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#                           CCDC
#
##############################################################################
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#
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#  from this site to visualise CIF-encoded structures and
#  to carry out CIF format checking respectively.
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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 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.
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C2 C 0.3054(4) 0.1805(6) 0.6589(4) 0.0416(13) Uani 1 1 d . . .
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C4 C 0.3542(5) 0.1773(8) 0.5095(4) 0.0584(17) Uani 1 1 d . . .
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H4B H 0.3513 0.2500 0.4611 0.070 Uiso 1 1 calc R . .
C5 C 0.4641(5) 0.1687(6) 0.5631(4) 0.0465(14) Uani 1 1 d . . .
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H5B H 0.5112 0.1505 0.5230 0.056 Uiso 1 1 calc R . .
C6 C 0.3615(4) 0.3466(6) 0.7927(4) 0.0375(12) Uani 1 1 d . . .
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C8 C 0.3814(6) 0.3393(9) 0.9527(5) 0.0647(19) Uani 1 1 d . . .
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H8B H 0.3059 0.3452 0.9449 0.097 Uiso 1 1 calc R . .
H8C H 0.4103 0.4404 0.9547 0.097 Uiso 1 1 calc R . .
C9 C 0.3592(5) 0.0968(7) 0.8700(4) 0.0499(15) Uani 1 1 d . . .
H9A H 0.3858 0.0435 0.9255 0.075 Uiso 1 1 calc R . .
H9B H 0.3774 0.0414 0.8213 0.075 Uiso 1 1 calc R . .
H9C H 0.2835 0.1053 0.8596 0.075 Uiso 1 1 calc R . .
C10 C 0.5252(5) 0.2399(8) 0.8875(4) 0.0547(17) Uani 1 1 d . . .
H10A H 0.5554 0.3396 0.8859 0.082 Uiso 1 1 calc R . .
H10B H 0.5408 0.1788 0.8398 0.082 Uiso 1 1 calc R . .
H10C H 0.5549 0.1926 0.9443 0.082 Uiso 1 1 calc R . .

C11 C 0.5820(4) 0.3953(6) 0.5934(4) 0.0394(13) Uani 1 1 d . . .
H11A H 0.5725 0.5006 0.6094 0.047 Uiso 1 1 calc R . .
H11B H 0.5760 0.3917 0.5289 0.047 Uiso 1 1 calc R . .
C12 C 0.6929(4) 0.3457(6) 0.6400(4) 0.0425(13) Uani 1 1 d . . .
C13 C 0.7163(5) 0.1870(7) 0.6141(5) 0.0559(17) Uani 1 1 d . . .
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H13B H 0.6961 0.1766 0.5498 0.084 Uiso 1 1 calc R . .
H13C H 0.6770 0.1155 0.6414 0.084 Uiso 1 1 calc R . .
C14 C 0.7680(5) 0.4521(7) 0.6091(5) 0.0613(18) Uani 1 1 d . . .
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H14B H 0.7580 0.4450 0.5449 0.092 Uiso 1 1 calc R . .
H14C H 0.8395 0.4247 0.6372 0.092 Uiso 1 1 calc R . .
C15 C 0.7090(5) 0.3537(10) 0.7392(4) 0.0621(19) Uani 1 1 d . . .
H15A H 0.6890 0.4529 0.7561 0.093 Uiso 1 1 calc R . .
H15B H 0.7823 0.3355 0.7666 0.093 Uiso 1 1 calc R . .
H15C H 0.6661 0.2777 0.7592 0.093 Uiso 1 1 calc R . .
N1 N 0.3812(3) 0.2870(5) 0.7101(3) 0.0324(9) Uani 1 1 d . . .
N2 N 0.4972(3) 0.3057(5) 0.6144(3) 0.0353(10) Uani 1 1 d . . .
