Supporting Information

A Novel Cyanide-Bridged Thulium-Nickel Heterobimetallic Polymeric Complex $\{(H_2O)_2(DMF)_{10}Tm_2[Ni(CN)_4]_2\}[Ni(CN)_4]$ including O-H···N Hydrogen Bond

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#₩#CIF_1.1

#   CIF produced by WinGX routine CIF_UPDATE
#   Created on 2011-07-14 at 14:10:01
#   Using CIFtbx version 2.6.2 16 Jun 1998

#   Dictionary name     : cif_core.dic
#   Dictionary vers     : 2.4
#   Request file        : c:₩wingx₩files₩archive.reqdat
#   CIF files read      : shelxl dreduc import struct

#------------------ SECTION 1. GLOBAL INFORMATION ---------------------------#

data_global

#------------------ AUDIT DETAILS -------------------------------------------#

_audit_creation_date                2011-07-14
_audit_creation_method              'WinGX routine CIF_UPDATE'
_audit_conform_dict_name            cif_core.dic
_audit_conform_dict_version         2.4
_audit_conform_dict_location        ftp://ftp.iucr.org/pub/cif_core.dic
_audit_update_record                ?

#------------------ AUTHOR DETAILS ------------------------------------------#

# Name and address of author for correspondence
```

```
_publ_contact_author_name         'Jang-Hoon Chung'
_publ_contact_author_address
;Department of Chemistry
The Ohio State University
100 West 18th Avenue
Columbus, OH 43210
USA
;
_publ_contact_author_email        'chungjh@mju.ac.kr'
_journal_name_full                'Bulletin of the Korean Chemical Society'

#------------------   SECTION 2. COMPOUND(S) DETAILS -------------------------#

data_Chung1826

_audit_creation_date                      2011-07-14T14:10:01-00:00
_audit_creation_method                    'WinGX routine CIF_UPDATE'
_audit_conform_dict_name                  cif_core.dic
_audit_conform_dict_version               2.4
_audit_conform_dict_location              ftp://ftp.iucr.org/pub/cif_core.dic
_publ_requested_category                  FM

#------------------------------------------------------------------------------#
#                         CHEMICAL INFORMATION                                 #
#------------------------------------------------------------------------------#

_chemical_name_systematic
;
 ?
;
_chemical_name_common                     'Jangchung-9'
_chemical_formula_moiety                  '2(C19 H37 N9 Ni O6 Tm), C4 N4 Ni'
_chemical_formula_sum                     'C42 H74 N22 Ni3 O12 Tm2'
_chemical_formula_weight                  1593.22
_chemical_compound_source                 'synthesis as described'

#------------------------------------------------------------------------------#
#                         UNIT CELL INFORMATION                                #
#------------------------------------------------------------------------------#
```

```
_symmetry_cell_setting                triclinic
_symmetry_space_group_name_H-M        P-1
 _symmetry_space_group_name_Hall      '-P 1'
_symmetry_Int_Tables_number           2

loop_
    _symmetry_equiv_pos_as_xyz
'x, y, z'
'-x, -y, -z'

_cell_length_a                        7.6095(1)
_cell_length_b                        9.7666(1)
_cell_length_c                        22.7764(2)
_cell_angle_alpha                     86.423(1)
_cell_angle_beta                      81.935(1)
_cell_angle_gamma                     74.204(1)
_cell_volume                          1612.17(3)
_cell_formula_units_Z                 1
_cell_measurement_temperature         180(2)
_cell_measurement_reflns_used         7351
_cell_measurement_theta_min           2.037
_cell_measurement_theta_max           27.485
_cell_measurement_wavelength          0.71073

#------------------------------------------------------------------------#
#                      CRYSTAL INFORMATION                               #
#------------------------------------------------------------------------#

_exptl_crystal_description            plate
_exptl_crystal_colour                 colourless
_exptl_crystal_size_max               0.38
_exptl_crystal_size_mid               0.23
_exptl_crystal_size_min               0.08
_exptl_crystal_density_diffrn         1.641
_exptl_crystal_density_method         'not measured'
_exptl_crystal_F_000                  798
_exptl_special_details
;All work was done at 180 K using an Oxford Cryosystems Cryostream Cooler.
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The data collection strategy was set up to measure a
  hemisphere of reciprocal space with a redundancy
  factor of 3.4, which means that 90% of the reflections were measured at
  least 3.4 times.  A combination of phi and omega scans with a frame width
  of 1.0 degree was used for data collection.  Data integration was done with
  Denzo, and scaling and merging of the data was done with Scalepack.
;

#---------------------------------------------------------------------------#
#                         ABSORPTION CORRECTION                             #
#---------------------------------------------------------------------------#

_exptl_absorpt_coefficient_mu                3.651
_exptl_absorpt_correction_type               multi-scan
_exptl_absorpt_correction_T_min              0.521
_exptl_absorpt_correction_T_max              0.747
_exptl_absorpt_process_details   'HKL Scalepack (Otwinowski & Minor, 1997)'

#---------------------------------------------------------------------------#
#                         DATA COLLECTION                                   #
#---------------------------------------------------------------------------#

