Balancing Hydrogen Bonding and van der Waals Interactions in Cyclohexane-Based Bisamide and Bisurea Organogelators

Zweep, Nick; Hopkinson, Andrew; Meetsma, Auke; Browne, Wesley R.; Feringa, Ben L.; van Esch, Jan H.; Zweep, Niek

Published in:
Langmuir

DOI:
10.1021/la9004714

```
# CIF-file generated for C14H26N2O2                  P21           q1001
#===============================================================================
data_global
#===============================================================================

# 0. AUDIT DETAILS

_audit_creation_date                 '2009-03-10  08:05:41'
_audit_creation_method
;
 PLATON <TABLE ACC> option    (version :: 170209)
 SHELXL97-2 & Manual Editing
;
_audit_update_record
;
  first edition was created 2005-05-17 10:02:34

  Now : the hydrogen atoms were refined in riding mode
;

#===============================================================================

# 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              'Langmuir AC'
# Publication choise 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 :  2009-01-20  13:02:34
 Consider this CIF submission for deposition of the
 X-ray structure of a manuscript to be submitted to : Langmuir ACS
  (Our Compound_Identification_Code : Q1001)

  Updated : 2009-03-10  08:07:00
;

#===============================================================================

# 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
;
;
_publ_section_title_footnote
;
;

# The loop structure below should contain the names and adresses 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
;
The chiral centers of C1 and C1 showed both the <i>R</i>-configuration. A
search of the distances yielded intermolecular and intramolecular contacts
shorter than the sum of the van der Waals radii for the atoms: the moieties
are linked by hydrogen bonds, forming an infinite one-dimensional
network along the [0 1 0] base vector.
;
_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: tertiary C-H = 1.00 \%A,
  secondary C-H~2~ = 0.99 \%A, methyl C-H~3~ = 0.98 \%A,
   aromatic C-H = 0.95 \%A and N-H = 0.91 \%A
;


_publ_section_related_literature
;
;

# Insert blank lines between references

_publ_section_references
;
Allen, F. H. (2000). <i>Acta Cryst.</i> B<b>58</b>, 380--388.
```

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). J. Appl. Cryst. 32, 115--119.

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

Bruker, (2000). SMART, SAINT, SADABS, XPREP and SHELXTL/NT. Software Reference Manual Bruker AXS Inc. Madison, Wisconsin, USA.

International Tables for Crystallography (1983). Vol. A. Space-group symmetry, edited by T. Hahn. Dordrecht: Reidel. (Present distributor Kluwer Academic Publishers, Dordrecht).

International Tables for Crystallography (1992). Vol. C. Edited by A.J.C Wilson, Kluwer Academic Publishers, Dordrecht, The Netherlands.

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

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

Meetsma, A. (2004). Extended version of the program PLUTO. Groningen University, The Netherlands. (unpublished).

Sheldrick, G.M. SHELXL97. Program for Crystal Structure Refinement. University of G\"ottingen, Germany, 1997.

Spek, A. L. (1990). Acta Cryst. A46 C-34.

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

_publ_section_figure_captions
;

Fig. 1. Perspective PLUTO drawing 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 are represented at the 50% probability level. The H-atoms are drawn with an arbitrary radius.
;

_publ_section_acknowledgements
;
;

