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Coexisting Ferromagnetic and Ferroelectric Order in a CuCl₄-based Organic-Inorganic Hybrid

Polyakov, Alexey O.; Arkenbout, Anne H.; Baas, Jacob; Blake, Graeme R.; Meetsma, Auke; Caretta, Antonio; van Loosdrecht, Paul H. M.; Palstra, Thomas T. M.

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1. SUBMISSION DETAILS

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Drs. A. Meetsma

;

_publ_contact_author_address # Address of author for correspondence

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Your Reference number :

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2. PROCESSING SUMMARY (JOURNAL OFFICE ONLY)

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_journal_date_printers_first ?

_journal_date_printers_final ?

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_journal_techeditor_code ?

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_journal_codен_ASTM ?

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_journal_volume ?

_journal_issue ?

_journal_page_first ?

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3. TITLE AND AUTHOR LIST

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_publ_section_title_footnote

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loop_

_publ_author_name

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_publ_author_footnote
_publ_author_address
'? ' # author name
; # author related footnote
;
; # Address of this author
;
'Meetsma, Auke'
;
? # author related footnote
;
;
Crystal Structure Center, Chemical Physics,
Zernike Institute for Advanced Materials,
University of Groningen,
Nijenborgh 4,
NL-9747 AG Groningen, The Netherlands.
;

#=====

# 4. TEXT

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_publ_section_abstract
;
?
;

# Insert blank lines between paragraphs

_publ_section_comment
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;
_publ_section_exptl_prep
;
;
_publ_section_exptl_refinement
;
The hydrogen atoms were generated by geometrical considerations,
constrained to idealized geometries, and allowed to ride on the
carrier atom with an isotropic displacement parameter related to the
equivalent displacement parameter of their carrier atoms,
with  $U_{iso}(H) = 1.2U_{eq}(C)$  or  $1.5U_{eq}(methyl\ C)$ .
The amine-group was refined as rigid groups, which were allowed
to rotate freely.
Assigned values of bond distances for secondary C-H2 = 0.97 \%,
aromatic C-H = 0.93 \% and N-H = 0.89 \%

;

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;
;

# Insert blank lines between references

_publ_section_references
;
Allen, F.H. (2002). Acta Cryst. B58, 380-388.

Beurskens, P.T., Beurskens, G., Gelder, R. de, Smits, J.M.M.,

```

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The DIRDIF08 program system, Technical Report of the Crystallography
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Wilson, A.J.C. (1992). Ed. *International Tables for Crystallography*,
Volume C, Kluwer Academic Publishers, Dordrecht, The Netherlands.

;

_publ_section_figure_captions

;

Fig. 1. Perspective PLUTO drawings of the molecule illustrating the
configuration and the adopted numbering scheme.

Fig. 2. Molecular packing viewed down unit cell axes.

Fig. 3. Perspective ORTEP drawing of the title compound.
Displacement ellipsoids for non-H atoms are represented at the 50%
probability level.
The H-atoms have been omitted to improve clarity.
The H-atoms are drawn with an arbitrary radius.

Fig. 4. Portion of the crystal packing, showing
N-H...Cl hydrogen bonds shown as dashed lines.

;

_publ_section_acknowledgements

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#####

5. CHEMICAL DATA

_chemical_name_systematic

;

;

_chemical_name_common ?

_chemical_melting_point ?

_chemical_formula_moiety

'2(C8 H12 N), Cl4 Cu'

Ex: 'C12 H16 N2 O6, H2 O' and '(Cd 2+)3, (C6 N6 Cr 3-)2, 2(H2 O)'

_chemical_formula_structural ?

_chemical_formula_sum

'C16 H24 Cl4 Cu N2'

_chemical_formula_iupac ?

_chemical_formula_weight 449.72

_chemical_compound_source 'see text'

loop_

_atom_type_symbol

_atom_type_description

_atom_type_scatter_dispersion_real

_atom_type_scatter_dispersion_imag

_atom_type_scatter_source

N N 0.0061 0.0033

'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'

Cl Cl 0.1484 0.1585

'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'

C C 0.0033 0.0016

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

#=====

6. CRYSTAL DATA

_symmetry_cell_setting orthorhombic

_symmetry_space_group_name_Hall '-C 2bc 2'

_symmetry_space_group_name_H-M 'C m c a'

_symmetry_Int_Tables_number 64

loop_

_symmetry_equiv_pos_site_id

_symmetry_equiv_pos_as_xyz

1 x, y, z

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

3 x, -y, -z

4 -x, 1/2+y, 1/2-z

5 -x, -y, -z

6 x, 1/2+y, 1/2-z

7 -x, y, z

8 x, 1/2-y, 1/2+z

9 1/2+x, 1/2+y, z

10 1/2-x, -y, 1/2+z

11 1/2+x, 1/2-y, -z

12 1/2-x, y, 1/2-z

13 1/2-x, 1/2-y, -z

14 1/2+x, y, 1/2-z

15 1/2-x, 1/2+y, z

16 1/2+x, -y, 1/2+z

_cell_length_a 39.021(8)

_cell_length_b 7.3430(15)

_cell_length_c 7.3939(15)

_cell_angle_alpha 90

_cell_angle_beta 90

_cell_angle_gamma 90

_cell_volume 2118.6(7)

_cell_formula_units_Z 4

_cell_measurement_temperature 373(1)

_cell_measurement_reflns_used 2107

_cell_measurement_theta_min 3.13

_cell_measurement_theta_max 23.02

_cell_special_details

;

The final unit cell was obtained from the xyz centroids of 2107 reflections after integration using the SAINTPLUS software package (Bruker, 2007).

Reduced cell calculations did not indicate any higher metric lattice symmetry and examination of the final atomic coordinates of the structure did not yield extra symmetry elements (Spek, 1988; Le Page 1987, 1988)

;