_diffrn_source                               'Enraf Nonius FR590'
_diffrn_ambient_temperature                  180(2)
_diffrn_radiation_wavelength                 0.71073
_diffrn_radiation_type                       MoK₩a
_diffrn_radiation_monochromator              graphite
_diffrn_radiation_probe                      x-ray
_diffrn_detector                             'CCD plate'
_diffrn_detector_area_resol_mean             9
_diffrn_orient_matrix_type                   'by Nonius Collect from scalepack cell'
_diffrn_orient_matrix_ub_11                  -0.133292
_diffrn_orient_matrix_ub_12                  0.151559E-1
_diffrn_orient_matrix_ub_13                  0.155366E-1
_diffrn_orient_matrix_ub_21                  -0.316119E-1
_diffrn_orient_matrix_ub_22                  0.131419E-1
_diffrn_orient_matrix_ub_23                  -0.415083E-1
_diffrn_orient_matrix_ub_31                  0.140865E-1
_diffrn_orient_matrix_ub_32                  -0.104535
```

```
_diffrn_orient_matrix_ub_33                -0.18185E-2
_diffrn_measurement_device                 '95mm CCD camera on ₩k-goniostat'
_diffrn_measurement_device_type            'Nonius KappaCCD'
_diffrn_measurement_method                 'phi and omega scans'
_diffrn_reflns_av_R_equivalents            0.057
_diffrn_reflns_av_unetI/netI               ?
_diffrn_reflns_number                      41439
_diffrn_reflns_limit_h_min                 -9
_diffrn_reflns_limit_h_max                 9
_diffrn_reflns_limit_k_min                 -12
_diffrn_reflns_limit_k_max                 12
_diffrn_reflns_limit_l_min                 -29
_diffrn_reflns_limit_l_max                 29
_diffrn_reflns_theta_min                   2.17
_diffrn_reflns_theta_max                   27.5
_diffrn_reflns_theta_full                  27.5
_diffrn_measured_fraction_theta_full
                                           0.997
_diffrn_measured_fraction_theta_max
                                           0.997
_reflns_number_total                       7383
_reflns_number_gt                          6090
_reflns_threshold_expression               >2₩s(I)

#-----------------------------------------------------------------------#
#                      COMPUTER PROGRAMS USED                           #
#-----------------------------------------------------------------------#

_computing_data_collection                 'Collect (Nonius BV, 1997-2000)'
_computing_cell_refinement
                                           'HKL Scalepack (Otwinowski & Minor 1997)'
_computing_data_reduction
                                  'HKL Denzo and Scalepack (Otwinowski & Minor 1997)'
_computing_structure_solution              'SHELXS-97 (Sheldrick, 2008)'
_computing_structure_refinement            'SHELXL-97 (Sheldrick, 2008)'
_computing_molecular_graphics              'Ortep-3 for Windows (Farrugia, 1997)'
_computing_publication_material
                                  'WinGX publication routines (Farrugia, 1999)'
```

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#------------------------------------------------------------------------------#
#                         STRUCTURE SOLUTION
#------------------------------------------------------------------------------#

_atom_sites_solution_primary                heavy
_atom_sites_solution_secondary              difmap
_atom_sites_solution_hydrogens              geom

#------------------------------------------------------------------------------#
#                         REFINEMENT INFORMATION                               #
#------------------------------------------------------------------------------#

_refine_special_details
;This structure is an extended chain consisting of alternating
 Tm and Ni complexes.  Within a chain, each Tm atom is bonded to five
 DMF molecules, one water molecule, and two Ni(CN)4 and each
 Ni(CN)4 complex is bonded to two Tm complexes.

 There is a Ni(CN)4 complex off by itself (with the Ni on an inversion
 center), and this complex is involved in hydrogen bonding with the water
 molecule bonded to Tm.  Hence this Ni(CN)4 complex, by virtue of its
 inversion center, serves as a link between two extended chains through
 this hydrogen bonding.  The other hydrogen atom of this water molecule is
 involved in a hydrogen bond with a nitrile group in a neighboring extended
 chain.

 One of the DMF ligands (containing atom O(5)) is disordered over two sites,
 with atoms O(5) and N(11) common to both sites.

 The hydrogen atoms bonded to the C=O atoms of the DMF ligands and the
 hydrogen atoms of the water molecule are located on electron density maps,
 and added to the model fixed at these positions.
 For most of the methyl groups, the hydrogen atoms were added at calculated
 positions using a riding model with U(H) = 1.5 *Ueq(bonded carbon atom).
 The torsion angle, which defines the orientation about the C-N bond,
 was allowed to refine.  The methyl groups of the disordered DMF ligand were
 treated in a similar manner, but their torsion angles were not allowed to
 refine.
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  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₩s(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
          'w=1/[₩s^2^(Fo^2^)+(0.0310P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
_refine_ls_hydrogen_treatment                 constr
_refine_ls_extinction_method                  none
_refine_ls_number_reflns                      7383
_refine_ls_number_parameters                  403
_refine_ls_number_restraints                  0
_refine_ls_R_factor_all                       0.0403
_refine_ls_R_factor_gt                        0.027
_refine_ls_wR_factor_ref                      0.0624
_refine_ls_wR_factor_gt                       0.0586
_refine_ls_goodness_of_fit_ref                1.024
_refine_ls_restrained_S_all                   1.024
_refine_ls_shift/su_max                       0.003
_refine_ls_shift/su_mean                      0
_refine_diff_density_max                      1.512
_refine_diff_density_min                      -1.129
_refine_diff_density_rms                      0.1

#---------------------------------------------------------------------------#
#      ATOMIC TYPES, COORDINATES AND THERMAL PARAMETERS      #
#---------------------------------------------------------------------------#

loop_
     _atom_type_symbol
     _atom_type_description
     _atom_type_scat_dispersion_real
     _atom_type_scat_dispersion_imag
```

