```
data_3a(EtOH)3

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             tris(ethanol) complex of tris-2,2'-
tris(2,6-pyridylene-5,5'-dipyrryl)dimethane cryptand
_chemical_melting_point           ?
_chemical_formula_moiety          'C71 H94 N9 O3'
_chemical_formula_sum
 'C71 H124 N9 O18'
_chemical_formula_weight          1391.79

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'   0.0000   0.0000
 '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   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            hexagonal
_symmetry_space_group_name_H-M    P6(3)/m

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

_cell_length_a                    18.0581(19)
```

```
_cell_length_b                    18.0581(19)
_cell_length_c                    15.2425(18)
_cell_angle_alpha                 90.00
_cell_angle_beta                  90.00
_cell_angle_gamma                 120.00
_cell_volume                      4304.6(8)
_cell_formula_units_Z             2
_cell_measurement_temperature     298(2)
_cell_measurement_reflns_used     5360
_cell_measurement_theta_min       2.256
_cell_measurement_theta_max       27.171

_exptl_crystal_description        plate
_exptl_crystal_colour             colorless
_exptl_crystal_size_max           0.55
_exptl_crystal_size_mid           0.35
_exptl_crystal_size_min           0.20
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.074
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              1514
_exptl_absorpt_coefficient_mu     0.077
_exptl_absorpt_correction_type    empirical
_exptl_absorpt_correction_T_min   ?
_exptl_absorpt_correction_T_max   ?
_exptl_absorpt_process_details    'program SADABS ver. 2.03(Sheldrick)'

_exptl_special_details
;
 ?
;

_diffrn_ambient_temperature       298(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK¥a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   'CCD area detector'
_diffrn_measurement_method        'phi and omega scanes'
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          ?
_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time   ?
_diffrn_standards_decay_%         ?
_diffrn_reflns_number             23950
_diffrn_reflns_av_R_equivalents   0.0462
_diffrn_reflns_av_sigmaI/netI     0.0352
_diffrn_reflns_limit_h_min        -17
_diffrn_reflns_limit_h_max        23
_diffrn_reflns_limit_k_min        -21
```

```
_diffrn_reflns_limit_k_max        19
_diffrn_reflns_limit_l_min        -19
_diffrn_reflns_limit_l_max        19
_diffrn_reflns_theta_min          1.30
_diffrn_reflns_theta_max          27.22
_reflns_number_total              3154
_reflns_number_gt                 1920
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        'Bruker SAINT+ ver.6.45'
_computing_cell_refinement        'Bruker SAINT+ ver.6.45'
_computing_data_reduction         'Bruker SAINT+ ver.6.45'
_computing_structure_solution     'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics     'Bruker XSHELL ver. 4.02'
_computing_publication_material   'Bruker SHELXTL ver.6.14'

_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^)+(0.1247P)^2^+0.4806P] 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     mixed
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          3154
_refine_ls_number_parameters      182
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.1240
_refine_ls_R_factor_gt            0.0676
_refine_ls_wR_factor_ref          0.2179
_refine_ls_wR_factor_gt           0.1837
_refine_ls_goodness_of_fit_ref    1.054
_refine_ls_restrained_S_all       1.054
_refine_ls_shift/su_max           0.001
_refine_ls_shift/su_mean          0.000
```

```
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 0.6667 0.3333 0.4874(2) 0.0472(8) Uani 1 3 d S . .
H1 H 0.6667 0.3333 0.417(2) 0.051(9) Uiso 1 3 d S . .
C2 C 0.64885(12) 0.24551(12) 0.51324(12) 0.0447(5) Uani 1 1 d . . .
C3 C 0.66382(13) 0.18939(13) 0.46551(12) 0.0513(5) Uani 1 1 d . . .
C4 C 0.63476(14) 0.11406(13) 0.51637(13) 0.0542(6) Uani 1 1 d . . .
C5 C 0.60363(13) 0.12741(12) 0.59437(13) 0.0493(5) Uani 1 1 d . . .
C6 C 0.57218(13) 0.07597(12) 0.67381(13) 0.0502(5) Uani 1 1 d . . .
C7 C 0.52138(15) -0.01219(14) 0.67193(15) 0.0607(6) Uani 1 1 d . . .
H7 H 0.5049(13) -0.0403(14) 0.6186(16) 0.061(6) Uiso 1 1 d . . .
C8 C 0.4964(2) -0.0562(2) 0.7500 0.0654(9) Uani 1 2 d S . .
H8 H 0.454(2) -0.121(3) 0.7500 0.079(10) Uiso 1 2 d S . .
C9 C 0.70913(18) 0.20848(18) 0.37885(15) 0.0672(7) Uani 1 1 d . . .
H9A H 0.6989(16) 0.2483(18) 0.3478(18) 0.086(8) Uiso 1 1 d . . .
H9B H 0.6895(16) 0.1560(17) 0.3461(17) 0.079(7) Uiso 1 1 d . . .
C10 C 0.8042(3) 0.2508(4) 0.3887(3) 0.1118(15) Uani 1 1 d . . .
H10A H 0.824(2) 0.304(3) 0.409(3) 0.137(16) Uiso 1 1 d . . .
H10B H 0.833(2) 0.2615(18) 0.334(2) 0.098(9) Uiso 1 1 d . . .
H10C H 0.808(4) 0.216(4) 0.407(4) 0.17(3) Uiso 1 1 d . . .
C11 C 0.6438(2) 0.03890(18) 0.4923(2) 0.0807(8) Uani 1 1 d . . .
H11A H 0.597(2) -0.015(2) 0.488(2) 0.109(11) Uiso 1 1 d . . .
H11B H 0.642(2) 0.023(2) 0.424(2) 0.114(10) Uiso 1 1 d . . .
C12 C 0.7116(3) 0.0336(3) 0.5362(3) 0.1392(16) Uani 1 1 d . . .
H12A H 0.7657 0.0816 0.5199 0.209 Uiso 1 1 calc R . .
H12B H 0.7101 -0.0184 0.5195 0.209 Uiso 1 1 calc R . .
H12C H 0.7041 0.0339 0.5985 0.209 Uiso 1 1 calc R . .
C13 C 0.8108(3) 0.2596(3) 0.7500 0.1244(19) Uani 1 2 d S . .
H13A H 0.8615 0.3018 0.7800 0.149 Uiso 0.50 1 d PR . .
H13B H 0.7786 0.2060 0.7800 0.149 Uiso 0.50 1 d PR . .
C14 C 0.8324(8) 0.2351(9) 0.6800(7) 0.190(5) Uani 0.50 1 d P . .
H14A H 0.8528 0.1969 0.6956 0.285 Uiso 0.50 1 calc PR . .
H14B H 0.8767 0.2840 0.6501 0.285 Uiso 0.50 1 calc PR . .
H14C H 0.7836 0.2060 0.6422 0.285 Uiso 0.50 1 calc PR . .
N1 N 0.61339(11) 0.20765(11) 0.59103(10) 0.0481(5) Uani 1 1 d . . .
H1N H 0.5970(13) 0.2294(14) 0.6299(15) 0.056(6) Uiso 1 1 d . . .
N2 N 0.59761(14) 0.11950(13) 0.7500 0.0479(6) Uani 1 2 d S . .
```

```
O1 O 0.74895(16) 0.28446(15) 0.7500 0.0686(7) Uani 1 2 d S . .
H1O H 0.702(3) 0.240(3) 0.7500 0.097(15) Uiso 1 2 d S . .
O2 O 0.8903 0.0018 0.2500 1.311 Uiso 1 2 d S . .
O3 O 0.8285 0.0570 0.2500 0.760 Uiso 1 2 d S . .
O4 O 0.9755 0.1732 0.2500 0.753 Uiso 1 2 d S . .
O5 O 0.9143 0.8985 0.5449 1.145 Uiso 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 0.0523(13) 0.0523(13) 0.0370(16) 0.000 0.000 0.0261(6)
C2 0.0442(10) 0.0464(11) 0.0402(10) -0.0031(8) -0.0036(8) 0.0201(9)
C3 0.0529(12) 0.0538(12) 0.0440(11) -0.0087(8) -0.0025(8) 0.0243(10)
C4 0.0617(13) 0.0469(12) 0.0514(11) -0.0077(9) -0.0014(9) 0.0253(10)
C5 0.0556(12) 0.0426(11) 0.0478(11) -0.0046(8) -0.0024(8) 0.0231(9)
C6 0.0545(12) 0.0447(12) 0.0521(12) -0.0028(8) -0.0011(9) 0.0253(10)
C7 0.0685(15) 0.0452(12) 0.0586(14) -0.0073(10) -0.0043(11) 0.0210(11)
C8 0.071(2) 0.0397(18) 0.074(2) 0.000 0.000 0.0193(16)
C9 0.0861(18) 0.0667(16) 0.0506(13) -0.0062(11) 0.0105(11) 0.0397(14)
C10 0.087(3) 0.112(3) 0.106(3) 0.002(3) 0.044(2) 0.027(2)
C11 0.108(2) 0.0637(18) 0.0811(19) -0.0070(13) 0.0172(16) 0.0505(18)
C12 0.145(3) 0.125(3) 0.187(4) -0.022(3) -0.019(3) 0.097(3)
C13 0.087(3) 0.096(3) 0.193(6) 0.000 0.000 0.048(3)
C14 0.195(10) 0.251(13) 0.191(10) -0.073(9) 0.003(8) 0.161(10)
N1 0.0574(11) 0.0475(10) 0.0413(9) -0.0030(7) 0.0026(7) 0.0276(8)
N2 0.0543(14) 0.0407(13) 0.0472(13) 0.000 0.000 0.0227(11)
O1 0.0615(15) 0.0563(14) 0.0733(16) 0.000 0.000 0.0185(13)

_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 1.504(2) . ?
C1 C2 1.504(2) 2_655 ?
C1 C2 1.504(2) 3_665 ?
C2 N1 1.359(2) . ?
C2 C3 1.380(3) . ?
C3 C4 1.419(3) . ?
C3 C9 1.500(3) . ?
C4 C5 1.387(3) . ?
C4 C11 1.492(3) . ?
C5 N1 1.370(3) . ?
C5 C6 1.457(3) . ?
C6 N2 1.348(2) . ?
C6 C7 1.384(3) . ?
C7 C8 1.376(3) . ?
C8 C7 1.376(3) 10_557 ?
C9 C10 1.497(5) . ?
C11 C12 1.439(5) . ?
C13 C14 1.288(8) . ?
C13 C14 1.288(8) 10_557 ?
C13 O1 1.397(6) . ?
N2 C6 1.348(2) 10_557 ?

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 C2 113.42(11) . 2_655 ?
C2 C1 C2 113.42(11) . 3_665 ?
C2 C1 C2 113.42(11) 2_655 3_665 ?
N1 C2 C3 107.86(17) . . ?
N1 C2 C1 123.87(18) . . ?
C3 C2 C1 128.27(19) . . ?
C2 C3 C4 107.56(17) . . ?
C2 C3 C9 125.5(2) . . ?
C4 C3 C9 126.80(19) . . ?
C5 C4 C3 106.83(17) . . ?
C5 C4 C11 127.4(2) . . ?
C3 C4 C11 125.5(2) . . ?
N1 C5 C4 107.63(17) . . ?
N1 C5 C6 120.53(17) . . ?
C4 C5 C6 131.62(18) . . ?
N2 C6 C7 121.68(19) . . ?
N2 C6 C5 115.70(17) . . ?
C7 C6 C5 122.60(19) . . ?
C8 C7 C6 118.9(2) . . ?
C7 C8 C7 119.7(3) . 10_557 ?
```

```
C10 C9 C3 112.6(3) . . ?
C12 C11 C4 115.5(3) . . ?
C14 C13 C14 111.9(12) . 10_557 ?
C14 C13 O1 122.6(6) . . ?
C14 C13 O1 122.6(6) 10_557 . ?
C2 N1 C5 110.11(16) . . ?
C6 N2 C6 119.0(2) 10_557 . ?

_diffrn_measured_fraction_theta_max    0.944
_diffrn_reflns_theta_full              27.22
_diffrn_measured_fraction_theta_full   0.944
_refine_diff_density_max    0.522
_refine_diff_density_min   -0.320
_refine_diff_density_rms    0.142


#===END


data_3a(DCA)3

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             tris(dichloroacetic acid) complex of
tris-2,2'-(2,6-pyridylene-5,5'-dipyrryl)dimethane cryptand
_chemical_melting_point           ?
_chemical_formula_moiety          'C71 H82 Cl6 N9 O6'
_chemical_formula_sum
 'C75 H86 Cl10 N9 O10'
_chemical_formula_weight          1628.03

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'   0.0000   0.0000
 '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   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Cl'  'Cl'   0.1484   0.1585
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

