

University of Groningen

## Coexisting Ferromagnetic and Ferroelectric Order in a $\text{CuCl}_4$ -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.

*Published in:*  
 Chemistry of Materials

*DOI:*  
[10.1021/cm2023696](https://doi.org/10.1021/cm2023696)

**IMPORTANT NOTE:** You are advised to consult the publisher's version (publisher's PDF) if you wish to cite from it. Please check the document version below.

*Document Version*  
 Publisher's PDF, also known as Version of record

*Publication date:*  
 2012

[Link to publication in University of Groningen/UMCG research database](#)

*Citation for published version (APA):*

Polyakov, A. O., Arkenbout, A. H., Baas, J., Blake, G. R., Meetsma, A., Caretta, A., van Loosdrecht, P. H. M., & Palstra, T. T. M. (2012). Coexisting Ferromagnetic and Ferroelectric Order in a  $\text{CuCl}_4$ -based Organic-Inorganic Hybrid. *Chemistry of Materials*, 24(1), 133-139. <https://doi.org/10.1021/cm2023696>

### Copyright

Other than for strictly personal use, it is not permitted to download or to forward/distribute the text or part of it without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license (like Creative Commons).

The publication may also be distributed here under the terms of Article 25fa of the Dutch Copyright Act, indicated by the "Taverne" license. More information can be found on the University of Groningen website: <https://www.rug.nl/library/open-access/self-archiving-pure/taverne-amendment>.

### Take-down policy

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

*Downloaded from the University of Groningen/UMCG research database (Pure): <http://www.rug.nl/research/portal>. For technical reasons the number of authors shown on this cover page is limited to 10 maximum.*

# CIF-file generated for C16H24Cl4CuN2 Cmca a0007

data\_global

# 0. AUDIT DETAILS

\_audit\_creation\_date '2009-09-21 13:30:04'

\_audit\_creation\_method

;

PLATON <TABLE ACC> option (version :: 60709)

SHELXL97-2 & Manual Editing

;

\_audit\_update\_record

;

?

;

# 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 'Organometallics Acta Crystallographica  
C'

# Publication choose 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

;

Date of submission : 2009-09-21 13:30:04

Please consider this CIF submission for publication as a  
Regular Structural Paper in Acta Crystallographica E.  
All authors have seen and approved this submission.

The CIF has passed the Chester CHECKCIF routines and gives  
a satisfactory PRINTCIF file.

The Structure Factor Listing (in CIF format) and any artwork  
(Schemes, Figures) as HPGL, Postscript, TIFF or encapsulated Postscript files  
will be transferred to Chester by ftp as instructed in Notes for Authors.

Consider this CIF submission for deposition of the Xray-structure quoted  
in Journal Ref: Inorganica Chimica Acta 259 (2000) ...

"A remarkable Lewis acid-base adduct: preparation..."

Compound : ....

Your Reference number : ....

Please consider this CIF submission for deposition at the Cambridge Data Base.

Consider this CIF submission for deposition of the first X-ray structure of a manuscript to be submitted to : Organometallics (Our Compound\_Identification\_Code : a0007)

;

#####

# 2. PROCESSING SUMMARY (JOURNAL OFFICE ONLY)

\_journal\_date\_recd\_electronic ?

\_journal\_date\_to\_coeditor ?

\_journal\_date\_from\_coeditor ?

\_journal\_date\_accepted ?

\_journal\_date\_printers\_first ?

\_journal\_date\_printers\_final ?

\_journal\_date\_proofs\_out ?

\_journal\_date\_proofs\_in ?

\_journal\_coeditor\_name ?

\_journal\_coeditor\_code ?

\_journal\_coeditor\_notes

;

;

\_journal\_techeditor\_code ?

\_journal\_techeditor\_notes

;

;

\_journal\_codен\_ASTM ?

\_journal\_name\_full ?

\_journal\_year ?

\_journal\_volume ?

\_journal\_issue ?

\_journal\_page\_first ?

\_journal\_page\_last ?

\_journal\_suppl\_publ\_number ?

\_journal\_suppl\_publ\_pages ?

