Supporting Information

Battlement shaped 1D coordination polymer, based on a bis(*N*-methylimidazol-2-yl)butadiyne ligand

Thomas Waidmann[a], Nico Fritsch[a], Johannes Tucher[a], Marc Rudolf[b], Felix Glaser[a], Dirk M. Guldi[b]* and Nicolai Burzlaff[a]*

Experimental Section

The synthesis of 2-iodo-1-methylimidazole (**4**) and 1-methyl-2-trimethylsilyethynylimidazole (**5**) were performed following literature procedures.[8-10] NMR spectra were recorded on a Bruker DPX 300 AVANCE, δ are given values relative to the residual solvent signal (1H: $CHCl_3$, 7.26 ppm; $^{13}C\{^1H\}$: $CDCl_3$, 77.2 ppm). Elemental analyses were carried out on a Euro EA 3000 (Euro Vector). Infrared spectra were recorded with a EXCALIBUR FTS-3500 FT-IR spectrometer. X-Ray structure analyses were carried out on a Bruker Kappa CCD (SADABS-2008/1 (Bruker, 2008)). All calculations were carried out with the SHELX97 software package.

2-(2-Chloroethenyl)-*N*-methylimidazole-2-yl (3a,b): To a solution of (chloromethyl)triphenylphosphonium chloride (25.0 g, 72.0 mmol) in THF (100 mL) *n*-butyllithium (45.0 mL, 72.0 mmol, 1.60 M in hexanes) was added drop wise at 0 °C, and stirred for 1 h. Subsequently 1-methylimidazole-2-carbaldehyde (**2**) (5.00 g, 45.0 mmol) was added and stirred for 12 hours at room temperature. The yellow-brown product containing a mixture of *E*/*Z*-isomers was purified by column chromatography (silica, 6 × 20 cm, hexane/EtOAc 5/1 and silica 6 × 40 cm, $CHCl_3$/MeOH 12/1) yielding a brown oil. (1.60 g, 11.2 mmol, 25 %). ^{1}H-NMR (300 MHz, $CDCl_3$): (*E*)-2-(2-Chloroethynyl)-*N*-methylimidazole-2-yl: δ = 3.62 (s, 3H, CH_3), 6.66 (d, 1H, $^2J_{HH}$ = 13.2 Hz, Cl-HC=*CH*-), 6.84 (s, 1H, C_{im}), 7.00 (d, 1H, $^2J_{HH}$ = 5.94 Hz, C_{im}), 7.08 (d, 1H, $^2J_{HH}$ =13.2 Hz, Cl-H*C*=CH-) ppm. ^{1}H−NMR (300 MHz, $CDCl_3$): (*Z*)-2-(2-Chloroethynyl)-*N*-methylimidazole-2-yl: δ = 3.62 (s, 3H, CH_3), 6.43 (d, 1H, $^2J_{HH}$ = 8.19 Hz, Cl-HC=*C*H-), 6.53 (d, 1H, $^2J_{HH}$ = 8.31 Hz, C_{im}), 6.89 (s, 1H, C_{im}), 7.20 (s, 1H, Cl-H*C*=CH-) ppm. IR : (KBr) $\tilde{\nu}$ = 3047 (m), 2956 (w), 2857 (w), 1720 (s), 1615 (m), 1485 (m), 1438 (s), 1418 (m), 1363 (w), 1290 (s), 1189 (s), 1123 (m), 1120 (m) cm^{-1}.

2-Ethynyl-*N*-methylimidazole-2-yl (6): Route A - A Schlenk flask was charged with 1-methyl-2-trimethylsilyethynylimidazole (**5**) (490 mg, 2.75 mmol) and MeOH (25 mL). After the addition of KOH (13.4 M, 2 mL) the reaction mixture was stirred 2 h. The mixture was diluted with water (100 mL), extracted with $CHCl_3$ (4 × 50 mL) and dried over Na_2SO_4. The solvent was removed by rotary evaporator and the crude product was purified by column chromatography (silica, 4 × 5 cm, EtOAc/hexane 3/1) to give yellow oil (254 mg, 2.39 mmol, 87 %). Characterization see below. **Route B** - A round bottom flask was charged with 2-(2-chloroethynyl)-*N*-methylimidazol-2-yl (**3a,b**) (400 mg, 2.80 mmol) in dried THF (10 mL), and potassium-*tert*-butoxide (670 mg, 59.0 mmol) in THF (10 mL) was added at 0 °C. The mixture was stirred for 12 hours at room temperature. Afterwards the mixture was poured on ice water and neutralized with a solution of ammonium chloride (20%). The brown mixture was extracted with CH_2Cl_2 (4 × 50 mL), dried over Na_2SO_4. The solvent was removed by rotary evaporator. The product was purified by column chromatography (silica 3 × 30cm, $CHCl_3$/MeOH/Et_3N, 12/1/1) to yield a yellow-brown oil (90.0 mg 0.80 mmol, 28.5%). ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.31 (s, 1H, H*C*≡C-), 3.73 (s, 3H, CH_3), 6.89 (s, 1H, C_{im}) 7.02 (s, 1H, C_{im}) ppm. $^{13}C\{^1H\}$-NMR (300 MHz, $CDCl_3$): δ = 33.7 (CH_3), 72.2 (H*C*≡C-), 73.1 (HC≡*C*-), 128.4 (C_{im}), 129.5 (C_{im}), 130.5 (C_{im}) ppm.