```
_exptl_crystal_description      'block'  
_exptl_crystal_colour          'orange'  
_exptl_crystal_size_max        0.20  
_exptl_crystal_size_mid        0.10  
_exptl_crystal_size_min        0.10  
_exptl_crystal_size_rad        ?  
_exptl_crystal_density_meas    ?  
_exptl_crystal_density_diffrn  1.410  
_exptl_crystal_density_method  'not measured'  
_exptl_crystal_F_000           924  
_exptl_absorpt_coefficient_mu   1.535  
_exptl_absorpt_correction_type 'multi-scan'  
_exptl_absorpt_process_details '(SADABS, (Bruker, 2007))'  
_exptl_absorpt_correction_T_min 0.7256  
_exptl_absorpt_correction_T_max 0.8577
```

#=====

7. EXPERIMENTAL DATA

```
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;  
;  
_diffrn_ambient_temperature      373(1)  
_diffrn_radiation_wavelength     0.71073  
_diffrn_radiation_type           'MoK\alpha'  
_diffrn_radiation_source         'fine focus sealed Siemens Mo tube '  
_diffrn_radiation_monochromator  'parallel mounted graphite'  
_diffrn_radiation_detector  
;  
  CCD area-detector  
;  
_diffrn_measurement_device_type  
;  
  Bruker Smart Apex; CCD area detector  
;  
_diffrn_measurement_method       '\f and \w scans'  
_diffrn_special_details  
;  
  Crystal into the cold nitrogen stream of the low-temperature unit  
  (KRYOFLEX, (Bruker, 2007)).  
;  
_diffrn_detector_area_resol_mean  66.06  
  
_diffrn_standards_number          0  
_diffrn_standards_interval_count  .  
_diffrn_standards_interval_time  .  
_diffrn_standards_decay_%        0  
  
loop_  
_diffrn_standard_refl_index_h     .  
_diffrn_standard_refl_index_k     .  
_diffrn_standard_refl_index_l     .  
? ? ?  
  
# number of measured reflections (redundant set)  
_diffrn_reflns_number             7895  
_diffrn_reflns_av_R_equivalents   0.0313  
_diffrn_reflns_av_sigmaI/netI     0.0218
```

```

_diffrn_reflms_limit_h_min      -49
_diffrn_reflms_limit_h_max      49
_diffrn_reflms_limit_k_min      -9
_diffrn_reflms_limit_k_max      9
_diffrn_reflms_limit_l_min      -9
_diffrn_reflms_limit_l_max      9
_diffrn_reflms_theta_min        3.13
_diffrn_reflms_theta_max        26.72
_diffrn_measured_fraction_theta_max  0.998
_diffrn_reflms_theta_full        25.00
_diffrn_measured_fraction_theta_full  0.998

_diffrn_reflms_reduction_process
;
Intensity data were corrected for Lorentz and polarization
effects, decay and absorption and reduced to  $F_o^2$ 
using SAINT-Plus & SADABS (Bruker, 2007).
;

# number of unique reflections
_reflms_number_total            1146
_reflms_number_gt               913
_reflms_threshold_expression     I>2\s(I)

_computing_data_collection      'SMART (Bruker, 2007)'
_computing_cell_refinement      'SAINT-Plus (Bruker, 2007)'
_computing_data_reduction       'SAINT-Plus'
_computing_structure_solution
;
DIRDIF-08 (Beurskens et al., 2008)
;
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 2008)'
_computing_molecular_graphics
;
PLATON (Spek, 2003)
PLUTO (Meetsma, 2009)
;
_computing_publication_material  'PLATON (Spek, 2003)'

#=====

# 8. REFINEMENT DATA

_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
 $F^2 > 2\sigma(F^2)$  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)+(0.1044P)^2+0.6794P] where P=(Fo^2+2Fc^2)/3'
_atom_sites_solution_primary     heavy
_atom_sites_solution_secondary   direct
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method     none

```



```

_refine_ls_extinction_coef      ?
_refine_ls_abs_structure_details ?
_chemical_absolute_configuration '.'

_refine_ls_abs_structure_Flack  ?
_refine_ls_number_reflns       1146
_refine_ls_number_parameters    68
_refine_ls_number_restraints    2
_refine_ls_number_constraints   ?
_refine_ls_R_factor_all         0.0653
_refine_ls_R_factor_gt         0.0544
_refine_ls_wR_factor_ref       0.1597
_refine_ls_wR_factor_gt       0.1481
_refine_ls_goodness_of_fit_ref  1.119
_refine_ls_restrained_S_all     1.128
_refine_ls_shift/su_max        0.000
_refine_ls_shift/su_mean       0.000

_refine_diff_density_max       0.756
_refine_diff_density_min      -0.723
_refine_diff_density_rms      0.104

_vrn_publ_code_void_volume     128.1
_vrn_publ_code_number_frames   1800
_vrn_publ_code_frame_time_sec  10.0
_vrn_publ_code_meas_time_hour  7.8

```

#=====

9. ATOMIC COORDINATES AND DISPLACEMENT PARAMETERS