#####

# 3. TITLE AND AUTHOR LIST

\_publ\_section\_title

;

;

\_publ\_section\_title\_footnote

;

;

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

loop\_

\_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
;
?
;
_publ_section_abstract
;
?
;

# Insert blank lines between paragraphs

_publ_section_comment
;
;
_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 \%

;

_publ_section_related_literature
;
;

# 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.,

```

Garc'ia-Granda, S. & Gould, R.O. (2008).  
The DIRDIF08 program system, Technical Report of the Crystallography  
Laboratory, University of Nijmegen, The Netherlands.

Boeyens, J.C.A. (1978). *J. Cryst. Mol. Struct.* 8, 317-320.

Bondi, A. (1964). *J. Phys. Chem.* 68, 441-451.

Bruker, (2007). SMART (Version 5.632), SAINT-Plus (Version 7.46a) and  
SADABS (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Hahn, T. (1983). Ed. *International Tables for Crystallography*,  
Volume A, Space-group symmetry, Kluwer Academic Publishers,  
Dordrecht, The Netherlands.

Meetsma, A. (2009). Extended version of the program PLUTO.  
University of Groningen, The Netherlands. (unpublished).

Sheldrick, G.M. (2008). *Acta Cryst.* A64, 112-122.

Spek, A.L. (2003). *J. Appl. Cryst.* 36, 7-13.

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

;

;

#####

# 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

```
_exptl_special_details  
;  
;  
_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
```

```

_diffrrn_reflms_limit_h_min      -49
_diffrrn_reflms_limit_h_max      49
_diffrrn_reflms_limit_k_min      -9
_diffrrn_reflms_limit_k_max      9
_diffrrn_reflms_limit_l_min      -9
_diffrrn_reflms_limit_l_max      9
_diffrrn_reflms_theta_min        3.13
_diffrrn_reflms_theta_max        26.72
_diffrrn_measured_fraction_theta_max  0.998
_diffrrn_reflms_theta_full       25.00
_diffrrn_measured_fraction_theta_full  0.998

_diffrrn_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

```

loop_
_atom_site_label
_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 . .

loop_
_atom_site_aniso_label

```

```

_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
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\_

| _geom_bond_atom_site_label_1 | _geom_bond_atom_site_label_2 | _geom_bond_distance | _geom_bond_site_symmetry_1 | _geom_bond_site_symmetry_2 | _geom_bond_publ_flag |     |
|------------------------------|------------------------------|---------------------|----------------------------|----------------------------|----------------------|-----|
| 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  |

```

loop_
_geom_angle_atom_site_label_1
_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 |

loop\_

\_geom\_torsion\_atom\_site\_label\_1

\_geom\_torsion\_atom\_site\_label\_2

\_geom\_torsion\_atom\_site\_label\_3

\_geom\_torsion\_atom\_site\_label\_4

\_geom\_torsion

\_geom\_torsion\_site\_symmetry\_1

\_geom\_torsion\_site\_symmetry\_2

\_geom\_torsion\_site\_symmetry\_3

\_geom\_torsion\_site\_symmetry\_4

\_geom\_torsion\_publ\_flag

|    |    |    |    |            |       |       |       |    |
|----|----|----|----|------------|-------|-------|-------|----|
| N1 | C1 | C2 | C3 | 179.98(3)  | .     | .     | .     | no |
| 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 |

loop\_

\_geom\_contact\_atom\_site\_label\_1

\_geom\_contact\_atom\_site\_label\_2

\_geom\_contact\_distance

\_geom\_contact\_site\_symmetry\_1

\_geom\_contact\_site\_symmetry\_2

\_geom\_contact\_publ\_flag

|     |     |            |   |       |    |
|-----|-----|------------|---|-------|----|
| C11 | C12 | 3.2387(15) | . | .     | no |
| 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 |

loop\_

\_geom\_hbond\_atom\_site\_label\_D

\_geom\_hbond\_atom\_site\_label\_H

\_geom\_hbond\_atom\_site\_label\_A

\_geom\_hbond\_distance\_DH

\_geom\_hbond\_distance\_HA

\_geom\_hbond\_distance\_DA

\_geom\_hbond\_angle\_DHA

\_geom\_hbond\_site\_symmetry\_A

\_geom\_hbond\_publ\_flag

#

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

#

|    |     |     |        |        |          |     |       |     |
|----|-----|-----|--------|--------|----------|-----|-------|-----|
| 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

# CIF-file generated for C16H24Cl4CuN2 Pbca q1311

data\_DATNAM

# 0. AUDIT DETAILS

\_audit\_creation\_date '2007-09-27 15:01:04'

\_audit\_creation\_method

;

PLATON <TABLE ACC> option (version :: 250307)  
SHELXL97-2 & Manual Editing

;

\_audit\_update\_record

;

?