#==

5. CHEMICAL DATA

_chemical_name_systematic
;
;
_chemical_name_common ?
_chemical_melting_point ?
_chemical_formula_moiety

```
'C14 H26 N2 O2'
# 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
'C14 H26 N2 O2'
_chemical_formula_iupac              ?
_chemical_formula_weight               254.37
_chemical_compound_source           'see text'

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
 O   O    0.0106    0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 N   N    0.0061    0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 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                monoclinic
_symmetry_space_group_name_Hall      'P 2yb'
_symmetry_space_group_name_H-M       'P 21'
_symmetry_Int_Tables_number                4

loop_
_symmetry_equiv_pos_site_id
_symmetry_equiv_pos_as_xyz
   1 x,y,z
   2 -x,1/2+y,-z

_cell_length_a                        11.776(2)
_cell_length_b                         4.777(1)
_cell_length_c                        13.250(3)
_cell_angle_alpha                     90
_cell_angle_beta                      99.185(3)
_cell_angle_gamma                     90
_cell_volume                         735.8(3)
_cell_formula_units_Z                 2

_cell_measurement_temperature    100(1)
_cell_measurement_reflns_used      3025
_cell_measurement_theta_min        2.52
_cell_measurement_theta_max       27.53
_cell_special_details
;
 The final unit cell was obtained from the xyz centroids of
   3025 reflections after integration using the SAINT software
   package (Bruker, 2000).
;

_exptl_crystal_description       'platelet'
_exptl_crystal_colour            'colorless'
```

```
_exptl_crystal_size_max          0.41
_exptl_crystal_size_mid          0.29
_exptl_crystal_size_min          0.04
_exptl_crystal_size_rad          ?
_exptl_crystal_density_meas      ?
_exptl_crystal_density_diffrn    1.148
_exptl_crystal_density_method    'not measured'
_exptl_crystal_F_000             280
_exptl_absorpt_coefficient_mu    0.076
_exptl_absorpt_correction_type   'multi-scan'
_exptl_absorpt_process_details   '(SADABS, (Bruker, 2000))'
_exptl_absorpt_correction_T_min   0.9774
_exptl_absorpt_correction_T_max   0.9969

#===============================================================================

# 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-temparature 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_refln_index_h
_diffrn_standard_refln_index_k
_diffrn_standard_refln_index_l
 ? ? ?

# number of measured reflections (redundant set)
_diffrn_reflns_number             5448
_diffrn_reflns_av_R_equivalents   0.0477
_diffrn_reflns_av_sigmaI/netI     0.0452
_diffrn_reflns_limit_h_min        -14
_diffrn_reflns_limit_h_max        14
_diffrn_reflns_limit_k_min        -5
```

```
_diffrn_reflns_limit_k_max        5
_diffrn_reflns_limit_l_min        -16
_diffrn_reflns_limit_l_max        16
_diffrn_reflns_theta_min          3.11
_diffrn_reflns_theta_max          25.67
_diffrn_measured_fraction_theta_max    0.997
_diffrn_reflns_theta_full              25.00
_diffrn_measured_fraction_theta_full   0.997

_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, 2000).
;

# number of unique reflections
_reflns_number_total              1574
_reflns_number_gt                 1324
_reflns_threshold_expression     I>2\s(I)


_computing_data_collection          'SMART (Bruker, 2000)'
_computing_cell_refinement          'SAINT-Plus (Bruker, 2000)'
_computing_data_reduction           'SAINT-Plus'
_computing_structure_solution
;
 SIR-97 (Altomare et al., 1999)
;
_computing_structure_refinement      'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics
;
 PLATON (Spek, 2003)
 PLUTO (Meetsma, 2004)
;
_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^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0719P)^2^+0.0P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary        direct
_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
;
  Enantiomorph twin refinement resulted in 0.47(2) so ultimately set to 0.5

  Because this light-atom structure does not reveal sufficient anomalous
   dispersion, the absolute structure could not be confirmed as known
   by synthese route.
;
_chemical_absolute_configuration  syn
_refine_ls_abs_structure_Flack    ?
_refine_ls_number_reflns          1574
_refine_ls_number_parameters      165
_refine_ls_number_restraints      1
_refine_ls_number_constraints     ?
_refine_ls_R_factor_all           0.0558