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C3 0.055(4) 0.046(4) 0.056(4) 0.007(3) -0.010(3) -0.006(3)
C4 0.076(5) 0.050(4) 0.044(4) -0.012(3) 0.001(3) -0.011(3)
C5 0.061(4) 0.027(3) 0.057(4) -0.012(3) 0.024(3) -0.008(3)
C6 0.038(3) 0.031(3) 0.048(3) -0.004(2) 0.017(2) -0.002(2)
C7 0.040(3) 0.049(3) 0.037(3) -0.002(3) 0.017(2) 0.000(2)
C8 0.079(5) 0.065(5) 0.061(4) -0.011(4) 0.037(4) -0.007(4)
C9 0.058(4) 0.045(3) 0.050(4) 0.016(3) 0.019(3) 0.003(3)
C10 0.036(3) 0.087(5) 0.042(3) 0.007(3) 0.011(3) -0.003(3)
C11 0.057(3) 0.025(3) 0.041(3) 0.004(2) 0.022(3) 0.003(2)
C12 0.050(3) 0.026(3) 0.059(4) 0.005(3) 0.026(3) 0.002(2)
C13 0.066(4) 0.037(3) 0.073(4) 0.015(3) 0.031(4) 0.012(3)
C14 0.063(4) 0.039(3) 0.092(5) 0.011(3) 0.036(4) 0.003(3)
C15 0.044(3) 0.098(6) 0.047(4) -0.007(4) 0.017(3) 0.002(4)
N1 0.028(2) 0.026(2) 0.044(3) 0.0009(18) 0.0109(19) 0.0000(16)
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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
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used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.
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C3 C4 1.520(10) . ?
C4 C5 1.505(9) . ?
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_refine_special_details
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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 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.
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C2 C -0.2360(4) 0.7609(6) 0.3094(4) 0.0219(16) Uani 1 1 d . . .
H2A H -0.2534 0.8077 0.3469 0.026 Uiso 1 1 calc R . .
H2B H -0.2768 0.7168 0.2935 0.026 Uiso 1 1 calc R . .
C3 C -0.2083(4) 0.8274(6) 0.2500(4) 0.0289(18) Uani 1 1 d . . .
H3A H -0.1711 0.8773 0.2666 0.035 Uiso 1 1 calc R . .
H3B H -0.2481 0.8688 0.2294 0.035 Uiso 1 1 calc R . .
C4 C -0.1766(4) 0.7530(6) 0.1966(4) 0.0262(17) Uani 1 1 d . . .
H4A H -0.2153 0.7093 0.1761 0.031 Uiso 1 1 calc R . .
H4B H -0.1539 0.7946 0.1592 0.031 Uiso 1 1 calc R . .
C5 C -0.1790(4) 0.6748(5) 0.4107(4) 0.0176(14) Uani 1 1 d . . .
H5A H -0.1773 0.7449 0.4337 0.021 Uiso 1 1 calc R . .
H5B H -0.1343 0.6369 0.4233 0.021 Uiso 1 1 calc R . .
C6 C -0.2434(4) 0.6122(6) 0.4414(4) 0.0226(16) Uani 1 1 d . . .
C7 C -0.3147(5) 0.6739(7) 0.4427(5) 0.041(2) Uani 1 1 d . . .
H7A H -0.3500 0.6350 0.4704 0.061 Uiso 1 1 calc R . .
H7B H -0.3327 0.6820 0.3954 0.061 Uiso 1 1 calc R . .
H7C H -0.3067 0.7440 0.4631 0.061 Uiso 1 1 calc R . .
C8 C -0.2531(5) 0.5080(7) 0.4022(5) 0.045(2) Uani 1 1 d . . .
H8A H -0.2078 0.4685 0.4026 0.068 Uiso 1 1 calc R . .
H8B H -0.2671 0.5231 0.3544 0.068 Uiso 1 1 calc R . .
H8C H -0.2907 0.4658 0.4246 0.068 Uiso 1 1 calc R . .
C9 C -0.2222(5) 0.5897(8) 0.5164(5) 0.041(2) Uani 1 1 d . . .