```

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_atom_site_type_symbol
_atom_site_thermal_displace_type
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
_atom_site_calc_flag
_atom_site_refinement_flags
N1 N Uani 0.05465(14) 0.500 0.00000 1.000 0.077(2) . .
C1 C Uani 0.0895(3) 0.500 0.00000 1.000 0.161(8) . .
C2 C Uani 0.1196(2) 0.455(2) -0.1085(16) 0.5 0.185(10) . .
C3 C Uani 0.1505(3) 0.500 0.00000 1.000 0.201(9) . .
C4 C Uani 0.1699(2) 0.6185(16) -0.1079(16) 1.0 0.210(7) . .
C5 C Uani 0.2045(2) 0.6148(16) -0.0941(17) 1.0 0.183(6) . .
C6 C Uani 0.2197(4) 0.500 0.00000 1.000 0.180(10) . .
Cu1 Cu Uani 0.000 0.50000 0.50000 1.000 0.0495(3) . .
Cl1 Cl Uani -0.05866(4) 0.500 0.50000 1.000 0.0764(6) . .
Cl2 Cl Uani 0.000 0.27901(16) 0.28124(16) 1.000 0.0671(5) . .
H1 H Uiso 0.09425 0.43231 0.10982 0.5 0.1945 . .
H1' H Uiso 0.09425 0.62571 0.03151 0.5 0.1945 . .
H2 H Uiso 0.11946 0.52552 -0.21974 0.5 0.2200 . .
H2' H Uiso 0.11946 0.32705 -0.13938 0.5 0.2200 . .
H4 H Uiso 0.15919 0.69821 -0.18753 1.0 0.2522 . .
H5 H Uiso 0.21733 0.70086 -0.15703 1.0 0.2197 . .
H6 H Uiso 0.24355 0.500 0.00000 1.000 0.2171 . .
H9 H Uiso 0.04704 0.54756 0.10319 0.5 0.1152 . .
H9' H Uiso 0.04704 0.38624 -0.01069 0.5 0.1152 . .
H9" H Uiso 0.04704 0.56620 -0.09250 0.5 0.1152 . .

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_atom_site_aniso_label

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_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
N1 0.070(4) 0.078(4) 0.082(4) -0.004(3) 0.036(8) -0.017(10)
C1 0.080(7) 0.163(13) 0.24(2) 0.074(10) 0.085(15) 0.043(15)
C2 0.152(15) 0.232(18) 0.17(2) -0.088(16) -0.050(14) 0.061(14)
C3 0.050(5) 0.262(19) 0.29(2) 0.134(14) 0.00 0.0000
C4 0.133(8) 0.260(14) 0.237(13) 0.153(13) 0.014(6) 0.003(7)
C5 0.097(7) 0.202(12) 0.250(13) 0.033(11) 0.038(7) -0.025(6)
C6 0.081(8) 0.22(2) 0.24(2) -0.036(12) 0.00 0.0000
Cu1 0.0626(6) 0.0447(5) 0.0412(5) -0.0076(3) 0.00 0.0000
Cl1 0.0604(9) 0.0866(11) 0.0822(11) 0.0013(6) 0.00 0.0000
Cl2 0.1076(10) 0.0485(6) 0.0451(7) -0.0088(5) 0.00 0.0000

```

#=====

10. MOLECULAR GEOMETRY

_geom_special_details

```

;
Bond distances, angles etc. have been calculated using the
rounded fractional coordinates. All su's are estimated
from the variances of the (full) variance-covariance matrix.
The cell esds are taken into account in the estimation of
distances, angles and torsion angles
;

```

loop_

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Cu1	Cl2	2.9192(13)	.	.	2_555	yes
Cu1	Cl1	2.2890(16)	.	.	.	yes
Cu1	Cl2	2.2912(13)	.	.	.	yes
Cu1	Cl1	2.2890(16)	.	.	5_566	yes
Cu1	Cl2	2.2912(13)	.	.	3_566	yes
Cu1	Cl2	2.9192(13)	.	.	4_555	yes
N1	C1	1.360(13)	.	.	.	yes
N1	H9"	0.89	.	.	3_565	no
N1	H9"	0.89	.	.	.	no
N1	H9	0.89	.	.	.	no
N1	H9'	0.89	.	.	.	no
N1	H9	0.89	.	.	3_565	no
N1	H9'	0.89	.	.	3_565	no
C1	C2	1.460(13)	.	.	.	no
C1	C2	1.460(13)	.	.	3_565	no
C2	C3	1.486(13)	.	.	.	no
C3	C4	1.402(12)	.	.	.	no
C3	C4	1.402(12)	.	.	3_565	no
C4	C5	1.354(11)	.	.	.	no
C5	C6	1.244(14)	.	.	.	no
C1	H1'	0.97	.	.	.	no
C1	H1	0.97	.	.	.	no
C1	H1	0.97	.	.	3_565	no
C1	H1'	0.97	.	.	3_565	no
C2	H2	0.97	.	.	.	no
C2	H2'	0.97	.	.	.	no
C4	H4	0.93	.	.	.	no
C5	H5	0.93	.	.	.	no
C6	H6	0.93	.	.	.	no