```
_symmetry_cell_setting            monoclinic
_symmetry_space_group_name_H-M    P2(1)/n

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

_cell_length_a                    22.0204(12)
_cell_length_b                    13.7305(8)
_cell_length_c                    26.5513(15)
_cell_angle_alpha                 90.00
_cell_angle_beta                  91.5070(10)
_cell_angle_gamma                 90.00
_cell_volume                      8025.0(8)
_cell_formula_units_Z             4
_cell_measurement_temperature     298(2)
_cell_measurement_reflns_used     13777
_cell_measurement_theta_min       2.302
_cell_measurement_theta_max       27.157

_exptl_crystal_description        prism
_exptl_crystal_colour             green
_exptl_crystal_size_max           0.5
_exptl_crystal_size_mid           0.4
_exptl_crystal_size_min           0.3
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.347
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              3396
_exptl_absorpt_coefficient_mu     0.409
_exptl_absorpt_correction_type    empirical
_exptl_absorpt_correction_T_min   ?
_exptl_absorpt_correction_T_max   ?
_exptl_absorpt_process_details    'program SADABS ver. 2.03(Sheldrick)'

_exptl_special_details
;
 ?
;

_diffrn_ambient_temperature       298(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK¥a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   'CCD area detector'
```

```
_diffrn_measurement_method        'phi and omega scanes'
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          ?
_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time   ?
_diffrn_standards_decay_%         ?
_diffrn_reflns_number             41285
_diffrn_reflns_av_R_equivalents   0.0340
_diffrn_reflns_av_sigmaI/netI     0.0534
_diffrn_reflns_limit_h_min        -27
_diffrn_reflns_limit_h_max        26
_diffrn_reflns_limit_k_min        -8
_diffrn_reflns_limit_k_max        17
_diffrn_reflns_limit_l_min        -32
_diffrn_reflns_limit_l_max        32
_diffrn_reflns_theta_min          1.19
_diffrn_reflns_theta_max          27.32
_reflns_number_total              15510
_reflns_number_gt                 9275
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        'Bruker SAINT+ ver.6.45'
_computing_cell_refinement        'Bruker SAINT+ ver.6.45'
_computing_data_reduction         'Bruker SAINT+ ver.6.45'
_computing_structure_solution     'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics     'Bruker XSHELL ver. 4.02'
_computing_publication_material   'Bruker SHELXTL ver.6.14'

_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^)+(0.0667P)^2^+17.8521P] 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     mixed
_refine_ls_extinction_method      none
```

```
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          15510
_refine_ls_number_parameters      1056
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.1386
_refine_ls_R_factor_gt            0.0893
_refine_ls_wR_factor_ref          0.2388
_refine_ls_wR_factor_gt           0.2170
_refine_ls_goodness_of_fit_ref    1.165
_refine_ls_restrained_S_all       1.165
_refine_ls_shift/su_max           0.000
_refine_ls_shift/su_mean          0.000

