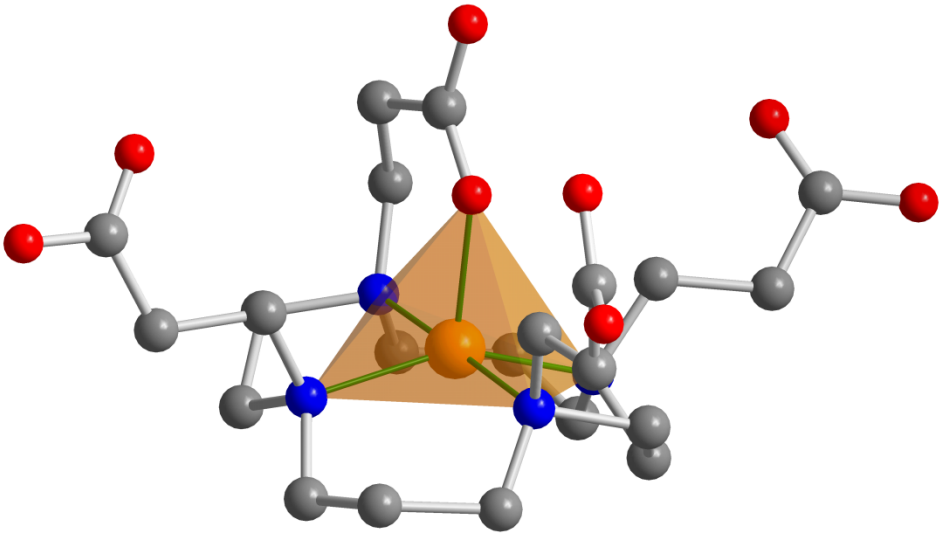

Figure S5. Coordination environment around the copper atom in Cu^{II}TETP.

Table S3. Crystal data and structure refinement of TETP and Cu^{II}TETP.

Formula	$C_{22}H_{50}N_4O_{13}$	$C_{22}H_{41}CuN_5O_{12}$
Formula weight	578.66	631.14
Temperature [K]	273(2)	273(2)
Wavelength [Å]	0.71073	0.71073
Crystal system	triclinic	orthorhombic
Space group	P-1 (No. 2)	Pbca (No. 61)
Lattice constants [Å, °]	a = 7.833(10) b = 9.223(12) c = 11.953(16) α = 106.960(14) β = 105.082(13) γ = 100.538(14)	a = 17.319(13) b = 15.007(13) c = 21.514(17)
Z	1	8
Volume [Å³]	765.5(17)	5592(8)
Calculated density [g/cm^3]	1.255	1.499
Absorption coefficient [mm^{-1}]	0.103	0.851
θ range for data collection [°]	1.89 to 27.49	1.89 to 27.50
Limiting indices	-10 ≤ h ≤9, -12 ≤ k≤ 9, -15 ≤ l≤ 11	-20 ≤ h ≤ 16, -18 ≤ k ≤ 12, -23 ≤ l ≤ 26
Reflections collected/unique	4000 / 3480 [R(int) = 0.1335]	15484 / 5378 [R(int) = 0.2335]
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	3301/ 0 / 202	5378 / 0 / 362
Goodness-of-fit on F^2	0.712	0.967
Final R indices [I>2σ(I)]	R1 = 0.0849, wR2 = 0.1808	R1 = 0.0635, wR2 = 0.0732
R indices (all data)	R1 = 0.4314, wR2 = 0.2488	R1 = 0.26378, wR2 = 0.0937
Largest diff. peak and hole [e / Å^3]	0.241 and -0.219	0.432 and -0.405

Table S4. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for TETP. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U(eq)
O1	823(8)	4646(7)	3073(6)	78(2)
O2	-254(8)	3612(7)	4338(6)	84(2)
O3	7267(11)	1868(8)	9541(7)	109(3)
O4	4420(10)	841(6)	9426(6)	88(2)
N1	3480(9)	2509(7)	3102(6)	41(2)
N2	5775(8)	4484(7)	7197(6)	42(2)
C1	193(12)	3448(11)	3358(10)	61(3)
C2	63(12)	1831(10)	2532(8)	69(3)
C3	1697(13)	1754(9)	2047(8)	70(3)
C4	5086(11)	2929(8)	2689(7)	47(2)
C5	3831(12)	1455(8)	3818(7)	57(3)
C6	5159(11)	2308(9)	5148(7)	56(3)
C7	4374(10)	3458(9)	5930(7)	46(2)
C8	4960(11)	4218(9)	2071(7)	54(3)
C9	6426(11)	3575(9)	8027(7)	58(3)
C10	4867(12)	2630(9)	8330(8)	72(3)
C11	5602(16)	1708(11)	9161(9)	65(3)
O5	-253(9)	7522(11)	3184(9)	187(4)
O6	842(12)	2817(10)	-718(8)	208(5)
O7	1129(18)	-327(15)	5305(14)	137(6)

Table S5. Bond lengths [Å] and angles [°] for TETP.

Atoms	**Bondlengths[Å]**	**Atoms**	**Bondlengths[Å]**
O1-C1	1.299(10)	N2-C5#1	1.560(9)
O2-C1	1.283(10)	C1-C2	1.498(11)
O3-C11	1.229(11)	C2-C3	1.541(11)
O4-C11	1.275(11)	C4-C5	1.577(9)
N1-C3	1.488(8)	C6-C7	1.529(9)
N1-C4	1.495(9)	C7-C8	1.528(10)
N1-C6	1.497(9)	C5-N2#1	1.560(9)
N2-C8	1.520(8)	C9-C10	1.552(10)
N2-C9	1.525(9)	C10-C11	1.554(12)
Atoms	**Angles[°]**	**Atoms**	**Angles[°]**
C6-N1-C4	110.1(7)	N1-C3-C2	110.2(8)
C6-N1-C3	112.9(6)	N1-C4-C5	112.7(6)
C4-N1-C3	114.3(6)	N1-C6-C7	113.7(7)
C8-N2-C9	111.7(7)	C8-C7-C6	111.6(7)
C8-N2-C5#1	112.4(6)	N2-C8-C7	112.8(6)
C9-N2-C5#1	110.2(6)	N2#1-C5-C4	110.1(6)
O2-C1-O1	114.2(7)	N2-C9-C10	114.4(7)
O2-C1-C2	112.0(8)	C9-C10-C11	110.7(8)
O1-C1-C2	123.8(10)	O3-C11-O4	125.3(11)
C1-C2-C3	119.1(10)	O3-C11-C10	122.0(10)
	117.1(10)	O4-C11-C10	112.8(10)