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    _atom_type_scat_source
C C 0.0033 0.0016 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
H H 0 0 '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'
Ni Ni 0.3393 1.1124 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
O O 0.0106 0.006 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Tm Tm -0.3139 5.2483 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

loop_
    _atom_site_label
    _atom_site_type_symbol
    _atom_site_fract_x
    _atom_site_fract_y
    _atom_site_fract_z
    _atom_site_U_iso_or_equiv
    _atom_site_adp_type
    _atom_site_occupancy
    _atom_site_symmetry_multiplicity
    _atom_site_calc_flag
    _atom_site_refinement_flags
    _atom_site_disorder_assembly
    _atom_site_disorder_group
Tm Tm 0.832138(17) 0.630181(12) 0.713129(6) 0.02423(5) Uani 1 1 d . . .
Ni1 Ni 0.5 1 1 0.02808(13) Uani 1 2 d S . .
Ni2 Ni 0.35512(5) 1.13693(3) 0.668690(16) 0.01994(9) Uani 1 1 d . . .
O1 O 0.5919(3) 0.6746(2) 0.79169(9) 0.0300(5) Uani 1 1 d . . .
O2 O 0.6667(3) 0.4704(2) 0.70601(10) 0.0325(5) Uani 1 1 d . . .
O3 O 0.9320(3) 0.4757(2) 0.79078(9) 0.0289(5) Uani 1 1 d . . .
O4 O 0.7961(3) 0.6384(2) 0.61199(9) 0.0373(6) Uani 1 1 d . . .
O5 O 1.0622(3) 0.7212(2) 0.66703(11) 0.0483(7) Uani 1 1 d . . .
O6 O 0.9056(3) 0.7796(2) 0.77684(9) 0.0322(5) Uani 1 1 d . . .
H1O6 H 0.9902 0.8209 0.7695 0.039 Uiso 1 1 d . . .
H2O6 H 0.865 0.8023 0.8106 0.039 Uiso 1 1 d . . .
N1 N 0.2818(4) 1.1237(3) 1.11440(13) 0.0440(7) Uani 1 1 d . . .
N2 N 0.5986(6) 0.7057(4) 1.05461(16) 0.0816(13) Uani 1 1 d . . .
N3 N 0.6461(4) 1.2331(3) 0.58535(14) 0.0470(8) Uani 1 1 d . . .
N4 N 0.6303(4) 0.8574(3) 0.68793(11) 0.0309(6) Uani 1 1 d . . .
N5 N 0.0771(4) 1.0064(3) 0.74169(13) 0.0406(7) Uani 1 1 d . . .
N6 N 0.0709(3) 1.4208(2) 0.67144(11) 0.0278(6) Uani 1 1 d . . .
```

N7 N 0.3558(3) 0.7546(3) 0.86375(11) 0.0310(6) Uani 1 1 d . . .
N8 N 0.5577(3) 0.3114(3) 0.76716(12) 0.0310(6) Uani 1 1 d . . .
N9 N 0.9184(4) 0.3961(3) 0.88664(11) 0.0352(7) Uani 1 1 d . . .
N10 N 0.7755(3) 0.5815(3) 0.51901(11) 0.0304(6) Uani 1 1 d . . .
N11 N 1.1853(4) 0.8530(3) 0.59696(11) 0.0309(6) Uani 1 1 d . . .
C1 C 0.3640(5) 1.0754(3) 1.07089(15) 0.0340(8) Uani 1 1 d . . .
C2 C 0.5593(6) 0.8170(4) 1.03438(15) 0.0450(9) Uani 1 1 d . . .
C3 C 0.5350(4) 1.2010(3) 0.61833(14) 0.0287(7) Uani 1 1 d . . .
C4 C 0.5271(4) 0.9643(3) 0.67886(13) 0.0248(7) Uani 1 1 d . A .
C5 C 0.1795(4) 1.0601(3) 0.71517(14) 0.0276(7) Uani 1 1 d . . .
C6 C 0.1795(4) 1.3137(3) 0.66661(12) 0.0234(6) Uani 1 1 d . . .
C7 C 0.4540(4) 0.7661(3) 0.81230(14) 0.0280(7) Uani 1 1 d . . .
HC7 H 0.4128 0.8551 0.7862 0.034 Uiso 1 1 d . . .
C8 C 0.4079(5) 0.6329(4) 0.90389(16) 0.0464(9) Uani 1 1 d . . .
H8A H 0.5071 0.5586 0.8833 0.07 Uiso 1 1 calc R . .
H8B H 0.3012 0.5956 0.9169 0.07 Uiso 1 1 calc R . .
H8C H 0.4506 0.6625 0.9385 0.07 Uiso 1 1 calc R . .
C9 C 0.1996(5) 0.8709(4) 0.88648(17) 0.0577(11) Uani 1 1 d . . .
H9A H 0.227 0.9075 0.9223 0.087 Uiso 1 1 calc R . .
H9B H 0.0897 0.8358 0.8961 0.087 Uiso 1 1 calc R . .