_refine_ls_R_factor_gt            0.0444
_refine_ls_wR_factor_ref          0.1119
_refine_ls_wR_factor_gt           0.1079
_refine_ls_goodness_of_fit_ref    1.031
_refine_ls_restrained_S_all       1.030
_refine_ls_shift/su_max           0.000
_refine_ls_shift/su_mean          0.000
_refine_diff_density_max          0.231
_refine_diff_density_min         -0.209
_refine_diff_density_rms          0.047


_vrn_publ_code_frame_time_sec      45.0
_vrn_publ_code_meas_time_hour      28.0

#===============================================================================

# 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
O1 O Uani 0.41406(17) 0.6096(4) 0.29015(14) 1.000 0.0240(7) . .
O2 O Uani 0.0765(2) -0.2027(5) 0.23427(16) 1.000 0.0433(8) . .
N1 N Uani 0.37912(16) 0.1758(5) 0.34469(15) 1.000 0.0149(7) . .
N2 N Uani 0.13379(17) 0.2293(5) 0.28674(15) 1.000 0.0152(6) . .
C1 C Uani 0.3063(2) 0.2646(6) 0.41736(17) 1.000 0.0147(7) . .
C2 C Uani 0.3576(2) 0.1802(6) 0.52582(18) 1.000 0.0199(8) . .
C3 C Uani 0.2823(2) 0.2731(7) 0.60284(18) 1.000 0.0199(8) . .
C4 C Uani 0.1608(2) 0.1582(6) 0.57482(18) 1.000 0.0199(8) . .
C5 C Uani 0.1105(2) 0.2397(7) 0.46660(18) 1.000 0.0200(8) . .
C6 C Uani 0.1848(2) 0.1468(6) 0.38904(18) 1.000 0.0144(7) . .
C7 C Uani 0.4268(2) 0.3549(6) 0.28660(19) 1.000 0.0160(8) . .
C8 C Uani 0.5017(2) 0.2280(6) 0.21646(17) 1.000 0.0171(8) . .
C9 C Uani 0.4679(3) 0.3256(7) 0.1075(2) 1.000 0.0271(9) . .
```

```
C10 C Uani 0.3483(3) 0.2300(11) 0.0613(2) 1.000 0.0507(13) . .
C11 C Uani 0.0814(2) 0.0507(6) 0.2172(2) 1.000 0.0203(9) . .
C12 C Uani 0.0252(2) 0.1725(6) 0.11754(18) 1.000 0.0192(8) . .
C13 C Uani -0.1029(2) 0.0996(7) 0.0951(2) 1.000 0.0230(8) . .
C14 C Uani -0.1690(3) 0.2255(8) 0.1735(2) 1.000 0.0356(10) . .
H1 H Uiso 0.30109 0.47344 0.41482 1.000 0.0176 . .
H2 H Uiso 0.36673 -0.02574 0.52922 1.000 0.0239 . .
H2' H Uiso 0.43482 0.26531 0.54385 1.000 0.0239 . .
H3 H Uiso 0.27950 0.48009 0.60471 1.000 0.0239 . .
H3' H Uiso 0.31612 0.20609 0.67181 1.000 0.0239 . .
H4 H Uiso 0.16233 -0.04839 0.58105 1.000 0.0238 . .
H4' H Uiso 0.11177 0.23290 0.62271 1.000 0.0238 . .
H5 H Uiso 0.10136 0.44561 0.46286 1.000 0.0240 . .
H5' H Uiso 0.03321 0.15488 0.44879 1.000 0.0240 . .
H6 H Uiso 0.18987 -0.06213 0.39110 1.000 0.0172 . .
H8 H Uiso 0.49561 0.02149 0.21884 1.000 0.0204 . .
H8' H Uiso 0.58288 0.27936 0.24082 1.000 0.0204 . .
H9 H Uiso 0.52392 0.25231 0.06574 1.000 0.0325 . .
H9' H Uiso 0.47113 0.53257 0.10554 1.000 0.0325 . .
H10 H Uiso 0.34276 0.02632 0.06809 1.000 0.0759 . .
H10' H Uiso 0.33302 0.28128 -0.01117 1.000 0.0759 . .
H10" H Uiso 0.29154 0.32042 0.09724 1.000 0.0759 . .
H12 H Uiso 0.06378 0.09984 0.06179 1.000 0.0230 . .
H12' H Uiso 0.03430 0.37853 0.11962 1.000 0.0230 . .
H13 H Uiso -0.13563 0.16971 0.02626 1.000 0.0276 . .
H13' H Uiso -0.11193 -0.10644 0.09485 1.000 0.0276 . .
H14 H Uiso -0.13852 0.15162 0.24139 1.000 0.0536 . .
H14' H Uiso -0.25056 0.17646 0.15589 1.000 0.0536 . .
H14" H Uiso -0.16056 0.42957 0.17367 1.000 0.0536 . .
H21 H Uiso 0.39238 -0.00423 0.33870 1.000 0.0179 . .
H22 H Uiso 0.13755 0.40660 0.26954 1.000 0.0183 . .

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
O1 0.0328(12) 0.0106(11) 0.0324(11) -0.0005(9) 0.0170(9) 0.0002(9)
O2 0.0594(16) 0.0136(13) 0.0463(13) 0.0020(11) -0.0242(12) -0.0019(11)
N1 0.0114(11) 0.0109(12) 0.0240(11) -0.0003(10) 0.0081(9) 0.0024(9)
N2 0.0136(11) 0.0102(12) 0.0214(10) 0.0016(10) 0.0012(9) 0.0002(10)
C1 0.0109(12) 0.0117(14) 0.0227(12) -0.0009(12) 0.0065(10) 0.0007(11)
C2 0.0122(13) 0.0218(16) 0.0247(13) -0.0018(13) -0.0005(10) -0.0006(12)
C3 0.0187(14) 0.0221(16) 0.0184(12) -0.0032(13) 0.0014(10) 0.0000(12)
C4 0.0186(14) 0.0236(16) 0.0190(13) 0.0016(12) 0.0080(11) 0.0013(13)
C5 0.0132(13) 0.0230(16) 0.0245(13) 0.0002(13) 0.0054(10) -0.0004(12)
C6 0.0118(12) 0.0114(14) 0.0200(12) 0.0030(11) 0.0030(10) -0.0007(11)
C7 0.0105(13) 0.0158(16) 0.0204(13) -0.0031(12) -0.0013(11) 0.0014(11)
C8 0.0104(12) 0.0167(15) 0.0254(13) 0.0010(12) 0.0070(10) 0.0009(12)
C9 0.0284(17) 0.0285(18) 0.0269(14) 0.0037(14) 0.0124(12) 0.0094(14)
C10 0.0307(18) 0.085(3) 0.0337(17) -0.009(2) -0.0031(14) 0.021(2)
C11 0.0189(14) 0.0141(17) 0.0283(14) 0.0000(12) 0.0047(12) 0.0020(12)
C12 0.0170(14) 0.0170(15) 0.0230(13) -0.0035(12) 0.0015(11) -0.0021(12)
C13 0.0178(14) 0.0241(16) 0.0261(14) 0.0011(13) 0.0003(11) -0.0014(13)
C14 0.0265(16) 0.046(2) 0.0361(16) 0.0033(17) 0.0104(13) 0.0067(17)

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