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Table S6. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for Cu^{II}TETP. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U(eq)
Cu1	235(1)	2411(1)	3725(1)	41(1)
N1	-473(4)	2803(5)	4443(3)	59(2)
N2	-187(3)	1129(3)	3727(3)	48(1)
N3	979(3)	1985(4)	3007(3)	50(2)
N4	997(3)	3384(4)	4055(3)	49(2)
O1	1426(3)	3825(3)	2654(2)	64(2)
O2	-477(3)	3074(3)	3078(2)	46(1)
O3	2435(3)	671(4)	3098(3)	110(2)
O4	2154(3)	-682(3)	2780(2)	72(2)
O5	1810(3)	2827(3)	950(2)	81(2)
O6	634(3)	3203(3)	1234(3)	66(2)
O7	1413(3)	6518(3)	3657(3)	113(2)
O8	378(4)	5906(3)	3471(3)	143(3)
C1	-555(6)	2186(7)	4955(4)	110(4)
C2	-850(5)	1294(7)	4731(4)	96(3)
C3	-327(5)	773(6)	4359(4)	74(3)
C4	448(5)	569(4)	3466(4)	67(3)
C5	721(5)	1012(6)	2880(4)	95(3)
C6	1745(5)	1885(6)	3206(4)	83(3)
C7	2068(4)	2814(5)	3435(4)	86(3)
C8	1809(4)	3112(5)	4037(4)	68(3)
C9	780(6)	3528(5)	4721(4)	87(3)
C10	-95(6)	3577(6)	4763(5)	118(4)
C11	1256(5)	3115(6)	4271(3)	84(3)
C12	1374(4)	3738(5)	3740(4)	84(3)

C13	1068(4)	3531(5)	3110(4)	45(2)
C14	-871(4)	1076(4)	3329(3)	49(2)
C15	1193(4)	129(4)	3179(3)	58(2)
C16	1995(4)	74(5)	2993(4)	52(2)
C17	882(4)	2485(6)	2426(3)	63(2)
C18	1378(4)	2236(5)	1889(3)	61(2)
C19	1217(4)	2792(5)	1324(4)	58(2)
C20	853(3)	4202(4)	3677(4)	57(2)
C21	1328(4)	5004(4)	3843(4)	73(2)
C22	1004(5)	5839(5)	3619(4)	63(3)
N5	1949(4)	588(6)	4869(4)	102(3)
O9	1518(4)	1202(4)	4923(3)	115(2)
O10A	2421(4)	399(4)	5236(3)	115(2)
O11	1914(4)	81(5)	4401(3)	115(2)
O12	901(3)	8060(3)	3419(2)	61(2)

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_computing_data_reduction          'SAINT'
_computing_structure_solution      'SHELXS-97 (Sheldrick, 2008)'
_computing_structure_refinement    'SHELXL-97 (Sheldrick, 2008)'
_computing_molecular_graphics
'SHELXTL (Bruker, 2001), ORTEPIII (Burnett & Johnson, 1996)'
_computing_publication_material    'SHELXTL (Sheldrick, 2008)'


_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR
and
 goodness of fit S are based on F^2^, conventional R-factors R are
based
 on F, with F set to zero for negative F^2^. The threshold expression
of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
```

```
 not relevant to the choice of reflections for refinement.  R-factors
based
 on F^2^ are statistically about twice as large as those based on F,
and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0959P)^2^+0.0000P] where
P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      SHELXL
_refine_ls_extinction_coef        0.045(8)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          3301
_refine_ls_number_parameters      202
_refine_ls_number_restraints      9
_refine_ls_R_factor_all           0.4343
_refine_ls_R_factor_gt            0.0849
_refine_ls_wR_factor_ref          0.2809
_refine_ls_wR_factor_gt           0.1808
_refine_ls_goodness_of_fit_ref    0.712
_refine_ls_restrained_S_all       0.711
_refine_ls_shift/su_max           0.088
_refine_ls_shift/su_mean          0.005

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 _atom_site_fract_z
 _atom_site_U_iso_or_equiv
 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_symmetry_multiplicity
 _atom_site_calc_flag
 _atom_site_refinement_flags
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O1 O 0.0823(8) 0.4646(7) 0.3073(6) 0.078(2) Uani 1 1 d . . .
H1 H 0.0114 0.5199 0.3076 0.118 Uiso 1 1 calc R . .
O2 O -0.0254(8) 0.3612(7) 0.4338(6) 0.084(2) Uani 1 1 d . . .
O3 O 0.7267(11) 0.1868(8) 0.9541(7) 0.109(3) Uani 1 1 d . . .
H3 H 0.7444 0.1073 0.9671 0.163 Uiso 1 1 calc R . .
O4 O 0.4420(10) 0.0841(6) 0.9426(6) 0.088(2) Uani 1 1 d . . .
N1 N 0.3480(9) 0.2509(7) 0.3102(6) 0.0407(19) Uani 1 1 d . . .
N2 N 0.5775(8) 0.4484(7) 0.7197(6) 0.0423(19) Uani 1 1 d . . .
C1 C 0.0193(12) 0.3448(11) 0.3358(10) 0.061(3) Uani 1 1 d . . .
C2 C 0.0063(12) 0.1831(10) 0.2532(8) 0.069(3) Uani 1 1 d . . .
H2A H -0.1066 0.1461 0.1829 0.083 Uiso 1 1 calc R . .
H2B H -0.0011 0.1119 0.2987 0.083 Uiso 1 1 calc R . .
C3 C 0.1697(13) 0.1754(9) 0.2047(8) 0.070(3) Uani 1 1 d . . .
```

