Controlled translocation of palladium(II) within a 22 ring atom macrocyclic ligand.

Michael G. Burgess, M. Naveed Zafar, Stephen T. Horner, George R. Clark,

and L. James Wright*

School of Chemical Sciences, University of Auckland , Private Bag 92019, Auckland 1142, New Zealand.

SUPPLEMENTARY INFORMATION

S2 Crystallographic data for **H_4L**, [PdCl(H_3L)] (**3a)**, [Pd(*N*-(3-aminopropyl)caprolactam)(H_2L)] **(5)** and [Pd(OH_2)(H_2L)] (**6)** in CIF format.

Crystallographic data for **H_4L**, [PdCl(H_3L)] (**3a)**, [Pd(*N*-(3-aminopropyl)caprolactam)(H_2L)] **(5)** and [Pd(OH_2)(H_2L)] (**6)** in CIF format.

1. Compound **H_4L.**

```
data_H4L

_audit_creation_method     SHELXL-97

_publ_section_title
;
;
_publ_section_abstract        ' Abstract  '
_publ_section_comment         ' Comment  '

_publ_section_related_literature
;

;

_publ_requested_journal  'Journal'



_publ_contact_letter
;
Please consider this CIF for publication. I certify that this
contibution is the
original work of those listed as authors; that it has not been
published
before (in any language or medium) and is not being considered for
publication
elsewhere; that all authors concur with and are aware of the
submission; that
all workers involved in the study are listed as authors or given
proper credit
in the acknowledgements; that I have obtained permission for and
acknowledged
the source of any excerpts from other copyright works; and that to
the best of
my knowledge the paper contains no statements which are libellous,
unlawful or
in any way actionable. All coauthors have made significant
scientific
contributions to the work reported, including the ideas and their
execution,
and share responsibility and accountability for the results.
;


_publ_contact_author_name        'George R. Clark'
_publ_contact_author_address
```

```
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;
_publ_contact_author_email       'g.clark@auckland.ac.nz'
_publ_contact_author_phone       '0064 9 3737599 Ext 88294'
_publ_contact_author_fax         '0064 9 3737422'

loop_
_publ_author_name
_publ_author_address


'Burgess, Michael G.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Zafar, M. Naveed'
;
Chemistry Department
University of Gujrat
H-H campus
Gujrat
Pakistan
;

'Horner, Stephen T.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Clark, George R.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Wright, L. James.'
;
```

Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

_publ_section_exptl_refinement
;

The X-ray data was “squeezed” by the method of Sluis and Spek (1990) to allow for the presence of disordered solvent of crystallization which could not be satisfactorily modelled. The SQUEEZE results showed that the total potential accessible void volume was 1553.4 Å3 (24.4% of the crystal unit cell volume). The estimated missing electron count per cell of 538 corresponds to 134 electrons per asymmetric unit. The two possible molecules of crystallization are n-hexane and dioxane, both of which happen to have an identical electron count of 48. Hence it is not possible to make a definitive determination of the solvent of crystallization, other than to note that both the void volume and electron count are consistent with the unit cell containing 12 solvent molecules (either n-hexane or dioxane or both) in addition to the 8 molecules of H4L.

The hydrogens were placed in calculated positions
and refined using the riding
model under the conditions:. C~aryl~-H~aryl~=0.93, C~methyl~-H~methyl~=0.96,
C~methylene~-H~methylene~ = 0.97, N-H = 0.86\%A, U~iso~(H) = 1.2U~eq~(C) except for the methyl hydrogens where
U~iso~(H) =
1.5 x U~eq~(C).

;

_publ_section_figure_captions
;
Fig. 1: Structure showing 50 % probability displacement ellipsoids for non-hydrogen atoms of Molecule A (Burnett & Johnson, 1996).

Fig. 1: Structure showing 50 % probability displacement ellipsoids for non-hydrogen atoms of Molecule B (Burnett & Johnson, 1996).

;

_publ_section_acknowledgements
;
No acknowledgements
;

```
_publ_section_references

;


Burnett, M.N. and Johnson, C.K. (1996). ORTEP-III: Report ORNL-6895.
Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G.M. (1997). <i>SADABS</i>. Univ. of G\"ottingen,
Germany.

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


Bruker (1995). <i>SHELXTL</i> (Version 5), <i>SMART</i> (Version
4.050) and
<i>SAINT</i> (Version 4.050). Bruker AXS Inc., Madison, Wisconsin,
USA.


Sluis, P. v.d. and Spek, A.L., Acta Crystallographica, (1990), A46,
194-201.
;




data_H4L;

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          'C27 H30 N8 O4'
_chemical_formula_sum
 'C27 H30 N8 O4'
_chemical_formula_weight          530.59

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            Monoclinic
_symmetry_space_group_name_H-M    P21/c
_symmetry_space_group_name_Hall -P_2ybc
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                    27.2057(4)
_cell_length_b                    24.9559(1)
_cell_length_c                    9.3707(2)
_cell_angle_alpha                 90.00
_cell_angle_beta                  91.032(1)
_cell_angle_gamma                 90.00
_cell_volume                      6361.14(17)
_cell_formula_units_Z             8
_cell_measurement_temperature     87(2)
_cell_measurement_reflns_used     8192
_cell_measurement_theta_min       1.63
_cell_measurement_theta_max       25.00

_exptl_crystal_description        plate
_exptl_crystal_colour             colourless
_exptl_crystal_size_max           0.32
_exptl_crystal_size_mid           0.26
_exptl_crystal_size_min           0.08
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.108
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              2240
_exptl_absorpt_coefficient_mu     0.077


_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_process_details   'SADABS; Sheldrick, 1997'


_exptl_absorpt_correction_T_min   0.874580
_exptl_absorpt_correction_T_max   0.979856

_exptl_special_details
;
 ?
;

_diffrn_ambient_temperature       87(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    'Siemens SMART CCD'
_diffrn_measurement_method         'Area detector \w scans'
_diffrn_standards_decay_%          0



_diffrn_detector_area_resol_mean   ?
_diffrn_standards_number           0
_diffrn_standards_interval_count   0
_diffrn_standards_interval_time    0
_diffrn_reflns_number              35496
_diffrn_reflns_av_R_equivalents    0.0384
_diffrn_reflns_av_sigmaI/netI      0.0802
_diffrn_reflns_limit_h_min         -32
_diffrn_reflns_limit_h_max         32
_diffrn_reflns_limit_k_min         0
_diffrn_reflns_limit_k_max         29
_diffrn_reflns_limit_l_min         0
_diffrn_reflns_limit_l_max         11
_diffrn_reflns_theta_min           1.63
_diffrn_reflns_theta_max           25.00
_reflns_number_total               11179
_reflns_number_gt                  6619
_reflns_threshold_expression       I>2\s(I)



_computing_data_collection         'SMART (Bruker, 1995)'
_computing_cell_refinement         'SAINT (Bruker, 1995)'
_computing_data_reduction          'SAINT'
_computing_structure_solution      'SHELXS97 (Sheldrick, 2008)'
_computing_structure_refinement    'SHELXL97 (Sheldrick, 2008)'
_computing_molecular_graphics      'Ortep-III (Burnett & Johnson,
1996)'
_computing_publication_material    'SHELXTL (Sheldrick, 2008)'



_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^)+(0.0851P)^2^+1.4208P] 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_extinction_coef        ?
_refine_ls_number_reflns          11179
_refine_ls_number_parameters      705
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.1138
_refine_ls_R_factor_gt            0.0679
_refine_ls_wR_factor_ref          0.1832
_refine_ls_wR_factor_gt           0.1660
_refine_ls_goodness_of_fit_ref    1.032
_refine_ls_restrained_S_all       1.032
_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
O1A O -0.07791(9) 0.40459(9) 0.4068(2) 0.0389(6) Uani 1 1 d . . .
O2A O 0.05901(8) 0.21330(8) 0.7838(3) 0.0410(6) Uani 1 1 d . . .
O3A O 0.01968(7) 0.60881(8) 1.0792(2) 0.0256(5) Uani 1 1 d . . .
O4A O 0.21432(8) 0.41273(9) 1.2856(2) 0.0387(6) Uani 1 1 d . . .
N1A N -0.04377(9) 0.42610(9) 0.6256(3) 0.0240(6) Uani 1 1 d . . .
H1A H -0.0319 0.4120 0.7026 0.029 Uiso 1 1 calc R . .
N2A N -0.00930(9) 0.32286(9) 0.6445(3) 0.0261(6) Uani 1 1 d . . .
N3A N 0.06097(9) 0.30215(9) 0.8423(3) 0.0278(6) Uani 1 1 d . . .
H3A H 0.0466 0.3322 0.8251 0.033 Uiso 1 1 calc R . .
```

```
N4A N -0.03335(8) 0.50507(9) 0.7484(3) 0.0235(6) Uani 1 1 d . . .
N5A N 0.10730(8) 0.35261(10) 0.9953(3) 0.0268(6) Uani 1 1 d . . .
N6A N -0.02472(8) 0.58343(9) 0.8879(3) 0.0230(6) Uani 1 1 d . . .
H6A H -0.0476 0.6034 0.9212 0.028 Uiso 1 1 calc R . .
N7A N 0.14912(8) 0.41439(10) 1.1285(3) 0.0276(6) Uani 1 1 d . . .
H7A H 0.1277 0.4358 1.0911 0.033 Uiso 1 1 calc R . .
N8A N 0.14278(8) 0.51502(10) 0.9766(3) 0.0243(6) Uani 1 1 d . . .
C1A C -0.05665(11) 0.39138(12) 0.5189(3) 0.0294(7) Uani 1 1 d . . .
C2A C -0.04395(11) 0.33404(12) 0.5444(3) 0.0272(7) Uani 1 1 d . . .
C3A C -0.06756(12) 0.29447(12) 0.4653(4) 0.0324(8) Uani 1 1 d . . .
H3A1 H -0.0911 0.3035 0.3964 0.039 Uiso 1 1 calc R . .
C4A C -0.05573(12) 0.24158(13) 0.4898(4) 0.0366(8) Uani 1 1 d . . .
H4A H -0.0716 0.2145 0.4387 0.044 Uiso 1 1 calc R . .
C5A C -0.02030(12) 0.22945(13) 0.5905(4) 0.0372(8) Uani 1 1 d . . .
H5A H -0.0117 0.1940 0.6092 0.045 Uiso 1 1 calc R . .
C6A C 0.00241(11) 0.27114(12) 0.6639(4) 0.0301(8) Uani 1 1 d . . .
C7A C 0.04336(11) 0.25902(12) 0.7704(4) 0.0298(7) Uani 1 1 d . . .
C8A C -0.04761(10) 0.48208(11) 0.6239(3) 0.0228(7) Uani 1 1 d . . .
C9A C -0.06455(10) 0.51087(12) 0.5055(3) 0.0253(7) Uani 1 1 d . . .
H9A H -0.0744 0.4937 0.4217 0.030 Uiso 1 1 calc R . .
C10A C -0.06609(11) 0.56605(12) 0.5180(4) 0.0317(8) Uani 1 1 d . . .
H10A H -0.0768 0.5866 0.4407 0.038 Uiso 1 1 calc R . .
C11A C -0.05209(11) 0.59074(12) 0.6419(3) 0.0272(7) Uani 1 1 d . . .
H11A H -0.0532 0.6278 0.6511 0.033 Uiso 1 1 calc R . .
C12A C -0.03609(10) 0.55845(11) 0.7536(3) 0.0223(7) Uani 1 1 d . . .
C13A C 0.10015(11) 0.30333(12) 0.9419(3) 0.0287(7) Uani 1 1 d . . .
C14A C 0.12886(12) 0.25956(13) 0.9811(4) 0.0406(9) Uani 1 1 d . . .
H14A H 0.1232 0.2256 0.9433 0.049 Uiso 1 1 calc R . .
C15A C 0.16655(12) 0.26914(14) 1.0801(4) 0.0456(10) Uani 1 1 d . . .
H15A H 0.1866 0.2409 1.1092 0.055 Uiso 1 1 calc R . .
C16A C 0.17485(12) 0.31905(13) 1.1355(4) 0.0366(8) Uani 1 1 d . . .
H16A H 0.1999 0.3252 1.2022 0.044 Uiso 1 1 calc R . .
C17A C 0.14447(11) 0.36016(12) 1.0886(3) 0.0287(7) Uani 1 1 d . . .
C18A C 0.01670(10) 0.58033(10) 0.9708(3) 0.0198(6) Uani 1 1 d . . .
C19A C 0.05702(10) 0.54339(11) 0.9267(3) 0.0240(7) Uani 1 1 d . . .
H19A H 0.0599 0.5447 0.8237 0.029 Uiso 1 1 calc R . .
H19B H 0.0484 0.5070 0.9527 0.029 Uiso 1 1 calc R . .
C20A C 0.10621(10) 0.55745(11) 0.9949(3) 0.0251(7) Uani 1 1 d . . .
H20A H 0.1183 0.5903 0.9529 0.030 Uiso 1 1 calc R . .
H20B H 0.1019 0.5638 1.0961 0.030 Uiso 1 1 calc R . .
C21A C 0.18298(10) 0.43755(12) 1.2186(3) 0.0254(7) Uani 1 1 d . . .
C22A C 0.17903(11) 0.49824(13) 1.2202(3) 0.0304(8) Uani 1 1 d . . .
H22A H 0.2037 0.5125 1.2857 0.036 Uiso 1 1 calc R . .
H22B H 0.1470 0.5083 1.2554 0.036 Uiso 1 1 calc R . .
C23A C 0.18587(10) 0.52361(12) 1.0729(3) 0.0270(7) Uani 1 1 d . . .
H23A H 0.1914 0.5618 1.0843 0.032 Uiso 1 1 calc R . .
H23B H 0.2148 0.5084 1.0296 0.032 Uiso 1 1 calc R . .
C24A C 0.15860(11) 0.50869(12) 0.8273(3) 0.0285(7) Uani 1 1 d . . .
H24A H 0.1890 0.5283 0.8139 0.034 Uiso 1 1 calc R . .
H24B H 0.1338 0.5238 0.7634 0.034 Uiso 1 1 calc R . .
C25A C 0.16643(11) 0.45009(12) 0.7901(3) 0.0295(7) Uani 1 1 d . . .
H25A H 0.1880 0.4344 0.8623 0.035 Uiso 1 1 calc R . .
H25B H 0.1350 0.4318 0.7950 0.035 Uiso 1 1 calc R . .
C26A C 0.18801(12) 0.43915(13) 0.6451(4) 0.0329(8) Uani 1 1 d . . .
```

```
H26A H 0.2204 0.4552 0.6407 0.040 Uiso 1 1 calc R . .
H26B H 0.1674 0.4556 0.5718 0.040 Uiso 1 1 calc R . .
C27A C 0.19196(14) 0.37923(14) 0.6158(4) 0.0454(9) Uani 1 1 d . . .
H27A H 0.2123 0.3628 0.6882 0.068 Uiso 1 1 calc R . .
H27B H 0.2062 0.3737 0.5241 0.068 Uiso 1 1 calc R . .
H27C H 0.1598 0.3635 0.6168 0.068 Uiso 1 1 calc R . .
O1B O 0.43829(8) 0.79058(8) 0.6367(2) 0.0379(6) Uani 1 1 d . . .
O2B O 0.56326(8) 0.61125(8) 1.0884(2) 0.0339(5) Uani 1 1 d . . .
O3B O 0.29196(8) 0.57061(10) 0.1742(3) 0.0477(7) Uani 1 1 d . . .
O4B O 0.48674(7) 0.39187(7) 0.4334(2) 0.0242(5) Uani 1 1 d . . .
N1B N 0.43117(9) 0.69949(10) 0.6175(3) 0.0300(6) Uani 1 1 d . . .
H1B H 0.4430 0.6703 0.6527 0.036 Uiso 1 1 calc R . .
N2B N 0.49975(9) 0.68506(9) 0.8228(3) 0.0260(6) Uani 1 1 d . . .
N3B N 0.53437(8) 0.58278(9) 0.8691(3) 0.0233(6) Uani 1 1 d . . .
H3B H 0.5202 0.5939 0.7919 0.028 Uiso 1 1 calc R . .
N4B N 0.38582(9) 0.64183(10) 0.4813(3) 0.0284(6) Uani 1 1 d . . .
N5B N 0.53034(8) 0.50131(9) 0.7575(2) 0.0204(5) Uani 1 1 d . . .
N6B N 0.34402(9) 0.57529(10) 0.3665(3) 0.0311(6) Uani 1 1 d . . .
H6B H 0.3592 0.5554 0.4278 0.037 Uiso 1 1 calc R . .
N7B N 0.52882(8) 0.42101(9) 0.6256(3) 0.0248(6) Uani 1 1 d . . .
H7B H 0.5521 0.4006 0.5967 0.030 Uiso 1 1 calc R . .
N8B N 0.35824(8) 0.47857(10) 0.5199(3) 0.0258(6) Uani 1 1 d . . .
C1B C 0.45046(11) 0.74524(12) 0.6719(3) 0.0296(7) Uani 1 1 d . . .
C2B C 0.48935(11) 0.73610(12) 0.7864(3) 0.0289(7) Uani 1 1 d . . .
C3B C 0.51235(12) 0.77998(12) 0.8497(4) 0.0327(8) Uani 1 1 d . . .
H3B1 H 0.5047 0.8146 0.8202 0.039 Uiso 1 1 calc R . .
C4B C 0.54693(12) 0.77121(12) 0.9577(4) 0.0342(8) Uani 1 1 d . . .
H4B H 0.5627 0.7999 1.0024 0.041 Uiso 1 1 calc R . .
C5B C 0.55758(12) 0.71920(12) 0.9979(4) 0.0334(8) Uani 1 1 d . . .
H5B H 0.5803 0.7120 1.0707 0.040 Uiso 1 1 calc R . .
C6B C 0.53328(11) 0.67762(11) 0.9260(3) 0.0259(7) Uani 1 1 d . . .
C7B C 0.54501(11) 0.62100(12) 0.9707(3) 0.0265(7) Uani 1 1 d . . .
C8B C 0.39464(11) 0.69319(12) 0.5118(3) 0.0293(8) Uani 1 1 d . . .
C9B C 0.37033(12) 0.73529(13) 0.4445(4) 0.0414(9) Uani 1 1 d . . .
H9B H 0.3773 0.7708 0.4677 0.050 Uiso 1 1 calc R . .
C10B C 0.33528(13) 0.72225(14) 0.3412(4) 0.0466(10) Uani 1 1 d . . .
H10B H 0.3188 0.7495 0.2924 0.056 Uiso 1 1 calc R . .
C11B C 0.32441(12) 0.66933(14) 0.3096(4) 0.0444(10) Uani 1 1 d . . .
H11B H 0.3007 0.6603 0.2411 0.053 Uiso 1 1 calc R . .
C12B C 0.35059(11) 0.63038(13) 0.3847(4) 0.0322(8) Uani 1 1 d . . .
C13B C 0.54401(10) 0.52808(11) 0.8773(3) 0.0203(6) Uani 1 1 d . . .
C14B C 0.56705(10) 0.50312(12) 0.9942(3) 0.0234(7) Uani 1 1 d . . .
H14B H 0.5760 0.5226 1.0752 0.028 Uiso 1 1 calc R . .
C15B C 0.57610(11) 0.44875(12) 0.9858(3) 0.0278(7) Uani 1 1 d . . .
H15B H 0.5915 0.4312 1.0619 0.033 Uiso 1 1 calc R . .
C16B C 0.56238(10) 0.42040(11) 0.8647(3) 0.0243(7) Uani 1 1 d . . .
H16B H 0.5682 0.3838 0.8574 0.029 Uiso 1 1 calc R . .
C17B C 0.53955(10) 0.44867(11) 0.7543(3) 0.0219(7) Uani 1 1 d . . .
C18B C 0.31681(11) 0.54840(13) 0.2648(4) 0.0336(8) Uani 1 1 d . . .
C19B C 0.32154(11) 0.48830(13) 0.2755(3) 0.0297(7) Uani 1 1 d . . .
H19C H 0.3535 0.4780 0.2402 0.036 Uiso 1 1 calc R . .
H19D H 0.2968 0.4721 0.2134 0.036 Uiso 1 1 calc R . .
C20B C 0.31603(11) 0.46547(13) 0.4253(3) 0.0305(8) Uani 1 1 d . . .
H20C H 0.2862 0.4796 0.4664 0.037 Uiso 1 1 calc R . .
```

```
H20D H 0.3127 0.4268 0.4191 0.037 Uiso 1 1 calc R . .
C21B C 0.48804(10) 0.42121(11) 0.5401(3) 0.0227(7) Uani 1 1 d . . .
C22B C 0.44500(10) 0.45587(11) 0.5812(3) 0.0250(7) Uani 1 1 d . . .
H22C H 0.4527 0.4931 0.5625 0.030 Uiso 1 1 calc R . .
H22D H 0.4394 0.4521 0.6826 0.030 Uiso 1 1 calc R . .
C23B C 0.39848(10) 0.44046(12) 0.4982(3) 0.0241(7) Uani 1 1 d . . .
H23C H 0.4057 0.4388 0.3973 0.029 Uiso 1 1 calc R . .
H23D H 0.3881 0.4051 0.5280 0.029 Uiso 1 1 calc R . .
C24B C 0.34284(11) 0.48266(13) 0.6709(3) 0.0293(7) Uani 1 1 d . . .
H24C H 0.3152 0.5071 0.6750 0.035 Uiso 1 1 calc R . .
H24D H 0.3696 0.4986 0.7260 0.035 Uiso 1 1 calc R . .
C25B C 0.32837(11) 0.43044(13) 0.7425(3) 0.0348(8) Uani 1 1 d . . .
H25C H 0.3571 0.4077 0.7531 0.042 Uiso 1 1 calc R . .
H25D H 0.3046 0.4118 0.6824 0.042 Uiso 1 1 calc R . .
C26B C 0.30641(13) 0.44030(15) 0.8888(4) 0.0421(9) Uani 1 1 d . . .
H26C H 0.2766 0.4613 0.8772 0.050 Uiso 1 1 calc R . .
H26D H 0.3295 0.4610 0.9465 0.050 Uiso 1 1 calc R . .
C27B C 0.29432(15) 0.38812(16) 0.9669(4) 0.0544(11) Uani 1 1 d . . .
H27D H 0.2716 0.3674 0.9100 0.082 Uiso 1 1 calc R . .
H27E H 0.2799 0.3964 1.0570 0.082 Uiso 1 1 calc R . .
H27F H 0.3239 0.3679 0.9827 0.082 Uiso 1 1 calc R . .

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
O1A 0.0542(15) 0.0347(13) 0.0272(13) -0.0041(11) -0.0153(11)
0.0011(11)
O2A 0.0518(15) 0.0191(12) 0.0522(17) -0.0017(11) 0.0011(12)
0.0055(10)
O3A 0.0294(12) 0.0239(11) 0.0235(12) -0.0005(10) -0.0011(9)
0.0010(9)
O4A 0.0341(13) 0.0446(14) 0.0370(14) 0.0081(12) -0.0101(11)
0.0041(11)
N1A 0.0314(14) 0.0215(13) 0.0190(14) -0.0012(11) -0.0016(11) -
0.0010(10)
N2A 0.0295(14) 0.0253(14) 0.0235(15) -0.0018(12) 0.0027(11)
0.0004(11)
N3A 0.0304(15) 0.0163(13) 0.0366(16) -0.0002(12) 0.0016(12)
0.0020(10)
N4A 0.0224(13) 0.0270(14) 0.0211(14) -0.0023(12) 0.0016(11) -
0.0013(10)
N5A 0.0235(14) 0.0252(14) 0.0319(16) 0.0027(12) 0.0039(12)
0.0043(11)
N6A 0.0218(13) 0.0232(13) 0.0240(14) -0.0053(11) 0.0006(11)
0.0049(10)
N7A 0.0204(13) 0.0293(15) 0.0329(16) 0.0030(12) -0.0031(11)
0.0047(10)
N8A 0.0198(13) 0.0309(14) 0.0221(14) -0.0025(12) 0.0000(11)
0.0017(10)
```