```

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_geom_angle_atom_site_label_2
_geom_angle_atom_site_label_3
_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_2
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
C11      Cu1      C12      90      .      .      .      yes
C11      Cu1      C12      90      .      .      2_555    yes
C11      Cu1      C12      90      .      .      3_566    yes
C11      Cu1      C12      90      .      .      4_555    yes
C11      Cu1      C11      180     .      .      5_566    yes
C12      Cu1      C12      90.33(4) .      .      2_555    yes
C12      Cu1      C12      180     .      .      3_566    yes
C12      Cu1      C12      89.67(4) .      .      4_555    yes
C11      Cu1      C12      90      5_566  .      .      yes
C12      Cu1      C12      89.67(4) 2_555  .      3_566    yes
C12      Cu1      C12      180     2_555  .      4_555    yes
C11      Cu1      C12      90      5_566  .      2_555    yes
C12      Cu1      C12      90.33(4) 3_566  .      4_555    yes
C11      Cu1      C12      90      5_566  .      3_566    yes
C11      Cu1      C12      90      5_566  .      4_555    yes
Cu1      C12      Cu1      179.48(5) .      .      2_554    yes
C1       N1       H9       109     .      .      3_565    no
C1       N1       H9'      109     .      .      3_565    no
C1       N1       H9''     109     .      .      3_565    no
H9       N1       H9'      109     .      .      .        no
H9       N1       H9''     109     .      .      .        no
H9       N1       H9'      141     .      .      3_565    no
H9       N1       H9'      56      .      .      3_565    no
H9       N1       H9''     56      .      .      3_565    no
H9'      N1       H9''     109     .      .      .        no
H9'      N1       H9'      56      3_565  .      .        no
H9'      N1       H9'      141     .      .      3_565    no
H9'      N1       H9''     56      .      .      3_565    no
H9       N1       H9''     56      3_565  .      .        no
H9'      N1       H9''     56      3_565  .      .        no
H9''     N1       H9''     141     .      .      3_565    no
H9       N1       H9'      109     3_565  .      3_565    no
H9       N1       H9''     109     3_565  .      3_565    no
H9'      N1       H9''     109     3_565  .      3_565    no
C1       N1       H9       109     .      .      .        no
C1       N1       H9'      109     .      .      .        no
C2       C1       C2       72.9(8) .      .      3_565    no
N1       C1       C2       143.6(5) .      .      3_565    yes
N1       C1       C2       143.6(5) .      .      .        yes
C1       C2       C3       107.8(9) .      .      .        no
C2       C3       C2       71.5(8) .      .      3_565    no
C2       C3       C4       105.6(7) .      .      .        no
C2       C3       C4       127.4(7) 3_565  .      .        no
C4       C3       C4       114.7(10) .      .      3_565    no
C2       C3       C4       127.4(8) .      .      3_565    no
C2       C3       C4       105.6(7) 3_565  .      3_565    no
C3       C4       C5       118.9(10) .      .      .        no
C4       C5       C6       122.1(12) .      .      .        no
C5       C6       C5       123.0(14) .      .      3_565    no
C2       C1       H1'      101     3_565  .      3_565    no
H1       C1       H1'      104     3_565  .      3_565    no
N1       C1       H1       101     .      .      3_565    no
N1       C1       H1'      101     .      .      3_565    no
C2       C1       H1       101     .      .      .        no
C2       C1       H1'      101     .      .      .        no

```

C2	C1	H1	60	.	.	3_565	no
C2	C1	H1'	60	.	.	3_565	no
H1	C1	H1'	104	.	.	.	no
C2	C1	H1	60	3_565	.	.	no
H1	C1	H1	158	.	.	3_565	no
H1	C1	H1'	71	.	.	3_565	no
C2	C1	H1'	60	3_565	.	.	no
H1'	C1	H1'	158	.	.	3_565	no
C2	C1	H1	101	3_565	.	3_565	no
N1	C1	H1	101	.	.	.	no
N1	C1	H1'	101	.	.	.	no
H1	C1	H1'	71	3_565	.	.	no
C1	C2	H2	110	.	.	.	no
C3	C2	H2	110	.	.	.	no
C3	C2	H2'	110	.	.	.	no
C1	C2	H2'	110	.	.	.	no
H2	C2	H2'	109	.	.	.	no
C5	C4	H4	121	.	.	.	no
C3	C4	H4	121	.	.	.	no
C4	C5	H5	119	.	.	.	no
C6	C5	H5	119	.	.	.	no
C5	C6	H6	118	.	.	.	no
C5	C6	H6	118	3_565	.	.	no

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C2	C1	C2	C3	0.0(8)	3_565	.	.	no
C2	C1	C2	C3	0.0(8)	.	3_565	.	no
C1	C2	C3	C4	-124.9(9)	.	.	.	no
C1	C2	C3	C2	0.0(8)	.	.	3_565	no
C1	C2	C3	C4	95.8(11)	.	.	3_565	no
C2	C3	C4	C5	-148.0(11)	.	.	.	no
C2	C3	C4	C5	133.6(11)	3_565	.	.	no
C4	C3	C4	C5	-2.8(13)	3_565	.	.	no
C3	C4	C5	C6	5.9(17)	.	.	.	no
C4	C5	C6	C5	-3.1(16)	.	.	3_565	no

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C11	C12	3.2387(15)	.	3_566	no
C11	C12	3.2387(15)	.	3_566	no
C12	N1	3.371(4)	.	2_555	no
C12	N1	3.371(4)	.	4_545	no
C12	N1	3.392(4)	.	.	no
C12	N1	3.392(4)	.	.	no
C12	N1	3.392(4)	.	5_565	no
C12	C11	3.2387(15)	.	.	no
C12	C11	3.2387(15)	.	5_566	no
C12	N1	3.392(4)	.	7_555	no
C12	C11	3.2387(15)	.	5_566	no

C11	H9"	3.09	.	5_565	no
C11	H9'	2.87	.	4_555	no
C11	H1'	3.09	.	4_545	no
C11	H9"	3.09	.	7_556	no
C11	H9	2.99	.	5_566	no
C11	H1'	3.09	.	2_565	no
C11	H9	2.99	.	7_555	no
C11	H9'	2.87	.	2_555	no
C12	H9	2.64	.	4_545	no
C12	H9'	2.94	.	.	no
C12	H9'	2.68	.	2_555	no
C12	H9"	2.57	.	5_565	no
C12	H9	3.00	.	.	no
C12	H9'	2.94	.	7_555	no
C12	H9	3.00	.	7_555	no
C12	H9"	2.57	.	3_565	no
N1	C12	3.392(4)	.	3_565	no
N1	C12	3.371(4)	.	2_554	no
N1	C12	3.392(4)	.	.	no
N1	C12	3.371(4)	.	2_554	no
N1	C12	3.392(4)	.	5_565	no
N1	C12	3.371(4)	.	4_555	no
N1	C12	3.371(4)	.	4_555	no
N1	C12	3.392(4)	.	.	no
C2	H4	2.87	.	6_544	no
C4	H1	2.98	.	3_565	no
H1	C4	2.98	.	3_565	no
H1'	C11	3.09	.	2_564	no
H1'	C11	3.09	.	4_555	no
H2	H4	2.02	.	.	no
H2'	H4	2.22	.	6_544	no
H4	C2	2.87	.	6_554	no
H4	H2'	2.22	.	6_554	no
H4	H2	2.02	.	.	no
H9	C11	2.99	.	5_566	no
H9	C12	3.00	.	.	no
H9	C11	2.99	.	5_566	no
H9	C12	3.00	.	.	no
H9	C12	2.64	.	4_555	no
H9	C12	2.64	.	4_555	no
H9'	C12	2.94	.	.	no
H9'	C12	2.68	.	2_554	no
H9'	C12	2.68	.	2_554	no
H9'	C11	2.87	.	2_554	no
H9'	C11	2.87	.	2_554	no
H9'	C12	2.94	.	.	no
H9"	C12	2.57	.	5_565	no
H9"	C11	3.09	.	7_554	no
H9"	C11	3.09	.	5_565	no
H9"	C12	2.57	.	3_565	no

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_geom_hbond_atom_site_label_H

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#D H A D - H H...A D...A D - H...A symm(A)

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N1	H9	C12	0.8900	2.6400	3.371(4)	140	4_555	yes
N1	H9'	C12	0.8900	2.6800	3.371(4)	135	2_554	yes

N1 H9" C12 0.8900 2.5700 3.392(4) 154 3_565 yes

===END of Crystallographic Information File

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SHELXL97-2 & Manual Editing

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2008-05-06 # Formatted by publCIF

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1. SUBMISSION DETAILS

_publ_contact_author_name # Name of author for correspondence

;

Drs. A. Meetsma

;

_publ_contact_author_address # Address of author for correspondence

;

Crystal Structure Center, Chemical Physics,
Zernike Institute for Advanced Materials,
University of Groningen,
Nijenborgh 4,
NL-9747 AG Groningen, The Netherlands.

;

_publ_contact_author_email A.Meetsma@rug.nl

_publ_contact_author_fax '+31 50 3634441'

_publ_contact_author_phone '+31 50 3634368'

_publ_requested_journal 'JACS'

Publication choice FI, CI or EI for Inorganic

FM, CM or EM for Metal-organic

FO, CO or EO for Organic

_publ_requested_category ?

_publ_requested_coeditor_name ?

_publ_contact_letter # Include date of submission

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Date of submission : 2008-05-06 15:01:04

Consider this CIF submission for deposition of the fifth
X-ray structure of a manuscript to be submitted to : JACS
(Our Compound_Identification_Code : q1311)

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2. PROCESSING SUMMARY (JOURNAL OFFICE ONLY)

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# 3. TITLE AND AUTHOR LIST

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Title (type here to add)
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_publ_section_title_footnote
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```

# The loop structure below should contain the names and addresses of all
# authors, in the required order of publication. Repeat as necessary.