Bis(*N*-methylimidazol-2-yl)butadiyne (bmib) (7): CuCl (0.049 g, 0.500 mmol) in pyridine (3 mL) was reacted with 2-ethynyl-*N*-methylimidazole (**6**) (0.530 g, 5.00 mmol) under oxygen atmosphere for 1.5 h at 45 °C. Pyridine was removed in vacuum, the brown residue was washed with aqueous NH_4Cl solution (10%, 70 mL), and extracted with CH_2Cl_2 (3 × 100 mL). The combined organic phases were washed (sat. NH_4Cl solution), and dried ($MgSO_4$). Solvent was removed and the product was purified by column chromatography (silica, Ø = 3 cm, 30 cm, $CHCl_3$/MeOH/Et_3N, 12/1/1, v/v/v) to yield a yellow-brown powder. Crystals suitable for X-Ray diffraction analysis were obtained by slow evaporation from a solution of **7** in acetone. Yield (0.490 g, 2.33 mmol, 93%). ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.70 (s, 3H, CH_3), 6.89 (s, 1H, C_{im}), 7.03 (s, 1H, C_{im}) ppm. $^{13}C\{^1H\}$-NMR (300 MHz, acetone-d6): δ = 33.9 (CH_3), 73.7 (-C≡*C*-*C*≡C-), 76.5 ((-*C*≡C-C≡*C*-), 124.6 (C_{im}), 131.0 (C_{im}), 132.2 (C_{im}) ppm. $C_{12}H_{10}N_4$ (210.09 g/mol): calculated C: 68.56; H: 4.79; N: 26.65, found C: 68.96; H: 4.43; N: 27.21. ESI/TOF MS (MeOH): m/z (%) = 443.1702 (100) $[2 \times M + Na]^+$. IR: (KBr) $\tilde{\nu}$ = 2153 (w), 1709 (w), 1619 (w), 1509 (s), 1477 (s), 1454 (m), 1445 (m), 1415 (m), 1401 (m), 1388 (m), 1355 (m),

1287 (s), 1192 (m), 1150 (m), 1139 (m), 1083 (w), 1046 (w), 921 (w), 911 (w), 863 (w), 771(w), 754 (s), 697(w), 691(w), 623 (w), 598 (w), 537 (w) cm^{-1}.

$[Zn_5(OAc)_{10}(bmib)_2]_n$ (8): A solution of bmib (50.0 mg, 0.236 mmol) in MeCN was layered with a solution of $Zn(OAc)_2$ in THF (35 mL). By slow evaporation of the solvents, single crystals suitable for X-Ray diffraction analysis were obtained after three days.

Steady-state absorption: Absorption spectra of all samples were recorded with a Lambda 2 UV/Vis-spectrometer from Perkin Elmer (190 to 1100 nm; double-beam-instrument) using a quartz cell and 0.5 nm resolution.

Steady-state emission: The spectra were recorded on a FluoroMax 3 fluorometer built by HORIBA JobinYvon.

Time resolved absorption: Femtosecond transient absorption studies were performed with 258 nm laser pulses (1 kHz, 150 fs pulse width) from an amplified Ti:Sapphire laser system (model CPA 2101, Clark- MXR Inc.) in the TAPPS, transient absorption pump/probe system, Helios from Ultrafast Systems with 1 μJ laser energy. Nanosecond laser flash photolysis experiments were performed with 266 nm laser pulses from a Brilliant CDR Nd:YAG system (4 ns pulse width, Quantel) in a front face excitation geometry with 5 mJ laser energy.