C1A 0.0307(18) 0.0331(18) 0.0244(18) -0.0057(15) 0.0018(14) -
0.0024(14)
C2A 0.0284(17) 0.0247(16) 0.0289(19) -0.0064(14) 0.0075(14) -
0.0028(13)
C3A 0.0368(19) 0.0323(19) 0.0282(19) -0.0115(15) 0.0032(15) -
0.0046(14)
C4A 0.042(2) 0.0332(19) 0.034(2) -0.0114(16) 0.0091(16) -0.0096(15)
C5A 0.046(2) 0.0243(17) 0.042(2) -0.0050(16) 0.0075(17) -0.0030(15)
C6A 0.0362(19) 0.0221(17) 0.0325(19) -0.0049(15) 0.0124(15) -
0.0044(13)
C7A 0.0333(18) 0.0220(17) 0.034(2) -0.0012(15) 0.0096(15) -
0.0003(14)
C8A 0.0225(16) 0.0259(16) 0.0200(16) -0.0055(14) 0.0035(13)
0.0023(12)
C9A 0.0274(17) 0.0323(18) 0.0160(16) -0.0013(14) -0.0067(13)
0.0020(13)
C10A 0.0323(18) 0.0337(19) 0.0289(19) 0.0062(16) -0.0055(14)
0.0074(14)
C11A 0.0280(17) 0.0216(16) 0.0318(19) -0.0008(14) -0.0033(14)
0.0026(13)
C12A 0.0203(15) 0.0258(17) 0.0208(16) -0.0051(13) -0.0016(12) -
0.0006(12)
C13A 0.0276(17) 0.0273(17) 0.0314(19) 0.0079(15) 0.0072(14)
0.0033(13)
C14A 0.041(2) 0.0287(18) 0.052(2) 0.0017(17) 0.0028(17) 0.0039(15)
C15A 0.032(2) 0.039(2) 0.065(3) 0.014(2) -0.0088(18) 0.0087(16)
C16A 0.0349(19) 0.0311(19) 0.044(2) 0.0057(17) -0.0087(16)
0.0045(14)
C17A 0.0223(16) 0.0318(18) 0.0320(19) 0.0032(15) 0.0047(14)
0.0015(13)
C18A 0.0217(15) 0.0144(14) 0.0234(17) 0.0033(13) 0.0045(13) -
0.0024(11)
C19A 0.0281(17) 0.0227(15) 0.0211(16) 0.0021(13) 0.0009(13)
0.0038(12)
C20A 0.0289(17) 0.0219(16) 0.0246(17) 0.0013(13) -0.0012(13)
0.0004(12)
C21A 0.0203(16) 0.0361(18) 0.0199(17) 0.0020(15) 0.0019(13)
0.0002(13)
C22A 0.0237(17) 0.044(2) 0.0230(18) 0.0010(16) -0.0003(13) -
0.0031(14)
C23A 0.0195(16) 0.0318(17) 0.0298(18) 0.0009(15) 0.0016(13) -
0.0004(12)
C24A 0.0217(16) 0.0376(18) 0.0261(18) 0.0047(15) 0.0015(13)
0.0008(13)
C25A 0.0287(17) 0.0382(19) 0.0217(17) -0.0047(15) 0.0041(14) -
0.0009(14)
C26A 0.0292(18) 0.0385(19) 0.0312(19) 0.0010(16) 0.0026(14)
0.0038(14)
C27A 0.053(2) 0.048(2) 0.035(2) -0.0015(18) 0.0002(18) 0.0095(18)
O1B 0.0482(15) 0.0242(12) 0.0411(15) -0.0014(11) -0.0041(11)
0.0056(10)
O2B 0.0466(14) 0.0324(12) 0.0224(12) -0.0033(10) -0.0070(10) -
0.0026(10)