H9A H -0.2574 0.5415 0.5372 0.061 Uiso 1 1 calc R . .
H9B H -0.2213 0.6563 0.5425 0.061 Uiso 1 1 calc R . .
H9C H -0.1745 0.5571 0.5176 0.061 Uiso 1 1 calc R . .
C10 C -0.0628(4) 0.6515(6) 0.1854(4) 0.0199(15) Uani 1 1 d . . .
H10A H -0.0251 0.6198 0.2151 0.024 Uiso 1 1 calc R . .
H10B H -0.0420 0.7160 0.1642 0.024 Uiso 1 1 calc R . .
C11 C -0.0805(4) 0.5716(6) 0.1264(4) 0.0233(16) Uani 1 1 d . . .
C12 C -0.0068(5) 0.5271(8) 0.1035(4) 0.042(2) Uani 1 1 d . . .
H12A H 0.0150 0.4878 0.1419 0.063 Uiso 1 1 calc R . .
H12B H 0.0249 0.5857 0.0902 0.063 Uiso 1 1 calc R . .
H12C H -0.0134 0.4796 0.0640 0.063 Uiso 1 1 calc R . .
C13 C -0.1158(5) 0.6242(7) 0.0631(4) 0.033(2) Uani 1 1 d . . .
H13A H -0.1192 0.5726 0.0253 0.049 Uiso 1 1 calc R . .
H13B H -0.0866 0.6846 0.0481 0.049 Uiso 1 1 calc R . .
H13C H -0.1641 0.6488 0.0755 0.049 Uiso 1 1 calc R . .
C14 C -0.1274(5) 0.4822(6) 0.1528(4) 0.0303(19) Uani 1 1 d . . .
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C15 C 0.0290(4) 0.7266(5) 0.3259(4) 0.0186(15) Uani 1 1 d . . .

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H16 H -0.0563 0.7462 0.3912 0.022 Uiso 1 1 calc R . .
C17 C 0.0289(4) 0.7077(7) 0.4602(4) 0.0285(18) Uani 1 1 d . . .
H17A H -0.0089 0.6968 0.4954 0.034 Uiso 1 1 calc R . .
H17B H 0.0573 0.7705 0.4739 0.034 Uiso 1 1 calc R . .
C18 C 0.0781(4) 0.6110(6) 0.4593(4) 0.0269(18) Uani 1 1 d . . .
H18A H 0.1270 0.6328 0.4447 0.032 Uiso 1 1 calc R . .
H18B H 0.0817 0.5815 0.5066 0.032 Uiso 1 1 calc R . .
C19 C 0.0507(4) 0.5265(6) 0.4109(4) 0.0208(16) Uani 1 1 d . . .
H19 H 0.0163 0.4779 0.4284 0.025 Uiso 1 1 calc R . .
C20 C 0.0722(4) 0.5147(6) 0.3426(4) 0.0234(16) Uani 1 1 d . . .
H20 H 0.0520 0.4575 0.3173 0.028 Uiso 1 1 calc R . .
C21 C 0.1257(4) 0.5863(6) 0.3050(5) 0.0289(18) Uani 1 1 d . . .
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H21B H 0.1748 0.5709 0.3217 0.035 Uiso 1 1 calc R . .
C22 C 0.1089(4) 0.7050(6) 0.3165(4) 0.0239(17) Uani 1 1 d . . .
H22A H 0.1350 0.7299 0.3581 0.029 Uiso 1 1 calc R . .
H22B H 0.1267 0.7459 0.2764 0.029 Uiso 1 1 calc R . .
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N2 N -0.1224(3) 0.6835(5) 0.2295(3) 0.0199(13) Uani 1 1 d . . .
Cl1 Cl -0.08311(10) 0.41376(13) 0.33235(9) 0.0225(4) Uani 1 1 d . . .
Ir1 Ir -0.033220(14) 0.586648(19) 0.341277(13) 0.01467(10) Uani 1 1 d . . .