```
# 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
 O1      C7         1.228(3)                    .      .                   yes
 O2      C11        1.235(4)                    .      .                   yes
 N1      C1         1.451(3)                    .      .                   yes
 N1      C7         1.333(3)                    .      .                   yes
 N2      C6         1.447(3)                    .      .                   yes
 N2      C11        1.333(3)                    .      .                   yes
 N1      H21          0.8800                    .      .                   no
 N2      H22          0.8800                    .      .                   no
 C1      C6         1.528(3)                    .      .                   no
 C1      C2         1.522(3)                    .      .                   no
 C2      C3         1.521(3)                    .      .                   no
 C3      C4         1.522(4)                    .      .                   no
 C4      C5         1.512(3)                    .      .                   no
 C5      C6         1.518(3)                    .      .                   no
 C7      C8         1.507(3)                    .      .                   no
 C8      C9         1.509(4)                    .      .                   no
 C9      C10        1.514(5)                    .      .                   no
 C11     C12        1.497(4)                    .      .                   no
 C12     C13        1.530(3)                    .      .                   no
 C13     C14        1.518(4)                    .      .                   no
 C1      H1           1.0000                    .      .                   no
 C2      H2           0.9900                    .      .                   no
 C2      H2'          0.9900                    .      .                   no
 C3      H3           0.9900                    .      .                   no
 C3      H3'          0.9900                    .      .                   no
 C4      H4           0.9900                    .      .                   no
 C4      H4'          0.9900                    .      .                   no
 C5      H5           0.9900                    .      .                   no
 C5      H5'          0.9900                    .      .                   no
 C6      H6           1.0000                    .      .                   no
 C8      H8           0.9900                    .      .                   no
 C8      H8'          0.9900                    .      .                   no
 C9      H9           0.9900                    .      .                   no
 C9      H9'          0.9900                    .      .                   no
 C10     H10          0.9800                    .      .                   no
 C10     H10'         0.9800                    .      .                   no
 C10     H10"         0.9800                    .      .                   no
 C12     H12          0.9900                    .      .                   no
 C12     H12'         0.9900                    .      .                   no
 C13     H13          0.9900                    .      .                   no
 C13     H13'         0.9900                    .      .                   no
 C14     H14          0.9800                    .      .                   no
 C14     H14'         0.9800                    .      .                   no
```