O3B 0.0378(14) 0.0502(15) 0.0541(17) 0.0128(14) -0.0232(12) -
0.0084(11)
O4B 0.0308(12) 0.0247(11) 0.0172(11) -0.0043(10) -0.0006(9)
0.0012(9)
N1B 0.0329(15) 0.0224(14) 0.0346(16) 0.0000(12) -0.0056(12)
0.0074(11)
N2B 0.0304(15) 0.0245(14) 0.0231(14) -0.0058(12) 0.0036(11) -
0.0001(11)
N3B 0.0275(14) 0.0219(13) 0.0203(14) -0.0014(11) -0.0011(11) -
0.0002(10)
N4B 0.0225(14) 0.0302(15) 0.0325(16) -0.0005(13) -0.0009(12)
0.0020(11)
N5B 0.0234(13) 0.0184(13) 0.0193(14) -0.0026(11) -0.0022(10) -
0.0008(10)
N6B 0.0295(15) 0.0329(15) 0.0306(16) 0.0015(13) -0.0078(12)
0.0018(12)
N7B 0.0233(13) 0.0233(13) 0.0277(15) -0.0033(12) -0.0022(11)
0.0029(10)
N8B 0.0232(14) 0.0309(14) 0.0231(14) -0.0049(12) -0.0048(11) -
0.0015(10)
C1B 0.0384(19) 0.0194(17) 0.0310(19) -0.0066(15) 0.0039(15)
0.0047(13)
C2B 0.0337(18) 0.0246(17) 0.0286(19) -0.0050(14) 0.0041(14)
0.0047(13)
C3B 0.042(2) 0.0218(16) 0.034(2) -0.0046(15) 0.0017(16) 0.0019(14)
C4B 0.046(2) 0.0262(18) 0.030(2) -0.0091(15) -0.0016(16) -0.0046(15)
C5B 0.043(2) 0.0276(18) 0.029(2) -0.0057(15) 0.0002(16) 0.0003(14)
C6B 0.0322(18) 0.0226(16) 0.0230(17) -0.0037(14) 0.0051(14) -
0.0039(13)
C7B 0.0311(17) 0.0251(16) 0.0235(18) -0.0086(14) 0.0028(14) -
0.0008(13)
C8B 0.0241(17) 0.0340(19) 0.0297(19) -0.0014(15) -0.0012(14)
0.0045(13)
C9B 0.038(2) 0.0290(19) 0.057(2) 0.0037(18) -0.0067(18) 0.0052(15)
C10B 0.042(2) 0.038(2) 0.060(3) 0.0105(19) -0.0140(19) 0.0060(16)
C11B 0.034(2) 0.044(2) 0.054(2) 0.0115(19) -0.0169(17) -0.0051(16)
C12B 0.0247(17) 0.0334(19) 0.039(2) 0.0037(16) -0.0005(15)
0.0026(14)
C13B 0.0193(15) 0.0204(15) 0.0213(16) 0.0025(13) -0.0004(12) -
0.0021(11)
C14B 0.0263(16) 0.0290(17) 0.0149(16) -0.0038(13) -0.0021(12) -
0.0040(12)
C15B 0.0291(17) 0.0279(17) 0.0263(18) 0.0030(14) -0.0022(14) -
0.0008(13)
C16B 0.0301(17) 0.0177(15) 0.0250(17) -0.0010(13) -0.0015(13) -
0.0018(12)
C17B 0.0187(15) 0.0196(15) 0.0274(18) -0.0025(13) 0.0000(13) -
0.0037(11)
C18B 0.0241(17) 0.039(2) 0.037(2) 0.0029(17) -0.0047(15) -0.0038(14)
C19B 0.0201(16) 0.0417(19) 0.0271(18) -0.0060(16) -0.0017(13) -
0.0049(13)
C20B 0.0231(17) 0.0385(19) 0.0298(19) -0.0010(15) -0.0042(14) -
0.0040(14)

```
C21B 0.0262(16) 0.0182(15) 0.0236(17) 0.0005(13) 0.0003(13)
0.0002(12)
C22B 0.0280(17) 0.0250(16) 0.0219(17) -0.0010(14) 0.0006(13) -
0.0030(12)
C23B 0.0222(16) 0.0300(17) 0.0200(16) -0.0049(14) -0.0014(13) -
0.0004(12)
C24B 0.0197(16) 0.0414(19) 0.0268(18) -0.0059(16) -0.0003(13) -
0.0017(13)
C25B 0.0266(17) 0.045(2) 0.033(2) -0.0049(17) 0.0008(15) -0.0003(15)
C26B 0.036(2) 0.065(2) 0.0254(19) -0.0106(18) 0.0079(15) -0.0130(17)
C27B 0.061(3) 0.079(3) 0.024(2) -0.004(2) 0.0145(18) -0.017(2)

_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
O1A C1A 1.235(4) . ?
O2A C7A 1.224(4) . ?
O3A C18A 1.241(3) . ?
O4A C21A 1.219(3) . ?
N1A C1A 1.364(4) . ?
N1A C8A 1.401(4) . ?
N1A H1A 0.8600 . ?
N2A C6A 1.341(4) . ?
N2A C2A 1.347(4) . ?
N3A C7A 1.353(4) . ?
N3A C13A 1.404(4) . ?
N3A H3A 0.8600 . ?
N4A C12A 1.335(4) . ?
N4A C8A 1.350(4) . ?
N5A C17A 1.338(4) . ?
N5A C13A 1.341(4) . ?
N6A C18A 1.359(4) . ?
N6A C12A 1.433(4) . ?
N6A H6A 0.8600 . ?
N7A C21A 1.367(4) . ?
```

```
N7A C17A 1.409(4) . ?
N7A H7A 0.8600 . ?
N8A C20A 1.465(4) . ?
N8A C24A 1.479(4) . ?
N8A C23A 1.482(4) . ?
C1A C2A 1.490(4) . ?
C2A C3A 1.386(4) . ?
C3A C4A 1.377(4) . ?
C3A H3A1 0.9300 . ?
C4A C5A 1.371(5) . ?
C4A H4A 0.9300 . ?
C5A C6A 1.386(4) . ?
C5A H5A 0.9300 . ?
C6A C7A 1.513(5) . ?
C8A C9A 1.394(4) . ?
C9A C10A 1.383(4) . ?
C9A H9A 0.9300 . ?
C10A C11A 1.363(4) . ?
C10A H10A 0.9300 . ?
C11A C12A 1.385(4) . ?
C11A H11A 0.9300 . ?
C13A C14A 1.389(4) . ?
C14A C15A 1.390(5) . ?
C14A H14A 0.9300 . ?
C15A C16A 1.367(5) . ?
C15A H15A 0.9300 . ?
C16A C17A 1.384(4) . ?
C16A H16A 0.9300 . ?
C18A C19A 1.497(4) . ?
C19A C20A 1.514(4) . ?
C19A H19A 0.9700 . ?
C19A H19B 0.9700 . ?
C20A H20A 0.9700 . ?
C20A H20B 0.9700 . ?
C21A C22A 1.518(4) . ?
C22A C23A 1.533(4) . ?
C22A H22A 0.9700 . ?
C22A H22B 0.9700 . ?
C23A H23A 0.9700 . ?
C23A H23B 0.9700 . ?
C24A C25A 1.519(4) . ?
C24A H24A 0.9700 . ?
C24A H24B 0.9700 . ?
C25A C26A 1.515(4) . ?
C25A H25A 0.9700 . ?
C25A H25B 0.9700 . ?
C26A C27A 1.524(4) . ?
C26A H26A 0.9700 . ?
C26A H26B 0.9700 . ?
C27A H27A 0.9600 . ?
C27A H27B 0.9600 . ?
C27A H27C 0.9600 . ?
O1B C1B 1.223(3) . ?
O2B C7B 1.226(4) . ?
```

```
O3B C18B 1.210(4) . ?
O4B C21B 1.239(3) . ?
N1B C1B 1.353(4) . ?
N1B C8B 1.399(4) . ?
N1B H1B 0.8600 . ?
N2B C6B 1.330(4) . ?
N2B C2B 1.347(4) . ?
N3B C7B 1.375(4) . ?
N3B C13B 1.392(3) . ?
N3B H3B 0.8600 . ?
N4B C8B 1.334(4) . ?
N4B C12B 1.337(4) . ?
N5B C17B 1.338(3) . ?
N5B C13B 1.353(4) . ?
N6B C18B 1.372(4) . ?
N6B C12B 1.396(4) . ?
N6B H6B 0.8600 . ?
N7B C21B 1.357(4) . ?
N7B C17B 1.415(4) . ?
N7B H7B 0.8600 . ?
N8B C23B 1.467(4) . ?
N8B C20B 1.475(4) . ?
N8B C24B 1.486(4) . ?
C1B C2B 1.511(4) . ?
C2B C3B 1.389(4) . ?
C3B C4B 1.387(5) . ?
C3B H3B1 0.9300 . ?
C4B C5B 1.381(4) . ?
C4B H4B 0.9300 . ?
C5B C6B 1.397(4) . ?
C5B H5B 0.9300 . ?
C6B C7B 1.506(4) . ?
C8B C9B 1.387(4) . ?
C9B C10B 1.385(5) . ?
C9B H9B 0.9300 . ?
C10B C11B 1.384(5) . ?
C10B H10B 0.9300 . ?
C11B C12B 1.389(4) . ?
C11B H11B 0.9300 . ?
C13B C14B 1.398(4) . ?
C14B C15B 1.382(4) . ?
C14B H14B 0.9300 . ?
C15B C16B 1.383(4) . ?
C15B H15B 0.9300 . ?
C16B C17B 1.389(4) . ?
C16B H16B 0.9300 . ?
C18B C19B 1.509(4) . ?
C19B C20B 1.525(4) . ?
C19B H19C 0.9700 . ?
C19B H19D 0.9700 . ?
C20B H20C 0.9700 . ?
C20B H20D 0.9700 . ?
C21B C22B 1.511(4) . ?
C22B C23B 1.522(4) . ?
```

```
C22B H22C 0.9700 . ?
C22B H22D 0.9700 . ?
C23B H23C 0.9700 . ?
C23B H23D 0.9700 . ?
C24B C25B 1.521(4) . ?
C24B H24C 0.9700 . ?
C24B H24D 0.9700 . ?
C25B C26B 1.525(4) . ?
C25B H25C 0.9700 . ?
C25B H25D 0.9700 . ?
C26B C27B 1.533(5) . ?
C26B H26C 0.9700 . ?
C26B H26D 0.9700 . ?
C27B H27D 0.9600 . ?
C27B H27E 0.9600 . ?
C27B H27F 0.9600 . ?

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
C1A N1A C8A 127.4(3) . . ?
C1A N1A H1A 116.3 . . ?
C8A N1A H1A 116.3 . . ?
C6A N2A C2A 117.1(3) . . ?
C7A N3A C13A 127.2(3) . . ?
C7A N3A H3A 116.4 . . ?
C13A N3A H3A 116.4 . . ?
C12A N4A C8A 116.1(3) . . ?
C17A N5A C13A 118.4(3) . . ?
C18A N6A C12A 129.8(2) . . ?
C18A N6A H6A 115.1 . . ?
C12A N6A H6A 115.1 . . ?
C21A N7A C17A 128.8(3) . . ?
C21A N7A H7A 115.6 . . ?
C17A N7A H7A 115.6 . . ?
C20A N8A C24A 113.5(2) . . ?
C20A N8A C23A 110.8(2) . . ?
C24A N8A C23A 110.5(2) . . ?
O1A C1A N1A 124.3(3) . . ?
O1A C1A C2A 119.8(3) . . ?
N1A C1A C2A 115.9(3) . . ?
N2A C2A C3A 122.5(3) . . ?
N2A C2A C1A 117.9(3) . . ?
C3A C2A C1A 119.6(3) . . ?
C4A C3A C2A 119.3(3) . . ?
C4A C3A H3A1 120.4 . . ?
C2A C3A H3A1 120.4 . . ?
C5A C4A C3A 119.1(3) . . ?
C5A C4A H4A 120.5 . . ?
```

```
C3A C4A H4A 120.5 . . ?
C4A C5A C6A 118.5(3) . . ?
C4A C5A H5A 120.7 . . ?
C6A C5A H5A 120.7 . . ?
N2A C6A C5A 123.6(3) . . ?
N2A C6A C7A 116.9(3) . . ?
C5A C6A C7A 119.6(3) . . ?
O2A C7A N3A 124.8(3) . . ?
O2A C7A C6A 120.3(3) . . ?
N3A C7A C6A 114.8(3) . . ?
N4A C8A C9A 123.7(3) . . ?
N4A C8A N1A 113.2(3) . . ?
C9A C8A N1A 123.1(3) . . ?
C10A C9A C8A 117.1(3) . . ?
C10A C9A H9A 121.4 . . ?
C8A C9A H9A 121.4 . . ?
C11A C10A C9A 120.9(3) . . ?
C11A C10A H10A 119.5 . . ?
C9A C10A H10A 119.5 . . ?
C10A C11A C12A 117.4(3) . . ?
C10A C11A H11A 121.3 . . ?
C12A C11A H11A 121.3 . . ?
N4A C12A C11A 124.7(3) . . ?
N4A C12A N6A 117.0(3) . . ?
C11A C12A N6A 118.1(2) . . ?
N5A C13A C14A 123.1(3) . . ?
N5A C13A N3A 111.7(2) . . ?
C14A C13A N3A 125.2(3) . . ?
C13A C14A C15A 116.5(3) . . ?
C13A C14A H14A 121.8 . . ?
C15A C14A H14A 121.8 . . ?
C16A C15A C14A 121.7(3) . . ?
C16A C15A H15A 119.2 . . ?
C14A C15A H15A 119.2 . . ?
C15A C16A C17A 117.5(3) . . ?
C15A C16A H16A 121.3 . . ?
C17A C16A H16A 121.3 . . ?
N5A C17A C16A 122.9(3) . . ?
N5A C17A N7A 111.8(3) . . ?
C16A C17A N7A 125.2(3) . . ?
O3A C18A N6A 118.4(2) . . ?
O3A C18A C19A 122.8(3) . . ?
N6A C18A C19A 118.8(3) . . ?
C18A C19A C20A 112.8(2) . . ?
C18A C19A H19A 109.0 . . ?
C20A C19A H19A 109.0 . . ?
C18A C19A H19B 109.0 . . ?
C20A C19A H19B 109.0 . . ?
H19A C19A H19B 107.8 . . ?
N8A C20A C19A 112.3(2) . . ?
N8A C20A H20A 109.1 . . ?
C19A C20A H20A 109.1 . . ?
N8A C20A H20B 109.1 . . ?
C19A C20A H20B 109.1 . . ?
```

```
H20A C20A H20B 107.9 . . ?
O4A C21A N7A 124.1(3) . . ?
O4A C21A C22A 123.4(3) . . ?
N7A C21A C22A 112.4(2) . . ?
C21A C22A C23A 113.2(3) . . ?
C21A C22A H22A 108.9 . . ?
C23A C22A H22A 108.9 . . ?
C21A C22A H22B 108.9 . . ?
C23A C22A H22B 108.9 . . ?
H22A C22A H22B 107.8 . . ?
N8A C23A C22A 112.4(2) . . ?
N8A C23A H23A 109.1 . . ?
C22A C23A H23A 109.1 . . ?
N8A C23A H23B 109.1 . . ?
C22A C23A H23B 109.1 . . ?
H23A C23A H23B 107.9 . . ?
N8A C24A C25A 111.4(2) . . ?
N8A C24A H24A 109.4 . . ?
C25A C24A H24A 109.4 . . ?
N8A C24A H24B 109.4 . . ?
C25A C24A H24B 109.4 . . ?
H24A C24A H24B 108.0 . . ?
C26A C25A C24A 116.0(3) . . ?
C26A C25A H25A 108.3 . . ?
C24A C25A H25A 108.3 . . ?
C26A C25A H25B 108.3 . . ?
C24A C25A H25B 108.3 . . ?
H25A C25A H25B 107.4 . . ?
C25A C26A C27A 111.6(3) . . ?
C25A C26A H26A 109.3 . . ?
C27A C26A H26A 109.3 . . ?
C25A C26A H26B 109.3 . . ?
C27A C26A H26B 109.3 . . ?
H26A C26A H26B 108.0 . . ?
C26A C27A H27A 109.5 . . ?
C26A C27A H27B 109.5 . . ?
H27A C27A H27B 109.5 . . ?
C26A C27A H27C 109.5 . . ?
H27A C27A H27C 109.5 . . ?
H27B C27A H27C 109.5 . . ?
C1B N1B C8B 128.9(3) . . ?
C1B N1B H1B 115.6 . . ?
C8B N1B H1B 115.6 . . ?
C6B N2B C2B 117.0(3) . . ?
C7B N3B C13B 127.2(3) . . ?
C7B N3B H3B 116.4 . . ?
C13B N3B H3B 116.4 . . ?
C8B N4B C12B 118.3(3) . . ?
C17B N5B C13B 117.1(2) . . ?
C18B N6B C12B 129.3(3) . . ?
C18B N6B H6B 115.4 . . ?
C12B N6B H6B 115.4 . . ?
C21B N7B C17B 130.9(2) . . ?
C21B N7B H7B 114.5 . . ?
```

```
C17B N7B H7B 114.5 . . ?
C23B N8B C20B 110.4(2) . . ?
C23B N8B C24B 113.6(2) . . ?
C20B N8B C24B 111.0(2) . . ?
O1B C1B N1B 125.3(3) . . ?
O1B C1B C2B 121.0(3) . . ?
N1B C1B C2B 113.7(3) . . ?
N2B C2B C3B 123.1(3) . . ?
N2B C2B C1B 117.6(3) . . ?
C3B C2B C1B 119.2(3) . . ?
C4B C3B C2B 118.8(3) . . ?
C4B C3B H3B1 120.6 . . ?
C2B C3B H3B1 120.6 . . ?
C5B C4B C3B 118.9(3) . . ?
C5B C4B H4B 120.5 . . ?
C3B C4B H4B 120.5 . . ?
C4B C5B C6B 118.2(3) . . ?
C4B C5B H5B 120.9 . . ?
C6B C5B H5B 120.9 . . ?
N2B C6B C5B 124.0(3) . . ?
N2B C6B C7B 118.2(3) . . ?
C5B C6B C7B 117.8(3) . . ?
O2B C7B N3B 124.2(3) . . ?
O2B C7B C6B 121.1(3) . . ?
N3B C7B C6B 114.7(3) . . ?
N4B C8B C9B 123.2(3) . . ?
N4B C8B N1B 112.5(3) . . ?
C9B C8B N1B 124.3(3) . . ?
C10B C9B C8B 117.1(3) . . ?
C10B C9B H9B 121.4 . . ?
C8B C9B H9B 121.4 . . ?
C11B C10B C9B 121.0(3) . . ?
C11B C10B H10B 119.5 . . ?
C9B C10B H10B 119.5 . . ?
C10B C11B C12B 117.0(3) . . ?
C10B C11B H11B 121.5 . . ?
C12B C11B H11B 121.5 . . ?
N4B C12B C11B 123.2(3) . . ?
N4B C12B N6B 112.4(3) . . ?
C11B C12B N6B 124.3(3) . . ?
N5B C13B N3B 112.9(2) . . ?
N5B C13B C14B 122.9(3) . . ?
N3B C13B C14B 124.2(3) . . ?
C15B C14B C13B 118.1(3) . . ?
C15B C14B H14B 121.0 . . ?
C13B C14B H14B 121.0 . . ?
C14B C15B C16B 120.2(3) . . ?
C14B C15B H15B 119.9 . . ?
C16B C15B H15B 119.9 . . ?
C15B C16B C17B 117.5(3) . . ?
C15B C16B H16B 121.3 . . ?
C17B C16B H16B 121.3 . . ?
N5B C17B C16B 124.3(3) . . ?
N5B C17B N7B 117.6(3) . . ?
```

```
C16B C17B N7B 118.0(2) . . ?
O3B C18B N6B 123.4(3) . . ?
O3B C18B C19B 123.2(3) . . ?
N6B C18B C19B 113.3(3) . . ?
C18B C19B C20B 115.0(3) . . ?
C18B C19B H19C 108.5 . . ?
C20B C19B H19C 108.5 . . ?
C18B C19B H19D 108.5 . . ?
C20B C19B H19D 108.5 . . ?
H19C C19B H19D 107.5 . . ?
N8B C20B C19B 112.5(2) . . ?
N8B C20B H20C 109.1 . . ?
C19B C20B H20C 109.1 . . ?
N8B C20B H20D 109.1 . . ?
C19B C20B H20D 109.1 . . ?
H20C C20B H20D 107.8 . . ?
O4B C21B N7B 119.0(3) . . ?
O4B C21B C22B 122.2(3) . . ?
N7B C21B C22B 118.7(3) . . ?
C21B C22B C23B 111.5(2) . . ?
C21B C22B H22C 109.3 . . ?
C23B C22B H22C 109.3 . . ?
C21B C22B H22D 109.3 . . ?
C23B C22B H22D 109.3 . . ?
H22C C22B H22D 108.0 . . ?
N8B C23B C22B 112.4(2) . . ?
N8B C23B H23C 109.1 . . ?
C22B C23B H23C 109.1 . . ?
N8B C23B H23D 109.1 . . ?
C22B C23B H23D 109.1 . . ?
H23C C23B H23D 107.9 . . ?
N8B C24B C25B 116.2(3) . . ?
N8B C24B H24C 108.2 . . ?
C25B C24B H24C 108.2 . . ?
N8B C24B H24D 108.2 . . ?
C25B C24B H24D 108.2 . . ?
H24C C24B H24D 107.4 . . ?
C24B C25B C26B 111.5(3) . . ?
C24B C25B H25C 109.3 . . ?
C26B C25B H25C 109.3 . . ?
C24B C25B H25D 109.3 . . ?
C26B C25B H25D 109.3 . . ?
H25C C25B H25D 108.0 . . ?
C25B C26B C27B 112.5(3) . . ?
C25B C26B H26C 109.1 . . ?
C27B C26B H26C 109.1 . . ?
C25B C26B H26D 109.1 . . ?
C27B C26B H26D 109.1 . . ?
H26C C26B H26D 107.8 . . ?
C26B C27B H27D 109.5 . . ?
C26B C27B H27E 109.5 . . ?
H27D C27B H27E 109.5 . . ?
C26B C27B H27F 109.5 . . ?
H27D C27B H27F 109.5 . . ?
```

```
H27E C27B H27F 109.5 . . ?

loop_
 _geom_torsion_atom_site_label_1
 _geom_torsion_atom_site_label_2
 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
 _geom_torsion
 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
C2A C1A N1A C8A 174.2(3) . . . . ?
C1A N1A C8A N4A 177.8(3) . . . . ?
N1A C8A N4A C12A 179.9(2) . . . . ?
C8A N4A C12A N6A 175.4(2) . . . . ?
N4A C12A N6A C18A 56.9(4) . . . . ?
C12A N6A C18A C19A -4.3(4) . . . . ?
N6A C18A C19A C20A 160.0(3) . . . . ?
C18A C19A C20A N8A 167.6(2) . . . . ?
C19A C20A N8A C23A -166.1(2) . . . . ?
C19A C20A N8A C24A 68.9(3) . . . . ?
C20A N8A C24A C25A -142.3(2) . . . . ?
C6A C7A N3A C13A -176.8(3) . . . . ?
C7A N3A C13A N5A -178.7(3) . . . . ?
N3A C13A N5A C17A -178.0(3) . . . . ?
C13A N5A C17A N7A 176.5(3) . . . . ?
N5A C17A N7A C21A -178.6(3) . . . . ?
C17A N7A C21A C22A 173.4(3) . . . . ?
N7A C21A C22A C23A -58.8(3) . . . . ?
C21A C22A C23A N8A 73.3(3) . . . . ?
C22A C23A N8A C20A 87.6(3) . . . . ?
C22A C23A N8A C24A -145.8(3) . . . . ?
C23A N8A C24A C25A 92.5(3) . . . . ?
N8A C24A C25A C26A -173.6(2) . . . . ?
C24A C25A C26A C27A -177.2(3) . . . . ?
C2B C1B N1B C8B 179.6(3) . . . . ?
C1B N1B C8B N4B 177.9(3) . . . . ?
N1B C8B N4B C12B 178.1(3) . . . . ?
C8B N4B C12B N6B -178.0(3) . . . . ?
N4B C12B N6B C18B -170.0(3) . . . . ?
C12B N6B C18B C19B 176.4(3) . . . . ?
N6B C18B C19B C20B 47.4(4) . . . . ?
C18B C19B C20B N8B -70.6(3) . . . . ?
C19B C20B N8B C23B -83.1(3) . . . . ?
C19B C20B N8B C24B 150.1(3) . . . . ?
C20B N8B C24B C25B 69.9(3) . . . . ?
C6B C7B N3B C13B 175.8(3) . . . . ?
C7B N3B C13B N5B -178.1(3) . . . . ?
N3B C13B N5B C17B 178.1(2) . . . . ?
C13B N5B C17B N7B -175.3(2) . . . . ?
N5B C17B N7B C21B -48.4(4) . . . . ?
C17B N7B C21B C22B 0.1(5) . . . . ?
```

```
N7B C21B C22B C23B -164.9(3) . . . . ?
C21B C22B C23B N8B -171.3(2) . . . . ?
C22B C23B N8B C20B 173.6(2) . . . . ?
C22B C23B N8B C24B -61.1(3) . . . . ?
C23B N8B C24B C25B -55.1(3) . . . . ?
N8B C24B C25B C26B -171.9(3) . . . . ?
C24B C25B C26B C27B -176.6(3) . . . . ?

_diffrn_measured_fraction_theta_max    0.998
_diffrn_reflns_theta_full              25.00
_diffrn_measured_fraction_theta_full   0.998
_refine_diff_density_max    0.300
_refine_diff_density_min   -0.315
_refine_diff_density_rms    0.056
```

2. Compound [PdCl(H_3L)] **(3a)**.

```
data_Cpd_3a


data_global
_audit_creation_method     SHELXL-97

_publ_section_title
;
;
_publ_section_abstract        ' Abstract  '
_publ_section_comment         ' Comment  '

_publ_section_related_literature
;
For related literature on the   .

;

_publ_requested_journal          'Journal'


_publ_contact_letter
;
Please consider this CIF for publication. I certify that this
contibution is the
original work of those listed as authors; that it has not been
published
before (in any language or medium) and is not being considered for
publication
elsewhere; that all authors concur with and are aware of the
submission; that
all workers involved in the study are listed as authors or given
proper credit
```

```
in the acknowledgements; that I have obtained permission for and
acknowledged
the source of any excerpts from other copyright works; and that to
the best of
my knowledge the paper contains no statements which are libellous,
unlawful or
in any way actionable. All coauthors have made significant
scientific
contributions to the work reported, including the ideas and their
execution,
and share responsibility and accountability for the results.
;

_publ_contact_author_name        'George R. Clark'
_publ_contact_author_address
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;
_publ_contact_author_email       'g.clark@auckland.ac.nz'
_publ_contact_author_phone       '0064 9 3737599 Ext 88294'
_publ_contact_author_fax         '0064 9 3737422'

loop_
_publ_author_name
_publ_author_address


'Burgess, Michael G.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Zafar, M. Naveed'
;
Chemistry Department
University of Gujrat
H-H campus
Gujrat
Pakistan
;

'Horner, Stephen T.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
```

Auckland
New Zealand
;

'Clark, George R.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Wright, L. James.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

_publ_section_exptl_refinement
;
Hydrogen atoms were placed in calculated positions and refined using the
riding model [C-H 0.93-0.97 \%A), with U~iso~(H) = 1.2 or 1.5 times U~eq~(C).