Crystallographic data for **7**. $C_{12}H_{10}N_4$, M = 210.24, monoclinic, $P2_1/c$, a = 7.0586(4), b = 12.6096(4), c = 12.8621(7) Å, β = 92.413(5)°, V = 1053.08(9)Å^3, T = 153(2) K, Z = 4, μ(Mo-Ka) = 0.085 mm^{-1}, 14414 data collected, 2146 independent reflections (R_{int} = 0.0230). Final R_1 [I > 2σ(I)] = 0.0387, wR_2[all data] = 0.0993. CCDC 949213. Crystallographic data for **8**. $C_{22}H_{25}N_4O_{10}Zn_{2.5}$, M = 668.89, triclinic, P-1, a = 8.0568(10), b = 10.3125(10), c = 16.0151(16) Å, α = 98.594(8), β = 94.875(9), γ = 93.583(9)°, V = 1307.1(2)Å^3, T = 153(2) K, Z = 2, μ(Mo-Ka) = 2.344 mm^{-1}, 13144 data collected, 5321 independent reflections (R_{int} = 0.0743). Final R_1 [I > 2σ(I)] = 0.0530, wR_2[all data] = 0.1285. CCDC 949214.

```
#------------------------------------------------------------------------
#SUBMISSION INFORMATION
#
#         For a journal send the form to the address specified by the journal
#
#         For a private communication to the CCDC send the form to the address
#            deposit@ccdc.cam.ac.uk
#
#         For up-to-date information on deposition procedures, check the website
#            http://www.ccdc.cam.ac.uk/

# 1. SUBMISSION DETAILS

_publ_contact_author
;
Prof. Dr. Nicolai Burzlaff
Department of Chemistry and Pharmacy
University of Erlangen-N\"urnberg
Egerlandstrasse 1
D-91058 Erlangen
Germany
;
_publ_contact_author_phone                49(9131)8528976
_publ_contact_author_fax                  49(9131)8527387
_publ_contact_author_email                burzlaff@chemie.uni-erlangen.de
_publ_requested_journal                   'Chem. Commun.'
_publ_requested_coeditor_name             ?

_publ_contact_letter
;
Please consider this CIF submission (2 compounds) for structural data
deposition. We are awaiting a notice about the assigned CSD numbers.
;

#------------------------------------------------------------------------

# 2. TITLE AND AUTHOR LIST
_publ_section_title
;
Battlement shaped 1D coordination polymer, based on a
bis(N-methyl-imidazol-2-yl)butadiyne ligand
;

loop_
_publ_author_name
_publ_author_address
'Waidmann, Thomas'
;
Department of Chemistry and Pharmacy
University of Erlangen-N\"urnberg
Egerlandstrasse 1
D-91058 Erlangen
Germany
;
'Fritsch, Nico'
;
Department of Chemistry and Pharmacy
University of Erlangen-N\"urnberg
Egerlandstrasse 1
D-91058 Erlangen
Germany
;
'Tucher, Johannes'
;
Department of Chemistry and Pharmacy
University of Erlangen-N\"urnberg
Egerlandstrasse 1
D-91058 Erlangen
```

```
Germany
;
'Rudolf, Marc'
;
Department of Chemistry and Pharmacy
University of Erlangen-N\"urnberg
Egerlandstrasse 3
D-91058 Erlangen
Germany
;
'Glaser, Felix'
;
Department of Chemistry and Pharmacy
University of Erlangen-N\"urnberg
Egerlandstrasse 1
D-91058 Erlangen
Germany
;
'Guldi, Dirk'
;
Department of Chemistry and Pharmacy
University of Erlangen-N\"urnberg
Egerlandstrasse 3
D-91058 Erlangen
Germany
;
'Burzlaff, Nicolai'
;
Department of Chemistry and Pharmacy
University of Erlangen-N\"urnberg
Egerlandstrasse 1
D-91058 Erlangen
Germany
;

#------------------------------------------------------------------------

data_compound7

_audit_creation_date                    2013-06-28T11:59:27-00:00
_audit_creation_method                  'WinGX routine CIF_UPDATE'

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

_chemical_name_systematic
;
  ?
;
_chemical_formula_moiety                'C12 H10 N4'
_chemical_formula_sum                   'C12 H10 N4'
_chemical_formula_weight                210.24
_chemical_compound_source               'synthesis as described'

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

_symmetry_cell_setting                  monoclinic
_symmetry_space_group_name_H-M          'P 21/c'
_symmetry_space_group_name_Hall         '-P 2ybc'
_symmetry_Int_Tables_number             14
loop_
    _symmetry_equiv_pos_as_xyz
'x, y, z'
'-x, y+1/2, -z+1/2'
'-x, -y, -z'
```

```
'x, -y-1/2, z-1/2'

_cell_length_a                          7.0586(4)
_cell_length_b                          11.6096(4)
_cell_length_c                          12.8621(7)
_cell_angle_alpha                       90
_cell_angle_beta                        92.413(5)
_cell_angle_gamma                         90
_cell_volume                              1053.08(9)
_cell_formula_units_Z                   4
_cell_measurement_temperature             150(2)
_cell_measurement_reflns_used           89
_cell_measurement_theta_min               6
_cell_measurement_theta_max               20
_cell_measurement_wavelength              0.71073

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

_exptl_crystal_description              block
_exptl_crystal_colour                     yellow
_exptl_crystal_size_max                   0.37
_exptl_crystal_size_mid                   0.184
_exptl_crystal_size_min                   0.158
_exptl_crystal_density_diffrn           1.326
_exptl_crystal_density_method             'not measured'
_exptl_crystal_F_000                      440
_exptl_special_details
;
  ?
;

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

_exptl_absorpt_coefficient_mu           0.085
_exptl_absorpt_correction_type          multi-scan
_exptl_absorpt_process_details          'SADABS, Bruker-AXS, 2002'
_exptl_absorpt_correction_T_min         0.6921
_exptl_absorpt_correction_T_max         0.7454

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

_diffrn_ambient_temperature               150(2)
_diffrn_radiation_wavelength            0.71073
_diffrn_radiation_type                  MoK\a
_diffrn_radiation_monochromator           graphite
_diffrn_radiation_probe                   x-ray
_diffrn_detector_area_resol_mean        9
_diffrn_measurement_device_type             'Bruker-Nonius KappaCCD'
_diffrn_measurement_method
;
  \f- and \w-rotations with 1.60 \% and 192 sec per frame
;
_diffrn_reflns_av_R_equivalents         0.023
_diffrn_reflns_av_unetI/netI            0.014
_diffrn_reflns_number                     14414
_diffrn_reflns_limit_h_min              -8
_diffrn_reflns_limit_h_max              8
_diffrn_reflns_limit_k_min              -14
_diffrn_reflns_limit_k_max              14
_diffrn_reflns_limit_l_min              -16
_diffrn_reflns_limit_l_max              16
_diffrn_reflns_theta_min                  6.25
```

```
_diffrn_reflns_theta_max                26.5
_diffrn_reflns_theta_full               26.5
_diffrn_measured_fraction_theta_full
                                        0.986
_diffrn_measured_fraction_theta_max
                                        0.986
_reflns_number_total                    2146
_reflns_number_gt                       1914
_reflns_threshold_expression            >2sigma(I)

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

_computing_data_collection              'COLLECT (Bruker-Nonius, 2002)'
_computing_cell_refinement              'EVALCCD (Duisenberg, 2003)'
_computing_data_reduction               'EVALCCD (Duisenberg, 2003)'
_computing_structure_solution           'SHELXS-86 (Sheldrick, 1986)'
_computing_structure_refinement         'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics           'Ortep-3 for Windows (Farrugia, 1997)'
_computing_publication_material
                                        'WinGX publication routines (Farrugia, 1999)'

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

_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^)+(0.0456P)^2^+0.4547P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary            direct
_atom_sites_solution_secondary          difmap
_atom_sites_solution_hydrogens          geom
_refine_ls_hydrogen_treatment           constr
_refine_ls_extinction_method            none
_refine_ls_number_reflns                2146
_refine_ls_number_parameters            147
_refine_ls_number_restraints            0
_refine_ls_R_factor_all                 0.0443
_refine_ls_R_factor_gt                  0.0387
_refine_ls_wR_factor_ref                0.0993
_refine_ls_wR_factor_gt                 0.0958
_refine_ls_goodness_of_fit_ref          1.068
_refine_ls_restrained_S_all             1.068
_refine_ls_shift/su_max                 0
_refine_ls_shift/su_mean                0
_refine_diff_density_max                0.285
_refine_diff_density_min                -0.222
_refine_diff_density_rms                0.037

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

loop_
    _atom_type_symbol
```

```
        _atom_type_description
        _atom_type_scat_dispersion_real
        _atom_type_scat_dispersion_imag
        _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'

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
C1 C 0.18554(17) 0.64825(11) 0.53301(10) 0.0218(3) Uani 1 1 d . . .
C2 C 0.22584(17) 0.56073(11) 0.48919(10) 0.0218(3) Uani 1 1 d . . .
C3 C 0.27293(17) 0.46293(11) 0.43701(10) 0.0214(3) Uani 1 1 d . . .
C4 C 0.31627(17) 0.37864(11) 0.38930(9) 0.0217(3) Uani 1 1 d . . .
C11 C 0.13725(17) 0.75229(11) 0.58303(9) 0.0192(3) Uani 1 1 d . . .
C12 C 0.06006(18) 0.87078(11) 0.70561(10) 0.0227(3) Uani 1 1 d . . .
H12 H 0.0304 0.9023 0.7694 0.027 Uiso 1 1 calc R . .
C13 C 0.06586(19) 0.92696(11) 0.61323(10) 0.0243(3) Uani 1 1 d . . .
H13 H 0.0403 1.0049 0.6039 0.029 Uiso 1 1 calc R . .
C14 C 0.1154(2) 0.66564(12) 0.76171(11) 0.0301(3) Uani 1 1 d . . .
H14A H -0.0108 0.6437 0.7786 0.045 Uiso 1 1 calc R . .
H14B H 0.1839 0.6907 0.8237 0.045 Uiso 1 1 calc R . .
H14C H 0.1789 0.6008 0.7326 0.045 Uiso 1 1 calc R . .
C21 C 0.36786(17) 0.28038(11) 0.33160(10) 0.0209(3) Uani 1 1 d . . .
C22 C 0.44779(18) 0.10325(11) 0.29782(10) 0.0256(3) Uani 1 1 d . . .
H22 H 0.4792 0.0257 0.3037 0.031 Uiso 1 1 calc R . .
C23 C 0.4393(2) 0.16719(13) 0.20951(11) 0.0301(3) Uani 1 1 d . . .
H23 H 0.4641 0.139 0.1438 0.036 Uiso 1 1 calc R . .
C24 C 0.3899(2) 0.14708(12) 0.48572(10) 0.0272(3) Uani 1 1 d . . .
H24A H 0.3437 0.0698 0.4926 0.041 Uiso 1 1 calc R . .
H24B H 0.3049 0.1996 0.5177 0.041 Uiso 1 1 calc R . .
H24C H 0.5135 0.1532 0.5192 0.041 Uiso 1 1 calc R . .
N11 N 0.11426(16) 0.85349(9) 0.53599(8) 0.0239(3) Uani 1 1 d . . .
N12 N 0.10631(14) 0.75914(9) 0.68633(8) 0.0198(2) Uani 1 1 d . . .
N21 N 0.38958(17) 0.27852(10) 0.22980(9) 0.0285(3) Uani 1 1 d . . .
N22 N 0.40109(14) 0.17538(9) 0.37607(8) 0.0205(2) Uani 1 1 d . . .

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
C1 0.0222(6) 0.0203(6) 0.0230(6) 0.0007(5) 0.0019(5) 0.0009(5)
C2 0.0236(6) 0.0195(6) 0.0223(6) 0.0012(5) 0.0024(5) 0.0010(5)
C3 0.0231(6) 0.0198(6) 0.0214(6) 0.0009(5) 0.0018(5) 0.0013(5)
C4 0.0224(6) 0.0216(6) 0.0212(6) 0.0012(5) 0.0019(5) 0.0016(5)
C11 0.0202(6) 0.0181(6) 0.0195(6) 0.0002(5) 0.0014(4) 0.0007(4)
C12 0.0278(6) 0.0205(6) 0.0199(6) -0.0041(5) 0.0025(5) 0.0024(5)
C13 0.0318(7) 0.0165(6) 0.0248(7) -0.0006(5) 0.0030(5) 0.0045(5)
C14 0.0401(8) 0.0254(7) 0.0247(7) 0.0099(6) 0.0015(6) 0.0008(6)
C21 0.0214(6) 0.0204(6) 0.0211(6) -0.0013(5) 0.0020(5) 0.0024(5)
C22 0.0256(6) 0.0212(6) 0.0302(7) -0.0071(5) 0.0018(5) 0.0038(5)
C23 0.0351(7) 0.0324(7) 0.0230(7) -0.0082(6) 0.0034(5) 0.0085(6)
```