```

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_publ_author_name
_publ_author_footnote
_publ_author_address
'?' # author name
; # author related footnote
;
; # Address of this author
;
'Meetsma, Auke'
;
? # author related footnote
;
;
Crystal Structure Center, Chemical Physics,
Zernike Institute for Advanced Materials,
University of Groningen,

```

Nijenborgh 4,
NL-9747 AG Groningen, The Netherlands.

;

#####

4. TEXT

_publ_section_synopsis

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_publ_section_abstract

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Insert blank lines between paragraphs

_publ_section_comment

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(type here to add)

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_publ_section_exptl_prep

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_publ_section_exptl_refinement

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The hydrogen atoms were generated by geometrical considerations, constrained to idealized geometries, and allowed to ride on the carrier atom with an isotropic displacement parameter related to the equivalent displacement parameter of their carrier atoms, with $U_{iso}(H) = 1.2 U_{eq}(C)$ or $1.5 U_{eq}(methyl\ C)$. The methyl-groups were refined as rigid groups, which were allowed to rotate freely. Assigned values of bond distances: secondary C-H₂ = 0.99 Å, methyl C-H₃ = 0.98 Å, aromatic C-H = 0.95 Å.

C-H distances were in the range of 0.95 -- 0.99(3) Å.

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Insert blank lines between references

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Beurskens, P. T., Beurskens, G., de Gelder, R., Garcia-Granda, S., Gould, R. O., Israel, R. & Smits, J. M. M. (1999). The *DIRDIF99* program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

Brandenburg, K. (2006). *DIAMOND*. Release 3.1. Crystal Impact GbR, Bonn, Germany

Bruker, (2006). *SMART* (Version 5.632), *SAINT-Plus* (Version 6.45) and *SADABS* (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264--269.

Le Page, Y. (1988). *J. Appl. Cryst.* **21**, 983--984.

Meetsma, A. (2007). Extended version of the program *PLUTO*. University of Groningen, The Netherlands. (unpublished).

Sheldrick, G. M. (1997). *SHELXL97*. Program for Crystal Structure Refinement. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7--13.
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Fig. 1. Perspective *PLUTO* drawings of the moieties of the asymmetric unit illustrating the configuration and the adopted numbering scheme.

Fig. 2. Molecular packing viewed down unit cell axes.

Fig. 3. Perspective *ORTEP* drawing of the anion and cation of the title compound. Displacement ellipsoids for non-H atoms are represented at the 50% probability level. The H-atoms have been omitted to improve clarity.

Fig. 4. Portion of the crystal packing, showing N---H...Cl hydrogen bonds shown as dashed lines.
;

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5. CHEMICAL DATA

_chemical_name_systematic
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;

_chemical_name_common ?
_chemical_melting_point ?
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'2(C8 H12 N), C14 Cu'
Ex: 'C12 H16 N2 O6, H2 O' and '(Cd 2+)3, (C6 N6 Cr 3-)2, 2(H2 O)'
_chemical_formula_structural ?
_chemical_formula_sum
'C16 H24 C14 Cu N2'
_chemical_formula_iupac ?
_chemical_formula_weight 449.74
_chemical_compound_source 'see text'

loop_
_atom_type_symbol
_atom_type_description
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_atom_type_scatter_dispersion_imag
_atom_type_scatter_source
N N 0.0061 0.0033
'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'
Cl Cl 0.1484 0.1585
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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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6. CRYSTAL DATA

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_symmetry_space_group_name_H-M 'P b c a'
_symmetry_Int_Tables_number 61

loop_

_symmetry_equiv_pos_site_id
_symmetry_equiv_pos_as_xyz
1 x, y, z
2 1/2-x, -y, 1/2+z
3 1/2+x, 1/2-y, -z
4 -x, 1/2+y, 1/2-z
5 -x, -y, -z
6 1/2+x, y, 1/2-z
7 1/2-x, 1/2+y, z
8 x, 1/2-y, 1/2+z

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_cell_length_b 7.2664(9)
_cell_length_c 38.238(5)
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_cell_volume 2003.3(4)
_cell_formula_units_Z 4

_cell_measurement_temperature 100(1)
_cell_measurement_reflms_used 8650
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The final unit cell was obtained from the xyz centroids of 8650 reflections after integration using the SAINTPLUS software package (Bruker, 2000).

Reduced cell calculations did not indicate any higher metric lattice symmetry and examination of the final atomic coordinates of the structure did not yield extra symmetry elements (Spek, 1988; Le Page 1987, 1988)

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7. EXPERIMENTAL DATA

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_diffrn_radiation_monochromator   'parallel mounted graphite'
_diffrn_radiation_detector