```
H3A H 0.1617 0.0661 0.1619 0.085 Uiso 1 1 calc R . .
H3B H 0.1647 0.2297 0.1460 0.085 Uiso 1 1 calc R . .
C4 C 0.5086(11) 0.2929(8) 0.2689(7) 0.047(2) Uani 1 1 d . . .
H4A H 0.5158 0.1984 0.2098 0.057 Uiso 1 1 calc R . .
H4B H 0.6209 0.3318 0.3399 0.057 Uiso 1 1 calc R . .
C6 C 0.3831(12) 0.1455(8) 0.3818(7) 0.057(3) Uani 1 1 d . . .
H6A H 0.4327 0.0664 0.3384 0.069 Uiso 1 1 calc R . .
H6B H 0.2667 0.0909 0.3843 0.069 Uiso 1 1 calc R . .
C7 C 0.5159(11) 0.2308(9) 0.5148(7) 0.056(3) Uani 1 1 d . . .
H7A H 0.5405 0.1536 0.5526 0.067 Uiso 1 1 calc R . .
H7B H 0.6317 0.2883 0.5135 0.067 Uiso 1 1 calc R . .
C8 C 0.4374(10) 0.3458(9) 0.5930(7) 0.046(2) Uani 1 1 d . . .
H8A H 0.3963 0.4131 0.5490 0.055 Uiso 1 1 calc R . .
H8B H 0.3312 0.2865 0.6039 0.055 Uiso 1 1 calc R . .
C5 C 0.4960(11) 0.4218(9) 0.2071(7) 0.054(3) Uani 1 1 d . . .
H5A H 0.6169 0.4696 0.2068 0.065 Uiso 1 1 calc R . .
H5B H 0.4140 0.3725 0.1218 0.065 Uiso 1 1 calc R . .
C9 C 0.6426(11) 0.3575(9) 0.8027(7) 0.058(3) Uani 1 1 d . . .
H9A H 0.7342 0.4315 0.8798 0.069 Uiso 1 1 calc R . .
H9B H 0.7019 0.2847 0.7623 0.069 Uiso 1 1 calc R . .
C10 C 0.4867(12) 0.2630(9) 0.8330(8) 0.072(3) Uani 1 1 d . . .
H10A H 0.3940 0.1891 0.7563 0.086 Uiso 1 1 calc R . .
H10B H 0.4285 0.3353 0.8752 0.086 Uiso 1 1 calc R . .
C11 C 0.5602(16) 0.1708(11) 0.9161(9) 0.065(3) Uani 1 1 d . . .
O5 O -0.0253(9) 0.7522(11) 0.3184(9) 0.187(4) Uani 1 1 d D . .
H11 H 0.024(7) 0.779(10) 0.270(4) 0.281 Uiso 1 1 d D . .
H12 H 0.053(7) 0.785(15) 0.391(3) 0.281 Uiso 1 1 d D . .
O6 O 0.0842(12) 0.2817(10) -0.0718(8) 0.208(5) Uani 1 1 d D . .
H13 H 0.038(5) 0.307(7) -0.014(3) 0.312 Uiso 1 1 d D . .
H14 H 0.113(14) 0.361(5) -0.093(7) 0.312 Uiso 1 1 d D . .
O7 O 0.1129(18) -0.0327(15) 0.5305(14) 0.137(6) Uani 0.50 1 d PD . .
H15 H 0.223(3) -0.031(8) 0.535(9) 0.205 Uiso 0.50 1 d PD . .
H16 H 0.064(11) -0.111(10) 0.547(13) 0.205 Uiso 0.50 1 d PD . .

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 _atom_site_aniso_U_12
O1 0.093(5) 0.077(5) 0.104(5) 0.058(4) 0.048(4) 0.050(4)
O2 0.067(5) 0.111(5) 0.101(5) 0.064(4) 0.044(4) 0.021(4)
O3 0.141(7) 0.099(5) 0.112(6) 0.087(5) 0.032(6) 0.028(5)
O4 0.123(6) 0.060(4) 0.080(5) 0.041(3) 0.040(4) -0.010(4)
N1 0.043(5) 0.047(4) 0.042(4) 0.031(3) 0.012(4) 0.012(3)
N2 0.056(5) 0.030(4) 0.045(4) 0.017(3) 0.016(4) 0.016(4)
C1 0.034(6) 0.061(7) 0.096(9) 0.043(7) 0.010(6) 0.024(5)
C2 0.043(7) 0.071(7) 0.088(8) 0.040(6) 0.013(6) 0.000(5)
C3 0.070(8) 0.062(6) 0.070(7) 0.037(5) 0.005(6) 0.002(5)
C4 0.053(6) 0.046(6) 0.051(6) 0.024(4) 0.017(5) 0.021(5)
C6 0.086(7) 0.042(6) 0.049(6) 0.020(5) 0.029(6) 0.014(5)
C7 0.071(7) 0.042(5) 0.056(6) 0.029(5) 0.019(6) 0.002(5)
C8 0.040(6) 0.049(5) 0.053(6) 0.027(5) 0.016(5) 0.010(5)
C5 0.062(7) 0.056(6) 0.068(6) 0.039(5) 0.036(5) 0.023(5)
C9 0.070(7) 0.059(6) 0.054(6) 0.025(5) 0.032(5) 0.018(5)
C10 0.104(8) 0.050(6) 0.085(7) 0.046(5) 0.048(6) 0.018(5)
C11 0.081(9) 0.063(7) 0.061(7) 0.030(6) 0.026(7) 0.022(7)
O5 0.122(7) 0.241(9) 0.361(12) 0.236(9) 0.151(8) 0.119(7)
```

```
O6 0.130(8) 0.256(10) 0.173(9) 0.129(8) -0.027(7) -0.070(7)
O7 0.212(18) 0.091(11) 0.169(14) 0.063(10) 0.147(14) 0.036(11)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters are
only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
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loop_
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 _geom_bond_site_symmetry_2
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O1 H1 0.8200 . ?
O2 C1 1.283(10) . ?
O3 C11 1.229(11) . ?
O3 H3 0.8200 . ?
O4 C11 1.275(11) . ?
N1 C6 1.488(8) . ?
N1 C4 1.495(9) . ?
N1 C3 1.497(9) . ?
N2 C8 1.520(8) . ?
N2 C9 1.525(9) . ?
N2 C5 1.560(9) 2_666 ?
C1 C2 1.498(11) . ?
C2 C3 1.541(11) . ?
C2 H2A 0.9700 . ?
C2 H2B 0.9700 . ?
C3 H3A 0.9700 . ?
C3 H3B 0.9700 . ?
C4 C5 1.577(9) . ?
C4 H4A 0.9700 . ?
C4 H4B 0.9700 . ?
C6 C7 1.529(9) . ?
C6 H6A 0.9700 . ?
C6 H6B 0.9700 . ?
C7 C8 1.528(10) . ?
C7 H7A 0.9700 . ?
C7 H7B 0.9700 . ?
C8 H8A 0.9700 . ?
C8 H8B 0.9700 . ?
C5 N2 1.560(9) 2_666 ?
C5 H5A 0.9700 . ?
C5 H5B 0.9700 . ?
C9 C10 1.552(10) . ?
C9 H9A 0.9700 . ?
C9 H9B 0.9700 . ?
```

```
C10 C11 1.554(12) . ?
C10 H10A 0.9700 . ?
C10 H10B 0.9700 . ?
O5 H11 0.847(10) . ?
O5 H12 0.849(10) . ?
O6 H13 0.850(10) . ?
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O7 H15 0.849(10) . ?
O7 H16 0.850(11) . ?