;

_publ_section_figure_captions
;
Fig. 1: Structure showing 50 % probability displacement ellipsoids for non-hydrogen atoms and hydrogen atoms as arbitary spheres (Burnett
 & Johnson, 1996). The dioxane molecule of crystallisation is not shown.
;

_publ_section_acknowledgements
;
No acknowledgements
;

_publ_section_references

```
;

Burnett, M.N. and Johnson, C.K. (1996). ORTEP-III: Report ORNL-6895.
Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G.M. (1996). <i>SADABS</i>. Univ. of G\"ottingen,
Germany.

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


Siemens (1995). <i>SMART</i> (Version 4.050) and
<i>SAINT</i> (Version 4.050). Siemens Analytical X-ray Instruments
Inc., Madison, Wisconsin, USA.


;
```

```
data_compound 3a

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          'C27 H29 Cl N8 O4 Pd,C4 H8 O2'
_chemical_formula_sum
 'C31 H37 Cl N8 O6 Pd'
_chemical_formula_weight          759.54

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'
 'Pd'  'Pd'  -0.9988   1.0072
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            Monoclinic
_symmetry_space_group_name_H-M    P21/c
_symmetry_space_group_name_Hall    -p_2ybc

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                    15.9930(2)
_cell_length_b                    8.5772(1)
_cell_length_c                    23.3577(2)
_cell_angle_alpha                 90.00
_cell_angle_beta                  105.163(1)
_cell_angle_gamma                 90.00
_cell_volume                      3092.55(6)
_cell_formula_units_Z             4
_cell_measurement_temperature     85(2)
_cell_measurement_reflns_used     8192
_cell_measurement_theta_min       1.94
_cell_measurement_theta_max       25.00

_exptl_crystal_description        needle
_exptl_crystal_colour             yellow
_exptl_crystal_size_max           0.22
_exptl_crystal_size_mid           0.18
_exptl_crystal_size_min           0.12
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.631
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              1560
_exptl_absorpt_coefficient_mu     0.746



_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_process_details   'SADABS; Sheldrick, 1996'

_exptl_absorpt_correction_T_min   0.75340
_exptl_absorpt_correction_T_max   0.90699

_exptl_special_details
;
 ?
;

_diffrn_ambient_temperature       85(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   'Siemens SMART CCD'
_diffrn_measurement_method        'Area detector \w scans'
_diffrn_standards_decay_%         '<2%'



_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          0
_diffrn_standards_interval_count  0
_diffrn_standards_interval_time   0

_diffrn_reflns_number             15898
_diffrn_reflns_av_R_equivalents   0.0456
_diffrn_reflns_av_sigmaI/netI     0.0563
_diffrn_reflns_limit_h_min        -19
_diffrn_reflns_limit_h_max        16
_diffrn_reflns_limit_k_min        -10
_diffrn_reflns_limit_k_max        9
_diffrn_reflns_limit_l_min        -26
_diffrn_reflns_limit_l_max        27
_diffrn_reflns_theta_min          1.94
_diffrn_reflns_theta_max          25.00
_reflns_number_total              5424
_reflns_number_gt                 4259
_reflns_threshold_expression      I>2\s(I)


_computing_data_collection        'SMART (Siemens, 1995)'
_computing_cell_refinement        'SAINT (Siemens, 1995)'
_computing_data_reduction         'SAINT'
_computing_structure_solution     'SHELXS97 (Sheldrick, 2008)'
_computing_structure_refinement   'SHELXL97 (Sheldrick, 2008)'
_computing_molecular_graphics     'Ortep-III (Burnett & Johnson,
1996)'
_computing_publication_material   'SHELXTL (Sheldrick, 2008)'

_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^)+(0.0536P)^2^+14.6616P] 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_extinction_coef        ?
_refine_ls_number_reflns          5424
_refine_ls_number_parameters      420
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0741
_refine_ls_R_factor_gt            0.0519
_refine_ls_wR_factor_ref          0.1305
_refine_ls_wR_factor_gt           0.1202
_refine_ls_goodness_of_fit_ref    1.034
_refine_ls_restrained_S_all       1.034
_refine_ls_shift/su_max           0.017
_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
Pd Pd 0.08338(3) 0.06063(4) 0.134264(17) 0.01668(13) Uani 1 1 d . .
.
Cl Cl 0.22471(8) 0.07387(14) 0.12427(5) 0.0199(3) Uani 1 1 d . . .
O1 O 0.1210(3) -0.3360(4) -0.05910(16) 0.0265(9) Uani 1 1 d . . .
N1 N 0.1207(3) -0.1474(5) 0.00994(19) 0.0213(10) Uani 1 1 d . . .
H1 H 0.1493 -0.0685 0.0277 0.026 Uiso 1 1 calc R . .
C1 C 0.1550(3) -0.2229(6) -0.0298(2) 0.0205(11) Uani 1 1 d . . .
O2 O 0.4354(3) 0.2503(5) -0.0089(2) 0.0363(10) Uani 1 1 d . . .
N2 N 0.2649(3) -0.0184(5) -0.01231(18) 0.0184(9) Uani 1 1 d . . .
C2 C 0.2390(3) -0.1594(6) -0.0358(2) 0.0208(11) Uani 1 1 d . . .
O3 O -0.1668(2) -0.0210(5) 0.15458(16) 0.0264(9) Uani 1 1 d . . .
```

```
N3 N 0.3218(3) 0.2738(5) 0.03288(19) 0.0212(10) Uani 1 1 d . . .
H3 H 0.2778 0.2253 0.0388 0.025 Uiso 1 1 calc R . .
C3 C 0.2866(4) -0.2493(7) -0.0654(3) 0.0296(13) Uani 1 1 d . . .
H3A H 0.2660 -0.3456 -0.0813 0.036 Uiso 1 1 calc R . .
O4 O 0.1060(2) 0.6809(4) 0.17040(16) 0.0237(8) Uani 1 1 d . . .
N4 N 0.0295(3) -0.0959(5) 0.06896(18) 0.0191(9) Uani 1 1 d . . .
C4 C 0.3646(4) -0.1939(8) -0.0707(3) 0.0376(15) Uani 1 1 d . . .
H4 H 0.3990 -0.2536 -0.0888 0.045 Uiso 1 1 calc R . .
N5 N 0.2739(3) 0.4759(5) 0.08045(18) 0.0183(9) Uani 1 1 d . . .
C5 C 0.3909(4) -0.0478(8) -0.0486(3) 0.0331(14) Uani 1 1 d . . .
H5 H 0.4428 -0.0062 -0.0526 0.040 Uiso 1 1 calc R . .
N6 N -0.0357(3) -0.0038(5) 0.1322(2) 0.0220(10) Uani 1 1 d . . .
C6 C 0.3393(3) 0.0362(6) -0.0205(2) 0.0235(12) Uani 1 1 d . . .
N7 N 0.2180(3) 0.6746(5) 0.12995(18) 0.0194(9) Uani 1 1 d . . .
H7 H 0.2170 0.7746 0.1325 0.023 Uiso 1 1 calc R . .
C7 C 0.3700(3) 0.1958(6) 0.0020(2) 0.0237(12) Uani 1 1 d . . .
N8 N 0.0992(3) 0.2053(5) 0.20650(18) 0.0180(9) Uani 1 1 d . . .
C8 C 0.0442(3) -0.1840(6) 0.0251(2) 0.0183(11) Uani 1 1 d . . .
C9 C -0.0149(3) -0.2980(6) -0.0006(2) 0.0231(12) Uani 1 1 d . . .
H9 H -0.0057 -0.3595 -0.0312 0.028 Uiso 1 1 calc R . .
C10 C -0.0883(4) -0.3192(6) 0.0201(2) 0.0249(12) Uani 1 1 d . . .
H10 H -0.1280 -0.3962 0.0031 0.030 Uiso 1 1 calc R . .
C11 C -0.1033(3) -0.2288(6) 0.0649(2) 0.0234(12) Uani 1 1 d . . .
H11 H -0.1520 -0.2436 0.0790 0.028 Uiso 1 1 calc R . .
C12 C -0.0430(3) -0.1157(6) 0.0880(2) 0.0201(11) Uani 1 1 d . . .
C13 C 0.3369(3) 0.4261(6) 0.0561(2) 0.0196(11) Uani 1 1 d . . .
C14 C 0.4079(3) 0.5157(7) 0.0542(2) 0.0250(12) Uani 1 1 d . . .
H14 H 0.4501 0.4775 0.0371 0.030 Uiso 1 1 calc R . .
C15 C 0.4143(4) 0.6635(6) 0.0782(2) 0.0255(12) Uani 1 1 d . . .
H15 H 0.4609 0.7269 0.0768 0.031 Uiso 1 1 calc R . .
C16 C 0.3521(3) 0.7175(6) 0.1044(2) 0.0224(12) Uani 1 1 d . . .
H16 H 0.3562 0.8160 0.1215 0.027 Uiso 1 1 calc R . .
C17 C 0.2828(3) 0.6185(6) 0.1041(2) 0.0188(11) Uani 1 1 d . . .
C18 C -0.0952(3) 0.0416(6) 0.1606(2) 0.0233(12) Uani 1 1 d . . .
C19 C -0.0650(3) 0.1804(6) 0.2004(2) 0.0216(11) Uani 1 1 d . . .
H19A H -0.1022 0.1912 0.2269 0.026 Uiso 1 1 calc R . .
H19B H -0.0721 0.2734 0.1759 0.026 Uiso 1 1 calc R . .
C20 C 0.0287(3) 0.1732(7) 0.2376(2) 0.0233(12) Uani 1 1 d . . .
H20A H 0.0354 0.2474 0.2699 0.028 Uiso 1 1 calc R . .
H20B H 0.0388 0.0702 0.2552 0.028 Uiso 1 1 calc R . .
C21 C 0.1565(3) 0.6015(6) 0.1518(2) 0.0174(11) Uani 1 1 d . . .
C22 C 0.1550(3) 0.4274(6) 0.1536(2) 0.0199(11) Uani 1 1 d . . .
H22A H 0.2126 0.3881 0.1722 0.024 Uiso 1 1 calc R . .
H22B H 0.1369 0.3861 0.1136 0.024 Uiso 1 1 calc R . .
C23 C 0.0921(4) 0.3745(6) 0.1889(2) 0.0210(11) Uani 1 1 d . . .
H23A H 0.1017 0.4374 0.2245 0.025 Uiso 1 1 calc R . .
H23B H 0.0334 0.3944 0.1654 0.025 Uiso 1 1 calc R . .
C24 C 0.1852(3) 0.1812(6) 0.2512(2) 0.0225(11) Uani 1 1 d . . .
H24A H 0.1887 0.2507 0.2845 0.027 Uiso 1 1 calc R . .
H24B H 0.2311 0.2100 0.2331 0.027 Uiso 1 1 calc R . .
C25 C 0.2010(4) 0.0145(6) 0.2745(2) 0.0257(12) Uani 1 1 d . . .
H25A H 0.1715 -0.0018 0.3054 0.031 Uiso 1 1 calc R . .
H25B H 0.1771 -0.0581 0.2426 0.031 Uiso 1 1 calc R . .
C26 C 0.2965(4) -0.0169(8) 0.2990(3) 0.0468(18) Uani 1 1 d . . .
```

```
H26A H 0.3253 -0.0017 0.2677 0.056 Uiso 1 1 calc R . .
H26B H 0.3044 -0.1250 0.3114 0.056 Uiso 1 1 calc R . .
C27 C 0.3394(5) 0.0867(11) 0.3512(4) 0.084(4) Uani 1 1 d . . .
H27A H 0.3388 0.1929 0.3381 0.127 Uiso 1 1 calc R . .
H27B H 0.3982 0.0536 0.3674 0.127 Uiso 1 1 calc R . .
H27C H 0.3083 0.0791 0.3811 0.127 Uiso 1 1 calc R . .
O5 O 0.4796(6) 0.9915(14) 0.2091(4) 0.134(3) Uani 1 1 d . . .
O6 O 0.6493(7) 0.8892(13) 0.2578(5) 0.146(4) Uani 1 1 d . . .
C28 C 0.4990(9) 0.833(2) 0.2443(6) 0.146(7) Uani 1 1 d . . .
H28A H 0.4547 0.8102 0.2646 0.175 Uiso 1 1 calc R . .
H28B H 0.5021 0.7472 0.2178 0.175 Uiso 1 1 calc R . .
C29 C 0.5786(6) 0.8576(14) 0.2846(6) 0.099(4) Uani 1 1 d . . .
H29A H 0.5932 0.7662 0.3097 0.118 Uiso 1 1 calc R . .
H29B H 0.5733 0.9449 0.3098 0.118 Uiso 1 1 calc R . .
C30 C 0.6343(10) 1.0281(15) 0.2216(5) 0.129(6) Uani 1 1 d . . .
H30A H 0.6772 1.0358 0.1990 0.155 Uiso 1 1 calc R . .
H30B H 0.6391 1.1198 0.2465 0.155 Uiso 1 1 calc R . .
C31 C 0.5478(10) 1.0196(19) 0.1814(7) 0.132(5) Uiso 1 1 d . . .
H31A H 0.5360 1.1169 0.1596 0.158 Uiso 1 1 calc R . .
H31B H 0.5472 0.9372 0.1529 0.158 Uiso 1 1 calc R . .

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
Pd 0.0226(2) 0.0119(2) 0.0177(2) -0.00074(17) 0.00917(15) -
0.00052(17)
Cl 0.0243(6) 0.0156(6) 0.0224(6) -0.0012(5) 0.0107(5) -0.0007(5)
O1 0.038(2) 0.018(2) 0.026(2) -0.0045(17) 0.0118(17) -0.0020(17)
N1 0.028(2) 0.014(2) 0.021(2) -0.0010(18) 0.0054(19) 0.0011(18)
C1 0.029(3) 0.016(3) 0.017(3) 0.002(2) 0.007(2) 0.005(2)
O2 0.032(2) 0.035(2) 0.052(3) -0.009(2) 0.029(2) -0.0062(19)
N2 0.023(2) 0.018(2) 0.015(2) 0.0002(17) 0.0062(18) 0.0031(18)
C2 0.025(3) 0.018(3) 0.017(3) 0.001(2) 0.002(2) 0.002(2)
O3 0.024(2) 0.034(2) 0.023(2) 0.0042(17) 0.0104(16) -0.0019(17)
N3 0.021(2) 0.018(2) 0.028(2) -0.0002(19) 0.0126(19) -0.0010(18)
C3 0.033(3) 0.025(3) 0.030(3) -0.010(3) 0.008(3) 0.005(3)
O4 0.036(2) 0.0152(18) 0.025(2) 0.0013(16) 0.0152(17) 0.0015(17)
N4 0.023(2) 0.016(2) 0.017(2) 0.0026(17) 0.0046(18) 0.0000(18)
C4 0.038(4) 0.040(4) 0.037(4) -0.016(3) 0.015(3) 0.005(3)
N5 0.023(2) 0.015(2) 0.019(2) 0.0021(17) 0.0098(18) 0.0022(18)
C5 0.027(3) 0.041(4) 0.035(3) -0.009(3) 0.014(3) 0.004(3)
N6 0.024(2) 0.018(2) 0.026(2) -0.0002(19) 0.0096(19) -0.0032(19)
C6 0.024(3) 0.026(3) 0.022(3) 0.004(2) 0.008(2) 0.008(2)
N7 0.027(2) 0.009(2) 0.026(2) 0.0014(18) 0.0137(19) 0.0010(18)
C7 0.025(3) 0.024(3) 0.024(3) -0.002(2) 0.010(2) 0.005(2)
N8 0.025(2) 0.014(2) 0.018(2) 0.0019(17) 0.0114(18) -0.0018(18)
C8 0.028(3) 0.012(2) 0.015(2) 0.003(2) 0.006(2) 0.003(2)
C9 0.027(3) 0.015(3) 0.026(3) -0.003(2) 0.005(2) 0.003(2)
C10 0.030(3) 0.014(3) 0.027(3) 0.004(2) 0.001(2) -0.004(2)
```

```
C11 0.023(3) 0.021(3) 0.025(3) 0.003(2) 0.003(2) 0.001(2)
C12 0.023(3) 0.014(3) 0.022(3) 0.002(2) 0.004(2) 0.004(2)
C13 0.022(3) 0.017(3) 0.021(3) 0.003(2) 0.009(2) 0.001(2)
C14 0.023(3) 0.026(3) 0.030(3) -0.001(2) 0.015(2) 0.001(2)
C15 0.025(3) 0.018(3) 0.035(3) 0.004(2) 0.012(2) -0.004(2)
C16 0.028(3) 0.014(3) 0.025(3) 0.003(2) 0.007(2) -0.004(2)
C17 0.023(3) 0.019(3) 0.016(3) 0.003(2) 0.008(2) 0.003(2)
C18 0.028(3) 0.022(3) 0.022(3) 0.011(2) 0.012(2) 0.007(2)
C19 0.029(3) 0.021(3) 0.021(3) 0.003(2) 0.016(2) 0.002(2)
C20 0.033(3) 0.021(3) 0.022(3) 0.000(2) 0.017(2) -0.006(2)
C21 0.024(3) 0.016(3) 0.014(2) 0.002(2) 0.007(2) 0.000(2)
C22 0.029(3) 0.015(3) 0.019(3) 0.000(2) 0.012(2) 0.001(2)
C23 0.031(3) 0.011(2) 0.025(3) 0.000(2) 0.014(2) 0.000(2)
C24 0.031(3) 0.017(3) 0.020(3) 0.002(2) 0.008(2) -0.004(2)
C25 0.035(3) 0.019(3) 0.024(3) 0.001(2) 0.009(2) -0.002(2)
C26 0.035(4) 0.045(4) 0.059(5) 0.029(4) 0.009(3) 0.004(3)
C27 0.061(5) 0.078(6) 0.083(6) 0.053(5) -0.036(5) -0.036(5)
O5 0.104(7) 0.170(9) 0.122(7) 0.018(7) 0.019(6) 0.008(7)
O6 0.147(9) 0.161(10) 0.141(8) -0.006(7) 0.058(7) -0.002(8)
C28 0.095(9) 0.24(2) 0.089(9) -0.025(12) -0.009(7) 0.039(12)
C29 0.059(6) 0.099(9) 0.136(10) -0.027(8) 0.023(7) 0.009(6)
C30 0.191(15) 0.091(9) 0.080(8) 0.035(7) -0.011(9) -0.003(9)

_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
Pd N6 1.972(4) . ?
Pd N4 2.045(4) . ?
Pd N8 2.056(4) . ?
Pd Cl 2.3349(13) . ?
O1 C1 1.229(6) . ?
N1 C1 1.359(7) . ?
N1 C8 1.397(7) . ?
N1 H1 0.8600 . ?
C1 C2 1.490(7) . ?
```

```
O2 C7 1.230(7) . ?
N2 C6 1.337(7) . ?
N2 C2 1.348(7) . ?
C2 C3 1.388(8) . ?
O3 C18 1.239(6) . ?
N3 C7 1.362(7) . ?
N3 C13 1.411(7) . ?
N3 H3 0.8600 . ?
C3 C4 1.369(9) . ?
C3 H3A 0.9300 . ?
O4 C21 1.220(6) . ?
N4 C8 1.342(6) . ?
N4 C12 1.358(7) . ?
C4 C5 1.378(9) . ?
C4 H4 0.9300 . ?
N5 C17 1.334(7) . ?
N5 C13 1.349(7) . ?
C5 C6 1.386(8) . ?
C5 H5 0.9300 . ?
N6 C18 1.353(7) . ?
N6 C12 1.392(7) . ?
C6 C7 1.502(8) . ?
N7 C21 1.372(6) . ?
N7 C17 1.412(6) . ?
N7 H7 0.8600 . ?
N8 C23 1.504(6) . ?
N8 C24 1.509(7) . ?
N8 C20 1.517(6) . ?
C8 C9 1.383(7) . ?
C9 C10 1.393(8) . ?
C9 H9 0.9300 . ?
C10 C11 1.374(8) . ?
C10 H10 0.9300 . ?
C11 C12 1.374(7) . ?
C11 H11 0.9300 . ?
C13 C14 1.382(7) . ?
C14 C15 1.380(8) . ?
C14 H14 0.9300 . ?
C15 C16 1.375(8) . ?
C15 H15 0.9300 . ?
C16 C17 1.396(7) . ?
C16 H16 0.9300 . ?
C18 C19 1.510(8) . ?
C19 C20 1.525(7) . ?
C19 H19A 0.9700 . ?
C19 H19B 0.9700 . ?
C20 H20A 0.9700 . ?
C20 H20B 0.9700 . ?
C21 C22 1.495(7) . ?
C22 C23 1.526(7) . ?
C22 H22A 0.9700 . ?
C22 H22B 0.9700 . ?
C23 H23A 0.9700 . ?
C23 H23B 0.9700 . ?
```

```
C24 C25 1.527(7) . ?
C24 H24A 0.9700 . ?
C24 H24B 0.9700 . ?
C25 C26 1.509(8) . ?
C25 H25A 0.9700 . ?
C25 H25B 0.9700 . ?
C26 C27 1.520(12) . ?
C26 H26A 0.9700 . ?
C26 H26B 0.9700 . ?
C27 H27A 0.9600 . ?
C27 H27B 0.9600 . ?
C27 H27C 0.9600 . ?
O5 C31 1.428(16) . ?
O5 C28 1.578(18) . ?
O6 C30 1.445(14) . ?
O6 C29 1.454(13) . ?
C28 C29 1.388(15) . ?
C28 H28A 0.9700 . ?
C28 H28B 0.9700 . ?
C29 H29A 0.9700 . ?
C29 H29B 0.9700 . ?
C30 C31 1.456(17) . ?
C30 H30A 0.9700 . ?
C30 H30B 0.9700 . ?
C31 H31A 0.9700 . ?
C31 H31B 0.9700 . ?

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
N6 Pd N4 65.31(18) . . ?
N6 Pd N8 95.73(17) . . ?
N4 Pd N8 161.01(17) . . ?
N6 Pd Cl 164.88(13) . . ?
N4 Pd Cl 100.78(12) . . ?
N8 Pd Cl 97.95(12) . . ?
C1 N1 C8 127.1(4) . . ?
C1 N1 H1 116.5 . . ?
C8 N1 H1 116.5 . . ?
O1 C1 N1 123.9(5) . . ?
O1 C1 C2 120.9(5) . . ?
N1 C1 C2 115.1(5) . . ?
C6 N2 C2 116.5(4) . . ?
N2 C2 C3 123.4(5) . . ?
N2 C2 C1 118.6(5) . . ?
C3 C2 C1 118.0(5) . . ?
C7 N3 C13 126.5(5) . . ?
C7 N3 H3 116.7 . . ?
C13 N3 H3 116.7 . . ?
```

```
C4 C3 C2 118.9(5) . . ?
C4 C3 H3A 120.6 . . ?
C2 C3 H3A 120.6 . . ?
C8 N4 C12 121.0(4) . . ?
C8 N4 Pd 144.6(4) . . ?
C12 N4 Pd 93.8(3) . . ?
C3 C4 C5 118.6(6) . . ?
C3 C4 H4 120.7 . . ?
C5 C4 H4 120.7 . . ?
C17 N5 C13 117.0(4) . . ?
C4 C5 C6 119.2(6) . . ?
C4 C5 H5 120.4 . . ?
C6 C5 H5 120.4 . . ?
C18 N6 C12 129.0(5) . . ?
C18 N6 Pd 135.0(4) . . ?
C12 N6 Pd 96.0(3) . . ?
N2 C6 C5 123.2(5) . . ?
N2 C6 C7 119.5(5) . . ?
C5 C6 C7 117.3(5) . . ?
C21 N7 C17 132.8(4) . . ?
C21 N7 H7 113.6 . . ?
C17 N7 H7 113.6 . . ?
O2 C7 N3 123.5(5) . . ?
O2 C7 C6 119.5(5) . . ?
N3 C7 C6 116.9(5) . . ?
C23 N8 C24 108.2(4) . . ?
C23 N8 C20 107.1(4) . . ?
C24 N8 C20 107.5(4) . . ?
C23 N8 Pd 111.9(3) . . ?
C24 N8 Pd 112.3(3) . . ?
C20 N8 Pd 109.6(3) . . ?
N4 C8 C9 119.6(5) . . ?
N4 C8 N1 114.0(4) . . ?
C9 C8 N1 126.4(5) . . ?
C8 C9 C10 118.9(5) . . ?
C8 C9 H9 120.6 . . ?
C10 C9 H9 120.6 . . ?
C11 C10 C9 121.5(5) . . ?
C11 C10 H10 119.3 . . ?
C9 C10 H10 119.3 . . ?
C12 C11 C10 116.9(5) . . ?
C12 C11 H11 121.6 . . ?
C10 C11 H11 121.6 . . ?
N4 C12 C11 122.1(5) . . ?
N4 C12 N6 104.1(4) . . ?
C11 C12 N6 133.8(5) . . ?
N5 C13 C14 123.3(5) . . ?
N5 C13 N3 112.6(4) . . ?
C14 C13 N3 124.2(5) . . ?
C15 C14 C13 118.1(5) . . ?
C15 C14 H14 120.9 . . ?
C13 C14 H14 120.9 . . ?
C16 C15 C14 120.3(5) . . ?
C16 C15 H15 119.8 . . ?
```

```
C14 C15 H15 119.8 . . ?
C15 C16 C17 117.3(5) . . ?
C15 C16 H16 121.3 . . ?
C17 C16 H16 121.3 . . ?
N5 C17 C16 123.9(5) . . ?
N5 C17 N7 118.6(5) . . ?
C16 C17 N7 117.5(5) . . ?
O3 C18 N6 124.6(5) . . ?
O3 C18 C19 123.3(5) . . ?
N6 C18 C19 112.1(5) . . ?
C18 C19 C20 115.3(4) . . ?
C18 C19 H19A 108.4 . . ?
C20 C19 H19A 108.4 . . ?
C18 C19 H19B 108.4 . . ?
C20 C19 H19B 108.4 . . ?
H19A C19 H19B 107.5 . . ?
N8 C20 C19 117.5(4) . . ?
N8 C20 H20A 107.9 . . ?
C19 C20 H20A 107.9 . . ?
N8 C20 H20B 107.9 . . ?
C19 C20 H20B 107.9 . . ?
H20A C20 H20B 107.2 . . ?
O4 C21 N7 118.9(5) . . ?
O4 C21 C22 122.0(5) . . ?
N7 C21 C22 119.1(4) . . ?
C21 C22 C23 109.3(4) . . ?
C21 C22 H22A 109.8 . . ?
C23 C22 H22A 109.8 . . ?
C21 C22 H22B 109.8 . . ?
C23 C22 H22B 109.8 . . ?
H22A C22 H22B 108.3 . . ?
N8 C23 C22 114.9(4) . . ?
N8 C23 H23A 108.5 . . ?
C22 C23 H23A 108.5 . . ?
N8 C23 H23B 108.5 . . ?
C22 C23 H23B 108.5 . . ?
H23A C23 H23B 107.5 . . ?
N8 C24 C25 114.2(4) . . ?
N8 C24 H24A 108.7 . . ?
C25 C24 H24A 108.7 . . ?
N8 C24 H24B 108.7 . . ?
C25 C24 H24B 108.7 . . ?
H24A C24 H24B 107.6 . . ?
C26 C25 C24 111.0(5) . . ?
C26 C25 H25A 109.4 . . ?
C24 C25 H25A 109.4 . . ?
C26 C25 H25B 109.4 . . ?
C24 C25 H25B 109.4 . . ?
H25A C25 H25B 108.0 . . ?
C25 C26 C27 113.6(7) . . ?
C25 C26 H26A 108.9 . . ?
C27 C26 H26A 108.9 . . ?
C25 C26 H26B 108.9 . . ?
C27 C26 H26B 108.9 . . ?
```

```
H26A C26 H26B 107.7 . . ?
C26 C27 H27A 109.5 . . ?
C26 C27 H27B 109.5 . . ?
H27A C27 H27B 109.5 . . ?
C26 C27 H27C 109.5 . . ?
H27A C27 H27C 109.5 . . ?
H27B C27 H27C 109.5 . . ?
C31 O5 C28 108.2(11) . . ?
C30 O6 C29 112.1(12) . . ?
C29 C28 O5 104.0(14) . . ?
C29 C28 H28A 111.0 . . ?
O5 C28 H28A 111.0 . . ?
C29 C28 H28B 111.0 . . ?
O5 C28 H28B 111.0 . . ?
H28A C28 H28B 109.0 . . ?
C28 C29 O6 114.6(12) . . ?
C28 C29 H29A 108.6 . . ?
O6 C29 H29A 108.6 . . ?
C28 C29 H29B 108.6 . . ?
O6 C29 H29B 108.6 . . ?
H29A C29 H29B 107.6 . . ?
O6 C30 C31 108.7(13) . . ?
O6 C30 H30A 109.9 . . ?
C31 C30 H30A 109.9 . . ?
O6 C30 H30B 109.9 . . ?
C31 C30 H30B 109.9 . . ?
H30A C30 H30B 108.3 . . ?
O5 C31 C30 115.2(13) . . ?
O5 C31 H31A 108.5 . . ?
C30 C31 H31A 108.5 . . ?
O5 C31 H31B 108.5 . . ?
C30 C31 H31B 108.5 . . ?
H31A C31 H31B 107.5 . . ?

_diffrn_measured_fraction_theta_max    0.995
_diffrn_reflns_theta_full              25.00
_diffrn_measured_fraction_theta_full   0.995
_refine_diff_density_max    1.235
_refine_diff_density_min   -1.071
_refine_diff_density_rms    0.121
```