H9C H 0.1774 0.9474 0.8563 0.087 Uiso 1 1 calc R . .
C10 C 0.6866(5) 0.3491(4) 0.73139(15) 0.0353(8) Uani 1 1 d . . .
HC10 H 0.8023 0.2685 0.7211 0.042 Uiso 1 1 d . . .
C11 C 0.3769(5) 0.4070(4) 0.78305(18) 0.0522(10) Uani 1 1 d . . .
H11A H 0.3707 0.4997 0.763 0.078 Uiso 1 1 calc R . .
H11B H 0.3546 0.4191 0.8261 0.078 Uiso 1 1 calc R . .
H11C H 0.2832 0.367 0.7708 0.078 Uiso 1 1 calc R . .
C12 C 0.5914(5) 0.1684(4) 0.79489(17) 0.0489(10) Uani 1 1 d . . .
H12A H 0.7143 0.1113 0.7793 0.073 Uiso 1 1 calc R . .
H12B H 0.4986 0.1229 0.786 0.073 Uiso 1 1 calc R . .
H12C H 0.5837 0.1757 0.8379 0.073 Uiso 1 1 calc R . .
C13 C 0.9019(4) 0.4961(3) 0.84457(15) 0.0328(8) Uani 1 1 d . . .
HC13 H 0.8634 0.586 0.8617 0.039 Uiso 1 1 d . . .
C14 C 0.9796(6) 0.2464(4) 0.87144(18) 0.0568(11) Uani 1 1 d . . .
H14A H 1.0076 0.2385 0.8283 0.085 Uiso 1 1 calc R . .
H14B H 0.882 0.2002 0.886 0.085 Uiso 1 1 calc R . .
H14C H 1.0904 0.2 0.8899 0.085 Uiso 1 1 calc R . .
C15 C 0.8734(6) 0.4281(5) 0.94905(16) 0.0649(13) Uani 1 1 d . . .
H15A H 0.8322 0.5314 0.9536 0.097 Uiso 1 1 calc R . .

H15B H 0.9827 0.3888 0.9692 0.097 Uiso 1 1 calc R . .
H15C H 0.7748 0.3855 0.9666 0.097 Uiso 1 1 calc R . .
C16 C 0.7717(4) 0.5544(3) 0.57715(14) 0.0319(8) Uani 1 1 d . . .
HC16 H 0.7314 0.4622 0.5895 0.038 Uiso 1 1 d . . .
C17 C 0.7434(5) 0.4838(4) 0.47931(15) 0.0369(8) Uani 1 1 d . . .
H17A H 0.7163 0.402 0.5022 0.055 Uiso 1 1 calc R . .
H17B H 0.8534 0.451 0.4505 0.055 Uiso 1 1 calc R . .
H17C H 0.6387 0.5321 0.4583 0.055 Uiso 1 1 calc R . .
C18 C 0.8080(5) 0.7143(3) 0.49304(14) 0.0372(8) Uani 1 1 d . . .
H18A H 0.8176 0.7746 0.5246 0.056 Uiso 1 1 calc R . .
H18B H 0.7054 0.7642 0.4714 0.056 Uiso 1 1 calc R . .
H18C H 0.9229 0.6933 0.4657 0.056 Uiso 1 1 calc R . .
C19A C 1.2049(7) 0.7471(5) 0.6377(2) 0.0267(12) Uani 0.641(5) 1 d P A 1
H19A H 1.3169 0.6788 0.6377 0.032 Uiso 0.641(5) 1 d P A 1
C20A C 1.0171(7) 0.9562(6) 0.5924(3) 0.0432(16) Uani 0.641(5) 1 d P A 1
H20A H 1.0342 1.0233 0.5596 0.065 Uiso 0.641(5) 1 calc PR A 1
H20B H 0.976 1.0078 0.6295 0.065 Uiso 0.641(5) 1 calc PR A 1
H20C H 0.9242 0.9093 0.5847 0.065 Uiso 0.641(5) 1 calc PR A 1
C21A C 1.3523(7) 0.8750(6) 0.5603(2) 0.0414(15) Uani 0.641(5) 1 d P A 1
H21A H 1.3171 0.9556 0.5326 0.062 Uiso 0.641(5) 1 calc PR A 1
H21B H 1.4155 0.789 0.538 0.062 Uiso 0.641(5) 1 calc PR A 1
H21C H 1.4348 0.895 0.586 0.062 Uiso 0.641(5) 1 calc PR A 1
C19B C 1.0287(12) 0.8286(9) 0.6272(4) 0.032(2) Uani 0.359(5) 1 d P A 2
H19B H 0.9229 0.9201 0.6197 0.038 Uiso 0.359(5) 1 d P A 2
C20B C 1.1632(15) 0.9837(11) 0.5609(5) 0.051(3) Uani 0.359(5) 1 d P A 2
H20D H 1.2842 0.9914 0.5421 0.077 Uiso 0.359(5) 1 calc PR A 2
H20E H 1.1051 1.0657 0.5862 0.077 Uiso 0.359(5) 1 calc PR A 2
H20F H 1.0855 0.9821 0.5303 0.077 Uiso 0.359(5) 1 calc PR A 2
C21B C 1.3594(14) 0.7580(12) 0.5948(5) 0.050(3) Uani 0.359(5) 1 d P A 2
H21D H 1.45 0.8004 0.5712 0.075 Uiso 0.359(5) 1 calc PR A 2
H21E H 1.359 0.6698 0.5766 0.075 Uiso 0.359(5) 1 calc PR A 2
H21F H 1.3919 0.7368 0.6352 0.075 Uiso 0.359(5) 1 calc PR A 2

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_atom_site_aniso_U_33