;
  CCD area-detector
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_diffrn_measurement_device_type
;
  Bruker Smart Apex; CCD area detector
;
_diffrn_measurement_method       '\f and \w scans'
_diffrn_special_details
;
  Crystal into the cold nitrogen stream of the low-temperature unit
  (KRYOFLEX, (Bruker, 2006)).
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loop_
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? ? ?

# number of measured reflections (redundant set)
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_diffrn_reflns_av_sigmaI/netI  0.0251
_diffrn_reflns_limit_h_min      -9
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_diffrn_reflns_reduction_process
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  Intensity data were corrected for Lorentz and polarization
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_computing_cell_refinement      'SAINT-Plus (Bruker, 2006)'
_computing_data_reduction       'SAINT-Plus (Bruker, 2006)'
_computing_structure_solution

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DIRDIF-99 (Beurskens et al., 1999)
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_computing_structure_refinement      'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics
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DIAMOND (Brandenburg, 2006)
PLATON (Spek, 2003)
PLUTO (Meetsma, 2007)
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_computing_publication_material      'PLATON (Spek, 2003)'

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# 8. REFINEMENT DATA

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Refinement of F2 against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F2, conventional R-factors R are based
on F, with F set to zero for negative F2. The threshold expression of
F2 > 2sigma(F2) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F2 are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
;

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_refine_ls_weighting_scheme          calc
_refine_ls_weighting_details
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_chemical_absolute_configuration      '.'

_refine_ls_abs_structure_Flack        ?
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_refine_ls_number_parameters           107
_refine_ls_number_restraints           0
_refine_ls_number_constraints          ?
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_refine_ls_R_factor_gt                 0.0400
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_refine_ls_wR_factor_gt                0.1106
_refine_ls_goodness_of_fit_ref         1.329
_refine_ls_restrained_S_all           1.329
_refine_ls_shift/su_max                0.000
_refine_ls_shift/su_mean               0.000

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N N Uani 1.0138(4) 0.0064(4) 0.55580(7) 1.000 0.0165(8) . .
C1 C Uani 0.9605(6) -0.0433(5) 0.59223(9) 1.000 0.0176(10) . .
C2 C Uani 1.0436(6) 0.0920(5) 0.61820(9) 1.000 0.0212(10) . .
C3 C Uani 1.0184(6) 0.0273(5) 0.65541(9) 1.000 0.0179(10) . .
C4 C Uani 0.8791(6) 0.0978(5) 0.67676(10) 1.000 0.0219(11) . .
C5 C Uani 0.8605(6) 0.0357(6) 0.71109(10) 1.000 0.0265(11) . .
C6 C Uani 0.9804(7) -0.0957(6) 0.72429(10) 1.000 0.0265(11) . .
C7 C Uani 1.1182(7) -0.1673(6) 0.70311(11) 1.000 0.0283(14) . .
C8 C Uani 1.1375(6) -0.1052(6) 0.66918(10) 1.000 0.0241(11) . .
Cu Cu Uani 0.00000 0.00000 0.00000 1.000 0.0123(2) . .
Cl1 Cl Uani 0.03423(12) 0.01365(12) 0.05984(2) 1.000 0.0160(2) . .
Cl2 Cl Uani 0.21493(11) 0.22950(11) -0.00597(2) 1.000 0.0149(2) . .
H1 H Uiso 1.00485 -0.16910 0.59760 1.000 0.0211 . .
H1' H Uiso 0.82362 -0.04248 0.59440 1.000 0.0211 . .
H2 H Uiso 1.17755 0.10695 0.61326 1.000 0.0254 . .
H2' H Uiso 0.98343 0.21355 0.61530 1.000 0.0254 . .
H4 H Uiso 0.79652 0.18842 0.66792 1.000 0.0264 . .
H5 H Uiso 0.76482 0.08388 0.72551 1.000 0.0315 . .
H6 H Uiso 0.96822 -0.13651 0.74778 1.000 0.0315 . .
H7 H Uiso 1.19945 -0.25918 0.71191 1.000 0.0340 . .
H8 H Uiso 1.23375 -0.15377 0.65495 1.000 0.0291 . .
H9 H Uiso 0.97516 0.12294 0.55104 1.000 0.0247 . .
H9' H Uiso 0.95965 -0.07338 0.54056 1.000 0.0247 . .
H9" H Uiso 1.13931 -0.00002 0.55354 1.000 0.0247 . .
```

```
loop_
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_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
N 0.0180(15) 0.0196(14) 0.0119(13) -0.0003(12) -0.0003(12) 0.0007(13)
C1 0.0199(19) 0.0210(17) 0.0120(15) 0.0011(13) 0.0006(14) -0.0029(15)
C2 0.032(2) 0.0196(17) 0.0120(16) -0.0007(14) 0.0014(15) -0.0050(16)
C3 0.0239(19) 0.0173(17) 0.0124(15) -0.0024(13) -0.0007(14) -0.0023(15)
C4 0.024(2) 0.0201(18) 0.0217(19) -0.0007(15) 0.0007(16) 0.0026(16)
C5 0.030(2) 0.031(2) 0.0184(18) -0.0044(16) 0.0072(16) -0.0008(18)
C6 0.037(2) 0.028(2) 0.0144(17) 0.0008(15) -0.0011(17) -0.0066(19)
C7 0.035(3) 0.028(2) 0.022(2) 0.0021(16) -0.0064(18) 0.0061(19)
C8 0.026(2) 0.027(2) 0.0193(18) -0.0031(16) 0.0014(16) 0.0056(17)
Cu 0.0132(3) 0.0118(3) 0.0120(3) -0.0002(2) 0.0001(2) -0.0022(2)
Cl1 0.0183(4) 0.0171(4) 0.0126(4) -0.0003(3) 0.0000(3) -0.0003(3)
Cl2 0.0133(4) 0.0136(4) 0.0179(4) 0.0003(3) 0.0001(3) -0.0017(3)
```