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C19 0.007(3) 0.025(4) 0.031(4) 0.008(3) -0.005(3) 0.003(3)
C20 0.008(3) 0.022(4) 0.040(5) -0.001(3) 0.002(3) 0.004(3)
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Cl1 0.0264(9) 0.0159(8) 0.0252(9) -0.0018(7) -0.0005(7) -0.0026(7)
Ir1 0.01362(15) 0.01377(14) 0.01661(15) -0.00053(10) 0.00026(11) 0.00077(10)

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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.
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N2 C4 H4B 109.7 . . ?
C3 C4 H4B 109.7 . . ?
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Ir1 C15 H15 87.4 . . ?
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'x-1/2, -y-1/2, z'

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_diffrn_radiation_source         'fine-focus sealed tube'
_diffrn_radiation_monochromator  graphite
_diffrn_measurement_device_type  'Nonius KappaCCD'
_diffrn_measurement_method       'CCD scans'
_diffrn_detector_area_resol_mean ?
_diffrn_standards_number         ?
_diffrn_standards_interval_count ?
_diffrn_standards_interval_time  ?
_diffrn_standards_decay_%        ?
_diffrn_reflns_number            11361
_diffrn_reflns_av_R_equivalents  0.0526
_diffrn_reflns_av_sigmaI/netI    0.0626
_diffrn_reflns_limit_h_min       -15
_diffrn_reflns_limit_h_max       15
_diffrn_reflns_limit_k_min       -20
_diffrn_reflns_limit_k_max       20
_diffrn_reflns_limit_l_min       -19
_diffrn_reflns_limit_l_max       18
_diffrn_reflns_theta_min         2.70
_diffrn_reflns_theta_max         28.27
_reflns_number_total             5876
_reflns_number_gt                4384
_reflns_threshold_expression     >2\s(I)

_computing_data_collection       'cOLLECT,Nonius BV, 1997-2001'
_computing_cell_refinement       DENZO/SCALEPACK
_computing_data_reduction        DENZO/SCALEPACK
_computing_structure_solution    SHELXs-97
_computing_structure_refinement  SHELXL-97

_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^ > 2\s(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type           full
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
```

'calc w=1/[\s^2^(Fo^2^)+(0.0909P)^2^+22.8119P] 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 SHELXL
_refine_ls_extinction_coef 0.0023(3)
_refine_ls_extinction_expression Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^
_refine_ls_number_reflns 5876
_refine_ls_number_parameters 251
_refine_ls_number_restraints 0
_refine_ls_R_factor_all 0.0928
_refine_ls_R_factor_gt 0.0628
_refine_ls_wR_factor_ref 0.1759
_refine_ls_wR_factor_gt 0.1515
_refine_ls_goodness_of_fit_ref 1.003
_refine_ls_restrained_S_all 1.003
_refine_ls_shift/su_max 0.017
_refine_ls_shift/su_mean 0.004

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_atom_site_symmetry_multiplicity
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
_atom_site_disorder_group
C1 C 0.0151(7) 0.4851(6) 0.2693(6) 0.0281(17) Uani 1 1 d . . .
C2 C 0.1391(8) 0.4315(6) 0.4298(7) 0.037(2) Uani 1 1 d . . .
H2A H 0.1290 0.3809 0.4706 0.044 Uiso 1 1 calc R . .
H2B H 0.1091 0.4852 0.4547 0.044 Uiso 1 1 calc R . .
C3 C 0.2747(8) 0.4433(7) 0.4433(7) 0.041(2) Uani 1 1 d . . .
H3A H 0.3204 0.4403 0.5137 0.049 Uiso 1 1 calc R . .
H3B H 0.3032 0.3941 0.4102 0.049 Uiso 1 1 calc R . .
C4 C 0.3012(9) 0.5294(7) 0.4031(8) 0.042(2) Uani 1 1 d . . .
H4A H 0.3891 0.5326 0.4092 0.051 Uiso 1 1 calc R . .