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C11 O3 H3 109.5 . . ?
C6 N1 C4 109.2(6) . . ?
C6 N1 C3 110.6(6) . . ?
C4 N1 C3 112.8(6) . . ?
C8 N2 C9 114.5(6) . . ?
C8 N2 C5 112.1(6) . 2_666 ?
C9 N2 C5 107.9(6) . 2_666 ?
O2 C1 O1 122.3(9) . . ?
O2 C1 C2 119.4(9) . . ?
O1 C1 C2 118.2(10) . . ?
C1 C2 C3 114.0(7) . . ?
C1 C2 H2A 108.8 . . ?
C3 C2 H2A 108.8 . . ?
C1 C2 H2B 108.8 . . ?
C3 C2 H2B 108.8 . . ?
H2A C2 H2B 107.7 . . ?
N1 C3 C2 110.1(7) . . ?
N1 C3 H3A 109.6 . . ?
C2 C3 H3A 109.6 . . ?
N1 C3 H3B 109.6 . . ?
C2 C3 H3B 109.6 . . ?
H3A C3 H3B 108.1 . . ?
N1 C4 C5 112.9(6) . . ?
N1 C4 H4A 109.0 . . ?
C5 C4 H4A 109.0 . . ?
N1 C4 H4B 109.0 . . ?
C5 C4 H4B 109.0 . . ?
H4A C4 H4B 107.8 . . ?
N1 C6 C7 114.3(6) . . ?
N1 C6 H6A 108.7 . . ?
C7 C6 H6A 108.7 . . ?
N1 C6 H6B 108.7 . . ?
C7 C6 H6B 108.7 . . ?
H6A C6 H6B 107.6 . . ?
C8 C7 C6 111.7(7) . . ?
C8 C7 H7A 109.3 . . ?
C6 C7 H7A 109.3 . . ?
C8 C7 H7B 109.3 . . ?
C6 C7 H7B 109.3 . . ?
H7A C7 H7B 108.0 . . ?
N2 C8 C7 112.4(6) . . ?
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N2 C8 H8A 109.1 . . ?
C7 C8 H8A 109.1 . . ?
N2 C8 H8B 109.1 . . ?
C7 C8 H8B 109.1 . . ?
H8A C8 H8B 107.8 . . ?
N2 C5 C4 110.2(6) 2_666 . ?
N2 C5 H5A 109.6 2_666 . ?
C4 C5 H5A 109.6 . . ?
N2 C5 H5B 109.6 2_666 . ?
C4 C5 H5B 109.6 . . ?
H5A C5 H5B 108.1 . . ?
N2 C9 C10 114.2(7) . . ?
N2 C9 H9A 108.7 . . ?
C10 C9 H9A 108.7 . . ?
N2 C9 H9B 108.7 . . ?
C10 C9 H9B 108.7 . . ?
H9A C9 H9B 107.6 . . ?
C9 C10 C11 112.0(8) . . ?
C9 C10 H10A 109.2 . . ?
C11 C10 H10A 109.2 . . ?
C9 C10 H10B 109.2 . . ?
C11 C10 H10B 109.2 . . ?
H10A C10 H10B 107.9 . . ?
O3 C11 O4 123.8(10) . . ?
O3 C11 C10 119.1(10) . . ?
O4 C11 C10 117.1(10) . . ?
H11 O5 H12 109.9(18) . . ?
H13 O6 H14 109.1(18) . . ?
H15 O7 H16 109.5(18) . . ?

loop_
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 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
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O1 C1 C2 C3 39.2(11) . . . . ?
C6 N1 C3 C2 75.2(8) . . . . ?
C4 N1 C3 C2 -162.2(6) . . . . ?
C1 C2 C3 N1 51.2(10) . . . . ?
C6 N1 C4 C5 -172.6(6) . . . . ?
C3 N1 C4 C5 64.1(8) . . . . ?
C4 N1 C6 C7 77.4(8) . . . . ?
C3 N1 C6 C7 -157.9(7) . . . . ?
N1 C6 C7 C8 64.2(9) . . . . ?
C9 N2 C8 C7 -63.3(8) . . . . ?
C5 N2 C8 C7 173.5(6) 2_666 . . . ?
C6 C7 C8 N2 -171.9(6) . . . . ?
N1 C4 C5 N2 40.7(8) . . . 2_666 ?
C8 N2 C9 C10 -60.4(8) . . . . ?
C5 N2 C9 C10 65.1(8) 2_666 . . . ?
N2 C9 C10 C11 179.1(7) . . . . ?
C9 C10 C11 O3 3.9(13) . . . . ?
C9 C10 C11 O4 -177.3(8) . . . . ?
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_diffrn_measured_fraction_theta_full   0.983
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_refine_diff_density_rms    0.055
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data_ro11

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;
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_chemical_formula_moiety          ?
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 'C22 H41 Cu N5 O12'
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 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Cu'  'Cu'   0.3201   1.2651
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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_symmetry_space_group_name_H-M    'p_b_c_a'

loop_
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 'x, y, z'
 '-x+1/2, -y, z+1/2'
 '-x, y+1/2, -z+1/2'
 'x+1/2, -y+1/2, -z'
 '-x, -y, -z'
 'x-1/2, y, -z-1/2'
 'x, -y-1/2, z-1/2'
 '-x-1/2, y-1/2, z'

_cell_length_a                    17.319(13)
_cell_length_b                    15.007(13)
_cell_length_c                    21.514(17)
_cell_angle_alpha                 90.00
_cell_angle_beta                  90.00
_cell_angle_gamma                 90.00
_cell_volume                      5592(8)
_cell_formula_units_Z             8
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_exptl_crystal_density_diffrn     1.499
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_exptl_special_details
;
 ?
;