2008-05-06 # Formatted by publCIF

;

#####

# 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

;

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)

;

#####

# 2. PROCESSING SUMMARY (JOURNAL OFFICE ONLY)

\_journal\_date\_recd\_electronic ?

```

_journal_date_to_coeditor      ?
_journal_date_from_coeditor   ?
_journal_date_accepted        ?

_journal_date_printers_first  ?
_journal_date_printers_final  ?
_journal_date_proofs_out      ?
_journal_date_proofs_in      ?

_journal_coeditor_name        ?
_journal_coeditor_code        ?
_journal_coeditor_notes
;
;

_journal_techeditor_code      ?
_journal_techeditor_notes
;
;

_journal_coden_ASTM           ?
_journal_name_full            ?
_journal_year                  ?
_journal_volume                ?
_journal_issue                 ?
_journal_page_first           ?
_journal_page_last            ?

_journal_suppl_publ_number    ?
_journal_suppl_publ_pages     ?

```

```
#=====
```

```
# 3. TITLE AND AUTHOR LIST
```

```

_publ_section_title
;
Title (type here to add)
;
_publ_section_title_footnote
.

```

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

```

loop_
_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

.

\_publ\_section\_abstract

;

(type here to add abstract)

;

# Insert blank lines between paragraphs

\_publ\_section\_comment

;

(type here to add)

;

\_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 methyl-groups were refined as rigid groups, which were allowed to rotate freely. Assigned values of bond distances: secondary C---H<sub>2</sub> = 0.99 Å, methyl C---H<sub>3</sub> = 0.98 Å, aromatic C---H = 0.95 Å.

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

;

\_publ\_section\_related\_literature

;

;

# Insert blank lines between references

\_publ\_section\_references

;

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.

;

\_publ\_section\_figure\_captions

;

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.

;

\_publ\_section\_acknowledgements

;

?

;

#####

## # 5. CHEMICAL DATA

\_chemical\_name\_systematic

;

;

\_chemical\_name\_common ?

\_chemical\_melting\_point ?

\_chemical\_formula\_moiety

'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

\_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        '-P 2ac 2ab'  
\_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

\_cell\_length\_a                         7.2099(9)  
\_cell\_length\_b                         7.2664(9)  
\_cell\_length\_c                         38.238(5)  
\_cell\_angle\_alpha                      90  
\_cell\_angle\_beta                       90  
\_cell\_angle\_gamma                      90  
\_cell\_volume                           2003.3(4)  
\_cell\_formula\_units\_Z                  4

\_cell\_measurement\_temperature        100(1)  
\_cell\_measurement\_reflns\_used        8650  
\_cell\_measurement\_theta\_min          2.83  
\_cell\_measurement\_theta\_max          29.53  
\_cell\_special\_details

;

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)

;

\_exptl\_crystal\_description            'platelet'  
\_exptl\_crystal\_colour                'orange'  
\_exptl\_crystal\_size\_max              0.51  
\_exptl\_crystal\_size\_mid              0.49  
\_exptl\_crystal\_size\_min              0.12  
\_exptl\_crystal\_size\_rad              ?  
\_exptl\_crystal\_density\_meas          ?  
\_exptl\_crystal\_density\_diffn        1.491  
\_exptl\_crystal\_density\_method        'not measured'  
\_exptl\_crystal\_F\_000                924  
\_exptl\_absorpt\_coefficient\_mu        1.623  
\_exptl\_absorpt\_correction\_type        'multi-scan'  
\_exptl\_absorpt\_process\_details        '(SADABS, (Bruker, 2006))'  
\_exptl\_absorpt\_correction\_T\_min      0.4470  
\_exptl\_absorpt\_correction\_T\_max      0.8230

#=====

# 7. EXPERIMENTAL DATA

\_exptl\_special\_details

;

```

;
_diffrn_ambient_temperature      100(1)
_diffrn_radiation_wavelength     0.71073
_diffrn_radiation_type           'MoK\a'
_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, 2006)).
;
_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_n_index_h
_diffrn_standard_refl_n_index_k
_diffrn_standard_refl_n_index_l
? ? ?