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 0.26335(19) 0.7200(3) 0.15702(16) 0.0352(10) Uani 1 1 d . . .
C2 C 0.31823(19) 0.7792(3) 0.14411(16) 0.0361(10) Uani 1 1 d . . .
C3 C 0.3474(2) 0.8511(4) 0.17133(16) 0.0409(11) Uani 1 1 d . . .
C4 C 0.3973(2) 0.8823(4) 0.14345(17) 0.0416(11) Uani 1 1 d . . .
C5 C 0.39611(19) 0.8292(3) 0.09879(16) 0.0378(11) Uani 1 1 d . . .
C6 C 0.43172(19) 0.8385(3) 0.05431(17) 0.0388(11) Uani 1 1 d . . .
C7 C 0.4913(2) 0.8711(4) 0.0539(2) 0.0526(14) Uani 1 1 d . . .
C8 C 0.5207(2) 0.8757(5) 0.0089(2) 0.0639(17) Uani 1 1 d . . .
C9 C 0.4927(2) 0.8480(4) -0.0353(2) 0.0566(15) Uani 1 1 d . . .
H9 H 0.5134 0.8518 -0.0653 0.068 Uiso 1 1 calc R . .
C10 C 0.4335(2) 0.8143(4) -0.03552(17) 0.0417(11) Uani 1 1 d . . .
C11 C 0.3997(2) 0.7816(4) -0.07919(17) 0.0426(11) Uani 1 1 d . . .
C12 C 0.4176(2) 0.7300(4) -0.12112(18) 0.0456(12) Uani 1 1 d . . .
C13 C 0.3652(2) 0.7126(4) -0.15125(18) 0.0466(12) Uani 1 1 d . . .
C14 C 0.3168(2) 0.7536(4) -0.12717(17) 0.0424(11) Uani 1 1 d . . .
C15 C 0.20479(19) 0.7708(3) 0.14322(16) 0.0353(10) Uani 1 1 d . . .
C16 C 0.1498(2) 0.7681(3) 0.16713(17) 0.0385(11) Uani 1 1 d . . .
C17 C 0.1094(2) 0.8291(3) 0.13988(17) 0.0398(11) Uani 1 1 d . . .
C18 C 0.14026(19) 0.8664(3) 0.09952(17) 0.0378(10) Uani 1 1 d . . .
C19 C 0.1200(2) 0.9260(3) 0.05815(18) 0.0400(11) Uani 1 1 d . . .
C20 C 0.0764(2) 0.9979(4) 0.06069(19) 0.0477(12) Uani 1 1 d . . .
H20 H 0.0563 1.0084 0.0906 0.057 Uiso 1 1 calc R . .
C21 C 0.0628(3) 1.0540(4) 0.0192(2) 0.0562(15) Uani 1 1 d . . .
```

```
C22 C 0.0924(2) 1.0408(4) -0.0251(2) 0.0517(13) Uani 1 1 d . . .
C23 C 0.1353(2) 0.9685(4) -0.02896(18) 0.0425(11) Uani 1 1 d . . .
C24 C 0.1692(2) 0.9466(4) -0.07350(17) 0.0406(11) Uani 1 1 d . . .
C25 C 0.1920(2) 1.0067(4) -0.11011(18) 0.0450(12) Uani 1 1 d . . .
C26 C 0.2265(2) 0.9474(4) -0.14211(18) 0.0470(12) Uani 1 1 d . . .
C27 C 0.2233(2) 0.8534(4) -0.12443(17) 0.0427(11) Uani 1 1 d . . .
C28 C 0.26624(19) 0.6181(3) 0.13668(16) 0.0356(10) Uani 1 1 d . . .
C29 C 0.2988(2) 0.5386(4) 0.15415(17) 0.0409(11) Uani 1 1 d . . .
C30 C 0.2857(2) 0.4598(3) 0.12173(17) 0.0413(11) Uani 1 1 d . . .
C31 C 0.2459(2) 0.4944(3) 0.08471(17) 0.0398(11) Uani 1 1 d . . .
C32 C 0.2204(2) 0.4486(3) 0.03978(18) 0.0424(11) Uani 1 1 d . . .
C33 C 0.1986(3) 0.3546(4) 0.0374(2) 0.0556(14) Uani 1 1 d . . .
C34 C 0.1721(3) 0.3213(4) -0.0065(2) 0.0640(17) Uani 1 1 d . . .
C35 C 0.1666(3) 0.3799(4) -0.0483(2) 0.0571(15) Uani 1 1 d . . .
C36 C 0.1886(2) 0.4738(4) -0.04673(18) 0.0449(12) Uani 1 1 d . . .
C37 C 0.1859(2) 0.5421(4) -0.08817(18) 0.0449(12) Uani 1 1 d . . .
C38 C 0.1428(2) 0.5556(4) -0.1266(2) 0.0544(14) Uani 1 1 d . . .
C39 C 0.1608(2) 0.6388(4) -0.1536(2) 0.0575(14) Uani 1 1 d . . .
C40 C 0.2137(2) 0.6730(4) -0.13147(18) 0.0448(12) Uani 1 1 d . . .
C41 C 0.2508(2) 0.7614(4) -0.14402(19) 0.0458(12) Uani 1 1 d . . .
C42 C 0.3275(3) 0.8925(4) 0.22058(19) 0.0564(14) Uani 1 1 d . . .
H42A H 0.3629 0.9159 0.2394 0.068 Uiso 1 1 calc R . .
H42B H 0.3092 0.8410 0.2401 0.068 Uiso 1 1 calc R . .
C43 C 0.2824(3) 0.9750(5) 0.2144(3) 0.086(2) Uani 1 1 d . . .
H43A H 0.3004 1.0269 0.1957 0.128 Uiso 1 1 calc R . .
H43B H 0.2715 0.9986 0.2470 0.128 Uiso 1 1 calc R . .
H43C H 0.2467 0.9520 0.1966 0.128 Uiso 1 1 calc R . .
C44 C 0.4415(2) 0.9608(4) 0.1594(2) 0.0572(14) Uani 1 1 d . . .
H44A H 0.4198 1.0107 0.1775 0.069 Uiso 1 1 calc R . .
H44B H 0.4578 0.9906 0.1296 0.069 Uiso 1 1 calc R . .
C45 C 0.4939(3) 0.9244(6) 0.1927(2) 0.087(2) Uani 1 1 d . . .
H45A H 0.4784 0.8984 0.2234 0.131 Uiso 1 1 calc R . .
H45B H 0.5210 0.9774 0.2004 0.131 Uiso 1 1 calc R . .
H45C H 0.5154 0.8743 0.1753 0.131 Uiso 1 1 calc R . .
C46 C 0.4807(3) 0.6949(5) -0.1321(2) 0.0655(16) Uani 1 1 d . . .
H46A H 0.4782 0.6290 -0.1452 0.079 Uiso 1 1 calc R . .
H46B H 0.5045 0.6928 -0.1008 0.079 Uiso 1 1 calc R . .
C47 C 0.5135(3) 0.7582(6) -0.1696(3) 0.095(2) Uani 1 1 d . . .
H47A H 0.4907 0.7595 -0.2009 0.142 Uiso 1 1 calc R . .
H47B H 0.5532 0.7320 -0.1750 0.142 Uiso 1 1 calc R . .
H47C H 0.5172 0.8232 -0.1565 0.142 Uiso 1 1 calc R . .
C48 C 0.3613(3) 0.6565(4) -0.2001(2) 0.0652(16) Uani 1 1 d . . .
H48A H 0.3285 0.6831 -0.2210 0.078 Uiso 1 1 calc R . .
H48B H 0.3988 0.6655 -0.2179 0.078 Uiso 1 1 calc R . .
C49 C 0.3508(5) 0.5502(6) -0.1929(3) 0.139(4) Uani 1 1 d . . .
H49A H 0.3807 0.5247 -0.1695 0.208 Uiso 1 1 calc R . .
H49B H 0.3539 0.5173 -0.2247 0.208 Uiso 1 1 calc R . .
H49C H 0.3110 0.5402 -0.1800 0.208 Uiso 1 1 calc R . .
C50 C 0.1347(2) 0.7049(4) 0.21133(19) 0.0551(14) Uani 1 1 d . . .
H50A H 0.1062 0.7390 0.2322 0.066 Uiso 1 1 calc R . .
```