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_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
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_diffrn_standards_interval_count  ?
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_diffrn_reflns_av_sigmaI/netI     0.3548
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_diffrn_reflns_limit_k_min        -18
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_reflns_number_gt                 2284
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_computing_cell_refinement        'SAINT (Bruker, 2001)'
_computing_data_reduction         'SAINT'
_computing_structure_solution     'SHELXS-97 (Sheldrick, 2008)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 2008)'
_computing_molecular_graphics
'SHELXTL (Bruker, 2001), ORTEPIII (Burnett & Johnson, 1996)'
_computing_publication_material   'SHELXTL (Sheldrick, 2008)'
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```
_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor
wR and
 goodness of fit S are based on F^2^, conventional R-factors R are
based
 on F, with F set to zero for negative F^2^. The threshold
expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
 not relevant to the choice of reflections for refinement.  R-
factors based
 on F^2^ are statistically about twice as large as those based on F,
and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0058P)^2^+0.0000P] where
P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      SHELXL
_refine_ls_extinction_coef        0.00039(5)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          5378
_refine_ls_number_parameters      362
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.2678
_refine_ls_R_factor_gt            0.0635
_refine_ls_wR_factor_ref          0.0937
_refine_ls_wR_factor_gt           0.0732
_refine_ls_goodness_of_fit_ref    0.671
_refine_ls_restrained_S_all       0.671
_refine_ls_shift/su_max           0.022
_refine_ls_shift/su_mean          0.001

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Cu1 Cu 0.02352(4) 0.24106(5) 0.37254(5) 0.0413(2) Uani 1 1 d . . .
N1 N -0.0473(4) 0.2803(5) 0.4443(3) 0.0587(19) Uani 1 1 d . . .
N2 N -0.0187(3) 0.1129(3) 0.3727(3) 0.0479(13) Uani 1 1 d . . .
N3 N 0.0979(3) 0.1985(4) 0.3007(3) 0.0499(17) Uani 1 1 d . . .
N4 N 0.0997(3) 0.3384(4) 0.4055(3) 0.0491(16) Uani 1 1 d . . .
O1 O -0.1426(3) 0.3825(3) 0.2654(2) 0.0636(16) Uani 1 1 d . . .
O2 O -0.0477(3) 0.3074(3) 0.3078(2) 0.0460(13) Uani 1 1 d . . .
O3 O -0.2435(3) 0.0671(4) 0.3098(3) 0.110(2) Uani 1 1 d . . .
O4 O -0.2154(3) -0.0682(3) 0.2780(2) 0.0720(17) Uani 1 1 d . . .
H4 H -0.2610 -0.0697 0.2678 0.108 Uiso 1 1 calc R . .
O5 O 0.1810(3) 0.2827(3) 0.0950(2) 0.0806(18) Uani 1 1 d . . .
H5 H 0.1702 0.3135 0.0647 0.121 Uiso 1 1 calc R . .
O6 O 0.0634(3) 0.3203(3) 0.1234(3) 0.0664(15) Uani 1 1 d . . .
O7 O 0.1413(3) 0.6518(3) 0.3657(3) 0.113(2) Uani 1 1 d . . .
H7 H 0.1176 0.6947 0.3519 0.169 Uiso 1 1 calc R . .
O8 O 0.0378(4) 0.5906(3) 0.3471(3) 0.143(3) Uani 1 1 d . . .
C1 C -0.0555(6) 0.2186(7) 0.4955(4) 0.110(4) Uani 1 1 d . . .
H1A H -0.0911 0.2430 0.5258 0.132 Uiso 1 1 calc R . .
H1B H -0.0059 0.2106 0.5157 0.132 Uiso 1 1 calc R . .
C2 C -0.0850(5) 0.1294(7) 0.4731(4) 0.096(3) Uani 1 1 d . . .
H2A H -0.0995 0.0944 0.5092 0.116 Uiso 1 1 calc R . .
H2B H -0.1316 0.1396 0.4490 0.116 Uiso 1 1 calc R . .
C3 C -0.0327(5) 0.0773(6) 0.4359(4) 0.074(3) Uani 1 1 d . . .
H3A H -0.0532 0.0174 0.4323 0.089 Uiso 1 1 calc R . .
H3B H 0.0163 0.0733 0.4576 0.089 Uiso 1 1 calc R . .
C4 C 0.0448(5) 0.0569(4) 0.3466(4) 0.067(3) Uani 1 1 d . . .
H4A H 0.0260 -0.0026 0.3377 0.080 Uiso 1 1 calc R . .
H4B H 0.0868 0.0524 0.3762 0.080 Uiso 1 1 calc R . .
C5 C 0.0721(5) 0.1012(6) 0.2880(4) 0.095(3) Uani 1 1 d . . .
H5A H 0.0306 0.1012 0.2577 0.114 Uiso 1 1 calc R . .
H5B H 0.1150 0.0678 0.2708 0.114 Uiso 1 1 calc R . .
C6 C 0.1745(5) 0.1885(6) 0.3206(4) 0.083(3) Uani 1 1 d . . .
H6A H 0.2060 0.1664 0.2867 0.100 Uiso 1 1 calc R . .
H6B H 0.1768 0.1456 0.3543 0.100 Uiso 1 1 calc R . .
C7 C 0.2068(4) 0.2814(5) 0.3435(4) 0.086(3) Uani 1 1 d . . .
H7A H 0.2627 0.2780 0.3443 0.104 Uiso 1 1 calc R . .
H7B H 0.1927 0.3262 0.3131 0.104 Uiso 1 1 calc R . .
C8 C 0.1809(4) 0.3112(5) 0.4037(4) 0.068(3) Uani 1 1 d . . .
H8A H 0.2126 0.3612 0.4167 0.082 Uiso 1 1 calc R . .
H8B H 0.1886 0.2635 0.4335 0.082 Uiso 1 1 calc R . .
C9 C 0.0780(6) 0.3528(5) 0.4721(4) 0.087(3) Uani 1 1 d . . .
H9A H 0.1007 0.4078 0.4873 0.104 Uiso 1 1 calc R . .
H9B H 0.0971 0.3040 0.4974 0.104 Uiso 1 1 calc R . .
C10 C -0.0095(6) 0.3577(6) 0.4763(5) 0.118(4) Uani 1 1 d . . .
H10A H -0.0248 0.3585 0.5196 0.142 Uiso 1 1 calc R . .
H10B H -0.0271 0.4128 0.4573 0.142 Uiso 1 1 calc R . .
C11 C -0.1256(5) 0.3115(6) 0.4271(3) 0.084(3) Uani 1 1 d . . .
H11A H -0.1476 0.3396 0.4636 0.101 Uiso 1 1 calc R . .
H11B H -0.1565 0.2589 0.4187 0.101 Uiso 1 1 calc R . .
C12 C -0.1374(4) 0.3738(5) 0.3740(4) 0.084(3) Uani 1 1 d . . .
H12A H -0.1926 0.3825 0.3697 0.101 Uiso 1 1 calc R . .
H12B H -0.1157 0.4308 0.3863 0.101 Uiso 1 1 calc R . .
C13 C -0.1068(4) 0.3531(5) 0.3110(4) 0.045(2) Uani 1 1 d . . .
C14 C -0.0871(4) 0.1076(4) 0.3329(3) 0.049(2) Uani 1 1 d . . .
H14A H -0.0752 0.1368 0.2939 0.059 Uiso 1 1 calc R . .
H14B H -0.1281 0.1416 0.3526 0.059 Uiso 1 1 calc R . .
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C15 C -0.1193(4) 0.0129(4) 0.3179(3) 0.058(2) Uani 1 1 d . . .
H15A H -0.0880 -0.0125 0.2850 0.070 Uiso 1 1 calc R . .
H15B H -0.1123 -0.0241 0.3544 0.070 Uiso 1 1 calc R . .
C16 C -0.1995(4) 0.0074(5) 0.2993(4) 0.052(2) Uani 1 1 d . . .
C17 C 0.0882(4) 0.2485(6) 0.2426(3) 0.063(2) Uani 1 1 d . . .
H17A H 0.0970 0.3111 0.2515 0.076 Uiso 1 1 calc R . .
H17B H 0.0348 0.2426 0.2297 0.076 Uiso 1 1 calc R . .
C18 C 0.1378(4) 0.2236(5) 0.1889(3) 0.061(2) Uani 1 1 d . . .
H18A H 0.1294 0.1613 0.1789 0.073 Uiso 1 1 calc R . .
H18B H 0.1916 0.2306 0.2006 0.073 Uiso 1 1 calc R . .
C19 C 0.1217(4) 0.2792(5) 0.1324(4) 0.058(2) Uani 1 1 d . . .
C20 C 0.0853(3) 0.4202(4) 0.3677(4) 0.057(2) Uani 1 1 d . . .
H20A H 0.0312 0.4359 0.3717 0.069 Uiso 1 1 calc R . .
H20B H 0.0946 0.4060 0.3244 0.069 Uiso 1 1 calc R . .
C21 C 0.1328(4) 0.5004(4) 0.3843(4) 0.073(2) Uani 1 1 d . . .
H21A H 0.1377 0.5035 0.4292 0.088 Uiso 1 1 calc R . .
H21B H 0.1842 0.4932 0.3671 0.088 Uiso 1 1 calc R . .
C22 C 0.1004(5) 0.5839(5) 0.3619(4) 0.063(3) Uani 1 1 d . . .
N5 N 0.1949(4) 0.0588(6) 0.4869(4) 0.102(3) Uani 1 1 d . . .
O9 O 0.1518(4) 0.1202(4) 0.4923(3) 0.1154(15) Uani 1 1 d . . .
O10 O 0.2421(4) 0.0399(4) 0.5236(3) 0.1154(15) Uani 1 1 d . . .
O11 O 0.1914(4) 0.0081(5) 0.4401(3) 0.1154(15) Uani 1 1 d . . .
O12 O 0.0901(3) 0.8060(3) 0.3419(2) 0.0613(15) Uani 1 1 d . . .
H12F H 0.0371 0.8087 0.3421 0.074 Uiso 1 1 d G . .
H12E H 0.1108 0.8275 0.3887 0.074 Uiso 1 1 d G . .