```
 C14     H14"         0.9800                    .       .                 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
 C1      N1      C7         122.9(2)             .       .       .          yes
 C6      N2      C11        123.4(2)             .       .       .          yes
 C7      N1      H21          119.00             .       .       .          no
 C1      N1      H21          119.00             .       .       .          no
 C11     N2      H22          118.00             .       .       .          no
 C6      N2      H22          118.00             .       .       .          no
 N1      C1      C2         111.2(2)             .       .       .          yes
 C2      C1      C6         110.5(2)             .       .       .          no
 N1      C1      C6         111.0(2)             .       .       .          yes
 C1      C2      C3         111.7(2)             .       .       .          no
 C2      C3      C4         110.9(2)             .       .       .          no
 C3      C4      C5         110.4(2)             .       .       .          no
 C4      C5      C6         112.7(2)             .       .       .          no
 C1      C6      C5         110.2(2)             .       .       .          no
 N2      C6      C1         111.2(2)             .       .       .          yes
 N2      C6      C5         110.9(2)             .       .       .          yes
 O1      C7      C8         120.6(2)             .       .       .          yes
 N1      C7      C8         116.1(2)             .       .       .          yes
 O1      C7      N1         123.3(2)             .       .       .          yes
 C7      C8      C9         112.0(2)             .       .       .          no
 C8      C9      C10        112.3(3)             .       .       .          no
 O2      C11     N2         122.1(2)             .       .       .          yes
 N2      C11     C12        116.9(2)             .       .       .          yes
 O2      C11     C12        121.0(2)             .       .       .          yes
 C11     C12     C13        111.5(2)             .       .       .          no
 C12     C13     C14        112.0(2)             .       .       .          no
 N1      C1      H1           108.00             .       .       .          no
 C2      C1      H1           108.00             .       .       .          no
 C6      C1      H1           108.00             .       .       .          no
 C1      C2      H2           109.00             .       .       .          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
 C2      C3      H3           109.00             .       .       .          no
 C2      C3      H3'          109.00             .       .       .          no
 C4      C3      H3           109.00             .       .       .          no
 C4      C3      H3'          109.00             .       .       .          no
 H3      C3      H3'          108.00             .       .       .          no
 C3      C4      H4           110.00             .       .       .          no
 C3      C4      H4'          110.00             .       .       .          no
 C5      C4      H4           110.00             .       .       .          no
 C5      C4      H4'          110.00             .       .       .          no
 H4      C4      H4'          108.00             .       .       .          no
 C4      C5      H5           109.00             .       .       .          no
 C4      C5      H5'          109.00             .       .       .          no
 C6      C5      H5           109.00             .       .       .          no
 C6      C5      H5'          109.00             .       .       .          no
 H5      C5      H5'          108.00             .       .       .          no
 N2      C6      H6           108.00             .       .       .          no
```