```
H50B H 0.1714 0.6935 0.2315 0.066 Uiso 1 1 calc R . .
C51 C 0.1076(3) 0.6075(5) 0.1955(3) 0.083(2) Uani 1 1 d . . .
H51A H 0.0715 0.6183 0.1751 0.125 Uiso 1 1 calc R . .
H51B H 0.0975 0.5709 0.2249 0.125 Uiso 1 1 calc R . .
H51C H 0.1366 0.5718 0.1764 0.125 Uiso 1 1 calc R . .
C52 C 0.0442(2) 0.8474(4) 0.1538(2) 0.0528(13) Uani 1 1 d . . .
H52A H 0.0261 0.7863 0.1639 0.063 Uiso 1 1 calc R . .
H52B H 0.0217 0.8706 0.1242 0.063 Uiso 1 1 calc R . .
C53 C 0.0378(3) 0.9210(5) 0.1960(2) 0.0774(19) Uani 1 1 d . . .
H53A H 0.0616 0.9002 0.2249 0.116 Uiso 1 1 calc R . .
H53B H -0.0041 0.9259 0.2048 0.116 Uiso 1 1 calc R . .
H53C H 0.0520 0.9834 0.1850 0.116 Uiso 1 1 calc R . .
C54 C 0.1834(3) 1.1139(4) -0.1153(2) 0.0671(16) Uani 1 1 d . . .
H54A H 0.2206 1.1426 -0.1272 0.081 Uiso 1 1 calc R . .
H54B H 0.1757 1.1413 -0.0824 0.081 Uiso 1 1 calc R . .
C55 C 0.1315(4) 1.1410(6) -0.1512(3) 0.112(3) Uani 1 1 d . . .
H55A H 0.1390 1.1148 -0.1839 0.167 Uiso 1 1 calc R . .
H55B H 0.1284 1.2106 -0.1533 0.167 Uiso 1 1 calc R . .
H55C H 0.0942 1.1148 -0.1390 0.167 Uiso 1 1 calc R . .
C56 C 0.2624(3) 0.9794(5) -0.1869(2) 0.0752(19) Uani 1 1 d . . .
H56A H 0.2547 0.9338 -0.2143 0.090 Uiso 1 1 calc R . .
H56B H 0.2476 1.0426 -0.1979 0.090 Uiso 1 1 calc R . .
C57 C 0.3282(4) 0.9861(8) -0.1779(4) 0.136(4) Uani 1 1 d . . .
H57A H 0.3367 1.0330 -0.1518 0.203 Uiso 1 1 calc R . .
H57B H 0.3473 1.0061 -0.2083 0.203 Uiso 1 1 calc R . .
H57C H 0.3437 0.9237 -0.1677 0.203 Uiso 1 1 calc R . .
C58 C 0.3438(2) 0.5400(4) 0.19820(19) 0.0515(13) Uani 1 1 d . . .
H58A H 0.3436 0.4767 0.2145 0.062 Uiso 1 1 calc R . .
H58B H 0.3306 0.5877 0.2225 0.062 Uiso 1 1 calc R . .
C59 C 0.4084(3) 0.5639(6) 0.1834(3) 0.082(2) Uani 1 1 d . . .
H59A H 0.4219 0.5167 0.1595 0.124 Uiso 1 1 calc R . .
H59B H 0.4347 0.5626 0.2128 0.124 Uiso 1 1 calc R . .
H59C H 0.4093 0.6276 0.1685 0.124 Uiso 1 1 calc R . .
C60 C 0.3114(3) 0.3585(4) 0.1260(2) 0.0546(13) Uani 1 1 d . . .
H60A H 0.3546 0.3628 0.1336 0.065 Uiso 1 1 calc R . .
H60B H 0.3061 0.3259 0.0938 0.065 Uiso 1 1 calc R . .
C61 C 0.2818(3) 0.2979(4) 0.1664(2) 0.0746(18) Uani 1 1 d . . .
H61A H 0.2882 0.3285 0.1986 0.112 Uiso 1 1 calc R . .
H61B H 0.2996 0.2340 0.1671 0.112 Uiso 1 1 calc R . .
H61C H 0.2390 0.2927 0.1590 0.112 Uiso 1 1 calc R . .
C62 C 0.0858(3) 0.4964(5) -0.1364(3) 0.084(2) Uani 1 1 d . . .
H62A H 0.0526 0.5402 -0.1452 0.100 Uiso 1 1 calc R . .
H62B H 0.0754 0.4626 -0.1057 0.100 Uiso 1 1 calc R . .
C63 C 0.0924(4) 0.4223(6) -0.1783(3) 0.121(3) Uani 1 1 d . . .
H63A H 0.0944 0.4554 -0.2101 0.182 Uiso 1 1 calc R . .
H63B H 0.0581 0.3792 -0.1789 0.182 Uiso 1 1 calc R . .
H63C H 0.1290 0.3854 -0.1726 0.182 Uiso 1 1 calc R . .
C64 C 0.1360(6) 0.6763(7) -0.2113(7) 0.198(10) Uani 1 1 d . . .
H64A H 0.1694 0.6841 -0.2341 0.237 Uiso 1 1 calc R . .
H64B H 0.1064 0.6315 -0.2259 0.237 Uiso 1 1 calc R . .
```

```
C65 C 0.1142(6) 0.7501(16) -0.2017(6) 0.251(11) Uani 1 1 d . . .
H65A H 0.0852 0.7411 -0.1757 0.376 Uiso 1 1 calc R . .
H65B H 0.0941 0.7760 -0.2313 0.376 Uiso 1 1 calc R . .
H65C H 0.1452 0.7946 -0.1902 0.376 Uiso 1 1 calc R . .
C67 C 0.3534(2) 0.5764(4) -0.0121(2) 0.0504(13) Uani 1 1 d . . .
C69 C 0.3000(2) -0.0162(4) 0.0103(2) 0.0535(14) Uani 1 1 d . . .
C71 C 0.1063(2) 0.6903(4) -0.0027(2) 0.0496(13) Uani 1 1 d . . .
C72 C 0.9316(5) 0.7452(8) 0.8416(4) 0.149(4) Uani 1 1 d . . .
C73 C 0.9833(4) 0.7702(7) 0.8796(3) 0.096(3) Uani 1 1 d . . .
C74 C 0.3455(3) 0.3203(5) 0.8933(2) 0.0730(17) Uani 1 1 d . . .
H74 H 0.3410 0.3807 0.9124 0.088 Uiso 1 1 calc . . .
C75 C 0.3312(3) 0.2354(5) 0.9283(3) 0.0735(18) Uani 1 1 d . . .
C66A C 0.4158(4) 0.5307(5) 0.0036(4) 0.053(3) Uani 0.81(3) 1 d P . .
H66A H 0.4412 0.5305 -0.0262 0.063 Uiso 0.81 1 calc P . .
C68A C 0.2908(4) 0.0840(9) 0.0387(4) 0.048(4) Uani 0.69(3) 1 d P . .
H68A H 0.2871 0.1351 0.0130 0.057 Uiso 0.69 1 calc P . .
C70A C 0.0509(3) 0.6284(5) 0.0126(3) 0.058(3) Uani 0.88(2) 1 d P . .
H70A H 0.0307 0.6039 -0.0182 0.070 Uiso 0.88 1 calc P . .
C66B C 0.3923(17) 0.525(3) 0.0331(19) 0.070(14) Uani 0.19(3) 1 d P . .
H66B H 0.3658 0.5265 0.0623 0.084 Uiso 0.19 1 calc P . .
C68B C 0.2970(8) 0.0337(19) 0.0630(11) 0.047(8) Uani 0.31(3) 1 d P . .
H68B H 0.3008 -0.0191 0.0877 0.057 Uiso 0.31 1 calc P . .
C70B C 0.070(2) 0.655(3) 0.043(2) 0.049(17) Uiso 0.12(2) 1 d P . .
H70B H 0.0912 0.6812 0.0727 0.058 Uiso 0.12 1 calc P . .
Cl1 Cl 0.45473(8) 0.59342(15) 0.05059(8) 0.0979(7) Uani 1 1 d . . .
Cl2 Cl 0.40321(9) 0.40787(13) 0.02086(7) 0.0873(5) Uani 1 1 d . . .
Cl3 Cl 0.22677(7) 0.08667(13) 0.07295(8) 0.0901(6) Uani 1 1 d . . .
Cl4 Cl 0.35639(7) 0.10805(15) 0.07502(7) 0.0887(6) Uani 1 1 d . . .
Cl5 Cl 0.07048(8) 0.52981(13) 0.05020(8) 0.0870(6) Uani 1 1 d . . .
Cl6 Cl -0.00048(8) 0.70594(15) 0.04368(9) 0.0995(7) Uani 1 1 d . . .
Cl9 Cl 0.41885(9) 0.31778(18) 0.87052(9) 0.1127(7) Uani 1 1 d . . .
Cl10 Cl 0.29042(11) 0.32038(19) 0.84460(9) 0.1249(9) Uani 1 1 d . . .
Cl7A Cl 0.9030(3) 0.6309(3) 0.84677(17) 0.166(2) Uani 0.70 1 d P . .
Cl8A Cl 0.87628(15) 0.8306(3) 0.8388(2) 0.1381(17) Uani 0.70 1 d P . .
Cl7B Cl 0.9514(8) 0.733(3) 0.7893(8) 0.45(3) Uani 0.30 1 d P . .
Cl8B Cl 0.9175(11) 0.8022(9) 0.7966(6) 0.271(13) Uani 0.30 1 d P . .
H1 H 0.2654(18) 0.715(3) 0.1919(16) 0.028(11) Uiso 1 1 d . . .
H7 H 0.511(2) 0.886(4) 0.0850(19) 0.050(14) Uiso 1 1 d . . .
H8 H 0.559(3) 0.901(4) 0.008(2) 0.068(17) Uiso 1 1 d . . .
H21 H 0.036(2) 1.101(4) 0.0216(18) 0.046(14) Uiso 1 1 d . . .
H22 H 0.080(2) 1.076(4) -0.053(2) 0.054(15) Uiso 1 1 d . . .
H33 H 0.201(2) 0.317(4) 0.065(2) 0.054(15) Uiso 1 1 d . . .
H34 H 0.157(2) 0.261(4) -0.0087(19) 0.056(16) Uiso 1 1 d . . .
H35 H 0.148(2) 0.357(4) -0.0782(19) 0.048(14) Uiso 1 1 d . . .
H41 H 0.252(2) 0.768(3) -0.1822(18) 0.046(13) Uiso 1 1 d . . .
H72A H 0.9497 0.7467 0.8085 0.055 Uiso 0.40 1 d P . .
H72B H 0.8996 0.7751 0.8618 0.055 Uiso 0.30 1 d P . .
H72C H 0.9128 0.6837 0.8526 0.055 Uiso 0.30 1 d P . .
H1N H 0.3403(16) 0.728(3) 0.0809(14) 0.009(10) Uiso 1 1 d . . .
H2N H 0.372(2) 0.791(4) 0.0081(19) 0.049(16) Uiso 1 1 d . . .
```