C24 0.0322(7) 0.0261(7) 0.0236(7) 0.0035(5) 0.0046(5) 0.0017(5)
N11 0.0321(6) 0.0190(5) 0.0208(5) 0.0017(4) 0.0044(4) 0.0046(4)
N12 0.0240(5) 0.0176(5) 0.0178(5) 0.0017(4) 0.0010(4) -0.0005(4)
N21 0.0356(6) 0.0291(6) 0.0211(6) -0.0011(5) 0.0048(5) 0.0082(5)
N22 0.0215(5) 0.0185(5) 0.0216(5) -0.0015(4) 0.0011(4) 0.0008(4)

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

_geom_special_details
;
All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
;
loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
C1 C2 1.2019(18) . ?
C1 C11 1.4169(17) . ?
C2 C3 1.3671(17) . ?
C3 C4 1.2014(18) . ?
C4 C21 1.4168(17) . ?
C11 N11 1.3284(16) . ?
C11 N12 1.3579(16) . ?
C12 C13 1.3576(18) . ?
C12 N12 1.3620(16) . ?
C13 N11 1.3638(16) . ?
C14 N12 1.4550(16) . ?
C21 N21 1.3249(17) . ?
C21 N22 1.3627(16) . ?
C22 C23 1.356(2) . ?
C22 N22 1.3605(16) . ?
C23 N21 1.3673(18) . ?
C24 N22 1.4535(17) . ?