_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

Tm 0.02818(8) 0.01641(7) 0.01800(8) 0.00294(5) 0.00503(5) 0.00635(5)
Ni1 0.0348(3) 0.0262(3) 0.0201(3) -0.0028(2) 0.0033(2) -0.0057(2)
Ni2 0.02058(19) 0.01254(17) 0.0216(2) 0.00090(15) 0.00014(15) 0.00243(14)
O1 0.0318(12) 0.0250(11) 0.0242(12) 0.0003(9) 0.0090(9) 0.0014(9)
O2 0.0371(13) 0.0293(12) 0.0268(12) 0.0012(10) -0.0006(10) -0.0041(10)
O3 0.0330(12) 0.0265(11) 0.0179(12) 0.0026(9) 0.0013(9) 0.0047(9)
O4 0.0538(15) 0.0241(11) 0.0198(12) 0.0028(10) 0.0002(11) 0.0105(10)
O5 0.0535(16) 0.0312(13) 0.0479(16) -0.0024(11) 0.0309(13) -0.0092(11)
O6 0.0394(13) 0.0331(12) 0.0230(12) -0.0011(10) 0.0088(10) -0.0139(10)
N1 0.0521(19) 0.0474(18) 0.0298(17) -0.0124(14) 0.0104(15) -0.0143(15)
N2 0.149(4) 0.039(2) 0.040(2) 0.0025(17) 0.005(2) -0.006(2)
N3 0.0412(18) 0.0493(19) 0.047(2) 0.0146(15) 0.0016(15) -0.0141(15)
N4 0.0354(15) 0.0217(13) 0.0236(15) 0.0024(11) 0.0063(12) 0.0068(12)
N5 0.0356(16) 0.0375(16) 0.0454(19) 0.0089(14) 0.0057(14) -0.0120(14)
N6 0.0299(14) 0.0202(13) 0.0229(14) 0.0021(11) 0.0042(11) 0.0063(11)
N7 0.0268(14) 0.0346(15) 0.0248(15) -0.0021(12) 0.0055(12) -0.0011(12)
N8 0.0287(14) 0.0365(15) 0.0288(15) -0.0024(12) -0.0002(12) -0.0121(12)
N9 0.0483(17) 0.0320(15) 0.0206(15) 0.0031(12) -0.0048(13) -0.0034(13)
N10 0.0315(14) 0.0302(14) 0.0211(14) 0.0042(11) 0.0015(11) 0.0023(12)
N11 0.0344(15) 0.0300(14) 0.0261(15) -0.0007(12) 0.0080(12) -0.0109(12)
C1 0.0401(19) 0.0298(17) 0.0301(19) -0.0009(15) 0.0005(16) -0.0087(15)
C2 0.072(3) 0.034(2) 0.0228(19) -0.0055(15) 0.0058(18) -0.0081(19)
C3 0.0290(17) 0.0219(15) 0.0315(18) 0.0057(13) -0.0073(15) 0.0000(13)
C4 0.0277(16) 0.0228(15) 0.0187(16) 0.0002(12) 0.0046(13) -0.0022(13)
C5 0.0285(17) 0.0193(15) 0.0294(18) -0.0004(13) -0.0040(14) 0.0031(13)
C6 0.0289(16) 0.0223(15) 0.0148(15) 0.0023(12) 0.0026(12) -0.0030(13)
C7 0.0249(16) 0.0251(16) 0.0269(18) 0.0000(13) 0.0015(14) 0.0026(13)
C8 0.055(2) 0.047(2) 0.031(2) 0.0045(17) 0.0116(17) -0.0136(19)
C9 0.044(2) 0.068(3) 0.039(2) -0.008(2) 0.0127(19) 0.015(2)
C10 0.0358(19) 0.040(2) 0.0278(19) -0.0064(15) -0.0012(15) -0.0068(15)
C11 0.032(2) 0.071(3) 0.052(3) -0.015(2) 0.0042(18) -0.0129(19)
C12 0.060(3) 0.045(2) 0.045(2) 0.0027(18) -0.003(2) -0.022(2)
C13 0.0310(18) 0.0284(17) 0.0295(19) -0.0018(14) 0.0002(15) 0.0061(14)
C14 0.093(3) 0.034(2) 0.044(2) 0.0124(18) -0.016(2) -0.017(2)
C15 0.096(4) 0.065(3) 0.022(2) -0.0007(19) -0.005(2) -0.003(3)
C16 0.0306(17) 0.0273(17) 0.0237(18) 0.0050(14) 0.0050(14) 0.0106(14)
C17 0.0380(19) 0.044(2) 0.0271(19) 0.0025(16) -0.0035(15) -0.0098(16)

C18 0.042(2) 0.0357(18) 0.0248(18) 0.0089(15) -0.0020(15) 0.0015(15)
C19A 0.032(3) 0.024(2) 0.022(3) -0.006(2) 0.007(2) -0.006(2)
C20A 0.037(3) 0.050(4) 0.039(3) 0.014(3) -0.004(3) -0.009(3)
C21A 0.036(3) 0.046(3) 0.041(3) 0.007(3) 0.006(3) -0.014(3)
C19B 0.037(5) 0.030(5) 0.029(5) -0.002(4) 0.005(4) -0.013(4)
C20B 0.062(8) 0.056(7) 0.047(7) 0.026(5) -0.015(6) -0.035(6)
C21B 0.044(6) 0.054(7) 0.050(7) -0.025(6) 0.002(5) -0.009(5)

#---#
MOLECULAR GEOMETRY
#---#