# number of measured reflections (redundant set)
_diffrn_reflns_number           14376
_diffrn_reflns_av_R_equivalents 0.0344
_diffrn_reflns_av_sigmaI/netI  0.0251
_diffrn_reflns_limit_h_min      -9
_diffrn_reflns_limit_h_max      8
_diffrn_reflns_limit_k_min      -9
_diffrn_reflns_limit_k_max      9
_diffrn_reflns_limit_l_min      -50
_diffrn_reflns_limit_l_max      50
_diffrn_reflns_theta_min        3.02
_diffrn_reflns_theta_max        28.27
_diffrn_measured_fraction_theta_max 0.939
_diffrn_reflns_theta_full       25.00
_diffrn_measured_fraction_theta_full 0.976

_diffrn_reflns_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, 2006).
;

# number of unique reflections
_reflns_number_total           2340
_reflns_number_gt              2163
_reflns_threshold_expression    I>2\s(I)

_computing_data_collection     'SMART (Bruker, 2006)'
_computing_cell_refinement     'SAINT-Plus (Bruker, 2006)'
_computing_data_reduction     'SAINT-Plus (Bruker, 2006)'
_computing_structure_solution

```

```

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

#=====

# 8. REFINEMENT DATA

_refine_special_details
;
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.
;

_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type              full
_refine_ls_weighting_scheme          calc
_refine_ls_weighting_details
'calc w=1/[s2(Fo2)+(0.0P)2+9.9068P] where P=(Fo2+2Fc2)/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             2340
_refine_ls_number_parameters         107
_refine_ls_number_restraints         0
_refine_ls_number_constraints        ?
_refine_ls_R_factor_all              0.0437
_refine_ls_R_factor_gt               0.0400
_refine_ls_wR_factor_ref             0.1120
_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

_refine_diff_density_max             0.733
_refine_diff_density_min             -0.605
_refine_diff_density_rms            0.121

_vrn_publ_code_void_volume          70.6
_vrn_publ_code_frame_time_sec        5.0
_vrn_publ_code_meas_time_hour        5.5

#=====

```

# 9. ATOMIC COORDINATES AND DISPLACEMENT PARAMETERS

```
loop_
_atom_site_label
_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
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_
_atom_site_aniso_label
_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
_geom_bond_distance
_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

```

```

loop_
_geom_angle_atom_site_label_1
_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
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  |

loop\_

\_geom\_torsion\_atom\_site\_label\_1  
 \_geom\_torsion\_atom\_site\_label\_2  
 \_geom\_torsion\_atom\_site\_label\_3  
 \_geom\_torsion\_atom\_site\_label\_4  
 \_geom\_torsion  
 \_geom\_torsion\_site\_symmetry\_1  
 \_geom\_torsion\_site\_symmetry\_2  
 \_geom\_torsion\_site\_symmetry\_3  
 \_geom\_torsion\_site\_symmetry\_4  
 \_geom\_torsion\_publ\_flag

|    |    |    |    |           |   |   |   |    |
|----|----|----|----|-----------|---|---|---|----|
| 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\_

\_geom\_contact\_atom\_site\_label\_1  
 \_geom\_contact\_atom\_site\_label\_2  
 \_geom\_contact\_distance  
 \_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 |

loop\_

\_geom\_hbond\_atom\_site\_label\_D  
 \_geom\_hbond\_atom\_site\_label\_H  
 \_geom\_hbond\_atom\_site\_label\_A  
 \_geom\_hbond\_distance\_DH  
 \_geom\_hbond\_distance\_HA  
 \_geom\_hbond\_distance\_DA  
 \_geom\_hbond\_angle\_DHA  
 \_geom\_hbond\_site\_symmetry\_A  
 \_geom\_hbond\_publ\_flag

| # | 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