3. Compound [Pd(*N*-(3-aminopropyl)caprolactam)(H_2L)] **(5).**

```
data_compound_5

data_global
_audit_creation_method     SHELXL-97
```

```
_publ_section_title
;
;
_publ_section_abstract        ' Abstract  '
_publ_section_comment         ' Comment  '

_publ_section_related_literature
;
For related literature on the

;

_publ_requested_journal

_publ_contact_author_name        'George R. Clark'
_publ_contact_author_address
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;
_publ_contact_author_email       'g.clark@auckland.ac.nz'
_publ_contact_author_phone       '0064 9 3737599 Ext 88294'
_publ_contact_author_fax         '0064 9 3737422'

loop_
_publ_author_name
_publ_author_address


'Burgess, Michael G.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Zafar, M. Naveed'
;
Chemistry Department
University of Gujrat
H-H campus
Gujrat
Pakistan
;

'Horner, Stephen T.'
;
Department of Chemistry
The University of Auckland
```

Private Bag 92019
Auckland
New Zealand
;

'Clark, George R.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Wright, L. James.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

_publ_section_exptl_refinement
;
Hydrogen atoms were placed in calculated positions and refined using the
riding model [C-H 0.93-0.97 \%A), with U~iso~(H) = 1.2 or 1.5 times U~eq~(C). The crystals contain a water molecule of crystallization, whose hydrogen atoms were not located. There is high thermal motion in the carbons at the end of the aliphatic chain.
;

_publ_section_figure_captions
;
Fig. 1: Structure showing 50 % probability displacement ellipsoids for non-hydrogen atoms and hydrogen atoms as arbitary spheres (Burnett
 & Johnson, 1996).
;

_publ_section_acknowledgements
;
No acknowledgements
;