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;
All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell s.u.'s are taken
into account individually in the estimation of s.u.'s in distances, angles
and torsion angles; correlations between s.u.'s in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.
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Tm O3 2.3240(19) . ?
Tm O1 2.3408(19) . ?
Tm O6 2.342(2) . ?
Tm O4 2.352(2) . ?
Tm N4 2.418(2) . ?
Tm N6 2.474(2) 1_645 ?
Ni1 C1 1.867(3) . ?
Ni1 C1 1.867(3) 2_677 ?
Ni1 C2 1.870(4) . ?

Ni1 C2 1.870(4) 2_677 ?
Ni2 C4 1.855(3) . ?
Ni2 C3 1.875(3) . ?
Ni2 C6 1.876(3) . ?
Ni2 C5 1.876(3) . ?
O1 C7 1.235(3) . ?
O2 C10 1.266(4) . ?
O3 C13 1.233(4) . ?
O4 C16 1.242(4) . ?
O5 C19A 1.271(5) . ?
O5 C19B 1.337(9) . ?
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O6 H2O6 0.808 . ?
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N2 C2 1.133(4) . ?
N3 C3 1.146(4) . ?
N4 C4 1.150(3) . ?
N5 C5 1.140(4) . ?
N6 C6 1.143(3) . ?
N6 Tm 2.474(2) 1_465 ?
N7 C7 1.314(4) . ?
N7 C8 1.454(4) . ?
N7 C9 1.462(4) . ?
N8 C10 1.302(4) . ?
N8 C11 1.449(4) . ?
N8 C12 1.468(4) . ?
N9 C13 1.318(4) . ?
N9 C15 1.448(4) . ?
N9 C14 1.456(4) . ?
N10 C16 1.332(4) . ?
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N10 C18 1.460(4) . ?
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N11 C19B 1.362(9) . ?
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N11 C20B 1.457(9) . ?
N11 C21A 1.479(5) . ?
C7 HC7 1.023 . ?

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C8 H8B 0.98 . ?
C8 H8C 0.98 . ?
C9 H9A 0.98 . ?
C9 H9B 0.98 . ?
C9 H9C 0.98 . ?
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C11 H11B 0.98 . ?
C11 H11C 0.98 . ?
C12 H12A 0.98 . ?
C12 H12B 0.98 . ?
C12 H12C 0.98 . ?
C13 HC13 0.938 . ?
C14 H14A 0.98 . ?
C14 H14B 0.98 . ?
C14 H14C 0.98 . ?
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C15 H15C 0.98 . ?
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C21B H21F 0.98 . ?

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O5 Tm O3 110.88(9) . . ?
O2 Tm O3 81.13(7) . . ?
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O3 Tm O1 73.13(7) . . ?
O5 Tm O6 69.78(8) . . ?
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O3 Tm O6 77.48(7) . . ?
O1 Tm O6 73.91(7) . . ?
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O2 Tm O4 75.76(8) . . ?
O3 Tm O4 142.95(7) . . ?
O1 Tm O4 125.41(8) . . ?
O6 Tm O4 135.31(7) . . ?
O5 Tm N4 84.99(9) . . ?
O2 Tm N4 104.42(8) . . ?
O3 Tm N4 144.66(8) . . ?
O1 Tm N4 75.13(8) . . ?
O6 Tm N4 79.15(8) . . ?
O4 Tm N4 70.28(8) . . ?
O5 Tm N6 75.54(8) . 1_645 ?
O2 Tm N6 77.45(8) . 1_645 ?
O3 Tm N6 71.43(7) . 1_645 ?
O1 Tm N6 136.92(7) . 1_645 ?
O6 Tm N6 120.19(8) . 1_645 ?
O4 Tm N6 75.51(8) . 1_645 ?
N4 Tm N6 143.88(8) . 1_645 ?
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C1 Ni1 C2 88.67(14) 2_677 . ?
C1 Ni1 C2 88.67(14) . 2_677 ?
C1 Ni1 C2 91.33(14) 2_677 2_677 ?
C2 Ni1 C2 180.0(2) . 2_677 ?
C4 Ni2 C3 89.54(12) . . ?
C4 Ni2 C6 174.26(13) . . ?
C3 Ni2 C6 93.22(12) . . ?
C4 Ni2 C5 87.76(12) . . ?
C3 Ni2 C5 175.40(13) . . ?