```
H3N H 0.319(2) 0.831(3) -0.0675(16) 0.027(13) Uiso 1 1 d . . .
H5N H 0.1707(19) 0.874(3) 0.0110(15) 0.021(12) Uiso 1 1 d . . .
H6N H 0.175(2) 0.806(4) -0.0697(17) 0.033(13) Uiso 1 1 d . . .
H8N H 0.229(2) 0.559(4) -0.0009(17) 0.034(13) Uiso 1 1 d . . .
H9N H 0.263(2) 0.615(4) -0.0781(19) 0.049(15) Uiso 1 1 d . . .
N1 N 0.34715(17) 0.7677(3) 0.10005(14) 0.0372(9) Uani 1 1 d . . .
N2 N 0.40566(19) 0.8129(3) 0.00980(15) 0.0419(10) Uani 1 1 d . . .
N3 N 0.33773(19) 0.7935(3) -0.08326(15) 0.0435(10) Uani 1 1 d . . .
N4 N 0.19867(16) 0.8294(3) 0.10230(13) 0.0362(9) Uani 1 1 d . . .
H4N H 0.2268 0.8417 0.0813 0.043 Uiso 1 1 calc R . .
N5 N 0.14677(19) 0.9144(3) 0.01288(15) 0.0403(10) Uani 1 1 d . . .
N6 N 0.19000(19) 0.8538(3) -0.08218(15) 0.0444(10) Uani 1 1 d . . .
N7 N 0.23500(17) 0.5911(3) 0.09421(13) 0.0380(9) Uani 1 1 d . . .
H7N H 0.2120 0.6286 0.0761 0.046 Uiso 1 1 calc R . .
N8 N 0.21502(19) 0.5033(3) -0.00267(15) 0.0416(10) Uani 1 1 d . . .
N9 N 0.2278(2) 0.6161(3) -0.09103(15) 0.0439(10) Uani 1 1 d . . .
O1 O 0.32863(15) 0.6397(3) 0.01375(13) 0.0523(9) Uani 1 1 d . . .
O2 O 0.33603(17) 0.5443(3) -0.05277(14) 0.0629(10) Uani 1 1 d . . .
O3 O 0.27922(16) -0.0960(3) 0.02224(15) 0.0605(10) Uani 1 1 d . . .
O4 O 0.32801(19) 0.0018(3) -0.02855(15) 0.0682(11) Uani 1 1 d . . .
O5 O 0.15464(17) 0.6897(3) 0.02040(14) 0.0579(10) Uani 1 1 d . . .
O6 O 0.09439(18) 0.7369(3) -0.04190(16) 0.0734(12) Uani 1 1 d . . .
O7 O 1.0040(3) 0.7053(5) 0.9026(3) 0.145(3) Uani 1 1 d . . .
H7O H 1.0309 0.7255 0.9219 0.218 Uiso 1 1 calc R . .
O8 O 1.0026(3) 0.8524(6) 0.8792(3) 0.136(2) Uani 1 1 d . . .
O9 O 0.3537(3) 0.1556(4) 0.9167(2) 0.1011(16) Uani 1 1 d . . .
H9O H 0.3439 0.1142 0.9372 0.152 Uiso 1 1 calc R . .
O10 O 0.2973(4) 0.2509(5) 0.9612(3) 0.146(3) 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 0.036(2) 0.041(3) 0.028(2) 0.003(2) -0.0014(19) -0.003(2)
C2 0.036(2) 0.041(3) 0.031(2) -0.001(2) 0.0011(19) 0.006(2)
C3 0.042(2) 0.045(3) 0.035(2) -0.007(2) -0.001(2) 0.000(2)
C4 0.036(2) 0.046(3) 0.042(3) -0.005(2) -0.001(2) -0.001(2)
C5 0.033(2) 0.044(3) 0.036(2) -0.005(2) 0.0001(19) -0.005(2)
C6 0.034(2) 0.042(3) 0.040(2) -0.005(2) 0.000(2) -0.003(2)
C7 0.039(3) 0.072(4) 0.047(3) -0.009(3) -0.003(2) -0.012(3)
C8 0.036(3) 0.098(5) 0.058(3) -0.006(3) 0.009(3) -0.020(3)
C9 0.039(3) 0.083(4) 0.048(3) -0.004(3) 0.008(2) -0.012(3)
C10 0.038(2) 0.048(3) 0.039(2) -0.002(2) 0.006(2) -0.002(2)
C11 0.037(2) 0.051(3) 0.039(2) 0.000(2) 0.006(2) -0.003(2)
C12 0.049(3) 0.046(3) 0.042(3) 0.000(2) 0.007(2) 0.001(2)
C13 0.056(3) 0.044(3) 0.040(3) -0.003(2) 0.007(2) -0.002(3)
```

C14 0.049(3) 0.042(3) 0.036(2) 0.004(2) -0.001(2) -0.007(2)
C15 0.037(2) 0.033(2) 0.036(2) -0.002(2) 0.0031(19) -0.004(2)
C16 0.038(2) 0.040(3) 0.038(2) -0.003(2) 0.004(2) -0.007(2)
C17 0.035(2) 0.040(3) 0.044(3) -0.004(2) 0.006(2) -0.007(2)
C18 0.035(2) 0.034(3) 0.045(3) -0.001(2) 0.003(2) 0.000(2)
C19 0.033(2) 0.040(3) 0.047(3) -0.001(2) 0.002(2) -0.003(2)
C20 0.044(3) 0.047(3) 0.053(3) 0.003(3) 0.002(2) 0.006(2)
C21 0.049(3) 0.051(3) 0.068(4) 0.002(3) -0.002(3) 0.018(3)
C22 0.052(3) 0.052(3) 0.052(3) 0.011(3) -0.007(3) 0.007(3)
C23 0.041(2) 0.037(3) 0.049(3) 0.008(2) -0.007(2) -0.003(2)
C24 0.040(2) 0.036(3) 0.045(3) 0.008(2) -0.003(2) -0.001(2)
C25 0.050(3) 0.041(3) 0.043(3) 0.009(2) -0.009(2) -0.006(2)
C26 0.052(3) 0.047(3) 0.041(3) 0.012(2) -0.004(2) -0.010(3)
C27 0.039(2) 0.050(3) 0.039(2) 0.009(2) -0.004(2) -0.007(2)
C28 0.036(2) 0.036(3) 0.035(2) 0.002(2) 0.0021(19) -0.004(2)
C29 0.038(2) 0.045(3) 0.039(2) 0.009(2) 0.002(2) 0.000(2)
C30 0.046(3) 0.037(3) 0.041(2) 0.007(2) 0.005(2) 0.007(2)
C31 0.048(3) 0.032(3) 0.039(2) 0.001(2) 0.002(2) -0.003(2)
C32 0.049(3) 0.034(3) 0.044(3) 0.002(2) 0.002(2) 0.003(2)
C33 0.082(4) 0.039(3) 0.046(3) 0.006(3) -0.005(3) -0.006(3)
C34 0.093(5) 0.038(3) 0.062(4) -0.005(3) 0.000(3) -0.018(3)
C35 0.076(4) 0.047(3) 0.048(3) -0.006(3) -0.009(3) -0.010(3)
C36 0.050(3) 0.040(3) 0.045(3) -0.006(2) -0.002(2) -0.004(2)
C37 0.049(3) 0.041(3) 0.045(3) -0.004(2) -0.004(2) -0.005(2)
C38 0.053(3) 0.049(3) 0.060(3) 0.002(3) -0.015(3) -0.008(3)
C39 0.057(3) 0.049(3) 0.065(3) 0.016(3) -0.024(3) -0.004(3)
C40 0.050(3) 0.040(3) 0.044(3) -0.001(2) -0.004(2) 0.000(2)
C41 0.050(3) 0.048(3) 0.038(3) 0.003(2) -0.004(2) -0.003(3)
C42 0.062(3) 0.066(4) 0.042(3) -0.019(3) 0.009(2) -0.016(3)
C43 0.075(4) 0.097(5) 0.086(5) -0.045(4) 0.018(4) 0.007(4)
C44 0.053(3) 0.065(4) 0.054(3) -0.021(3) 0.003(3) -0.021(3)
C45 0.066(4) 0.120(6) 0.073(4) -0.017(4) -0.024(3) -0.029(4)
C46 0.058(3) 0.074(4) 0.064(4) -0.017(3) 0.006(3) 0.010(3)
C47 0.068(4) 0.140(7) 0.078(4) -0.007(5) 0.032(4) -0.007(5)
C48 0.076(4) 0.066(4) 0.054(3) -0.015(3) 0.001(3) 0.001(3)
C49 0.240(12) 0.075(6) 0.101(6) -0.030(5) -0.005(7) -0.014(7)
C50 0.049(3) 0.069(4) 0.048(3) 0.011(3) 0.009(2) 0.000(3)
C51 0.094(5) 0.069(4) 0.087(5) 0.037(4) 0.000(4) -0.024(4)
C52 0.039(3) 0.061(4) 0.059(3) 0.003(3) 0.010(2) 0.001(3)
C53 0.063(4) 0.093(5) 0.076(4) -0.016(4) 0.019(3) 0.015(4)
C54 0.089(4) 0.044(3) 0.068(4) 0.010(3) 0.006(3) -0.006(3)
C55 0.157(8) 0.092(6) 0.085(5) 0.030(5) -0.015(5) 0.055(6)
C56 0.093(5) 0.065(4) 0.069(4) 0.017(3) 0.025(4) -0.013(4)
C57 0.127(8) 0.142(9) 0.140(8) 0.030(7) 0.062(7) -0.025(7)
C58 0.050(3) 0.058(3) 0.047(3) 0.009(3) -0.008(2) 0.001(3)
C59 0.052(3) 0.113(6) 0.082(4) 0.025(4) -0.016(3) -0.006(4)
C60 0.063(3) 0.047(3) 0.054(3) 0.005(3) 0.000(3) 0.009(3)
C61 0.112(5) 0.048(4) 0.064(4) 0.015(3) 0.002(4) 0.005(4)
C62 0.062(4) 0.070(4) 0.117(6) 0.026(4) -0.036(4) -0.019(3)
C63 0.165(8) 0.095(6) 0.101(6) 0.004(5) -0.057(6) -0.068(6)