_publ_section_references

```
;

Burnett, M.N. and Johnson, C.K. (1996). ORTEP-III: Report ORNL-6895.
Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G.M. (1997). <i>SADABS</i>. Univ. of G\"ottingen,
Germany.

Sheldrick, G.M. (1997). <i>SHELXS97</i> and <i>SHELXL97</i>. Release
97-1,
University of G\"ottingen, Germany.

Siemens (1995). <i>SHELXTL</i> (Version 5), <i>SMART</i> (Version
4.050)
 and <i>SAINT</i> (Version 4.050). Siemens Analytical X-ray
Instruments
Inc., Madison, Wisconsin, USA.


;




data_compound 5

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety     'C36 H46 N10 O5 Pd, H2 O'

_chemical_formula_sum
 'C36 H48 N10 O6 Pd'
_chemical_formula_weight          823.26

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0030   0.0020
 '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'   0.0060   0.0030
```

```
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0110   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Pd'  'Pd'  -0.9990   1.0070
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            Monoclinic
_symmetry_space_group_name_H-M    P21/c
_symmetry_space_group_name_Hall    -P_2ybc

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                    12.2234(4)
_cell_length_b                    15.7777(6)
_cell_length_c                    19.3023(7)
_cell_angle_alpha                 90.00
_cell_angle_beta                  102.824(1)
_cell_angle_gamma                 90.00
_cell_volume                      3629.7(2)
_cell_formula_units_Z             4
_cell_measurement_temperature     87(2)
_cell_measurement_reflns_used     8192
_cell_measurement_theta_min       1.68
_cell_measurement_theta_max       25.02

_exptl_crystal_description        block
_exptl_crystal_colour             yellow
_exptl_crystal_size_max           0.18
_exptl_crystal_size_mid           0.18
_exptl_crystal_size_min           0.10
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.507
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              1712
_exptl_absorpt_coefficient_mu     0.572



_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_process_details   'SADABS; Sheldrick, 1997'


_exptl_absorpt_correction_T_min   0.662482
_exptl_absorpt_correction_T_max   0.925825



_diffrn_ambient_temperature       87(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   'Siemens SMART CCD'
_diffrn_measurement_method        'Area detector \w scans'
_diffrn_standards_decay_%         2


_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          0
_diffrn_standards_interval_count  0
_diffrn_standards_interval_time   0
_diffrn_reflns_number             32058
_diffrn_reflns_av_R_equivalents   0.0693
_diffrn_reflns_av_sigmaI/netI     0.1143
_diffrn_reflns_limit_h_min        -14
_diffrn_reflns_limit_h_max        14
_diffrn_reflns_limit_k_min        -18
_diffrn_reflns_limit_k_max        18
_diffrn_reflns_limit_l_min        -22
_diffrn_reflns_limit_l_max        22
_diffrn_reflns_theta_min          1.68
_diffrn_reflns_theta_max          25.02
_reflns_number_total              6403
_reflns_number_gt                 3900
_reflns_threshold_expression      I>2\s(I)


_computing_data_collection        'SADABS (Sheldrick, 1997)'
_computing_cell_refinement        'SMART (Siemens, 1995)'
_computing_data_reduction         'SAINT (Siemens, 1995)'
_computing_structure_solution     'SHELXS97 (Sheldrick, 1997)'
_computing_structure_refinement   'SHELXL97 (Sheldrick, 1997)'
_computing_molecular_graphics     'Ortep-III (Burnett & Johnson,
1996)'
_computing_publication_material   'SHELXTL (Siemens, 1995)'

_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^)+(0.0757P)^2^+0.0000P] 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_extinction_coef        ?
_refine_ls_number_reflns          6403
_refine_ls_number_parameters      474
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.1276
_refine_ls_R_factor_gt            0.0642
_refine_ls_wR_factor_ref          0.1569
_refine_ls_wR_factor_gt           0.1337
_refine_ls_goodness_of_fit_ref    0.986
_refine_ls_restrained_S_all       0.986
_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
Pd Pd 0.44176(4) 0.49318(3) 0.11158(2) 0.01780(16) Uani 1 1 d . . .
O1 O 0.2910(4) 0.2861(3) -0.0008(2) 0.0288(11) Uani 1 1 d . . .
O2 O 0.7744(4) 0.5373(3) 0.1069(2) 0.0266(11) Uani 1 1 d . . .
O3 O 0.1487(4) 0.6644(3) 0.0785(2) 0.0260(11) Uani 1 1 d . . .
O4 O 0.4288(4) 0.7524(3) 0.3199(3) 0.0446(14) Uani 1 1 d . . .
O5 O 0.5792(6) 0.3755(3) 0.4184(3) 0.066(2) Uani 1 1 d . . .
O6 O 0.2777(11) 0.1642(9) -0.1036(7) 0.199(5) Uiso 1 1 d . . .
N1 N 0.3176(4) 0.4077(3) 0.0719(3) 0.0200(12) Uani 1 1 d . . .
N2 N 0.5242(4) 0.4170(3) 0.0630(3) 0.0191(12) Uani 1 1 d . . .
N3 N 0.5966(4) 0.5545(3) 0.1336(3) 0.0226(12) Uani 1 1 d . . .
N4 N 0.1601(4) 0.4874(3) 0.0819(2) 0.0191(11) Uani 1 1 d . . .
N5 N 0.5423(4) 0.6804(3) 0.1789(3) 0.0219(12) Uani 1 1 d . . .
N6 N 0.0088(4) 0.5749(3) 0.0963(3) 0.0221(13) Uani 1 1 d . . .
```

```
H6 H -0.0579 0.5755 0.1038 0.027 Uiso 1 1 calc R . .
N7 N 0.4660(5) 0.8032(3) 0.2167(3) 0.0316(14) Uani 1 1 d . . .
H7 H 0.4478 0.8400 0.1833 0.038 Uiso 1 1 calc R . .
N8 N 0.1002(5) 0.8468(3) 0.1710(3) 0.0251(13) Uani 1 1 d . . .
N9 N 0.3525(4) 0.5722(3) 0.1630(3) 0.0214(12) Uani 1 1 d . . .
H9A H 0.3872 0.6229 0.1689 0.026 Uiso 1 1 calc R . .
H9B H 0.2841 0.5804 0.1347 0.026 Uiso 1 1 calc R . .
N10 N 0.5447(5) 0.5177(4) 0.4139(3) 0.0370(15) Uani 1 1 d . . .
C1 C 0.3496(5) 0.3435(4) 0.0322(3) 0.0207(15) Uani 1 1 d . . .
C2 C 0.4717(5) 0.3497(4) 0.0289(3) 0.0179(14) Uani 1 1 d . . .
C3 C 0.5273(5) 0.2956(4) -0.0073(3) 0.0230(16) Uani 1 1 d . . .
H3 H 0.4916 0.2479 -0.0301 0.028 Uiso 1 1 calc R . .
C4 C 0.6373(6) 0.3135(4) -0.0090(4) 0.0261(16) Uani 1 1 d . . .
H4 H 0.6767 0.2766 -0.0322 0.031 Uiso 1 1 calc R . .
C5 C 0.6903(5) 0.3863(4) 0.0236(3) 0.0238(15) Uani 1 1 d . . .
H5 H 0.7627 0.4006 0.0202 0.029 Uiso 1 1 calc R . .
C6 C 0.6304(5) 0.4363(4) 0.0612(3) 0.0188(14) Uani 1 1 d . . .
C7 C 0.6762(5) 0.5151(4) 0.1038(3) 0.0216(14) Uani 1 1 d . . .
C8 C 0.2030(5) 0.4083(4) 0.0785(3) 0.0175(14) Uani 1 1 d . . .
C9 C 0.1406(5) 0.3350(4) 0.0823(3) 0.0227(16) Uani 1 1 d . . .
H9 H 0.1707 0.2816 0.0783 0.027 Uiso 1 1 calc R . .
C10 C 0.0322(5) 0.3445(4) 0.0923(3) 0.0223(15) Uani 1 1 d . . .
H10 H -0.0106 0.2966 0.0960 0.027 Uiso 1 1 calc R . .
C11 C -0.0124(6) 0.4232(4) 0.0969(3) 0.0232(15) Uani 1 1 d . . .
H11 H -0.0850 0.4298 0.1036 0.028 Uiso 1 1 calc R . .
C12 C 0.0552(5) 0.4937(4) 0.0911(3) 0.0202(13) Uani 1 1 d . . .
C13 C 0.6286(6) 0.6293(4) 0.1752(3) 0.0247(16) Uani 1 1 d . . .
C14 C 0.7376(6) 0.6486(5) 0.2111(3) 0.0291(17) Uani 1 1 d . . .
H14 H 0.7972 0.6133 0.2079 0.035 Uiso 1 1 calc R . .
C15 C 0.7554(6) 0.7215(5) 0.2516(4) 0.041(2) Uani 1 1 d . . .
H15 H 0.8276 0.7353 0.2761 0.050 Uiso 1 1 calc R . .
C16 C 0.6667(6) 0.7740(5) 0.2562(4) 0.0336(18) Uani 1 1 d . . .
H16 H 0.6773 0.8230 0.2836 0.040 Uiso 1 1 calc R . .
C17 C 0.5626(6) 0.7506(4) 0.2183(3) 0.0253(16) Uani 1 1 d . . .
C18 C 0.0564(6) 0.6533(4) 0.0910(3) 0.0240(16) Uani 1 1 d . . .
C19 C -0.0208(6) 0.7256(4) 0.1026(4) 0.0296(17) Uani 1 1 d . . .
H19A H -0.0431 0.7162 0.1472 0.035 Uiso 1 1 calc R . .
H19B H -0.0881 0.7240 0.0649 0.035 Uiso 1 1 calc R . .
C20 C 0.0321(6) 0.8146(4) 0.1042(3) 0.0268(16) Uani 1 1 d . . .
H20A H 0.0785 0.8151 0.0694 0.032 Uiso 1 1 calc R . .
H20B H -0.0284 0.8547 0.0880 0.032 Uiso 1 1 calc R . .
C21 C 0.4016(6) 0.7970(4) 0.2667(4) 0.0324(18) Uani 1 1 d . . .
C22 C 0.2937(6) 0.8489(4) 0.2487(4) 0.0316(17) Uani 1 1 d . . .
H22A H 0.3073 0.9018 0.2263 0.038 Uiso 1 1 calc R . .
H22B H 0.2691 0.8622 0.2918 0.038 Uiso 1 1 calc R . .
C23 C 0.2027(6) 0.7983(4) 0.1983(4) 0.0295(17) Uani 1 1 d . . .
H23A H 0.2324 0.7786 0.1585 0.035 Uiso 1 1 calc R . .
H23B H 0.1841 0.7488 0.2232 0.035 Uiso 1 1 calc R . .
C24 C 0.0352(6) 0.8602(5) 0.2265(4) 0.0369(19) Uani 1 1 d . . .
H24A H 0.0865 0.8735 0.2711 0.044 Uiso 1 1 calc R . .
H24B H -0.0034 0.8080 0.2330 0.044 Uiso 1 1 calc R . .
C25 C -0.0509(7) 0.9316(5) 0.2076(4) 0.046(2) Uani 1 1 d . . .
H25A H -0.1053 0.9168 0.1646 0.056 Uiso 1 1 calc R . .
H25B H -0.0906 0.9382 0.2455 0.056 Uiso 1 1 calc R . .
```

```
C26 C 0.0054(9) 1.0157(6) 0.1962(7) 0.087(4) Uani 1 1 d . . .
H26A H 0.0678 1.0250 0.2364 0.104 Uiso 1 1 calc R . .
H26B H 0.0362 1.0104 0.1542 0.104 Uiso 1 1 calc R . .
C27 C -0.0684(9) 1.0919(6) 0.1877(6) 0.083(4) Uani 1 1 d . . .
H27A H -0.1345 1.0816 0.1513 0.124 Uiso 1 1 calc R . .
H27B H -0.0287 1.1397 0.1747 0.124 Uiso 1 1 calc R . .
H27C H -0.0896 1.1036 0.2318 0.124 Uiso 1 1 calc R . .
C28 C 0.3375(6) 0.5423(5) 0.2338(4) 0.0350(18) Uani 1 1 d . . .
H28A H 0.2989 0.4882 0.2283 0.042 Uiso 1 1 calc R . .
H28B H 0.2917 0.5825 0.2527 0.042 Uiso 1 1 calc R . .
C29 C 0.4511(6) 0.5327(5) 0.2858(4) 0.0332(18) Uani 1 1 d . . .
H29A H 0.4941 0.4890 0.2687 0.040 Uiso 1 1 calc R . .
H29B H 0.4923 0.5855 0.2876 0.040 Uiso 1 1 calc R . .
C30 C 0.4392(6) 0.5098(5) 0.3608(4) 0.0419(19) Uani 1 1 d . . .
H30A H 0.3835 0.5466 0.3739 0.050 Uiso 1 1 calc R . .
H30B H 0.4123 0.4520 0.3607 0.050 Uiso 1 1 calc R . .
C31 C 0.6074(8) 0.4492(5) 0.4386(4) 0.043(2) Uani 1 1 d . . .
C32 C 0.7181(7) 0.4615(5) 0.4920(4) 0.049(2) Uani 1 1 d . . .
H32A H 0.7428 0.4072 0.5135 0.059 Uiso 1 1 calc R . .
H32B H 0.7061 0.4990 0.5294 0.059 Uiso 1 1 calc R . .
C33 C 0.8116(7) 0.4986(5) 0.4588(4) 0.050(2) Uani 1 1 d . . .
H33A H 0.8836 0.4864 0.4900 0.061 Uiso 1 1 calc R . .
H33B H 0.8096 0.4702 0.4140 0.061 Uiso 1 1 calc R . .
C34 C 0.8029(7) 0.5930(6) 0.4457(4) 0.053(2) Uani 1 1 d . . .
H34A H 0.8629 0.6103 0.4233 0.064 Uiso 1 1 calc R . .
H34B H 0.8142 0.6218 0.4912 0.064 Uiso 1 1 calc R . .
C35 C 0.6905(7) 0.6218(5) 0.3990(4) 0.044(2) Uani 1 1 d . . .
H35A H 0.6944 0.6824 0.3912 0.053 Uiso 1 1 calc R . .
H35B H 0.6808 0.5943 0.3532 0.053 Uiso 1 1 calc R . .
C36 C 0.5873(6) 0.6039(5) 0.4287(4) 0.0373(19) Uani 1 1 d . . .
H36A H 0.6060 0.6127 0.4797 0.045 Uiso 1 1 calc R . .
H36B H 0.5286 0.6438 0.4083 0.045 Uiso 1 1 calc R . .

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
Pd 0.0157(2) 0.0216(3) 0.0181(2) -0.0009(2) 0.00816(17) 0.0005(2)
O1 0.023(3) 0.031(3) 0.035(3) -0.008(2) 0.013(2) -0.004(2)
O2 0.022(3) 0.029(3) 0.033(3) -0.006(2) 0.016(2) -0.005(2)
O3 0.022(3) 0.023(2) 0.037(3) -0.006(2) 0.015(2) -0.003(2)
O4 0.045(3) 0.057(4) 0.038(3) 0.013(3) 0.021(3) 0.011(3)
O5 0.133(6) 0.034(3) 0.040(4) -0.001(3) 0.037(4) -0.022(4)
N1 0.021(3) 0.019(3) 0.023(3) 0.000(2) 0.012(2) 0.003(2)
N2 0.016(3) 0.025(3) 0.018(3) 0.007(2) 0.007(2) 0.001(2)
N3 0.023(3) 0.025(3) 0.022(3) -0.002(2) 0.009(2) 0.002(2)
N4 0.017(3) 0.025(3) 0.016(3) -0.004(2) 0.006(2) 0.001(2)
N5 0.020(3) 0.022(3) 0.024(3) -0.001(2) 0.006(2) 0.000(2)
N6 0.015(3) 0.028(3) 0.027(3) -0.004(2) 0.013(2) -0.001(2)
N7 0.042(4) 0.027(3) 0.029(3) 0.001(3) 0.014(3) 0.002(3)
```

```
N8 0.028(3) 0.028(3) 0.022(3) -0.006(2) 0.011(3) 0.004(3)
N9 0.017(3) 0.024(3) 0.025(3) 0.000(2) 0.009(2) -0.002(2)
N10 0.046(4) 0.040(4) 0.026(3) -0.002(3) 0.009(3) -0.012(3)
C1 0.022(4) 0.022(3) 0.019(4) 0.003(3) 0.005(3) 0.000(3)
C2 0.027(4) 0.017(3) 0.014(3) 0.002(3) 0.013(3) -0.004(3)
C3 0.025(4) 0.025(4) 0.021(4) 0.002(3) 0.010(3) -0.001(3)
C4 0.029(4) 0.025(4) 0.027(4) -0.001(3) 0.013(3) 0.011(3)
C5 0.020(4) 0.026(4) 0.028(4) -0.001(3) 0.012(3) 0.005(3)
C6 0.022(4) 0.019(3) 0.017(3) 0.000(3) 0.007(3) 0.003(3)
C7 0.030(4) 0.018(3) 0.018(3) 0.001(3) 0.009(3) 0.005(3)
C8 0.013(3) 0.028(4) 0.013(3) 0.002(3) 0.005(3) 0.001(3)
C9 0.029(4) 0.021(3) 0.019(4) 0.004(3) 0.006(3) 0.003(3)
C10 0.018(4) 0.027(4) 0.025(4) -0.003(3) 0.010(3) -0.010(3)
C11 0.023(4) 0.032(4) 0.018(4) 0.000(3) 0.011(3) -0.002(3)
C12 0.016(3) 0.029(4) 0.014(3) 0.005(3) 0.002(2) 0.006(3)
C13 0.031(4) 0.029(4) 0.015(3) 0.000(3) 0.006(3) -0.005(3)
C14 0.025(4) 0.043(4) 0.022(4) -0.004(3) 0.010(3) 0.004(3)
C15 0.025(4) 0.056(5) 0.041(5) -0.018(4) 0.001(4) -0.010(4)
C16 0.034(5) 0.039(4) 0.028(4) -0.011(3) 0.007(3) -0.008(4)
C17 0.035(4) 0.027(4) 0.017(4) 0.001(3) 0.012(3) 0.005(3)
C18 0.022(4) 0.034(4) 0.015(4) -0.006(3) 0.002(3) -0.003(3)
C19 0.029(4) 0.033(4) 0.030(4) -0.006(3) 0.013(3) -0.004(3)
C20 0.027(4) 0.034(4) 0.021(4) 0.001(3) 0.010(3) 0.003(3)
C21 0.034(4) 0.028(4) 0.036(5) -0.009(3) 0.011(4) 0.000(3)
C22 0.032(4) 0.031(4) 0.032(4) -0.009(3) 0.009(3) 0.005(3)
C23 0.029(4) 0.029(4) 0.031(4) -0.006(3) 0.009(3) 0.005(3)
C24 0.040(5) 0.042(5) 0.032(4) -0.009(3) 0.014(4) 0.005(4)
C25 0.041(5) 0.064(6) 0.039(5) -0.007(4) 0.019(4) 0.015(4)
C26 0.062(7) 0.042(6) 0.171(12) -0.012(6) 0.055(7) 0.006(5)
C27 0.098(9) 0.053(6) 0.113(10) -0.027(6) 0.056(8) -0.003(6)
C28 0.029(4) 0.046(5) 0.034(4) -0.007(4) 0.017(3) -0.007(4)
C29 0.043(5) 0.035(4) 0.027(4) -0.004(3) 0.020(3) -0.010(3)
C30 0.043(5) 0.054(5) 0.033(4) -0.001(4) 0.017(3) -0.013(4)
C31 0.078(7) 0.039(5) 0.019(4) -0.002(4) 0.023(4) -0.013(5)
C32 0.074(6) 0.038(5) 0.031(5) 0.003(4) 0.001(4) 0.003(4)
C33 0.055(5) 0.060(6) 0.038(4) -0.007(5) 0.016(4) 0.007(5)
C34 0.056(6) 0.062(6) 0.043(5) -0.008(4) 0.013(5) -0.002(5)
C35 0.050(5) 0.045(5) 0.039(5) -0.003(4) 0.014(4) -0.014(4)
C36 0.041(5) 0.038(4) 0.030(4) -0.009(4) 0.002(4) -0.004(4)

_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
Pd N2 1.940(5) . ?
Pd N1 2.048(5) . ?
Pd N9 2.053(5) . ?
Pd N3 2.084(5) . ?
O1 C1 1.240(7) . ?
O2 C7 1.239(7) . ?
O3 C18 1.217(7) . ?
O4 C21 1.227(8) . ?
O5 C31 1.250(9) . ?
N1 C1 1.378(8) . ?
N1 C8 1.435(7) . ?
N2 C2 1.335(7) . ?
N2 C6 1.341(8) . ?
N3 C7 1.382(8) . ?
N3 C13 1.432(8) . ?
N4 C12 1.336(7) . ?
N4 C8 1.361(7) . ?
N5 C17 1.334(8) . ?
N5 C13 1.343(8) . ?
N6 C18 1.381(8) . ?
N6 C12 1.413(8) . ?
N6 H6 0.8600 . ?
N7 C21 1.377(9) . ?
N7 C17 1.438(8) . ?
N7 H7 0.8600 . ?
N8 C20 1.462(8) . ?
N8 C23 1.463(8) . ?
N8 C24 1.483(8) . ?
N9 C28 1.495(8) . ?
N9 H9A 0.9000 . ?
N9 H9B 0.9000 . ?
N10 C31 1.349(10) . ?
N10 C36 1.462(9) . ?
N10 C30 1.464(9) . ?
C1 C2 1.511(8) . ?
C2 C3 1.376(8) . ?
C3 C4 1.382(9) . ?
C3 H3 0.9300 . ?
C4 C5 1.398(9) . ?
C4 H4 0.9300 . ?
C5 C6 1.385(8) . ?
C5 H5 0.9300 . ?
C6 C7 1.528(8) . ?
C8 C9 1.395(8) . ?
C9 C10 1.389(9) . ?
C9 H9 0.9300 . ?
```

```
C10 C11 1.367(9) . ?
C10 H10 0.9300 . ?
C11 C12 1.405(9) . ?
C11 H11 0.9300 . ?
C13 C14 1.393(9) . ?
C14 C15 1.380(10) . ?
C14 H14 0.9300 . ?
C15 C16 1.383(10) . ?
C15 H15 0.9300 . ?
C16 C17 1.370(9) . ?
C16 H16 0.9300 . ?
C18 C19 1.528(9) . ?
C19 C20 1.543(9) . ?
C19 H19A 0.9700 . ?
C19 H19B 0.9700 . ?
C20 H20A 0.9700 . ?
C20 H20B 0.9700 . ?
C21 C22 1.526(9) . ?
C22 C23 1.530(9) . ?
C22 H22A 0.9700 . ?
C22 H22B 0.9700 . ?
C23 H23A 0.9700 . ?
C23 H23B 0.9700 . ?
C24 C25 1.530(10) . ?
C24 H24A 0.9700 . ?
C24 H24B 0.9700 . ?
C25 C26 1.532(12) . ?
C25 H25A 0.9700 . ?
C25 H25B 0.9700 . ?
C26 C27 1.491(12) . ?
C26 H26A 0.9700 . ?
C26 H26B 0.9700 . ?
C27 H27A 0.9600 . ?
C27 H27B 0.9600 . ?
C27 H27C 0.9600 . ?
C28 C29 1.529(10) . ?
C28 H28A 0.9700 . ?
C28 H28B 0.9700 . ?
C29 C30 1.529(9) . ?
C29 H29A 0.9700 . ?
C29 H29B 0.9700 . ?
C30 H30A 0.9700 . ?
C30 H30B 0.9700 . ?
C31 C32 1.519(11) . ?
C32 C33 1.545(11) . ?
C32 H32A 0.9700 . ?
C32 H32B 0.9700 . ?
C33 C34 1.510(11) . ?
C33 H33A 0.9700 . ?
C33 H33B 0.9700 . ?
C34 C35 1.536(11) . ?
C34 H34A 0.9700 . ?
C34 H34B 0.9700 . ?
C35 C36 1.523(10) . ?
```

```
C35 H35A 0.9700 . ?
C35 H35B 0.9700 . ?
C36 H36A 0.9700 . ?
C36 H36B 0.9700 . ?

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
N2 Pd N1 80.3(2) . . ?
N2 Pd N9 179.0(2) . . ?
N1 Pd N9 98.7(2) . . ?
N2 Pd N3 80.8(2) . . ?
N1 Pd N3 161.1(2) . . ?
N9 Pd N3 100.1(2) . . ?
C1 N1 C8 117.2(5) . . ?
C1 N1 Pd 114.5(4) . . ?
C8 N1 Pd 128.3(4) . . ?
C2 N2 C6 122.0(5) . . ?
C2 N2 Pd 119.1(4) . . ?
C6 N2 Pd 118.8(4) . . ?
C7 N3 C13 118.3(5) . . ?
C7 N3 Pd 113.4(4) . . ?
C13 N3 Pd 128.3(4) . . ?
C12 N4 C8 117.8(5) . . ?
C17 N5 C13 118.7(6) . . ?
C18 N6 C12 128.7(6) . . ?
C18 N6 H6 115.7 . . ?
C12 N6 H6 115.7 . . ?
C21 N7 C17 122.6(6) . . ?
C21 N7 H7 118.7 . . ?
C17 N7 H7 118.7 . . ?
C20 N8 C23 114.2(5) . . ?
C20 N8 C24 113.2(5) . . ?
C23 N8 C24 111.5(5) . . ?
C28 N9 Pd 115.8(4) . . ?
C28 N9 H9A 108.3 . . ?
Pd N9 H9A 108.3 . . ?
C28 N9 H9B 108.3 . . ?
Pd N9 H9B 108.3 . . ?
H9A N9 H9B 107.4 . . ?
C31 N10 C36 121.7(6) . . ?
C31 N10 C30 121.6(6) . . ?
C36 N10 C30 115.9(6) . . ?
O1 C1 N1 128.3(6) . . ?
O1 C1 C2 119.3(6) . . ?
N1 C1 C2 112.4(5) . . ?
N2 C2 C3 120.4(6) . . ?
N2 C2 C1 113.7(5) . . ?
C3 C2 C1 125.8(6) . . ?
```

```
C2 C3 C4 118.6(6) . . ?
C2 C3 H3 120.7 . . ?
C4 C3 H3 120.7 . . ?
C3 C4 C5 120.9(6) . . ?
C3 C4 H4 119.6 . . ?
C5 C4 H4 119.6 . . ?
C6 C5 C4 117.3(6) . . ?
C6 C5 H5 121.4 . . ?
C4 C5 H5 121.4 . . ?
N2 C6 C5 120.7(6) . . ?
N2 C6 C7 114.3(5) . . ?
C5 C6 C7 124.9(6) . . ?
O2 C7 N3 128.0(6) . . ?
O2 C7 C6 119.4(5) . . ?
N3 C7 C6 112.5(5) . . ?
N4 C8 C9 122.4(5) . . ?
N4 C8 N1 113.9(5) . . ?
C9 C8 N1 123.7(6) . . ?
C10 C9 C8 117.9(6) . . ?
C10 C9 H9 121.1 . . ?
C8 C9 H9 121.1 . . ?
C11 C10 C9 120.9(6) . . ?
C11 C10 H10 119.6 . . ?
C9 C10 H10 119.6 . . ?
C10 C11 C12 117.6(6) . . ?
C10 C11 H11 121.2 . . ?
C12 C11 H11 121.2 . . ?
N4 C12 C11 123.4(6) . . ?
N4 C12 N6 119.2(6) . . ?
C11 C12 N6 117.4(5) . . ?
N5 C13 C14 121.2(6) . . ?
N5 C13 N3 113.9(6) . . ?
C14 C13 N3 124.9(6) . . ?
C15 C14 C13 118.5(7) . . ?
C15 C14 H14 120.7 . . ?
C13 C14 H14 120.7 . . ?
C16 C15 C14 120.6(7) . . ?
C16 C15 H15 119.7 . . ?
C14 C15 H15 119.7 . . ?
C17 C16 C15 116.9(7) . . ?
C17 C16 H16 121.6 . . ?
C15 C16 H16 121.6 . . ?
N5 C17 C16 124.2(6) . . ?
N5 C17 N7 114.8(6) . . ?
C16 C17 N7 121.0(6) . . ?
O3 C18 N6 124.6(6) . . ?
O3 C18 C19 123.5(6) . . ?
N6 C18 C19 112.0(6) . . ?
C18 C19 C20 114.3(6) . . ?
C18 C19 H19A 108.7 . . ?
C20 C19 H19A 108.7 . . ?
C18 C19 H19B 108.7 . . ?
C20 C19 H19B 108.7 . . ?
H19A C19 H19B 107.6 . . ?
```

```
N8 C20 C19 119.2(6) . . ?
N8 C20 H20A 107.5 . . ?
C19 C20 H20A 107.5 . . ?
N8 C20 H20B 107.5 . . ?
C19 C20 H20B 107.5 . . ?
H20A C20 H20B 107.0 . . ?
O4 C21 N7 122.4(7) . . ?
O4 C21 C22 123.9(7) . . ?
N7 C21 C22 113.7(6) . . ?
C21 C22 C23 109.5(5) . . ?
C21 C22 H22A 109.8 . . ?
C23 C22 H22A 109.8 . . ?
C21 C22 H22B 109.8 . . ?
C23 C22 H22B 109.8 . . ?
H22A C22 H22B 108.2 . . ?
N8 C23 C22 113.6(5) . . ?
N8 C23 H23A 108.9 . . ?
C22 C23 H23A 108.9 . . ?
N8 C23 H23B 108.9 . . ?
C22 C23 H23B 108.9 . . ?
H23A C23 H23B 107.7 . . ?
N8 C24 C25 112.5(6) . . ?
N8 C24 H24A 109.1 . . ?
C25 C24 H24A 109.1 . . ?
N8 C24 H24B 109.1 . . ?
C25 C24 H24B 109.1 . . ?
H24A C24 H24B 107.8 . . ?
C26 C25 C24 111.4(7) . . ?
C26 C25 H25A 109.4 . . ?
C24 C25 H25A 109.4 . . ?
C26 C25 H25B 109.4 . . ?
C24 C25 H25B 109.4 . . ?
H25A C25 H25B 108.0 . . ?
C27 C26 C25 115.7(8) . . ?
C27 C26 H26A 108.4 . . ?
C25 C26 H26A 108.4 . . ?
C27 C26 H26B 108.4 . . ?
C25 C26 H26B 108.4 . . ?
H26A C26 H26B 107.4 . . ?
C26 C27 H27A 109.5 . . ?
C26 C27 H27B 109.5 . . ?
H27A C27 H27B 109.5 . . ?
C26 C27 H27C 109.5 . . ?
H27A C27 H27C 109.5 . . ?
H27B C27 H27C 109.5 . . ?
N9 C28 C29 110.7(5) . . ?
N9 C28 H28A 109.5 . . ?
C29 C28 H28A 109.5 . . ?
N9 C28 H28B 109.5 . . ?
C29 C28 H28B 109.5 . . ?
H28A C28 H28B 108.1 . . ?
C30 C29 C28 112.4(6) . . ?
C30 C29 H29A 109.1 . . ?
C28 C29 H29A 109.1 . . ?
```

```
C30 C29 H29B 109.1 . . ?
C28 C29 H29B 109.1 . . ?
H29A C29 H29B 107.9 . . ?
N10 C30 C29 112.9(6) . . ?
N10 C30 H30A 109.0 . . ?
C29 C30 H30A 109.0 . . ?
N10 C30 H30B 109.0 . . ?
C29 C30 H30B 109.0 . . ?
H30A C30 H30B 107.8 . . ?
O5 C31 N10 122.5(8) . . ?
O5 C31 C32 118.2(8) . . ?
N10 C31 C32 119.3(7) . . ?
C31 C32 C33 113.3(6) . . ?
C31 C32 H32A 108.9 . . ?
C33 C32 H32A 108.9 . . ?
C31 C32 H32B 108.9 . . ?
C33 C32 H32B 108.9 . . ?
H32A C32 H32B 107.7 . . ?
C34 C33 C32 114.4(7) . . ?
C34 C33 H33A 108.7 . . ?
C32 C33 H33A 108.7 . . ?
C34 C33 H33B 108.7 . . ?
C32 C33 H33B 108.7 . . ?
H33A C33 H33B 107.6 . . ?
C33 C34 C35 114.2(7) . . ?
C33 C34 H34A 108.7 . . ?
C35 C34 H34A 108.7 . . ?
C33 C34 H34B 108.7 . . ?
C35 C34 H34B 108.7 . . ?
H34A C34 H34B 107.6 . . ?
C36 C35 C34 115.6(7) . . ?
C36 C35 H35A 108.4 . . ?
C34 C35 H35A 108.4 . . ?
C36 C35 H35B 108.4 . . ?
C34 C35 H35B 108.4 . . ?
H35A C35 H35B 107.4 . . ?
N10 C36 C35 112.9(6) . . ?
N10 C36 H36A 109.0 . . ?
C35 C36 H36A 109.0 . . ?
N10 C36 H36B 109.0 . . ?
C35 C36 H36B 109.0 . . ?
H36A C36 H36B 107.8 . . ?

loop_
 _geom_hbond_atom_site_label_D
 _geom_hbond_atom_site_label_H
 _geom_hbond_atom_site_label_A
 _geom_hbond_distance_DH
 _geom_hbond_distance_HA
 _geom_hbond_distance_DA
 _geom_hbond_angle_DHA
 _geom_hbond_site_symmetry_A
N6 H6 O2  0.86 2.15 2.978(7) 161.4 1_455
N7 H7 O5  0.86 2.00 2.788(8) 152.4 2_655
```

```
N9 H9A N5  0.90 2.07 2.842(7) 142.8 .

_diffrn_measured_fraction_theta_max    0.997
_diffrn_reflns_theta_full              25.02
_diffrn_measured_fraction_theta_full   0.997
_refine_diff_density_max    1.144
_refine_diff_density_min   -1.390
_refine_diff_density_rms    0.124
```