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_3
_geom_angle_publ_flag
C2 C1 C11 179.03(13) . . ?
C1 C2 C3 178.43(14) . . ?
C4 C3 C2 178.38(14) . . ?
C3 C4 C21 179.06(13) . . ?
N11 C11 N12 111.86(11) . . ?
N11 C11 C1 125.05(11) . . ?
N12 C11 C1 123.08(11) . . ?
C13 C12 N12 106.35(11) . . ?
C12 C13 N11 110.78(11) . . ?
N21 C21 N22 112.04(11) . . ?
N21 C21 C4 125.02(12) . . ?
N22 C21 C4 122.95(11) . . ?
C23 C22 N22 106.21(11) . . ?
C22 C23 N21 111.11(12) . . ?
C11 N11 C13 104.53(10) . . ?
C11 N12 C12 106.47(10) . . ?

```
C11 N12 C14 127.15(11) . . ?
C12 N12 C14 126.36(11) . . ?
C21 N21 C23 104.21(11) . . ?
C22 N22 C21 106.43(11) . . ?
C22 N22 C24 127.12(11) . . ?
C21 N22 C24 126.45(11) . . ?

#------------------------------------------------------------------------

data_compound8

_audit_creation_date                    2013-06-28T11:26:43-00:00
_audit_creation_method                  'WinGX routine CIF_UPDATE'

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

_chemical_name_systematic
;
  ?
;
_chemical_formula_moiety                'C44 H50 N8 O20 Zn5'
_chemical_formula_sum                   'C44 H50 N8 O20 Zn5'
_chemical_formula_weight                1337.87
_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                          8.0568(10)
_cell_length_b                          10.3125(10)
_cell_length_c                          16.0151(16)
_cell_angle_alpha                       98.594(8)
_cell_angle_beta                        94.875(9)
_cell_angle_gamma                       93.583(9)
_cell_volume                            1307.1(2)
_cell_formula_units_Z                   1
_cell_measurement_temperature           150(2)
_cell_measurement_reflns_used           51
_cell_measurement_theta_min             6
_cell_measurement_theta_max             20
_cell_measurement_wavelength            0.71073

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

_exptl_crystal_description              block
_exptl_crystal_colour                   brown
_exptl_crystal_size_max                 0.15
_exptl_crystal_size_mid                 0.103
_exptl_crystal_size_min                 0.056
_exptl_crystal_density_diffrn           1.700
_exptl_crystal_density_method           'not measured'
_exptl_crystal_F_000                    680
_exptl_special_details
;
```

```
 ?
;

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

_exptl_absorpt_coefficient_mu          2.344
_exptl_absorpt_correction_type         multi-scan
_exptl_absorpt_process_details         'SADABS, Bruker-AXS, 2002'
_exptl_absorpt_correction_T_min        0.5815
_exptl_absorpt_correction_T_max        0.7454

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

_diffrn_ambient_temperature            150(2)
_diffrn_radiation_wavelength           0.71073
_diffrn_radiation_type                 MoK\a
_diffrn_radiation_monochromator        graphite
_diffrn_radiation_probe                x-ray
_diffrn_detector_area_resol_mean       9
_diffrn_measurement_device_type        'Bruker-Nonius KappaCCD'
_diffrn_measurement_method
;
 \f- and \w-rotations with 1.60 \% and 192 sec per frame
;
_diffrn_reflns_av_R_equivalents        0.0743
_diffrn_reflns_av_unetI/netI           0.1017
_diffrn_reflns_number                  13144
_diffrn_reflns_limit_h_min             -10
_diffrn_reflns_limit_h_max             10
_diffrn_reflns_limit_k_min             -12
_diffrn_reflns_limit_k_max             11
_diffrn_reflns_limit_l_min             -20
_diffrn_reflns_limit_l_max             19
_diffrn_reflns_theta_min               6.24
_diffrn_reflns_theta_max               26.5
_diffrn_reflns_theta_full              26.5
_diffrn_measured_fraction_theta_full
                                       0.985
_diffrn_measured_fraction_theta_max
                                       0.985
_reflns_number_total                   5321
_reflns_number_gt                      3632
_reflns_threshold_expression           >2sigma(I)

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

_computing_data_collection             'COLLECT (Bruker-Nonius, 2002)'
_computing_cell_refinement             'EVALCCD (Duisenberg, 2003)'
_computing_data_reduction              'EVALCCD (Duisenberg, 2003)'
_computing_structure_solution          'SHELXS-86 (Sheldrick, 1986)'
_computing_structure_refinement        'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics          'Ortep-3 for Windows (Farrugia, 1997)'
_computing_publication_material
                                       'WinGX publication routines (Farrugia, 1999)'

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

_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^)+(0.0500P)^2^+0.3497P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary                  direct
_atom_sites_solution_secondary                difmap
_atom_sites_solution_hydrogens                geom
_refine_ls_hydrogen_treatment                 constr
_refine_ls_extinction_method                  none
_refine_ls_number_reflns                      5321
_refine_ls_number_parameters                  356
_refine_ls_number_restraints                  0
_refine_ls_R_factor_all                       0.0931
_refine_ls_R_factor_gt                        0.053
_refine_ls_wR_factor_ref                      0.1285
_refine_ls_wR_factor_gt                       0.1122
_refine_ls_goodness_of_fit_ref                1.015
_refine_ls_restrained_S_all                   1.015
_refine_ls_shift/su_max                       0
_refine_ls_shift/su_mean                      0
_refine_diff_density_max                      0.618
_refine_diff_density_min                      -0.711
_refine_diff_density_rms                      0.119

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

loop_
      _atom_type_symbol
      _atom_type_description
      _atom_type_scat_dispersion_real
      _atom_type_scat_dispersion_imag
      _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'
O O 0.0106 0.006 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Zn Zn 0.2839 1.4301 '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
C1 C -0.2924(7) 0.0679(4) -0.0085(3) 0.0273(12) Uani 1 1 d . . .
C2 C -0.2285(7) 0.0445(5) 0.0570(3) 0.0289(12) Uani 1 1 d . . .
C3 C -0.1527(7) 0.0174(5) 0.1313(3) 0.0301(13) Uani 1 1 d . . .
C4 C -0.0839(7) -0.0095(5) 0.1954(3) 0.0290(13) Uani 1 1 d . . .
C11 C -0.3618(7) 0.0880(5) -0.0896(3) 0.0257(12) Uani 1 1 d . . .
C12 C -0.4466(7) 0.0376(5) -0.2248(3) 0.0350(14) Uani 1 1 d . . .
```