```
C64 0.122(10) 0.054(5) 0.41(3) 0.021(9) -0.147(13) -0.002(5)
C65 0.108(10) 0.49(4) 0.154(12) -0.109(19) -0.020(8) -0.040(16)
C67 0.046(3) 0.047(3) 0.058(3) -0.001(3) 0.000(3) -0.002(3)
C69 0.037(3) 0.061(4) 0.062(3) -0.016(3) 0.003(3) -0.005(3)
C71 0.054(3) 0.040(3) 0.055(3) 0.002(3) 0.000(3) -0.006(3)
C72 0.176(10) 0.134(9) 0.132(8) -0.022(7) -0.090(8) 0.022(8)
C73 0.130(7) 0.076(6) 0.082(5) -0.010(5) -0.017(5) 0.039(6)
C74 0.080(4) 0.064(4) 0.075(4) 0.005(3) 0.003(3) -0.008(3)
C75 0.081(5) 0.059(4) 0.080(5) -0.005(4) -0.009(4) 0.003(4)
C66A 0.049(5) 0.062(5) 0.047(6) -0.001(4) 0.003(4) 0.007(4)
C68A 0.050(4) 0.041(7) 0.053(6) 0.001(5) 0.004(4) -0.005(4)
C70A 0.057(4) 0.061(5) 0.057(5) 0.007(4) -0.001(3) -0.014(3)
C66B 0.047(18) 0.10(3) 0.06(3) 0.01(2) 0.022(19) 0.006(18)
C68B 0.042(9) 0.035(13) 0.065(15) 0.005(12) 0.011(9) 0.005(8)
Cl1 0.0665(10) 0.1054(15) 0.1200(15) -0.0367(12) -0.0351(10) 0.0243(10)
Cl2 0.0965(13) 0.0689(11) 0.0962(13) 0.0192(10) -0.0043(10) 0.0155(10)
Cl3 0.0528(8) 0.0749(11) 0.1438(17) -0.0420(11) 0.0257(10) -0.0105(8)
Cl4 0.0512(8) 0.1129(15) 0.1021(13) -0.0506(12) 0.0026(8) -0.0135(9)
Cl5 0.0787(11) 0.0648(11) 0.1178(14) 0.0368(10) 0.0063(10) -0.0069(9)
Cl6 0.0635(10) 0.0870(13) 0.1493(18) 0.0169(13) 0.0312(11) 0.0087(9)
Cl9 0.0870(13) 0.1201(18) 0.1319(18) 0.0251(15) 0.0176(12) -0.0095(12)
Cl10 0.1167(16) 0.1249(19) 0.1307(18) 0.0639(15) -0.0421(14) -0.0365(14)
Cl7A 0.248(5) 0.087(2) 0.156(3) 0.012(2) -0.129(4) -0.023(3)
Cl8A 0.0789(19) 0.123(3) 0.210(5) -0.032(3) -0.048(2) 0.019(2)
Cl7B 0.183(13) 0.91(7) 0.26(2) -0.40(3) -0.110(13) 0.21(3)
Cl8B 0.45(3) 0.123(9) 0.222(15) 0.124(11) -0.254(18) -0.119(13)
N1 0.041(2) 0.038(2) 0.032(2) -0.0113(19) -0.0020(17) -0.0054(19)
N2 0.033(2) 0.053(3) 0.040(2) -0.0032(19) 0.0033(18) -0.008(2)
N3 0.042(2) 0.051(3) 0.038(2) -0.007(2) 0.0049(19) 0.002(2)
N4 0.0326(18) 0.037(2) 0.039(2) 0.0042(17) 0.0068(16) -0.0036(17)
N5 0.035(2) 0.035(2) 0.051(3) 0.006(2) 0.0016(19) 0.008(2)
N6 0.047(2) 0.041(3) 0.046(2) 0.013(2) 0.006(2) -0.009(2)
N7 0.043(2) 0.034(2) 0.037(2) 0.0032(17) -0.0050(17) 0.0001(18)
N8 0.051(2) 0.030(2) 0.044(2) 0.000(2) -0.0022(19) -0.007(2)
N9 0.045(2) 0.046(3) 0.039(2) 0.0000(19) -0.0070(19) -0.007(2)
O1 0.0479(19) 0.057(2) 0.052(2) -0.0074(19) 0.0031(16) -0.0029(18)
O2 0.062(2) 0.068(3) 0.059(2) -0.018(2) -0.0072(19) 0.009(2)
O3 0.0437(19) 0.064(3) 0.073(3) 0.010(2) 0.0048(18) -0.0040(19)
O4 0.082(3) 0.055(2) 0.069(3) -0.001(2) 0.019(2) -0.005(2)
O5 0.058(2) 0.052(2) 0.063(2) -0.0024(19) -0.0129(19) -0.0031(19)
O6 0.063(2) 0.084(3) 0.072(3) 0.025(2) -0.018(2) -0.026(2)
O7 0.152(6) 0.129(5) 0.150(6) 0.050(5) -0.084(5) -0.033(5)
O8 0.138(6) 0.121(6) 0.147(6) -0.026(5) -0.027(4) 0.048(5)
O9 0.122(4) 0.076(4) 0.105(4) 0.009(3) -0.002(3) 0.000(3)
O10 0.183(7) 0.115(5) 0.142(6) 0.032(4) 0.063(5) 0.015(5)