4. Compound $[Pd(OH_2)(H_2L)]$ (**6**).

```
data_Cpd_6
data_global
_audit_creation_method     SHELXL-97

_publ_section_title
;
;
_publ_section_abstract        ' Abstract  '
_publ_section_comment         ' Comment  '

_publ_section_related_literature
;
For related literature on the   .

;

_publ_requested_journal          'Journal'


_publ_contact_letter
;
Please consider this CIF for publication. I certify that this
contibution is the
original work of those listed as authors; that it has not been
published
before (in any language or medium) and is not being considered for
publication
elsewhere; that all authors concur with and are aware of the
submission; that
all workers involved in the study are listed as authors or given
proper credit
in the acknowledgements; that I have obtained permission for and
acknowledged
the source of any excerpts from other copyright works; and that to
the best of
my knowledge the paper contains no statements which are libellous,
unlawful or
```

```
in any way actionable. All coauthors have made significant
scientific
contributions to the work reported, including the ideas and their
execution,
and share responsibility and accountability for the results.
;

_publ_contact_author_name        'George R. Clark'
_publ_contact_author_address
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;
_publ_contact_author_email       'g.clark@auckland.ac.nz'
_publ_contact_author_phone       '0064 9 3737599 Ext 88294'
_publ_contact_author_fax         '0064 9 3737422'

loop_
_publ_author_name
_publ_author_address


'Burgess, Michael G.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Zafar, M. Naveed'
;
Chemistry Department
University of Gujrat
H-H campus
Gujrat
Pakistan
;

'Horner, Stephen T.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Clark, George R.'
;
```

Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

'Wright, L. James.'
;
Department of Chemistry
The University of Auckland
Private Bag 92019
Auckland
New Zealand
;

_publ_section_exptl_refinement
;
Hydrogen atoms were placed in calculated positions and refined using the
riding model [C-H 0.93-0.97 \%A), with U~iso~(H) = 1.2 or 1.5 times U~eq~(C).
The hydrogen atoms of the coordinated water molecule were refined as a
 quasi-methyl group with O-H distances set at 0.82
\%A. One hydrogen was assigned zero occupancy.
;

_publ_section_figure_captions
;
Fig. 1: Structure showing 50 % probability displacement ellipsoids for non-hydrogen atoms and hydrogen atoms as arbitary spheres (Burnett
 & Johnson, 1996). The dichloromethane molecule of crystallisation is
not shown. There is a hydrogen bond between O5 and N5, and bifurcated
hydrogen bonds between O5 and O3, and O5 and N4. There is an
intermolecular hydrogen bond between N6 and O2.
;

_publ_section_acknowledgements
;
No acknowledgements
;