H12 H -0.4729 -0.0079 -0.2794 0.042 Uiso 1 1 calc R . .
C13 C -0.4657(7) 0.1675(5) -0.1979(3) 0.0331(14) Uani 1 1 d . . .
H13 H -0.5082 0.226 -0.2316 0.04 Uiso 1 1 calc R . .
C14 C -0.3425(9) -0.1474(5) -0.1560(4) 0.0456(17) Uani 1 1 d . . .
H14A H -0.3336 -0.1896 -0.2129 0.068 Uiso 1 1 calc R . .
H14B H -0.4291 -0.1935 -0.1319 0.068 Uiso 1 1 calc R . .
H14C H -0.2381 -0.1483 -0.1224 0.068 Uiso 1 1 calc R . .
C21 C -0.0062(6) -0.0549(4) 0.2665(3) 0.0247(12) Uani 1 1 d . . .
C22 C 0.0972(7) -0.1947(5) 0.3456(3) 0.0303(13) Uani 1 1 d . . .
H22 H 0.1315 -0.2709 0.3642 0.036 Uiso 1 1 calc R . .
C23 C 0.1109(7) -0.0718(5) 0.3911(3) 0.0305(13) Uani 1 1 d . . .
H23 H 0.1577 -0.0497 0.447 0.037 Uiso 1 1 calc R . .
C24 C -0.0127(8) -0.2894(5) 0.1940(3) 0.0388(15) Uani 1 1 d . . .
H24A H 0.086 -0.3008 0.1649 0.058 Uiso 1 1 calc R . .
H24B H -0.1007 -0.2661 0.1561 0.058 Uiso 1 1 calc R . .
H24C H -0.0466 -0.37 0.2132 0.058 Uiso 1 1 calc R . .
C31 C -0.2753(7) 0.2680(5) 0.4119(3) 0.0280(12) Uani 1 1 d . . .
C32 C -0.4198(7) 0.3415(5) 0.4422(4) 0.0385(14) Uani 1 1 d . . .
H32A H -0.4247 0.4206 0.4175 0.058 Uiso 1 1 calc R . .
H32B H -0.4052 0.3635 0.5029 0.058 Uiso 1 1 calc R . .
H32C H -0.5219 0.2872 0.4255 0.058 Uiso 1 1 calc R . .
C41 C 0.1878(6) 0.4301(5) 0.3324(3) 0.0232(11) Uani 1 1 d . . .
C42 C 0.2989(8) 0.4903(5) 0.2755(3) 0.0337(13) Uani 1 1 d . . .
H42A H 0.3228 0.5823 0.2966 0.051 Uiso 1 1 calc R . .
H42B H 0.2435 0.4795 0.2192 0.051 Uiso 1 1 calc R . .
H42C H 0.4013 0.4475 0.2744 0.051 Uiso 1 1 calc R . .
C51 C 0.2205(6) 0.2859(5) 0.5539(3) 0.0253(12) Uani 1 1 d . . .
C52 C 0.3286(7) 0.2456(5) 0.6245(3) 0.0376(14) Uani 1 1 d . . .
H52A H 0.4426 0.2483 0.6111 0.056 Uiso 1 1 calc R . .
H52B H 0.2927 0.1578 0.6318 0.056 Uiso 1 1 calc R . .
H52C H 0.3201 0.3048 0.6759 0.056 Uiso 1 1 calc R . .
C61 C -0.2172(6) 0.6081(5) -0.0299(3) 0.0240(11) Uani 1 1 d . . .
C62 C -0.0508(7) 0.6690(5) -0.0461(4) 0.0408(15) Uani 1 1 d . . .
H62A H -0.0411 0.7607 -0.0227 0.061 Uiso 1 1 calc R . .
H62B H -0.0423 0.6592 -0.1061 0.061 Uiso 1 1 calc R . .
H62C H 0.037 0.6258 -0.0198 0.061 Uiso 1 1 calc R . .
C71 C -0.6428(6) 0.5574(5) -0.1406(3) 0.0254(12) Uani 1 1 d . . .
C72 C -0.7324(7) 0.6002(5) -0.2173(3) 0.0339(13) Uani 1 1 d . . .
H72A H -0.8472 0.6107 -0.2079 0.051 Uiso 1 1 calc R . .
H72B H -0.7267 0.5348 -0.2662 0.051 Uiso 1 1 calc R . .
H72C H -0.6803 0.6824 -0.2268 0.051 Uiso 1 1 calc R . .
N11 N -0.4123(5) 0.1978(4) -0.1133(2) 0.0233(9) Uani 1 1 d . . .
N12 N -0.3829(6) -0.0115(4) -0.1571(3) 0.0284(10) Uani 1 1 d . . .
N21 N 0.0454(6) 0.0156(4) 0.3423(3) 0.0269(10) Uani 1 1 d . . .
N22 N 0.0228(5) -0.1844(4) 0.2672(3) 0.0263(10) Uani 1 1 d . . .
O31 O -0.1287(4) 0.3186(3) 0.4409(2) 0.0280(8) Uani 1 1 d . . .
O32 O -0.2938(5) 0.1667(4) 0.3607(3) 0.0561(13) Uani 1 1 d . . .
O41 O 0.1475(4) 0.5001(3) 0.3967(2) 0.0271(8) Uani 1 1 d . . .
O42 O 0.1411(5) 0.3074(3) 0.3098(2) 0.0295(9) Uani 1 1 d . . .
O51 O 0.1637(4) 0.3980(3) 0.5654(2) 0.0273(8) Uani 1 1 d . . .
O52 O 0.1937(5) 0.2030(3) 0.4867(2) 0.0297(9) Uani 1 1 d . . .
O61 O -0.2545(4) 0.4906(3) -0.0621(2) 0.0308(9) Uani 1 1 d . . .
O62 O -0.3080(5) 0.6768(3) 0.0159(2) 0.0348(9) Uani 1 1 d . . .
O71 O -0.5851(5) 0.4467(3) -0.1482(2) 0.0313(9) Uani 1 1 d . . .
O72 O -0.6335(5) 0.6366(3) -0.0725(2) 0.0329(9) Uani 1 1 d . . .
Zn1 Zn 0 0.5 0.5 0.0213(2) Uani 1 2 d S . .
Zn2 Zn -0.46055(7) 0.37417(5) -0.04817(3) 0.02167(17) Uani 1 1 d . . .
Zn3 Zn 0.03528(8) 0.20684(5) 0.38644(4) 0.02283(17) Uani 1 1 d . . .