```
 C1      C6      H6          108.00              .       .       .          no
 C5      C6      H6          108.00              .       .       .          no
 C7      C8      H8          109.00              .       .       .          no
 C7      C8      H8'         109.00              .       .       .          no
 C9      C8      H8          109.00              .       .       .          no
 C9      C8      H8'         109.00              .       .       .          no
 H8      C8      H8'         108.00              .       .       .          no
 C8      C9      H9          109.00              .       .       .          no
 C8      C9      H9'         109.00              .       .       .          no
 C10     C9      H9          109.00              .       .       .          no
 C10     C9      H9'         109.00              .       .       .          no
 H9      C9      H9'         108.00              .       .       .          no
 C9      C10     H10         109.00              .       .       .          no
 C9      C10     H10'        110.00              .       .       .          no
 C9      C10     H10"        109.00              .       .       .          no
 H10     C10     H10'        109.00              .       .       .          no
 H10     C10     H10"        109.00              .       .       .          no
 H10'    C10     H10"        109.00              .       .       .          no
 C11     C12     H12         109.00              .       .       .          no
 C11     C12     H12'        109.00              .       .       .          no
 C13     C12     H12         109.00              .       .       .          no
 C13     C12     H12'        109.00              .       .       .          no
 H12     C12     H12'        108.00              .       .       .          no
 C12     C13     H13         109.00              .       .       .          no
 C12     C13     H13'        109.00              .       .       .          no
 C14     C13     H13         109.00              .       .       .          no
 C14     C13     H13'        109.00              .       .       .          no
 H13     C13     H13'        108.00              .       .       .          no
 C13     C14     H14         109.00              .       .       .          no
 C13     C14     H14'        109.00              .       .       .          no
 C13     C14     H14"        109.00              .       .       .          no
 H14     C14     H14'        110.00              .       .       .          no
 H14     C14     H14"        110.00              .       .       .          no
 H14'    C14     H14"        109.00              .       .       .          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
 C7      N1      C1      C2        -118.9(3)     .       .       .       .   no
 C7      N1      C1      C6         117.7(3)     .       .       .       .   no
 C1      N1      C7      O1           0.6(4)     .       .       .       .   no
 C1      N1      C7      C8         179.0(2)     .       .       .       .   no
 C11     N2      C6      C1         131.8(2)     .       .       .       .   no
 C11     N2      C6      C5        -105.3(3)     .       .       .       .   no
 C6      N2      C11     O2          -2.8(4)     .       .       .       .   no
 C6      N2      C11     C12        175.5(2)     .       .       .       .   no
 N1      C1      C2      C3         179.8(2)     .       .       .       .   no
 C6      C1      C2      C3         -56.5(3)     .       .       .       .   no
 N1      C1      C6      N2         -57.2(3)     .       .       .       .   no
 N1      C1      C6      C5         179.4(2)     .       .       .       .   no
 C2      C1      C6      N2         178.9(2)     .       .       .       .   no
 C2      C1      C6      C5          55.6(3)     .       .       .       .   no
 C1      C2      C3      C4          56.2(3)     .       .       .       .   no
```

C2	C3	C4	C5	-54.9(3)	.	.	.	.	no
C3	C4	C5	C6	55.9(3)	.	.	.	.	no
C4	C5	C6	N2	-179.9(2)	.	.	.	.	no
C4	C5	C6	C1	-56.3(3)	.	.	.	.	no
O1	C7	C8	C9	-53.4(3)	.	.	.	.	no
N1	C7	C8	C9	128.2(3)	.	.	.	.	no
C7	C8	C9	C10	-64.4(4)	.	.	.	.	no
O2	C11	C12	C13	55.7(3)	.	.	.	.	no
N2	C11	C12	C13	-122.6(3)	.	.	.	.	no
C11	C12	C13	C14	63.6(3)	.	.	.	.	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

O1	N1	2.846(3)	.	1_565	no
O1	C8	3.327(3)	.	1_565	no
O1	C2	3.346(3)	.	2_656	no
O2	C12	3.372(4)	.	1_545	no
O2	N2	2.855(3)	.	1_545	no
O1	H9'	2.6600	.	.	no
O1	H8	2.4400	.	1_565	no
O1	H2'	2.7000	.	2_656	no
O1	H21	1.9800	.	1_565	no
O1	H1	2.3700	.	.	no
O2	H6	2.3800	.	.	no
O2	H12'	2.5100	.	1_545	no
O2	H13'	2.6900	.	.	no
O2	H22	2.0300	.	1_545	no
N1	O1	2.846(3)	.	1_545	no
N1	N2	2.880(3)	.	.	no
N2	O2	2.855(3)	.	1_565	no
N2	N1	2.880(3)	.	.	no
C2	O1	3.346(3)	.	2_646	no
C8	O1	3.327(3)	.	1_545	no
C12	O2	3.372(4)	.	1_565	no
C5	H5'	2.9400	.	2_556	no
C7	H10"	2.7500	.	.	no
C9	H9	3.0800	.	2_655	no
C11	H14	2.7000	.	.	no
H1	O1	2.3700	.	.	no
H1	H3	2.5700	.	.	no
H1	H5	2.5400	.	.	no
H1	H6	2.5700	.	1_565	no
H1	H22	2.5200	.	.	no
H2	H6	2.5500	.	.	no
H2	H21	2.5900	.	.	no
H2'	O1	2.7000	.	2_646	no
H3	H1	2.5700	.	.	no
H3	H5	2.5900	.	.	no
H3'	H8'	2.5400	.	2_646	no
H4	H6	2.5900	.	.	no
H5	H1	2.5400	.	.	no
H5	H3	2.5900	.	.	no
H5	H5'	2.3400	.	2_556	no
H5'	C5	2.9400	.	2_546	no
H5'	H5	2.3400	.	2_546	no
H6	O2	2.3800	.	.	no