#=====

10. MOLECULAR GEOMETRY

```
_geom_special_details
;
Bond distances, angles etc. have been calculated using the
```

rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

;

```

loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
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_geom_bond_site_symmetry_1
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
Cu      C12      2.2879(9)      .      .      yes
Cu      C12      2.8531(9)      .      3_455      yes
Cu      C11      2.3036(8)      .      5_555      yes
Cu      C12      2.2879(9)      .      5_555      yes
Cu      C12      2.8531(9)      .      7_545      yes
Cu      C11      2.3036(8)      .      .      yes
N       C1       1.490(4)      .      .      yes
N       H9"      0.9100      .      .      no
N       H9'      0.9100      .      .      no
N       H9       0.9100      .      .      no
C1      C2       1.520(5)      .      .      no
C2      C3       1.510(5)      .      .      no
C3      C8       1.393(6)      .      .      no
C3      C4       1.392(6)      .      .      no
C4      C5       1.395(5)      .      .      no
C5      C6       1.383(6)      .      .      no
C6      C7       1.383(7)      .      .      no
C7      C8       1.381(6)      .      .      no
C1      H1'      0.9900      .      .      no
C1      H1       0.9900      .      .      no
C2      H2       0.9900      .      .      no
C2      H2'      0.9900      .      .      no
C4      H4       0.9500      .      .      no
C5      H5       0.9500      .      .      no
C6      H6       0.9500      .      .      no
C7      H7       0.9500      .      .      no
C8      H8       0.9500      .      .      no

```

```

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_geom_angle_site_symmetry_3
_geom_angle_publ_flag
C12     Cu      C12      90.36(3)      3_455      .      5_555      yes
C12     Cu      C12      180.00      3_455      .      7_545      yes
C11     Cu      C12      89.72(3)      5_555      .      5_555      yes
C11     Cu      C12      88.17(3)      5_555      .      7_545      yes
C12     Cu      C12      89.64(3)      5_555      .      7_545      yes
C11     Cu      C12      89.72(3)      .      .      .      yes
C11     Cu      C12      88.17(3)      .      .      3_455      yes
C11     Cu      C11      180.00      .      .      5_555      yes
C11     Cu      C12      90.28(3)      .      .      5_555      yes
C11     Cu      C12      91.83(3)      .      .      7_545      yes
C12     Cu      C12      89.64(3)      .      .      3_455      yes
C11     Cu      C12      90.28(3)      5_555      .      .      yes
C12     Cu      C12      180.00      .      .      5_555      yes
C12     Cu      C12      90.36(3)      .      .      7_545      yes
C11     Cu      C12      91.83(3)      5_555      .      3_455      yes
Cu      C12     Cu      169.15(3)      .      .      3_555      yes

```

H9	N	H9"	109.00	.	.	.	no
H9'	N	H9"	109.00	.	.	.	no
C1	N	H9"	109.00	.	.	.	no
C1	N	H9	109.00	.	.	.	no
C1	N	H9'	109.00	.	.	.	no
H9	N	H9'	109.00	.	.	.	no
N	C1	C2	110.6(3)	.	.	.	yes
C1	C2	C3	111.5(3)	.	.	.	no
C2	C3	C8	119.8(4)	.	.	.	no
C4	C3	C8	118.5(3)	.	.	.	no
C2	C3	C4	121.7(3)	.	.	.	no
C3	C4	C5	120.2(4)	.	.	.	no
C4	C5	C6	120.4(4)	.	.	.	no
C5	C6	C7	119.7(4)	.	.	.	no
C6	C7	C8	120.0(4)	.	.	.	no
C3	C8	C7	121.3(4)	.	.	.	no
N	C1	H1	110.00	.	.	.	no
N	C1	H1'	110.00	.	.	.	no
C2	C1	H1	110.00	.	.	.	no
C2	C1	H1'	110.00	.	.	.	no
H1	C1	H1'	108.00	.	.	.	no
C1	C2	H2'	109.00	.	.	.	no
C3	C2	H2	109.00	.	.	.	no
C3	C2	H2'	109.00	.	.	.	no
H2	C2	H2'	108.00	.	.	.	no
C1	C2	H2	109.00	.	.	.	no
C5	C4	H4	120.00	.	.	.	no
C3	C4	H4	120.00	.	.	.	no
C4	C5	H5	120.00	.	.	.	no
C6	C5	H5	120.00	.	.	.	no
C7	C6	H6	120.00	.	.	.	no
C5	C6	H6	120.00	.	.	.	no
C6	C7	H7	120.00	.	.	.	no
C8	C7	H7	120.00	.	.	.	no
C3	C8	H8	119.00	.	.	.	no
C7	C8	H8	119.00	.	.	.	no