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
C1 0.032(3) 0.020(2) 0.031(3) 0.007(2) 0.004(3) 0.002(2)

C2 0.033(3) 0.023(2) 0.031(3) 0.004(2) 0.003(3) 0.001(2)
C3 0.036(3) 0.024(3) 0.028(3) 0.000(2) 0.002(3) 0.002(2)
C4 0.035(3) 0.022(3) 0.027(3) -0.003(2) 0.001(3) -0.002(2)
C11 0.025(3) 0.027(3) 0.027(3) 0.008(2) 0.009(2) 0.002(2)
C12 0.045(4) 0.035(3) 0.022(3) -0.006(2) 0.006(3) 0.001(3)
C13 0.051(4) 0.029(3) 0.020(3) 0.005(2) -0.001(3) 0.008(3)
C14 0.065(5) 0.017(3) 0.053(4) -0.002(3) 0.014(3) 0.003(3)
C21 0.025(3) 0.023(2) 0.026(3) 0.004(2) 0.003(2) 0.001(2)
C22 0.036(3) 0.019(2) 0.037(3) 0.008(2) 0.004(3) 0.003(2)
C23 0.040(4) 0.027(3) 0.024(3) 0.006(2) 0.000(2) 0.002(2)
C24 0.047(4) 0.025(3) 0.037(3) -0.009(3) -0.011(3) 0.001(3)
C31 0.029(3) 0.029(3) 0.025(3) 0.002(2) 0.002(2) -0.002(2)
C32 0.021(3) 0.046(3) 0.047(4) 0.001(3) 0.006(3) 0.000(2)
C41 0.019(3) 0.028(3) 0.022(3) 0.007(2) -0.002(2) 0.000(2)
C42 0.046(4) 0.030(3) 0.028(3) 0.010(2) 0.019(3) 0.000(2)
C51 0.022(3) 0.027(3) 0.027(3) 0.008(2) 0.002(2) 0.001(2)
C52 0.041(4) 0.039(3) 0.031(3) 0.003(3) -0.005(3) 0.008(3)
C61 0.022(3) 0.030(3) 0.021(3) 0.010(2) -0.002(2) -0.004(2)
C62 0.031(3) 0.042(3) 0.051(4) 0.011(3) 0.013(3) -0.004(3)
C71 0.019(3) 0.029(3) 0.029(3) 0.008(2) 0.001(2) 0.001(2)
C72 0.036(3) 0.041(3) 0.025(3) 0.004(2) 0.000(3) 0.011(3)
N11 0.029(3) 0.023(2) 0.018(2) 0.0051(17) 0.0028(18) 0.0022(18)
N12 0.040(3) 0.022(2) 0.023(2) 0.0014(18) 0.006(2) 0.0017(19)
N21 0.036(3) 0.022(2) 0.022(2) 0.0011(18) 0.001(2) 0.0025(19)
N22 0.031(3) 0.022(2) 0.024(2) 0.0021(18) 0.000(2) 0.0009(18)
O31 0.023(2) 0.0239(17) 0.034(2) -0.0030(16) 0.0015(16) -0.0004(15)
O32 0.045(3) 0.044(2) 0.067(3) -0.023(2) -0.005(2) -0.002(2)
O41 0.037(2) 0.0251(17) 0.0188(18) -0.0014(15) 0.0100(16) -0.0003(15)
O42 0.041(2) 0.0217(17) 0.0263(19) 0.0033(15) 0.0088(17) 0.0001(15)
O51 0.029(2) 0.0248(17) 0.0259(19) 0.0000(15) -0.0037(16) 0.0039(15)
O52 0.038(2) 0.0240(17) 0.0246(19) -0.0025(15) -0.0035(17) 0.0039(16)
O61 0.029(2) 0.0292(19) 0.035(2) 0.0055(17) 0.0077(17) 0.0000(16)
O62 0.031(2) 0.0330(19) 0.041(2) 0.0039(18) 0.0125(18) -0.0001(17)
O71 0.037(2) 0.0308(19) 0.027(2) 0.0057(16) 0.0020(17) 0.0054(17)
O72 0.043(2) 0.0347(19) 0.0218(19) 0.0040(16) 0.0021(17) 0.0112(17)
Zn1 0.0255(5) 0.0185(4) 0.0194(4) 0.0002(3) 0.0043(3) 0.0022(3)
Zn2 0.0249(3) 0.0189(3) 0.0213(3) 0.0020(2) 0.0039(2) 0.0033(2)
Zn3 0.0293(4) 0.0183(3) 0.0206(3) 0.0010(2) 0.0038(3) 0.0030(2)

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

_geom_special_details
;
All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
;
loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
C1 C2 1.193(7) . ?
C1 C11 1.420(7) . ?
C2 C3 1.365(8) . ?
C3 C4 1.202(7) . ?
C4 C21 1.407(7) . ?
C11 N11 1.323(6) . ?
C11 N12 1.366(6) . ?
C12 N12 1.340(7) . ?
C12 C13 1.366(7) . ?

```
C13 N11 1.369(6) . ?
C14 N12 1.460(6) . ?
C21 N21 1.338(6) . ?
C21 N22 1.371(6) . ?
C22 C23 1.357(6) . ?
C22 N22 1.367(6) . ?
C23 N21 1.379(6) . ?
C24 N22 1.467(6) . ?
C31 O32 1.221(5) . ?
C31 O31 1.281(6) . ?
C31 C32 1.503(7) . ?
C41 O41 1.245(5) . ?
C41 O42 1.286(5) . ?
C41 C42 1.501(7) . ?
C51 O51 1.263(6) . ?
C51 O52 1.264(5) . ?
C51 C52 1.493(7) . ?
C61 O61 1.251(5) . ?
C61 O62 1.255(6) . ?
C61 C62 1.503(7) . ?
C71 O71 1.252(6) . ?
C71 O72 1.255(5) . ?
C71 C72 1.510(7) . ?
N11 Zn2 2.034(4) . ?
N21 Zn3 2.002(4) . ?
O31 Zn3 1.980(3) . ?
O31 Zn1 2.129(3) . ?
O32 Zn3 2.645(4) . ?
O41 Zn1 2.118(3) . ?
O42 Zn3 1.938(4) . ?
O51 Zn1 2.052(3) . ?
O52 Zn3 1.971(3) . ?
O61 Zn2 2.035(3) . ?
O62 Zn2 2.036(4) 2_465 ?
O71 Zn2 2.077(4) . ?
O72 Zn2 2.036(3) 2_465 ?
Zn1 O51 2.052(3) 2_566 ?
Zn1 O41 2.118(3) 2_566 ?
Zn1 O31 2.129(3) 2_566 ?
Zn1 Zn3 3.3287(6) . ?
Zn2 O62 2.036(4) 2_465 ?
Zn2 O72 2.036(3) 2_465 ?
Zn2 Zn2 2.9489(10) 2_465 ?

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_3
    _geom_angle_publ_flag
C2 C1 C11 175.6(5) . . ?
C1 C2 C3 178.9(6) . . ?
C4 C3 C2 178.1(6) . . ?
C3 C4 C21 174.0(5) . . ?
N11 C11 N12 109.9(4) . . ?
N11 C11 C1 128.4(4) . . ?
N12 C11 C1 121.6(4) . . ?
N12 C12 C13 106.8(4) . . ?
C12 C13 N11 109.2(5) . . ?
N21 C21 N22 109.7(4) . . ?
N21 C21 C4 127.3(4) . . ?
N22 C21 C4 122.9(4) . . ?
C23 C22 N22 106.4(4) . . ?
C22 C23 N21 110.0(4) . . ?
O32 C31 O31 120.5(5) . . ?
O32 C31 C32 122.7(5) . . ?
```