_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 C28 1.501(6) . ?
C1 C15 1.503(6) . ?
C1 C2 1.504(6) . ?
C2 N1 1.356(6) . ?
C2 C3 1.373(6) . ?
C3 C4 1.408(6) . ?
C3 C42 1.502(6) . ?
C4 C5 1.392(6) . ?
C4 C44 1.505(7) . ?
C5 N1 1.371(6) . ?
C5 C6 1.440(6) . ?
C6 N2 1.347(6) . ?
C6 C7 1.386(6) . ?
C7 C8 1.375(7) . ?
C8 C9 1.367(7) . ?
C9 C10 1.382(6) . ?
C10 N2 1.365(6) . ?
C10 C11 1.433(6) . ?
C11 N3 1.377(6) . ?
C11 C12 1.386(7) . ?
C12 C13 1.407(7) . ?
C12 C46 1.506(7) . ?
C13 C14 1.378(7) . ?
C13 C48 1.509(7) . ?
C14 N3 1.357(6) . ?
C14 C41 1.513(7) . ?
C15 N4 1.356(5) . ?
C15 C16 1.382(6) . ?
C16 C17 1.409(7) . ?
C16 C50 1.504(7) . ?
C17 C18 1.383(6) . ?
C17 C52 1.513(6) . ?
C18 N4 1.383(5) . ?
C18 C19 1.431(6) . ?
C19 N5 1.362(6) . ?
C19 C20 1.381(7) . ?
C20 C21 1.371(7) . ?
```

C21 C22 1.371(8) . ?
C22 C23 1.376(7) . ?
C23 N5 1.355(6) . ?
C23 C24 1.448(7) . ?
C24 N6 1.374(6) . ?
C24 C25 1.379(6) . ?
C25 C26 1.412(7) . ?
C25 C54 1.490(7) . ?
C26 C27 1.376(7) . ?
C26 C56 1.512(7) . ?
C27 N6 1.356(6) . ?
C27 C41 1.500(7) . ?
C28 N7 1.357(5) . ?
C28 C29 1.380(6) . ?
C29 C30 1.407(7) . ?
C29 C58 1.513(6) . ?
C30 C31 1.383(6) . ?
C30 C60 1.504(7) . ?
C31 N7 1.374(6) . ?
C31 C32 1.449(6) . ?
C32 N8 1.357(6) . ?
C32 C33 1.379(7) . ?
C33 C34 1.368(8) . ?
C34 C35 1.374(8) . ?
C35 C36 1.378(7) . ?
C36 N8 1.354(6) . ?
C36 C37 1.446(7) . ?
C37 N9 1.377(6) . ?
C37 C38 1.386(7) . ?
C38 C39 1.410(7) . ?
C38 C62 1.513(8) . ?
C39 C40 1.373(7) . ?
C39 C64 1.693(16) . ?
C40 N9 1.357(6) . ?
C40 C41 1.506(7) . ?
C42 C43 1.513(9) . ?
C44 C45 1.520(8) . ?
C46 C47 1.518(9) . ?
C48 C49 1.491(10) . ?
C50 C51 1.520(8) . ?
C52 C53 1.519(8) . ?
C54 C55 1.515(9) . ?
C56 C57 1.464(10) . ?
C58 C59 1.523(7) . ?
C60 C61 1.518(8) . ?
C62 C63 1.518(11) . ?
C64 C65 1.151(19) . ?
C67 O2 1.219(6) . ?
C67 O1 1.243(6) . ?
C67 C66A 1.558(9) . ?

```
C67 C66B 1.62(4) . ?
C69 O3 1.232(6) . ?
C69 O4 1.241(6) . ?
C69 C68B 1.56(2) . ?
C69 C68A 1.583(11) . ?
C71 O5 1.214(6) . ?
C71 O6 1.244(6) . ?
C71 C70B 1.55(4) . ?
C71 C70A 1.549(8) . ?
C72 Cl8B 1.455(13) . ?
C72 Cl7B 1.47(2) . ?
C72 C73 1.539(12) . ?
C72 Cl8A 1.691(11) . ?
C72 Cl7A 1.698(12) . ?
C73 O7 1.167(9) . ?
C73 O8 1.207(10) . ?
C74 C75 1.529(9) . ?
C74 Cl9 1.739(7) . ?
C74 Cl10 1.750(7) . ?
C75 O10 1.184(9) . ?
C75 O9 1.244(8) . ?
C66A Cl1 1.725(7) . ?
C66A Cl2 1.771(9) . ?
C68A Cl3 1.698(8) . ?
C68A Cl4 1.747(9) . ?
C70A Cl5 1.731(7) . ?
C70A Cl6 1.773(7) . ?
C66B Cl2 1.66(4) . ?
C66B Cl1 1.72(4) . ?
C68B Cl4 1.683(18) . ?
C68B Cl3 1.735(18) . ?
C70B Cl6 1.69(4) . ?
C70B Cl5 1.73(5) . ?
Cl7A Cl7B 2.34(4) . ?
Cl8A Cl8B 1.51(3) . ?
Cl7B Cl8B 1.23(3) . ?

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
C28 C1 C15 112.9(4) . . ?
C28 C1 C2 112.3(4) . . ?
C15 C1 C2 112.5(4) . . ?
N1 C2 C3 108.3(4) . . ?
N1 C2 C1 122.1(4) . . ?
```

```
C3 C2 C1 129.5(4) . . ?
C2 C3 C4 107.7(4) . . ?
C2 C3 C42 125.9(4) . . ?
C4 C3 C42 126.3(4) . . ?
C5 C4 C3 106.9(4) . . ?
C5 C4 C44 127.8(4) . . ?
C3 C4 C44 125.3(4) . . ?
N1 C5 C4 107.4(4) . . ?
N1 C5 C6 121.3(4) . . ?
C4 C5 C6 130.8(4) . . ?
N2 C6 C7 117.3(4) . . ?
N2 C6 C5 117.9(4) . . ?
C7 C6 C5 124.8(4) . . ?
C8 C7 C6 119.4(5) . . ?
C9 C8 C7 121.3(5) . . ?
C8 C9 C10 120.2(5) . . ?
N2 C10 C9 116.5(4) . . ?
N2 C10 C11 118.3(4) . . ?
C9 C10 C11 125.3(4) . . ?
N3 C11 C12 107.5(4) . . ?
N3 C11 C10 121.2(4) . . ?
C12 C11 C10 131.2(4) . . ?
C11 C12 C13 107.3(4) . . ?
C11 C12 C46 127.0(5) . . ?
C13 C12 C46 125.6(5) . . ?
C14 C13 C12 107.4(4) . . ?
C14 C13 C48 125.5(5) . . ?
C12 C13 C48 127.0(5) . . ?
N3 C14 C13 108.3(4) . . ?
N3 C14 C41 121.8(4) . . ?
C13 C14 C41 129.8(4) . . ?
N4 C15 C16 108.3(4) . . ?
N4 C15 C1 122.5(4) . . ?
C16 C15 C1 129.2(4) . . ?
C15 C16 C17 107.3(4) . . ?
C15 C16 C50 126.0(4) . . ?
C17 C16 C50 126.5(4) . . ?
C18 C17 C16 107.5(4) . . ?
C18 C17 C52 128.1(4) . . ?
C16 C17 C52 124.4(4) . . ?
C17 C18 N4 107.4(4) . . ?
C17 C18 C19 131.0(4) . . ?
N4 C18 C19 121.4(4) . . ?
N5 C19 C20 116.4(4) . . ?
N5 C19 C18 118.5(4) . . ?
C20 C19 C18 125.1(4) . . ?
C21 C20 C19 120.0(5) . . ?
C22 C21 C20 121.0(5) . . ?
C21 C22 C23 120.2(5) . . ?
N5 C23 C22 116.6(5) . . ?
```