```
 H6      H1          2.5700                    .     1_545                no
 H6      H2          2.5500                    .       .                  no
 H6      H4          2.5900                    .       .                  no
 H8      O1          2.4400                    .     1_545                no
 H8      H10         2.4700                    .       .                  no
 H8      H21         2.1500                    .       .                  no
 H8'     H14'        2.4600                    .     1_655                no
 H8'     H3'         2.5400                    .     2_656                no
 H9      C9          3.0800                    .     2_645                no
 H9      H9'         2.5100                    .     2_645                no
 H9'     O1          2.6600                    .       .                  no
 H9'     H9          2.5100                    .     2_655                no
 H10     H8          2.4700                    .       .                  no
 H10"    C7          2.7500                    .       .                  no
 H12     H13         2.5700                    .     2_545                no
 H12'    O2          2.5100                    .     1_565                no
 H12'    H14"        2.5200                    .       .                  no
 H12'    H22         2.1600                    .       .                  no
 H13     H12         2.5700                    .     2_555                no
 H13'    O2          2.6900                    .       .                  no
 H13'    H14"        2.5500                    .     1_545                no
 H14     C11         2.7000                    .       .                  no
 H14'    H8'         2.4600                    .     1_455                no
 H14"    H12'        2.5200                    .       .                  no
 H14"    H13'        2.5500                    .     1_565                no
 H21     O1          1.9800                    .     1_545                no
 H21     H2          2.5900                    .       .                  no
 H21     H8          2.1500                    .       .                  no
 H22     O2          2.0300                    .     1_565                no
 H22     H1          2.5200                    .       .                  no
 H22     H12'        2.1600                    .       .                  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      H21     O1          0.8800     1.9800   2.846(3)     166.00   1_545 yes
N2      H22     O2          0.8800     2.0300   2.855(3)     156.00   1_565 yes
C1      H1      O1          1.0000     2.3700   2.802(3)     105.00     .   yes
C6      H6      O2          1.0000     2.3800   2.790(3)     104.00     .   yes
C8      H8      O1          0.9900     2.4400   3.327(3)     148.00   1_545 yes
C12     H12'    O2          0.9900     2.5100   3.372(4)     145.00   1_565 yes

#===END of Crystallographic Information File
```