H4B H 0.2819 0.5788 0.4412 0.051 Uiso 1 1 calc R . .
C5 C 0.2257(8) 0.5390(7) 0.2957(7) 0.037(2) Uani 1 1 d . . .
H5A H 0.2589 0.5894 0.2677 0.045 Uiso 1 1 calc R . .
H5B H 0.2368 0.4848 0.2607 0.045 Uiso 1 1 calc R . .
C6 C -0.0092(7) 0.3318(6) 0.3133(7) 0.036(2) Uani 1 1 d . . .
H6A H -0.0811 0.3388 0.2548 0.043 Uiso 1 1 calc R . .
H6B H -0.0397 0.3223 0.3697 0.043 Uiso 1 1 calc R . .
C7 C 0.0611(8) 0.2475(6) 0.2998(7) 0.036(2) Uani 1 1 d . . .
C8 C 0.1214(9) 0.2591(7) 0.2202(8) 0.043(2) Uani 1 1 d . . .
H8A H 0.0585 0.2645 0.1568 0.064 Uiso 1 1 calc R . .
H8B H 0.1729 0.2076 0.2194 0.064 Uiso 1 1 calc R . .

H8C H 0.1718 0.3129 0.2330 0.064 Uiso 1 1 calc R . .
C9 C 0.1563(10) 0.2173(7) 0.3956(8) 0.054(3) Uani 1 1 d . . .
H9A H 0.1864 0.1582 0.3866 0.080 Uiso 1 1 calc R . .
H9B H 0.1182 0.2154 0.4478 0.080 Uiso 1 1 calc R . .
H9C H 0.2245 0.2593 0.4131 0.080 Uiso 1 1 calc R . .
C10 C -0.0351(9) 0.1734(6) 0.2696(8) 0.045(2) Uani 1 1 d . . .
H10A H -0.0943 0.1882 0.2066 0.067 Uiso 1 1 calc R . .
H10B H -0.0773 0.1676 0.3190 0.067 Uiso 1 1 calc R . .
H10C H 0.0051 0.1172 0.2641 0.067 Uiso 1 1 calc R . .
C11 C 0.0443(8) 0.6398(6) 0.2348(6) 0.0340(19) Uani 1 1 d . . .
H11A H -0.0441 0.6322 0.2014 0.041 Uiso 1 1 calc R . .
H11B H 0.0829 0.6552 0.1842 0.041 Uiso 1 1 calc R . .
C12 C 0.0603(8) 0.7202(6) 0.3049(7) 0.0348(19) Uani 1 1 d . . .
C13 C 0.0239(9) 0.6945(7) 0.3950(8) 0.045(2) Uani 1 1 d . . .
H13A H 0.0159 0.7481 0.4310 0.068 Uiso 1 1 calc R . .
H13B H 0.0867 0.6559 0.4371 0.068 Uiso 1 1 calc R . .
H13C H -0.0543 0.6629 0.3746 0.068 Uiso 1 1 calc R . .
C14 C 0.1901(9) 0.7563(7) 0.3336(7) 0.043(2) Uani 1 1 d . . .
H14A H 0.2096 0.7750 0.2749 0.065 Uiso 1 1 calc R . .
H14B H 0.2472 0.7099 0.3670 0.065 Uiso 1 1 calc R . .
H14C H 0.1969 0.8072 0.3772 0.065 Uiso 1 1 calc R . .
C15 C -0.0247(9) 0.7918(7) 0.2462(9) 0.051(3) Uani 1 1 d . . .
H15A H -0.1095 0.7720 0.2311 0.076 Uiso 1 1 calc R . .
H15B H -0.0056 0.8028 0.1854 0.076 Uiso 1 1 calc R . .
H15C H -0.0134 0.8466 0.2845 0.076 Uiso 1 1 calc R . .
C16 C -0.1108(9) 0.5157(6) 0.0521(7) 0.036(2) Uani 1 1 d . . .
H16 H -0.0402 0.5448 0.0925 0.043 Uiso 1 1 calc R . .