C6 Ni2 C5 89.80(12) . . ?
C7 O1 Tm 142.9(2) . . ?
C10 O2 Tm 127.4(2) . . ?
C13 O3 Tm 129.14(19) . . ?
C16 O4 Tm 135.3(2) . . ?
C19A O5 C19B 65.1(4) . . ?
C19A O5 Tm 168.6(3) . . ?
C19B O5 Tm 121.2(4) . . ?
Tm O6 H1O6 126.1 . . ?
Tm O6 H2O6 132.0 . . ?
H1O6 O6 H2O6 101.8 . . ?
C4 N4 Tm 175.9(2) . . ?
C6 N6 Tm 163.0(2) . 1_465 ?
C7 N7 C8 122.1(3) . . ?
C7 N7 C9 121.4(3) . . ?
C8 N7 C9 116.2(3) . . ?
C10 N8 C11 122.8(3) . . ?
C10 N8 C12 120.9(3) . . ?
C11 N8 C12 116.3(3) . . ?
C13 N9 C15 122.6(3) . . ?
C13 N9 C14 120.4(3) . . ?
C15 N9 C14 117.0(3) . . ?
C16 N10 C17 121.9(3) . . ?
C16 N10 C18 120.9(3) . . ?
C17 N10 C18 117.2(3) . . ?
C19A N11 C19B 62.7(4) . . ?
C19A N11 C21B 63.8(5) . . ?
C19B N11 C21B 124.7(7) . . ?

C19A N11 C20A 123.2(3) . . ?
C19B N11 C20A 61.8(4) . . ?
C21B N11 C20A 172.9(6) . . ?
C19A N11 C20B 170.2(6) . . ?
C19B N11 C20B 116.4(6) . . ?
C21B N11 C20B 118.7(7) . . ?
C20A N11 C20B 54.7(5) . . ?
C19A N11 C21A 118.5(4) . . ?
C19B N11 C21A 175.8(5) . . ?
C21B N11 C21A 55.5(5) . . ?
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C20B N11 C21A 63.1(5) . . ?
N1 C1 Ni1 178.8(3) . . ?
N2 C2 Ni1 178.6(4) . . ?
N3 C3 Ni2 175.7(3) . . ?
N4 C4 Ni2 176.8(3) . . ?
N5 C5 Ni2 176.1(3) . . ?
N6 C6 Ni2 173.1(3) . . ?
O1 C7 N7 124.7(3) . . ?
O1 C7 HC7 116.4 . . ?
N7 C7 HC7 118.9 . . ?
N7 C8 H8A 109.5 . . ?
N7 C8 H8B 109.5 . . ?
H8A C8 H8B 109.5 . . ?
N7 C8 H8C 109.5 . . ?
H8A C8 H8C 109.5 . . ?
H8B C8 H8C 109.5 . . ?
N7 C9 H9A 109.5 . . ?
N7 C9 H9B 109.5 . . ?
H9A C9 H9B 109.5 . . ?
N7 C9 H9C 109.5 . . ?
H9A C9 H9C 109.5 . . ?
H9B C9 H9C 109.5 . . ?
O2 C10 N8 124.1(3) . . ?
O2 C10 HC10 121.3 . . ?
N8 C10 HC10 114.2 . . ?
N8 C11 H11A 109.5 . . ?
N8 C11 H11B 109.5 . . ?
H11A C11 H11B 109.5 . . ?

N8 C11 H11C 109.5 . . ?
H11A C11 H11C 109.5 . . ?
H11B C11 H11C 109.5 . . ?
N8 C12 H12A 109.5 . . ?
N8 C12 H12B 109.5 . . ?
H12A C12 H12B 109.5 . . ?
N8 C12 H12C 109.5 . . ?
H12A C12 H12C 109.5 . . ?
H12B C12 H12C 109.5 . . ?
O3 C13 N9 125.7(3) . . ?
O3 C13 HC13 124.7 . . ?
N9 C13 HC13 109.7 . . ?
N9 C14 H14A 109.5 . . ?
N9 C14 H14B 109.5 . . ?
H14A C14 H14B 109.5 . . ?
N9 C14 H14C 109.5 . . ?
H14A C14 H14C 109.5 . . ?
H14B C14 H14C 109.5 . . ?
N9 C15 H15A 109.5 . . ?
N9 C15 H15B 109.5 . . ?
H15A C15 H15B 109.5 . . ?
N9 C15 H15C 109.5 . . ?
H15A C15 H15C 109.5 . . ?
H15B C15 H15C 109.5 . . ?
O4 C16 N10 122.7(3) . . ?
O4 C16 HC16 125.2 . . ?
N10 C16 HC16 111.7 . . ?
N10 C17 H17A 109.5 . . ?
N10 C17 H17B 109.5 . . ?
H17A C17 H17B 109.5 . . ?
N10 C17 H17C 109.5 . . ?
H17A C17 H17C 109.5 . . ?
H17B C17 H17C 109.5 . . ?
N10 C18 H18A 109.5 . . ?
N10 C18 H18B 109.5 . . ?
H18A C18 H18B 109.5 . . ?
N10 C18 H18C 109.5 . . ?
H18A C18 H18C 109.5 . . ?
H18B C18 H18C 109.5 . . ?

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O5 C19A H19A 118.7 . . ?
N11 C19A H19A 120.1 . . ?
N11 C20A H20A 109.5 . . ?
N11 C20A H20B 109.5 . . ?
H20A C20A H20B 109.5 . . ?
N11 C20A H20C 109.5 . . ?
H20A C20A H20C 109.5 . . ?
H20B C20A H20C 109.5 . . ?
N11 C21A H21A 109.5 . . ?
N11 C21A H21B 109.5 . . ?
H21A C21A H21B 109.5 . . ?
N11 C21A H21C 109.5 . . ?