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Cu1 0.0400(4) 0.0407(5) 0.0432(4) 0.0023(5) -0.0033(6) -0.0013(5)
N1 0.047(5) 0.073(5) 0.057(4) 0.013(4) 0.000(4) 0.008(4)
N2 0.040(3) 0.043(3) 0.061(4) 0.018(4) -0.023(4) -0.009(3)
N3 0.032(4) 0.057(4) 0.061(5) 0.015(4) 0.004(4) 0.016(3)
N4 0.055(4) 0.035(4) 0.058(4) 0.010(3) -0.016(4) -0.002(3)
O1 0.055(4) 0.077(4) 0.059(4) 0.003(3) -0.005(3) 0.027(3)
O2 0.041(3) 0.057(3) 0.040(3) 0.013(3) 0.021(3) 0.017(3)
O3 0.082(5) 0.065(4) 0.185(6) -0.052(4) -0.069(5) 0.013(4)
O4 0.040(3) 0.065(4) 0.111(5) -0.031(4) 0.014(3) -0.008(3)
O5 0.074(4) 0.102(5) 0.066(3) 0.005(3) 0.016(3) 0.021(3)
O6 0.037(3) 0.095(4) 0.067(3) 0.022(4) 0.003(4) 0.006(3)
O7 0.078(4) 0.044(4) 0.216(7) 0.030(5) -0.013(5) -0.010(3)
O8 0.106(5) 0.056(4) 0.268(10) 0.022(4) -0.095(6) -0.016(4)
C1 0.126(10) 0.127(10) 0.077(7) 0.025(8) 0.020(7) 0.034(8)
C2 0.089(8) 0.120(10) 0.080(8) 0.010(7) -0.001(7) -0.023(8)
C3 0.053(6) 0.098(7) 0.071(7) 0.036(6) -0.011(6) -0.026(6)
C4 0.081(7) 0.026(4) 0.092(7) 0.009(5) -0.034(6) -0.012(4)
C5 0.098(8) 0.066(7) 0.121(9) -0.051(7) 0.002(7) 0.003(6)
C6 0.059(6) 0.129(9) 0.061(6) 0.004(6) 0.002(6) 0.016(6)
C7 0.043(5) 0.053(7) 0.164(10) 0.027(6) 0.001(7) -0.014(5)
C8 0.047(6) 0.054(6) 0.103(7) -0.001(5) -0.022(6) 0.002(5)
C9 0.124(9) 0.064(7) 0.072(7) -0.011(5) -0.038(7) -0.029(7)
C10 0.128(10) 0.107(9) 0.120(9) -0.054(7) 0.059(9) -0.018(8)
```