C17 C -0.1221(9) 0.4228(6) 0.0636(7) 0.038(2) Uani 1 1 d . . .
H17 H -0.0572 0.3937 0.1109 0.045 Uiso 1 1 calc R . .
C18 C -0.2286(11) 0.3663(9) 0.0068(10) 0.066(4) Uani 1 1 d . . .
H18A H -0.2287 0.3637 -0.0619 0.079 Uiso 1 1 calc R . .
H18B H -0.2158 0.3052 0.0331 0.079 Uiso 1 1 calc R . .
C19 C -0.3505(10) 0.3970(8) 0.0083(8) 0.053(3) Uani 1 1 d . . .
H19A H -0.4011 0.3445 0.0107 0.063 Uiso 1 1 calc R . .
H19B H -0.3905 0.4288 -0.0534 0.063 Uiso 1 1 calc R . .
C20 C -0.3479(7) 0.4571(7) 0.0924(7) 0.038(2) Uani 1 1 d . . .
H20 H -0.3582 0.4309 0.1494 0.045 Uiso 1 1 calc R . .
C21 C -0.3317(8) 0.5473(7) 0.0922(8) 0.045(2) Uani 1 1 d . . .
H21 H -0.3335 0.5784 0.1491 0.054 Uiso 1 1 calc R . .
C22 C -0.3116(11) 0.6012(9) 0.0119(10) 0.068(4) Uani 1 1 d . . .
H22A H -0.2973 0.6636 0.0339 0.082 Uiso 1 1 calc R . .
H22B H -0.3869 0.5998 -0.0445 0.082 Uiso 1 1 calc R . .
C23 C -0.2062(12) 0.5708(9) -0.0217(9) 0.063(3) Uani 1 1 d . . .
H23A H -0.1658 0.6237 -0.0385 0.076 Uiso 1 1 calc R . .
H23B H -0.2389 0.5354 -0.0819 0.076 Uiso 1 1 calc R . .
N1 N 0.0614(6) 0.4157(5) 0.3289(5) 0.0336(16) Uani 1 1 d . . .
N2 N 0.0934(6) 0.5536(5) 0.2767(6) 0.0362(17) Uani 1 1 d . . .
Cl1 Cl -0.2263(2) 0.50356(14) 0.32177(17) 0.0334(5) Uani 1 1 d . . .
Ir1 Ir -0.16259(3) 0.48846(2) 0.18006(2) 0.03090(16) Uani 1 1 d . . .

loop_
_atom_site_aniso_label
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_atom_site_aniso_U_22

_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
C1 0.018(3) 0.043(5) 0.018(4) -0.005(3) -0.002(3) 0.005(3)
C2 0.036(4) 0.036(5) 0.034(5) -0.003(4) 0.005(4) 0.001(4)
C3 0.030(4) 0.048(6) 0.041(6) 0.000(4) 0.004(4) 0.001(4)
C4 0.031(5) 0.050(6) 0.043(6) 0.001(5) 0.007(4) -0.005(4)
C5 0.027(4) 0.043(5) 0.044(6) -0.004(4) 0.015(4) -0.001(4)
C6 0.024(4) 0.037(5) 0.044(6) 0.000(4) 0.008(4) -0.001(3)
C7 0.039(5) 0.030(4) 0.041(5) -0.001(4) 0.018(4) -0.001(4)
C8 0.049(5) 0.042(5) 0.044(6) 0.000(4) 0.025(5) 0.001(4)
C9 0.061(7) 0.042(6) 0.049(7) -0.002(5) 0.005(5) 0.015(5)
C10 0.039(5) 0.039(5) 0.062(7) -0.004(5) 0.022(5) -0.005(4)
C11 0.032(4) 0.046(5) 0.029(5) 0.000(4) 0.017(4) 0.002(4)
C12 0.026(4) 0.040(5) 0.038(5) 0.003(4) 0.009(4) -0.004(3)
C13 0.050(6) 0.043(5) 0.047(6) -0.005(5) 0.022(5) 0.006(4)