H21A C21A H21C 109.5 . . ?
H21B C21A H21C 109.5 . . ?
O5 C19B N11 112.9(7) . . ?
O5 C19B H19B 138.5(8) . . ?
N11 C19B H19B 105.5(7) . . ?
N11 C20B H20D 109.5 . . ?
N11 C20B H20E 109.5 . . ?
H20D C20B H20E 109.5 . . ?
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N11 C21B H21D 109.5 . . ?
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O6 Tm O1 C7 75.3(3) . . . . ?
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N4 Tm O1 C7 -7.5(3) . . . . ?
N6 Tm O1 C7 -167.5(3) 1_645 . . . ?
O5 Tm O2 C10 101.6(3) . . . . ?
O3 Tm O2 C10 -12.2(3) . . . . ?
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O4 Tm O2 C10 138.6(3) . . . . ?
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N6 Tm O2 C10 60.6(3) 1_645 . . . ?
O5 Tm O3 C13 102.8(3) . . . . ?
O2 Tm O3 C13 -111.7(3) . . . . ?
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O5 Tm O4 C16 132.9(3) . . . . ?
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N4 Tm O4 C16 -137.3(3) . . . . ?
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O2 Tm O5 C19A -34.8(16) . . . . ?
O3 Tm O5 C19A 69.8(15) . . . . ?
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O2 Tm O5 C19B 85.9(5) . . . . ?
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O6 Tm O5 C19B -102.3(5) . . . . ?
O4 Tm O5 C19B 48.8(5) . . . . ?
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N6 Tm O5 C19B 127.2(5) 1_645 . . . ?
O5 Tm N4 C4 -152(4) . . . . ?
O2 Tm N4 C4 63(4) . . . . ?
O3 Tm N4 C4 -32(4) . . . . ?
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O6 Tm N4 C4 -82(4) . . . . ?
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N6 Tm N4 C4 151(4) 1_645 . . . ?
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C2 Ni1 C1 N1 134(17) . . . . ?
C2 Ni1 C1 N1 -46(17) 2_677 . . . ?
C1 Ni1 C2 N2 -140(17) . . . . ?
C1 Ni1 C2 N2 40(17) 2_677 . . . ?
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C6 Ni2 C5 N5 143(4) . . . . ?
Tm N6 C6 Ni2 -8(3) 1_465 . . . ?
C4 Ni2 C6 N6 -7(3) . . . . ?
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C5 Ni2 C6 N6 58(2) . . . . ?
Tm O1 C7 N7 -173.5(2) . . . . ?
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C9 N7 C7 O1 176.8(3) . . . . ?
Tm O2 C10 N8 121.1(3) . . . . ?
C11 N8 C10 O2 -1.6(5) . . . . ?
C12 N8 C10 O2 179.9(3) . . . . ?

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C15 N9 C13 O3 -177.4(4) . . . . ?
C14 N9 C13 O3 1.0(5) . . . . ?
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C17 N10 C16 O4 -178.8(3) . . . . ?
C18 N10 C16 O4 -0.4(5) . . . . ?
C19B O5 C19A N11 0.2(5) . . . . ?
Tm O5 C19A N11 126.0(14) . . . . ?
C19B N11 C19A O5 -0.2(5) . . . . ?
C21B N11 C19A O5 -165.8(7) . . . . ?
C20A N11 C19A O5 12.9(6) . . . . ?
C20B N11 C19A O5 87(3) . . . . ?
C21A N11 C19A O5 -175.7(4) . . . . ?
C19A O5 C19B N11 -0.2(5) . . . . ?
Tm O5 C19B N11 -169.4(4) . . . . ?
C19A N11 C19B O5 0.2(5) . . . . ?
C21B N11 C19B O5 16.0(10) . . . . ?
C20A N11 C19B O5 -167.4(8) . . . . ?
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O6 H1O6 N5 0.842 2.124 2.883(3) 149.8 1_655
O6 H2O6 N1 0.808 1.967 2.762(4) 167.9 2_677

# END of CIF
```