```
C11 0.068(7) 0.132(9) 0.052(6) 0.002(6) 0.014(6) 0.016(6)
C12 0.081(6) 0.100(7) 0.071(6) 0.029(7) -0.005(7) 0.026(5)
C13 0.032(5) 0.070(6) 0.032(5) -0.007(5) 0.010(5) 0.005(4)
C14 0.053(5) 0.033(5) 0.062(5) -0.006(4) -0.020(4) -0.009(4)
C15 0.058(5) 0.039(5) 0.077(6) -0.002(4) -0.014(5) -0.006(4)
C16 0.037(6) 0.043(6) 0.077(7) -0.003(5) -0.015(5) 0.003(5)
C17 0.040(5) 0.083(6) 0.067(5) 0.002(6) 0.011(4) 0.003(5)
C18 0.068(6) 0.063(6) 0.052(5) 0.010(5) 0.021(5) 0.007(4)
C19 0.046(5) 0.088(7) 0.040(5) -0.025(5) 0.018(5) 0.000(5)
C20 0.050(5) 0.028(4) 0.093(6) -0.005(5) -0.027(5) 0.001(4)
C21 0.082(6) 0.041(5) 0.097(7) -0.010(5) -0.031(6) 0.005(5)
C22 0.048(5) 0.047(6) 0.094(7) -0.004(5) -0.042(6) -0.016(5)
N5 0.061(6) 0.128(8) 0.116(7) 0.058(6) -0.069(6) -0.046(5)
O9 0.110(3) 0.118(4) 0.118(4) -0.024(3) -0.018(3) 0.022(3)
O10 0.110(3) 0.118(4) 0.118(4) -0.024(3) -0.018(3) 0.022(3)
O11 0.110(3) 0.118(4) 0.118(4) -0.024(3) -0.018(3) 0.022(3)
O12 0.044(3) 0.081(4) 0.059(3) -0.012(3) -0.007(3) -0.007(3)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters
are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;