_publ_section_references

```
;

Bruker (2006). <i>APEX2</i> and <i>SAINT</i>. Bruker AXS Inc.,
Madison,
Wisconsin, USA.


Burnett, M.N. and Johnson, C.K. (1996). ORTEP-III: Report ORNL-6895.
Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G.M. (1996). <i>SADABS</i>. Univ. of G\"ottingen,
Germany.

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


Siemens (1995). <i>SMART</i> (Version 4.050) and
<i>SAINT</i> (Version 4.050). Siemens Analytical X-ray Instruments
Inc., Madison, Wisconsin, USA.



;




data_compound 6

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          'C27 H30 N8 O5 Pd,C H2 Cl2'
_chemical_formula_sum
 'C28 H32 Cl2 N8 O5 Pd'
_chemical_formula_weight          737.92

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'
 'Pd'  'Pd'  -0.9988   1.0072
 '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            Triclinic
_symmetry_space_group_name_H-M    P-1
_symmetry_space_group_name_Hall    -P_1

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

_cell_length_a                    7.5642(2)
_cell_length_b                    11.5133(3)
_cell_length_c                    16.9769(5)
_cell_angle_alpha                 90.1220(10)
_cell_angle_beta                  98.3350(10)
_cell_angle_gamma                 93.2520(10)
_cell_volume                      1460.45(7)
_cell_formula_units_Z             2
_cell_measurement_temperature     89(2)
_cell_measurement_reflns_used     8192
_cell_measurement_theta_min       1.77
_cell_measurement_theta_max       26.43

_exptl_crystal_description        rhomboids
_exptl_crystal_colour             yellow
_exptl_crystal_size_max           0.24
_exptl_crystal_size_mid           0.12
_exptl_crystal_size_min           0.10
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.678
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              752
_exptl_absorpt_coefficient_mu     0.873
_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_correction_T_min   0.7162
_exptl_absorpt_correction_T_max   0.9406
_exptl_absorpt_process_details    'SADABS; Sheldrick, 1996'

_diffrn_ambient_temperature       89(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   'Siemens SMART CCD'
_diffrn_measurement_method        'Area detector \w scans'
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          0
_diffrn_standards_interval_count  0
```

```
_diffrn_standards_interval_time   0
_diffrn_reflns_number             14202
_diffrn_reflns_av_R_equivalents   0.0302
_diffrn_reflns_av_sigmaI/netI     0.0811
_diffrn_reflns_limit_h_min        -9
_diffrn_reflns_limit_h_max        9
_diffrn_reflns_limit_k_min        -14
_diffrn_reflns_limit_k_max        14
_diffrn_reflns_limit_l_min        -21
_diffrn_reflns_limit_l_max        21
_diffrn_reflns_theta_min          1.77
_diffrn_reflns_theta_max          26.43
_reflns_number_total              5955
_reflns_number_gt                 4801
_reflns_threshold_expression      >2\s(I)

_computing_data_collection        'SMART (Siemens, 1995)'
_computing_cell_refinement        'SAINT (Siemens, 1995)'
_computing_data_reduction         'SAINT'
_computing_structure_solution     'SHELXS97 (Sheldrick, 2008)'
_computing_structure_refinement   'SHELXL97 (Sheldrick, 2008)'
_computing_molecular_graphics     'Ortep-III (Burnett & Johnson,
1996)'
_computing_publication_material   'SHELXTL (Sheldrick, 2008)'


_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^)+(0.0678P)^2^+1.9359P] 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_extinction_coef        ?
_refine_ls_number_reflns          5955
_refine_ls_number_parameters      399
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0754
_refine_ls_R_factor_gt            0.0563
_refine_ls_wR_factor_ref          0.1388
_refine_ls_wR_factor_gt           0.1279
_refine_ls_goodness_of_fit_ref    1.048
_refine_ls_restrained_S_all       1.048
_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
Pd Pd 0.19351(5) 0.08092(3) 0.00337(2) 0.01753(12) Uani 1 1 d . . .
Cl1 Cl 0.36315(19) 0.21635(13) 0.59422(9) 0.0402(3) Uani 1 1 d . . .
Cl2 Cl 0.0441(2) 0.34252(13) 0.60440(9) 0.0416(4) Uani 1 1 d . . .
O1 O 0.2465(5) 0.1817(3) -0.2226(2) 0.0294(8) Uani 1 1 d . . .
O2 O 0.3606(5) -0.2405(3) 0.0751(2) 0.0253(8) Uani 1 1 d . . .
O3 O 0.2651(5) 0.3602(3) 0.15735(19) 0.0225(7) Uani 1 1 d . . .
O4 O 0.0826(6) 0.1595(3) 0.4097(2) 0.0366(9) Uani 1 1 d . . .
O5 O 0.0717(4) 0.1862(3) 0.07340(19) 0.0186(7) Uani 1 1 d . . .
H5A H 0.1170 0.2526 0.0730 0.028 Uiso 1 1 calc R . .
H5B H 0.0845 0.1619 0.1191 0.028 Uiso 1 1 calc R . .
N1 N 0.1925(5) 0.1886(3) -0.0906(2) 0.0194(8) Uani 1 1 d . . .
N2 N 0.2851(5) -0.0216(3) -0.0673(2) 0.0176(8) Uani 1 1 d . . .
N3 N 0.2441(5) -0.0570(3) 0.0796(2) 0.0177(8) Uani 1 1 d . . .
N4 N 0.2462(5) 0.3525(3) -0.0120(2) 0.0193(8) Uani 1 1 d . . .
N5 N 0.1866(5) 0.0475(3) 0.1879(2) 0.0188(8) Uani 1 1 d . . .
N6 N 0.3192(5) 0.5115(3) 0.0757(2) 0.0209(8) Uani 1 1 d . . .
H6 H 0.3626 0.5822 0.0765 0.025 Uiso 1 1 calc R . .
N7 N 0.1459(6) 0.1803(3) 0.2838(2) 0.0226(9) Uani 1 1 d . . .
H7 H 0.1543 0.2304 0.2468 0.027 Uiso 1 1 calc R . .
N8 N 0.3891(5) 0.4233(3) 0.3417(2) 0.0232(9) Uani 1 1 d . . .
C1 C 0.2382(7) 0.1375(4) -0.1578(3) 0.0226(10) Uani 1 1 d . . .
C2 C 0.2904(6) 0.0137(4) -0.1423(3) 0.0210(10) Uani 1 1 d . . .
C3 C 0.3534(7) -0.0588(4) -0.1950(3) 0.0236(10) Uani 1 1 d . . .
H3 H 0.3553 -0.0368 -0.2476 0.028 Uiso 1 1 calc R . .
C4 C 0.4143(6) -0.1658(4) -0.1683(3) 0.0237(10) Uani 1 1 d . . .
H4 H 0.4586 -0.2153 -0.2032 0.028 Uiso 1 1 calc R . .
C5 C 0.4093(6) -0.1992(4) -0.0897(3) 0.0208(10) Uani 1 1 d . . .
```

```
H5 H 0.4529 -0.2694 -0.0710 0.025 Uiso 1 1 calc R . .
C6 C 0.3371(6) -0.1244(4) -0.0404(3) 0.0183(9) Uani 1 1 d . . .
C7 C 0.3145(6) -0.1471(4) 0.0454(3) 0.0198(10) Uani 1 1 d . . .
C8 C 0.1829(6) 0.3096(4) -0.0848(3) 0.0193(10) Uani 1 1 d . . .
C9 C 0.1172(7) 0.3801(4) -0.1483(3) 0.0231(10) Uani 1 1 d . . .
H9 H 0.0784 0.3490 -0.1988 0.028 Uiso 1 1 calc R . .
C10 C 0.1123(6) 0.4983(4) -0.1328(3) 0.0222(10) Uani 1 1 d . . .
H10 H 0.0675 0.5474 -0.1733 0.027 Uiso 1 1 calc R . .
C11 C 0.1732(7) 0.5435(4) -0.0578(3) 0.0230(10) Uani 1 1 d . . .
H11 H 0.1688 0.6223 -0.0466 0.028 Uiso 1 1 calc R . .
C12 C 0.2415(6) 0.4667(4) 0.0002(3) 0.0191(9) Uani 1 1 d . . .
C13 C 0.2276(6) -0.0562(4) 0.1608(3) 0.0187(9) Uani 1 1 d . . .
C14 C 0.2512(7) -0.1496(4) 0.2129(3) 0.0247(11) Uani 1 1 d . . .
H14 H 0.2738 -0.2226 0.1944 0.030 Uiso 1 1 calc R . .
C15 C 0.2403(7) -0.1312(4) 0.2926(3) 0.0252(11) Uani 1 1 d . . .
H15 H 0.2571 -0.1926 0.3279 0.030 Uiso 1 1 calc R . .
C16 C 0.2051(7) -0.0238(4) 0.3211(3) 0.0259(11) Uani 1 1 d . . .
H16 H 0.1986 -0.0112 0.3747 0.031 Uiso 1 1 calc R . .
C17 C 0.1798(6) 0.0642(4) 0.2657(3) 0.0205(10) Uani 1 1 d . . .
C18 C 0.3341(6) 0.4575(4) 0.1470(3) 0.0198(10) Uani 1 1 d . . .
C19 C 0.4452(7) 0.5253(4) 0.2156(3) 0.0226(10) Uani 1 1 d . . .
H19A H 0.4662 0.6049 0.1992 0.027 Uiso 1 1 calc R . .
H19B H 0.5606 0.4916 0.2277 0.027 Uiso 1 1 calc R . .
C20 C 0.3568(7) 0.5259(4) 0.2908(3) 0.0249(11) Uani 1 1 d . . .
H20A H 0.4004 0.5952 0.3218 0.030 Uiso 1 1 calc R . .
H20B H 0.2287 0.5304 0.2756 0.030 Uiso 1 1 calc R . .
C21 C 0.1014(7) 0.2213(4) 0.3533(3) 0.0246(11) Uani 1 1 d . . .
C22 C 0.0813(7) 0.3518(4) 0.3569(3) 0.0257(11) Uani 1 1 d . . .
H22A H -0.0148 0.3675 0.3868 0.031 Uiso 1 1 calc R . .
H22B H 0.0502 0.3808 0.3034 0.031 Uiso 1 1 calc R . .
C23 C 0.2546(7) 0.4147(4) 0.3963(3) 0.0268(11) Uani 1 1 d . . .
H23A H 0.2298 0.4922 0.4126 0.032 Uiso 1 1 calc R . .
H23B H 0.3019 0.3730 0.4436 0.032 Uiso 1 1 calc R . .
C24 C 0.5718(7) 0.4361(5) 0.3874(3) 0.0272(11) Uani 1 1 d . . .
H24A H 0.5776 0.5021 0.4235 0.033 Uiso 1 1 calc R . .
H24B H 0.6564 0.4533 0.3507 0.033 Uiso 1 1 calc R . .
C25 C 0.6293(7) 0.3292(4) 0.4356(3) 0.0269(11) Uani 1 1 d . . .
H25A H 0.7518 0.3439 0.4611 0.032 Uiso 1 1 calc R . .
H25B H 0.5548 0.3185 0.4772 0.032 Uiso 1 1 calc R . .
C26 C 0.6176(7) 0.2171(5) 0.3873(3) 0.0317(12) Uani 1 1 d . . .
H26A H 0.4978 0.2052 0.3581 0.038 Uiso 1 1 calc R . .
H26B H 0.7005 0.2246 0.3487 0.038 Uiso 1 1 calc R . .
C27 C 0.6603(9) 0.1118(5) 0.4386(4) 0.0423(15) Uani 1 1 d . . .
H27A H 0.7790 0.1230 0.4675 0.063 Uiso 1 1 calc R . .
H27B H 0.6533 0.0437 0.4053 0.063 Uiso 1 1 calc R . .
H27C H 0.5757 0.1021 0.4754 0.063 Uiso 1 1 calc R . .
C28 C 0.1333(7) 0.2040(5) 0.5987(3) 0.0298(12) Uani 1 1 d . . .
H28A H 0.1120 0.1591 0.6451 0.036 Uiso 1 1 calc R . .
H28B H 0.0723 0.1627 0.5518 0.036 Uiso 1 1 calc R . .

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
Pd 0.0249(2) 0.01626(19) 0.01190(18) 0.00004(13) 0.00361(13)
0.00260(13)
Cl1 0.0405(8) 0.0376(8) 0.0448(8) 0.0039(6) 0.0135(7) 0.0037(6)
Cl2 0.0425(8) 0.0406(8) 0.0426(8) 0.0061(7) 0.0057(7) 0.0116(6)
O1 0.048(2) 0.0292(19) 0.0133(17) 0.0047(15) 0.0079(16) 0.0134(17)
O2 0.036(2) 0.0166(17) 0.0233(18) 0.0018(14) 0.0028(15) 0.0025(14)
O3 0.039(2) 0.0161(16) 0.0134(16) 0.0001(13) 0.0066(14) 0.0011(14)
O4 0.061(3) 0.036(2) 0.0165(19) 0.0036(16) 0.0159(18) 0.0044(19)
O5 0.0256(17) 0.0144(15) 0.0154(16) 0.0020(13) 0.0023(13) -
0.0009(13)
N1 0.024(2) 0.021(2) 0.0131(19) -0.0012(16) 0.0012(16) 0.0019(16)
N2 0.020(2) 0.0139(18) 0.019(2) 0.0026(15) 0.0033(16) 0.0016(15)
N3 0.024(2) 0.0103(17) 0.020(2) -0.0033(15) 0.0068(16) -0.0018(15)
N4 0.028(2) 0.0190(19) 0.0115(19) 0.0019(15) 0.0041(16) 0.0059(16)
N5 0.024(2) 0.0176(19) 0.0151(19) 0.0029(15) 0.0060(16) -0.0010(16)
N6 0.029(2) 0.0133(18) 0.020(2) 0.0008(16) 0.0045(17) 0.0006(16)
N7 0.036(2) 0.021(2) 0.0124(19) 0.0030(16) 0.0078(17) 0.0030(17)
N8 0.031(2) 0.025(2) 0.014(2) -0.0002(16) 0.0037(17) 0.0012(17)
C1 0.032(3) 0.022(2) 0.014(2) -0.0010(19) 0.002(2) 0.005(2)
C2 0.025(3) 0.019(2) 0.019(2) -0.0003(19) 0.0028(19) 0.0009(19)
C3 0.031(3) 0.021(2) 0.018(2) -0.0035(19) 0.003(2) -0.003(2)
C4 0.024(3) 0.028(3) 0.020(2) -0.004(2) 0.005(2) 0.003(2)
C5 0.019(2) 0.018(2) 0.025(3) -0.0029(19) 0.001(2) 0.0012(18)
C6 0.021(2) 0.018(2) 0.016(2) -0.0018(18) 0.0018(18) -0.0013(18)
C7 0.024(2) 0.019(2) 0.015(2) 0.0006(18) 0.0016(19) -0.0036(19)
C8 0.027(3) 0.019(2) 0.013(2) 0.0038(18) 0.0097(19) 0.0018(19)
C9 0.030(3) 0.026(2) 0.014(2) 0.0025(19) 0.003(2) 0.003(2)
C10 0.027(3) 0.022(2) 0.018(2) 0.0074(19) 0.003(2) 0.007(2)
C11 0.032(3) 0.015(2) 0.024(3) 0.0027(19) 0.008(2) 0.0042(19)
C12 0.020(2) 0.020(2) 0.017(2) 0.0034(18) 0.0018(18) 0.0001(18)
C13 0.025(2) 0.020(2) 0.011(2) 0.0040(18) 0.0031(18) 0.0013(19)
C14 0.033(3) 0.020(2) 0.020(2) -0.002(2) 0.004(2) 0.000(2)
C15 0.035(3) 0.022(2) 0.019(2) 0.011(2) 0.006(2) 0.005(2)
C16 0.033(3) 0.029(3) 0.016(2) 0.003(2) 0.007(2) 0.001(2)
C17 0.023(2) 0.019(2) 0.019(2) -0.0024(19) 0.0036(19) -0.0009(19)
C18 0.026(3) 0.017(2) 0.017(2) -0.0050(18) 0.0035(19) 0.0026(19)
C19 0.033(3) 0.017(2) 0.017(2) 0.0011(19) 0.003(2) 0.000(2)
C20 0.036(3) 0.021(2) 0.018(2) -0.0034(19) 0.003(2) 0.005(2)
C21 0.028(3) 0.028(3) 0.018(2) -0.003(2) 0.004(2) 0.002(2)
C22 0.032(3) 0.030(3) 0.015(2) -0.003(2) 0.002(2) 0.006(2)
C23 0.038(3) 0.026(3) 0.018(2) -0.004(2) 0.005(2) 0.006(2)
C24 0.030(3) 0.033(3) 0.019(2) 0.001(2) 0.004(2) 0.000(2)
C25 0.029(3) 0.035(3) 0.017(2) 0.001(2) 0.004(2) 0.004(2)
C26 0.032(3) 0.031(3) 0.031(3) 0.004(2) 0.001(2) 0.005(2)
C27 0.054(4) 0.031(3) 0.045(4) 0.007(3) 0.012(3) 0.012(3)
C28 0.032(3) 0.035(3) 0.022(3) -0.004(2) 0.006(2) 0.001(2)

_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
Pd N2 1.908(4) . ?
Pd N1 2.022(4) . ?
Pd O5 2.042(3) . ?
Pd N3 2.068(4) . ?
Cl1 C28 1.749(5) . ?
Cl2 C28 1.774(5) . ?
O1 C1 1.221(6) . ?
O2 C7 1.236(6) . ?
O3 C18 1.233(5) . ?
O4 C21 1.213(6) . ?
O5 H5A 0.8200 . ?
O5 H5B 0.8200 . ?
N1 C1 1.379(6) . ?
N1 C8 1.404(6) . ?
N2 C6 1.330(6) . ?
N2 C2 1.343(6) . ?
N3 C7 1.359(6) . ?
N3 C13 1.403(6) . ?
N4 C12 1.334(6) . ?
N4 C8 1.340(6) . ?
N5 C17 1.342(6) . ?
N5 C13 1.347(6) . ?
N6 C18 1.355(6) . ?
N6 C12 1.413(6) . ?
N6 H6 0.8600 . ?
N7 C21 1.365(6) . ?
N7 C17 1.416(6) . ?
N7 H7 0.8600 . ?
N8 C23 1.473(6) . ?
N8 C20 1.475(6) . ?
N8 C24 1.484(7) . ?
C1 C2 1.514(6) . ?
C2 C3 1.375(6) . ?
C3 C4 1.394(7) . ?
```

```
C3 H3 0.9300 . ?
C4 C5 1.395(7) . ?
C4 H4 0.9300 . ?
C5 C6 1.387(6) . ?
C5 H5 0.9300 . ?
C6 C7 1.511(6) . ?
C8 C9 1.400(6) . ?
C9 C10 1.389(7) . ?
C9 H9 0.9300 . ?
C10 C11 1.379(7) . ?
C10 H10 0.9300 . ?
C11 C12 1.388(7) . ?
C11 H11 0.9300 . ?
C13 C14 1.397(6) . ?
C14 C15 1.385(7) . ?
C14 H14 0.9300 . ?
C15 C16 1.380(7) . ?
C15 H15 0.9300 . ?
C16 C17 1.387(7) . ?
C16 H16 0.9300 . ?
C18 C19 1.520(6) . ?
C19 C20 1.525(7) . ?
C19 H19A 0.9700 . ?
C19 H19B 0.9700 . ?
C20 H20A 0.9700 . ?
C20 H20B 0.9700 . ?
C21 C22 1.521(7) . ?
C22 C23 1.525(7) . ?
C22 H22A 0.9700 . ?
C22 H22B 0.9700 . ?
C23 H23A 0.9700 . ?
C23 H23B 0.9700 . ?
C24 C25 1.527(7) . ?
C24 H24A 0.9700 . ?
C24 H24B 0.9700 . ?
C25 C26 1.519(7) . ?
C25 H25A 0.9700 . ?
C25 H25B 0.9700 . ?
C26 C27 1.517(7) . ?
C26 H26A 0.9700 . ?
C26 H26B 0.9700 . ?
C27 H27A 0.9600 . ?
C27 H27B 0.9600 . ?
C27 H27C 0.9600 . ?
C28 H28A 0.9700 . ?
C28 H28B 0.9700 . ?

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
N2 Pd N1 81.72(15) . . ?
N2 Pd O5 174.53(14) . . ?
N1 Pd O5 97.42(14) . . ?
N2 Pd N3 81.10(15) . . ?
N1 Pd N3 162.24(14) . . ?
O5 Pd N3 100.11(13) . . ?
Pd O5 H5A 109.5 . . ?
Pd O5 H5B 109.5 . . ?
H5A O5 H5B 109.5 . . ?
C1 N1 C8 121.1(4) . . ?
C1 N1 Pd 114.2(3) . . ?
C8 N1 Pd 123.4(3) . . ?
C6 N2 C2 123.6(4) . . ?
C6 N2 Pd 118.6(3) . . ?
C2 N2 Pd 117.8(3) . . ?
C7 N3 C13 121.6(4) . . ?
C7 N3 Pd 113.1(3) . . ?
C13 N3 Pd 125.0(3) . . ?
C12 N4 C8 118.1(4) . . ?
C17 N5 C13 120.6(4) . . ?
C18 N6 C12 128.5(4) . . ?
C18 N6 H6 115.8 . . ?
C12 N6 H6 115.8 . . ?
C21 N7 C17 127.2(4) . . ?
C21 N7 H7 116.4 . . ?
C17 N7 H7 116.4 . . ?
C23 N8 C20 108.8(4) . . ?
C23 N8 C24 110.2(4) . . ?
C20 N8 C24 109.4(4) . . ?
O1 C1 N1 127.9(4) . . ?
O1 C1 C2 120.5(4) . . ?
N1 C1 C2 111.5(4) . . ?
N2 C2 C3 119.3(4) . . ?
N2 C2 C1 114.3(4) . . ?
C3 C2 C1 126.3(4) . . ?
C2 C3 C4 118.8(5) . . ?
C2 C3 H3 120.6 . . ?
C4 C3 H3 120.6 . . ?
C3 C4 C5 120.5(4) . . ?
C3 C4 H4 119.8 . . ?
C5 C4 H4 119.8 . . ?
C6 C5 C4 117.9(4) . . ?
C6 C5 H5 121.0 . . ?
C4 C5 H5 121.0 . . ?
N2 C6 C5 119.8(4) . . ?
N2 C6 C7 114.5(4) . . ?
C5 C6 C7 125.7(4) . . ?
O2 C7 N3 128.7(4) . . ?
O2 C7 C6 118.6(4) . . ?
N3 C7 C6 112.7(4) . . ?
N4 C8 C9 122.7(4) . . ?
N4 C8 N1 113.3(4) . . ?
C9 C8 N1 124.1(4) . . ?
```

```
C10 C9 C8 117.4(4) . . ?
C10 C9 H9 121.3 . . ?
C8 C9 H9 121.3 . . ?
C11 C10 C9 120.7(4) . . ?
C11 C10 H10 119.7 . . ?
C9 C10 H10 119.7 . . ?
C10 C11 C12 117.3(4) . . ?
C10 C11 H11 121.3 . . ?
C12 C11 H11 121.3 . . ?
N4 C12 C11 123.7(4) . . ?
N4 C12 N6 117.3(4) . . ?
C11 C12 N6 118.8(4) . . ?
N5 C13 C14 119.8(4) . . ?
N5 C13 N3 113.7(4) . . ?
C14 C13 N3 126.4(4) . . ?
C15 C14 C13 118.6(4) . . ?
C15 C14 H14 120.7 . . ?
C13 C14 H14 120.7 . . ?
C16 C15 C14 121.6(5) . . ?
C16 C15 H15 119.2 . . ?
C14 C15 H15 119.2 . . ?
C15 C16 C17 116.6(4) . . ?
C15 C16 H16 121.7 . . ?
C17 C16 H16 121.7 . . ?
N5 C17 C16 122.6(4) . . ?
N5 C17 N7 112.6(4) . . ?
C16 C17 N7 124.8(4) . . ?
O3 C18 N6 123.9(4) . . ?
O3 C18 C19 120.9(4) . . ?
N6 C18 C19 115.2(4) . . ?
C18 C19 C20 113.3(4) . . ?
C18 C19 H19A 108.9 . . ?
C20 C19 H19A 108.9 . . ?
C18 C19 H19B 108.9 . . ?
C20 C19 H19B 108.9 . . ?
H19A C19 H19B 107.7 . . ?
N8 C20 C19 114.3(4) . . ?
N8 C20 H20A 108.7 . . ?
C19 C20 H20A 108.7 . . ?
N8 C20 H20B 108.7 . . ?
C19 C20 H20B 108.7 . . ?
H20A C20 H20B 107.6 . . ?
O4 C21 N7 123.1(5) . . ?
O4 C21 C22 121.5(4) . . ?
N7 C21 C22 115.4(4) . . ?
C21 C22 C23 110.8(4) . . ?
C21 C22 H22A 109.5 . . ?
C23 C22 H22A 109.5 . . ?
C21 C22 H22B 109.5 . . ?
C23 C22 H22B 109.5 . . ?
H22A C22 H22B 108.1 . . ?
N8 C23 C22 111.2(4) . . ?
N8 C23 H23A 109.4 . . ?
C22 C23 H23A 109.4 . . ?
```

```
N8 C23 H23B 109.4 . . ?
C22 C23 H23B 109.4 . . ?
H23A C23 H23B 108.0 . . ?
N8 C24 C25 114.4(4) . . ?
N8 C24 H24A 108.7 . . ?
C25 C24 H24A 108.7 . . ?
N8 C24 H24B 108.7 . . ?
C25 C24 H24B 108.7 . . ?
H24A C24 H24B 107.6 . . ?
C26 C25 C24 114.6(4) . . ?
C26 C25 H25A 108.6 . . ?
C24 C25 H25A 108.6 . . ?
C26 C25 H25B 108.6 . . ?
C24 C25 H25B 108.6 . . ?
H25A C25 H25B 107.6 . . ?
C27 C26 C25 112.7(5) . . ?
C27 C26 H26A 109.1 . . ?
C25 C26 H26A 109.1 . . ?
C27 C26 H26B 109.1 . . ?
C25 C26 H26B 109.1 . . ?
H26A C26 H26B 107.8 . . ?
C26 C27 H27A 109.5 . . ?
C26 C27 H27B 109.5 . . ?
H27A C27 H27B 109.5 . . ?
C26 C27 H27C 109.5 . . ?
H27A C27 H27C 109.5 . . ?
H27B C27 H27C 109.5 . . ?
Cl1 C28 Cl2 111.5(3) . . ?
Cl1 C28 H28A 109.3 . . ?
Cl2 C28 H28A 109.3 . . ?
Cl1 C28 H28B 109.3 . . ?
Cl2 C28 H28B 109.3 . . ?
H28A C28 H28B 108.0 . . ?

loop_
 _geom_hbond_atom_site_label_D
 _geom_hbond_atom_site_label_H
 _geom_hbond_atom_site_label_A
 _geom_hbond_distance_DH
 _geom_hbond_distance_HA
 _geom_hbond_distance_DA
 _geom_hbond_angle_DHA
 _geom_hbond_site_symmetry_A
O5 H5A O3  0.82 2.05 2.690(4) 134.4 .
O5 H5A N4  0.82 2.16 2.792(5) 134.2 .
O5 H5B N5  0.82 1.89 2.602(5) 145.2 .
N6 H6 O2  0.86 2.04 2.855(5) 157.3 1_565

_diffrn_measured_fraction_theta_max    0.989
_diffrn_reflns_theta_full              26.43
_diffrn_measured_fraction_theta_full   0.989
_refine_diff_density_max    1.265
_refine_diff_density_min   -1.522
_refine_diff_density_rms    0.136
```