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N	C1	C2	C3	-170.6(3)	.	.	.	no
C1	C2	C3	C4	-100.5(4)	.	.	.	no
C1	C2	C3	C8	80.0(5)	.	.	.	no
C2	C3	C4	C5	-179.6(4)	.	.	.	no
C8	C3	C4	C5	-0.1(6)	.	.	.	no
C2	C3	C8	C7	180.0(4)	.	.	.	no
C4	C3	C8	C7	0.5(6)	.	.	.	no
C3	C4	C5	C6	0.3(6)	.	.	.	no
C4	C5	C6	C7	-0.8(7)	.	.	.	no
C5	C6	C7	C8	1.1(7)	.	.	.	no
C6	C7	C8	C3	-1.0(7)	.	.	.	no

loop_

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 _geom_contact_site_symmetry_1
 _geom_contact_site_symmetry_2

_geom_contact_publ_flag

Cu	H9'	3.7000	.	2_554	no
Cu	H9"	3.3100	.	2_654	no
Cu	H9	3.3700	.	4_645	no
Cu	H9'	3.4800	.	4_655	no
Cu	H9'	3.7000	.	6_455	no
Cu	H9"	3.3100	.	6_355	no
Cu	H9	3.3700	.	8_454	no
C11	C12	3.2388(12)	.	.	no
C11	N	3.266(3)	.	2_654	no
C11	C12	3.6092(12)	.	3_455	no
C11	C12	3.2545(12)	.	5_555	no
C11	N	3.494(3)	.	8_454	no
C11	C2	3.633(4)	.	8_454	no
C12	C11	3.2388(12)	.	.	no
C12	C11	3.6092(12)	.	3_555	no
C12	N	3.302(3)	.	6_455	no
C12	C12	3.6459(12)	.	3_455	no
C12	C12	3.6459(12)	.	3_555	no
C12	N	3.225(3)	.	4_655	no
C12	N	3.371(3)	.	8_454	no
C12	C11	3.2545(12)	.	5_555	no
C12	C12	3.6682(12)	.	7_545	no
C12	C12	3.6682(12)	.	7_555	no
C11	H1'	2.9100	.	2_554	no
C11	H9"	2.3700	.	2_654	no
C11	H2	3.0400	.	2_654	no
C11	H2'	2.9300	.	8_454	no
C11	H9	2.7000	.	8_454	no
C12	H9'	3.1200	.	6_455	no
C12	H9	2.6600	.	6_455	no
C12	H9'	2.3200	.	4_655	no
C12	H9"	3.0100	.	2_654	no
C12	H9"	3.0600	.	8_454	no
C12	H9	2.9800	.	8_454	no
N	C12	3.302(3)	.	6_555	no
N	C11	3.494(3)	.	8_655	no
N	C12	3.225(3)	.	4_645	no
N	C11	3.266(3)	.	2_655	no
N	C12	3.371(3)	.	8_655	no
C2	C11	3.633(4)	.	8_655	no
C2	H8	2.8200	.	7_755	no
C3	H8	2.9300	.	7_755	no
C6	H5	2.9200	.	7_645	no
C6	H5	3.1000	.	6_556	no
C8	H1	2.9400	.	.	no
H1	C8	2.9400	.	.	no
H1	C11	2.9000	.	8_645	no
H1'	C11	2.9100	.	2_555	no
H2	H8	2.4400	.	7_755	no
H2	C11	3.0400	.	2_655	no
H2	H9"	2.4300	.	.	no
H2	H8	2.5100	.	.	no
H2'	C11	2.9300	.	8_655	no
H2'	H9	2.5400	.	.	no
H2'	H4	2.4300	.	.	no
H4	H2'	2.4300	.	.	no
H5	C6	3.1000	.	6_456	no
H5	C6	2.9200	.	7_655	no
H8	H2	2.4400	.	7_745	no
H8	H2	2.5100	.	.	no
H8	C2	2.8200	.	7_745	no
H8	C3	2.9300	.	7_745	no
H9	H2'	2.5400	.	.	no
H9	Cu	3.3700	.	4_655	no
H9	C12	2.6600	.	6_555	no

H9	C12	2.9800	.	8_655	no
H9	Cu	3.3700	.	8_655	no
H9	C11	2.7000	.	8_655	no
H9'	Cu	3.4800	.	4_645	no
H9'	C12	3.1200	.	6_555	no
H9'	C12	2.3200	.	4_645	no
H9'	Cu	3.7000	.	6_555	no
H9'	Cu	3.7000	.	2_555	no
H9'	Cu	3.4800	.	8_645	no
H9''	C11	2.3700	.	2_655	no
H9''	Cu	3.3100	.	6_655	no
H9''	C12	3.0600	.	8_655	no
H9''	C12	3.0100	.	2_655	no
H9''	H2	2.4300	.	.	no
H9''	Cu	3.3100	.	2_655	no

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 _geom_hbond_distance_HA
 _geom_hbond_distance_DA
 _geom_hbond_angle_DHA
 _geom_hbond_site_symmetry_A
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#	D	H	A	D - H	H...A	D...A	D - H...A	symm(A)		
#										
N		H9	C12	0.9100	2.6600	3.302(3)	128.00	6_555	yes	
N		H9	C11	0.9100	2.7000	3.494(3)	147.00	8_655	yes	
N		H9'	C12	0.9100	2.3200	3.225(3)	172.00	4_645	yes	
N		H9''	C11	0.9100	2.3700	3.266(3)	169.00	2_655	yes	

#===END of Crystallographic Information File