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Cu1 N1 2.058(6) . ?
Cu1 N2 2.058(5) . ?
Cu1 N4 2.092(6) . ?
Cu1 O2 2.110(5) . ?
Cu1 N3 2.112(6) . ?
N1 C1 1.445(9) . ?
N1 C11 1.482(8) . ?
N1 C10 1.501(10) . ?
N2 C14 1.465(7) . ?
N2 C3 1.482(8) . ?
N2 C4 1.492(8) . ?
N3 C6 1.402(8) . ?
N3 C17 1.468(7) . ?
N3 C5 1.552(9) . ?
N4 C8 1.464(8) . ?
N4 C20 1.494(7) . ?
N4 C9 1.497(8) . ?
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O1 C13 1.243(8) . ?
O2 C13 1.234(7) . ?
O3 C16 1.197(7) . ?
O4 C16 1.254(8) . ?
O4 H4 0.8200 . ?
O5 C19 1.306(8) . ?
O5 H5 0.8200 . ?
O6 C19 1.200(7) . ?
O7 C22 1.243(7) . ?
O7 H7 0.8200 . ?
O8 C22 1.135(7) . ?
C1 C2 1.512(10) . ?
C1 H1A 0.9700 . ?
C1 H1B 0.9700 . ?
C2 C3 1.439(10) . ?
C2 H2A 0.9700 . ?
C2 H2B 0.9700 . ?
C3 H3A 0.9700 . ?
C3 H3B 0.9700 . ?
C4 C5 1.502(9) . ?
C4 H4A 0.9700 . ?
C4 H4B 0.9700 . ?
C5 H5A 0.9700 . ?
C5 H5B 0.9700 . ?
C6 C7 1.580(10) . ?
C6 H6A 0.9700 . ?
C6 H6B 0.9700 . ?
C7 C8 1.443(9) . ?
C7 H7A 0.9700 . ?
C7 H7B 0.9700 . ?
C8 H8A 0.9700 . ?
C8 H8B 0.9700 . ?
C9 C10 1.520(10) . ?
C9 H9A 0.9700 . ?
C9 H9B 0.9700 . ?
C10 H10A 0.9700 . ?
C10 H10B 0.9700 . ?
C11 C12 1.490(9) . ?
C11 H11A 0.9700 . ?
C11 H11B 0.9700 . ?
C12 C13 1.488(9) . ?
C12 H12A 0.9700 . ?
C12 H12B 0.9700 . ?
C14 C15 1.559(7) . ?
C14 H14A 0.9700 . ?
C14 H14B 0.9700 . ?
C15 C16 1.449(8) . ?
C15 H15A 0.9700 . ?
C15 H15B 0.9700 . ?
C17 C18 1.487(7) . ?
C17 H17A 0.9700 . ?
C17 H17B 0.9700 . ?
C18 C19 1.501(9) . ?
C18 H18A 0.9700 . ?
C18 H18B 0.9700 . ?
C20 C21 1.501(7) . ?
C20 H20A 0.9700 . ?
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C20 H20B 0.9700 . ?
C21 C22 1.455(9) . ?
C21 H21A 0.9700 . ?
C21 H21B 0.9700 . ?
N5 O10 1.171(8) . ?
N5 O9 1.191(9) . ?
N5 O11 1.263(9) . ?
O12 H12F 0.92(6) . ?
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N1 Cu1 N4 85.5(3) . . ?
N2 Cu1 N4 151.2(2) . . ?
N1 Cu1 O2 90.7(2) . . ?
N2 Cu1 O2 103.6(2) . . ?
N4 Cu1 O2 105.2(2) . . ?
N1 Cu1 N3 178.4(3) . . ?
N2 Cu1 N3 86.3(2) . . ?
N4 Cu1 N3 94.3(2) . . ?
O2 Cu1 N3 90.9(2) . . ?
C1 N1 C11 107.6(7) . . ?
C1 N1 C10 100.9(8) . . ?
C11 N1 C10 105.6(7) . . ?
C1 N1 Cu1 116.6(5) . . ?
C11 N1 Cu1 116.6(5) . . ?
C10 N1 Cu1 107.8(5) . . ?
C14 N2 C3 112.6(6) . . ?
C14 N2 C4 110.3(6) . . ?
C3 N2 C4 105.3(6) . . ?
C14 N2 Cu1 109.7(4) . . ?
C3 N2 Cu1 113.3(5) . . ?
C4 N2 Cu1 105.3(4) . . ?
C6 N3 C17 115.1(6) . . ?
C6 N3 C5 103.1(6) . . ?
C17 N3 C5 107.4(7) . . ?
C6 N3 Cu1 112.7(5) . . ?
C17 N3 Cu1 113.5(4) . . ?
C5 N3 Cu1 103.7(4) . . ?
C8 N4 C20 112.0(6) . . ?
C8 N4 C9 107.8(7) . . ?
C20 N4 C9 111.1(6) . . ?
C8 N4 Cu1 113.7(5) . . ?
C20 N4 Cu1 106.5(4) . . ?
C9 N4 Cu1 105.5(4) . . ?
C13 O2 Cu1 135.2(5) . . ?
C16 O4 H4 109.5 . . ?
C19 O5 H5 109.5 . . ?
C22 O7 H7 109.5 . . ?
N1 C1 C2 110.9(8) . . ?
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N1 C1 H1A 109.5 . . ?
C2 C1 H1A 109.5 . . ?
N1 C1 H1B 109.5 . . ?
C2 C1 H1B 109.5 . . ?
H1A C1 H1B 108.0 . . ?
C3 C2 C1 116.4(8) . . ?
C3 C2 H2A 108.2 . . ?
C1 C2 H2A 108.2 . . ?
C3 C2 H2B 108.2 . . ?
C1 C2 H2B 108.2 . . ?
H2A C2 H2B 107.3 . . ?
C2 C3 N2 114.7(7) . . ?
C2 C3 H3A 108.6 . . ?
N2 C3 H3A 108.6 . . ?
C2 C3 H3B 108.6 . . ?
N2 C3 H3B 108.6 . . ?
H3A C3 H3B 107.6 . . ?
N2 C4 C5 107.3(6) . . ?
N2 C4 H4A 110.2 . . ?
C5 C4 H4A 110.2 . . ?
N2 C4 H4B 110.2 . . ?
C5 C4 H4B 110.2 . . ?
H4A C4 H4B 108.5 . . ?
C4 C5 N3 111.1(6) . . ?
C4 C5 H5A 109.4 . . ?
N3 C5 H5A 109.4 . . ?
C4 C5 H5B 109.4 . . ?
N3 C5 H5B 109.4 . . ?
H5A C5 H5B 108.0 . . ?
N3 C6 C7 109.6(7) . . ?
N3 C6 H6A 109.7 . . ?
C7 C6 H6A 109.7 . . ?
N3 C6 H6B 109.7 . . ?
C7 C6 H6B 109.7 . . ?
H6A C6 H6B 108.2 . . ?
C8 C7 C6 116.4(7) . . ?
C8 C7 H7A 108.2 . . ?
C6 C7 H7A 108.2 . . ?
C8 C7 H7B 108.2 . . ?
C6 C7 H7B 108.2 . . ?
H7A C7 H7B 107.3 . . ?
C7 C8 N4 114.1(7) . . ?
C7 C8 H8A 108.7 . . ?
N4 C8 H8A 108.7 . . ?
C7 C8 H8B 108.7 . . ?
N4 C8 H8B 108.7 . . ?
H8A C8 H8B 107.6 . . ?
N4 C9 C10 108.3(7) . . ?
N4 C9 H9A 110.0 . . ?
C10 C9 H9A 110.0 . . ?
N4 C9 H9B 110.0 . . ?
C10 C9 H9B 110.0 . . ?
H9A C9 H9B 108.4 . . ?
N1 C10 C9 111.7(7) . . ?
N1 C10 H10A 109.3 . . ?
C9 C10 H10A 109.3 . . ?
N1 C10 H10B 109.3 . . ?
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C9 C10 H10B 109.3 . . ?
H10A C10 H10B 107.9 . . ?
N1 C11 C12 121.0(7) . . ?
N1 C11 H11A 107.1 . . ?
C12 C11 H11A 107.1 . . ?
N1 C11 H11B 107.1 . . ?
C12 C11 H11B 107.1 . . ?
H11A C11 H11B 106.8 . . ?
C13 C12 C11 121.2(7) . . ?
C13 C12 H12A 107.0 . . ?
C11 C12 H12A 107.0 . . ?
C13 C12 H12B 107.0 . . ?
C11 C12 H12B 107.0 . . ?
H12A C12 H12B 106.8 . . ?
O2 C13 O1 124.5(7) . . ?
O2 C13 C12 117.6(7) . . ?
O1 C13 C12 117.9(7) . . ?
N2 C14 C15 117.3(5) . . ?
N2 C14 H14A 108.0 . . ?
C15 C14 H14A 108.0 . . ?
N2 C14 H14B 108.0 . . ?
C15 C14 H14B 108.0 . . ?
H14A C14 H14B 107.2 . . ?
C16 C15 C14 116.9(6) . . ?
C16 C15 H15A 108.1 . . ?
C14 C15 H15A 108.1 . . ?
C16 C15 H15B 108.1 . . ?
C14 C15 H15B 108.1 . . ?
H15A C15 H15B 107.3 . . ?
O3 C16 O4 127.3(7) . . ?
O3 C16 C15 121.0(8) . . ?
O4 C16 C15 111.3(7) . . ?
N3 C17 C18 117.7(6) . . ?
N3 C17 H17A 107.9 . . ?
C18 C17 H17A 107.9 . . ?
N3 C17 H17B 107.9 . . ?
C18 C17 H17B 107.9 . . ?
H17A C17 H17B 107.2 . . ?
C17 C18 C19 112.4(6) . . ?
C17 C18 H18A 109.1 . . ?
C19 C18 H18A 109.1 . . ?
C17 C18 H18B 109.1 . . ?
C19 C18 H18B 109.1 . . ?
H18A C18 H18B 107.9 . . ?
O6 C19 O5 122.7(9) . . ?
O6 C19 C18 125.0(8) . . ?
O5 C19 C18 112.1(7) . . ?
N4 C20 C21 116.0(6) . . ?
N4 C20 H20A 108.3 . . ?
C21 C20 H20A 108.3 . . ?
N4 C20 H20B 108.3 . . ?
C21 C20 H20B 108.3 . . ?
H20A C20 H20B 107.4 . . ?
C22 C21 C20 113.6(6) . . ?
C22 C21 H21A 108.8 . . ?
C20 C21 H21A 108.8 . . ?
C22 C21 H21B 108.8 . . ?
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C20 C21 H21B 108.8 . . ?
H21A C21 H21B 107.7 . . ?
O8 C22 O7 119.4(8) . . ?
O8 C22 C21 122.5(8) . . ?
O7 C22 C21 117.7(7) . . ?
O10 N5 O9 123.9(11) . . ?
O10 N5 O11 115.2(11) . . ?
O9 N5 O11 120.9(9) . . ?
H12F O12 H12E 107.8 . . ?

_diffrn_measured_fraction_theta_max    0.976
_diffrn_reflns_theta_full              26.00
_diffrn_measured_fraction_theta_full   0.976
_refine_diff_density_max    0.436
_refine_diff_density_min   -0.419
_refine_diff_density_rms    0.084
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