O31 C31 C32 116.8(4) . . ?
O41 C41 O42 124.9(5) . . ?
O41 C41 C42 119.1(4) . . ?
O42 C41 C42 116.0(4) . . ?
O51 C51 O52 125.4(5) . . ?
O51 C51 C52 118.4(4) . . ?
O52 C51 C52 116.1(4) . . ?
O61 C61 O62 124.3(5) . . ?
O61 C61 C62 117.3(5) . . ?
O62 C61 C62 118.4(4) . . ?
O71 C71 O72 124.6(5) . . ?
O71 C71 C72 118.9(4) . . ?
O72 C71 C72 116.5(4) . . ?
C11 N11 C13 106.2(4) . . ?
C11 N11 Zn2 133.3(3) . . ?
C13 N11 Zn2 119.3(3) . . ?
C12 N12 C11 107.9(4) . . ?
C12 N12 C14 126.0(4) . . ?
C11 N12 C14 126.1(5) . . ?
C21 N21 C23 106.0(4) . . ?
C21 N21 Zn3 131.1(3) . . ?
C23 N21 Zn3 122.9(3) . . ?
C22 N22 C21 107.8(4) . . ?
C22 N22 C24 127.3(4) . . ?
C21 N22 C24 124.8(4) . . ?
C31 O31 Zn3 108.0(3) . . ?
C31 O31 Zn1 141.3(3) . . ?
Zn3 O31 Zn1 108.16(16) . . ?
C31 O32 Zn3 77.8(3) . . ?
C41 O41 Zn1 143.2(3) . . ?
C41 O42 Zn3 121.1(3) . . ?
C51 O51 Zn1 136.6(3) . . ?
C51 O52 Zn3 128.7(3) . . ?
C61 O61 Zn2 128.1(3) . . ?
C61 O62 Zn2 128.2(3) . 2_465 ?
C71 O71 Zn2 123.2(3) . . ?
C71 O72 Zn2 132.8(3) . 2_465 ?
O51 Zn1 O51 180.00(16) 2_566 . ?
O51 Zn1 O41 86.43(14) 2_566 . ?
O51 Zn1 O41 93.57(14) . . ?
O51 Zn1 O41 93.57(14) 2_566 2_566 ?
O51 Zn1 O41 86.43(14) . 2_566 ?
O41 Zn1 O41 180.0000(10) . 2_566 ?
O51 Zn1 O31 90.50(13) 2_566 . ?
O51 Zn1 O31 89.50(13) . . ?
O41 Zn1 O31 90.54(13) . . ?
O41 Zn1 O31 89.46(13) 2_566 . ?
O51 Zn1 O31 89.50(13) 2_566 2_566 ?
O51 Zn1 O31 90.50(13) . 2_566 ?
O41 Zn1 O31 89.46(13) . 2_566 ?
O41 Zn1 O31 90.54(13) 2_566 2_566 ?
O31 Zn1 O31 180.00(18) . 2_566 ?
O51 Zn1 Zn3 109.54(8) 2_566 . ?
O51 Zn1 Zn3 70.46(8) . . ?
O41 Zn1 Zn3 64.42(8) . . ?
O41 Zn1 Zn3 115.58(8) 2_566 . ?
O31 Zn1 Zn3 34.41(9) . . ?
O31 Zn1 Zn3 145.59(9) 2_566 . ?
N11 Zn2 O61 102.06(15) . . ?
N11 Zn2 O62 98.77(15) . 2_465 ?
O61 Zn2 O62 158.97(14) . 2_465 ?
N11 Zn2 O72 102.81(15) . 2_465 ?
O61 Zn2 O72 89.47(15) . 2_465 ?
O62 Zn2 O72 88.74(16) 2_465 2_465 ?
N11 Zn2 O71 97.71(14) . . ?
O61 Zn2 O71 88.53(15) . . ?
O62 Zn2 O71 85.82(15) 2_465 . ?
O72 Zn2 O71 159.35(14) 2_465 . ?

N11 Zn2 Zn2 178.24(12) . 2_465 ?
O61 Zn2 Zn2 79.68(10) . 2_465 ?
O62 Zn2 Zn2 79.51(10) 2_465 2_465 ?
O72 Zn2 Zn2 76.86(10) 2_465 2_465 ?
O71 Zn2 Zn2 82.56(9) . 2_465 ?
O42 Zn3 O52 108.98(16) . . ?
O42 Zn3 O31 106.70(15) . . ?
O52 Zn3 O31 99.03(14) . . ?
O42 Zn3 N21 108.03(16) . . ?
O52 Zn3 N21 93.80(15) . . ?
O31 Zn3 N21 136.42(16) . . ?
O42 Zn3 O32 116.02(16) . . ?
O52 Zn3 O32 132.16(16) . . ?
O31 Zn3 O32 53.72(12) . . ?
N21 Zn3 O32 87.35(15) . . ?
O42 Zn3 Zn1 84.41(9) . . ?
O52 Zn3 Zn1 78.36(9) . . ?
O31 Zn3 Zn1 37.43(9) . . ?
N21 Zn3 Zn1 167.06(12) . . ?
O32 Zn3 Zn1 90.35(8) . . ?