```
N5 C23 C24 117.8(4) . . ?
C22 C23 C24 125.6(4) . . ?
N6 C24 C25 107.9(4) . . ?
N6 C24 C23 120.7(4) . . ?
C25 C24 C23 131.0(5) . . ?
C24 C25 C26 106.9(4) . . ?
C24 C25 C54 127.5(5) . . ?
C26 C25 C54 125.6(5) . . ?
C27 C26 C25 107.6(4) . . ?
C27 C26 C56 125.0(5) . . ?
C25 C26 C56 127.4(5) . . ?
N6 C27 C26 108.1(5) . . ?
N6 C27 C41 121.5(4) . . ?
C26 C27 C41 130.3(4) . . ?
N7 C28 C29 108.1(4) . . ?
N7 C28 C1 121.9(4) . . ?
C29 C28 C1 130.0(4) . . ?
C28 C29 C30 107.8(4) . . ?
C28 C29 C58 125.0(5) . . ?
C30 C29 C58 127.0(4) . . ?
C31 C30 C29 106.7(4) . . ?
C31 C30 C60 126.9(5) . . ?
C29 C30 C60 126.4(4) . . ?
N7 C31 C30 108.2(4) . . ?
N7 C31 C32 120.3(4) . . ?
C30 C31 C32 131.3(4) . . ?
N8 C32 C33 117.2(5) . . ?
N8 C32 C31 117.8(4) . . ?
C33 C32 C31 124.9(5) . . ?
C34 C33 C32 119.5(5) . . ?
C33 C34 C35 121.3(6) . . ?
C34 C35 C36 120.0(5) . . ?
N8 C36 C35 116.7(5) . . ?
N8 C36 C37 118.1(4) . . ?
C35 C36 C37 125.2(5) . . ?
N9 C37 C38 107.9(4) . . ?
N9 C37 C36 120.4(4) . . ?
C38 C37 C36 131.4(5) . . ?
C37 C38 C39 106.7(4) . . ?
C37 C38 C62 127.1(5) . . ?
C39 C38 C62 126.1(5) . . ?
C40 C39 C38 107.8(4) . . ?
C40 C39 C64 122.1(6) . . ?
C38 C39 C64 128.2(6) . . ?
N9 C40 C39 108.4(4) . . ?
N9 C40 C41 121.6(4) . . ?
C39 C40 C41 129.9(5) . . ?
C27 C41 C40 112.0(4) . . ?
C27 C41 C14 110.5(4) . . ?
C40 C41 C14 113.6(4) . . ?
```

```
C3 C42 C43 113.3(5) . . ?
C4 C44 C45 113.8(5) . . ?
C12 C46 C47 113.6(5) . . ?
C49 C48 C13 113.3(6) . . ?
C16 C50 C51 112.6(5) . . ?
C17 C52 C53 113.4(4) . . ?
C25 C54 C55 113.1(5) . . ?
C57 C56 C26 115.2(6) . . ?
C29 C58 C59 113.6(4) . . ?
C30 C60 C61 113.2(5) . . ?
C38 C62 C63 113.1(6) . . ?
C65 C64 C39 101.2(17) . . ?
O2 C67 O1 127.6(5) . . ?
O2 C67 C66A 110.2(5) . . ?
O1 C67 C66A 122.1(5) . . ?
O2 C67 C66B 130.4(16) . . ?
O1 C67 C66B 97.2(18) . . ?
C66A C67 C66B 34.9(15) . . ?
O3 C69 O4 125.9(5) . . ?
O3 C69 C68B 97.8(12) . . ?
O4 C69 C68B 133.8(10) . . ?
O3 C69 C68A 126.7(6) . . ?
O4 C69 C68A 107.2(7) . . ?
C68B C69 C68A 35.3(8) . . ?
O5 C71 O6 126.1(5) . . ?
O5 C71 C70B 94(2) . . ?
O6 C71 C70B 135.3(18) . . ?
O5 C71 C70A 123.4(5) . . ?
O6 C71 C70A 110.5(5) . . ?
C70B C71 C70A 36(2) . . ?
Cl8B C72 Cl7B 49.6(13) . . ?
Cl8B C72 C73 123.8(11) . . ?
Cl7B C72 C73 114.3(12) . . ?
Cl8B C72 Cl8A 56.9(11) . . ?
Cl7B C72 Cl8A 105.7(12) . . ?
C73 C72 Cl8A 113.2(7) . . ?
Cl8B C72 Cl7A 119.4(7) . . ?
Cl7B C72 Cl7A 95.0(16) . . ?
C73 C72 Cl7A 115.0(7) . . ?
Cl8A C72 Cl7A 112.0(7) . . ?
O7 C73 O8 125.9(10) . . ?
O7 C73 C72 116.5(9) . . ?
O8 C73 C72 117.2(9) . . ?
C75 C74 Cl9 113.9(5) . . ?
C75 C74 Cl10 107.5(5) . . ?
Cl9 C74 Cl10 112.1(4) . . ?
O10 C75 O9 127.1(8) . . ?
O10 C75 C74 117.2(7) . . ?
O9 C75 C74 115.5(7) . . ?
C67 C66A Cl1 114.2(5) . . ?
```

```
C67 C66A Cl2 108.0(6) . . ?
Cl1 C66A Cl2 111.5(5) . . ?
C69 C68A Cl3 113.1(6) . . ?
C69 C68A Cl4 108.2(7) . . ?
Cl3 C68A Cl4 112.7(6) . . ?
C71 C70A Cl5 113.2(5) . . ?
C71 C70A Cl6 108.0(5) . . ?
Cl5 C70A Cl6 110.7(4) . . ?
C67 C66B Cl2 111(3) . . ?
C67 C66B Cl1 112(2) . . ?
Cl2 C66B Cl1 118(2) . . ?
C69 C68B Cl4 112.6(13) . . ?
C69 C68B Cl3 112.3(14) . . ?
Cl4 C68B Cl3 114.1(12) . . ?
C71 C70B Cl6 112(3) . . ?
C71 C70B Cl5 113(3) . . ?
Cl6 C70B Cl5 114(3) . . ?
C66B Cl1 C66A 32.2(16) . . ?
C66B Cl2 C66A 32.1(16) . . ?
C68A Cl3 C68B 32.2(7) . . ?
C68B Cl4 C68A 32.2(8) . . ?
C70A Cl5 C70B 32.4(18) . . ?
C70B Cl6 C70A 32.3(19) . . ?
C72 Cl7A Cl7B 38.8(6) . . ?
Cl8B Cl8A C72 53.7(6) . . ?
Cl8B Cl7B C72 64.3(10) . . ?
Cl8B Cl7B Cl7A 94.2(19) . . ?
C72 Cl7B Cl7A 46.2(12) . . ?
Cl7B Cl8B C72 66.0(14) . . ?
Cl7B Cl8B Cl8A 134(2) . . ?
C72 Cl8B Cl8A 69.4(10) . . ?
C2 N1 C5 109.7(4) . . ?
C6 N2 C10 125.4(4) . . ?
C14 N3 C11 109.4(4) . . ?
C15 N4 C18 109.5(4) . . ?
C23 N5 C19 125.7(4) . . ?
C27 N6 C24 109.4(4) . . ?
C28 N7 C31 109.2(4) . . ?
C36 N8 C32 125.3(4) . . ?
C40 N9 C37 109.1(4) . . ?

_diffrn_measured_fraction_theta_max    0.855
_diffrn_reflns_theta_full              27.32
_diffrn_measured_fraction_theta_full   0.855
_refine_diff_density_max    0.621
_refine_diff_density_min   -0.591
_refine_diff_density_rms    0.060

#===END
```