C14 0.046(5) 0.037(5) 0.043(6) -0.003(4) 0.009(4) -0.005(4)
C15 0.040(5) 0.039(5) 0.068(8) 0.007(5) 0.008(5) 0.004(4)
C16 0.034(4) 0.052(5) 0.021(4) 0.014(4) 0.008(4) -0.001(4)
C17 0.043(5) 0.043(5) 0.029(5) -0.002(4) 0.015(4) 0.006(4)
C18 0.058(7) 0.063(7) 0.068(8) -0.038(6) 0.008(6) -0.003(6)
C19 0.055(6) 0.064(7) 0.037(6) -0.015(5) 0.011(5) -0.022(5)
C20 0.023(4) 0.052(5) 0.035(5) 0.003(4) 0.004(4) -0.002(4)
C21 0.027(4) 0.055(6) 0.039(6) 0.012(5) -0.010(4) -0.002(4)
C22 0.051(7) 0.072(8) 0.076(9) 0.032(7) 0.011(6) 0.000(6)
C23 0.075(8) 0.068(8) 0.046(7) 0.027(6) 0.019(6) 0.021(6)
N1 0.030(4) 0.031(4) 0.032(4) 0.001(3) 0.000(3) -0.001(3)
N2 0.027(3) 0.036(4) 0.045(5) 0.000(3) 0.010(3) -0.003(3)
Cl1 0.0356(11) 0.0378(11) 0.0300(11) -0.0039(8) 0.0147(9) -0.0016(8)
Ir1 0.0259(2) 0.0346(2) 0.0308(2) 0.00006(13) 0.00664(14) -0.00051(12)

_geom_special_details
;
All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell s.u.'s are taken
into account individually in the estimation of s.u.'s in distances, angles
and torsion angles; correlations between s.u.'s in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.
;

loop_
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_geom_bond_site_symmetry_2
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C1 N2 1.354(11) . ?
C1 N1 1.356(11) . ?
C1 Ir1 2.057(8) . ?
C2 N1 1.474(11) . ?
C2 C3 1.524(12) . ?
C2 H2A 0.9900 . ?

C2 H2B 0.9900 . ?
C3 C4 1.491(14) . ?
C3 H3A 0.9900 . ?
C3 H3B 0.9900 . ?
C4 C5 1.532(14) . ?
C4 H4A 0.9900 . ?
C4 H4B 0.9900 . ?
C5 N2 1.480(11) . ?
C5 H5A 0.9900 . ?
C5 H5B 0.9900 . ?
C6 N1 1.484(11) . ?
C6 C7 1.551(12) . ?
C6 H6A 0.9900 . ?
C6 H6B 0.9900 . ?
C7 C8 1.522(13) . ?
C7 C10 1.539(12) . ?
C7 C9 1.545(14) . ?
C8 H8A 0.9800 . ?
C8 H8B 0.9800 . ?
C8 H8C 0.9800 . ?
C9 H9A 0.9800 . ?
C9 H9B 0.9800 . ?
C9 H9C 0.9800 . ?
C10 H10A 0.9800 . ?
C10 H10B 0.9800 . ?
C10 H10C 0.9800 . ?
C11 N2 1.471(12) . ?
C11 C12 1.551(13) . ?
C11 H11A 0.9900 . ?
C11 H11B 0.9900 . ?
C12 C14 1.525(12) . ?
C12 C13 1.531(13) . ?
C12 C15 1.527(13) . ?
C13 H13A 0.9800 . ?
C13 H13B 0.9800 . ?
C13 H13C 0.9800 . ?
C14 H14A 0.9800 . ?
C14 H14B 0.9800 . ?
C14 H14C 0.9800 . ?
C15 H15A 0.9800 . ?
C15 H15B 0.9800 . ?
C15 H15C 0.9800 . ?
C16 C17 1.422(13) . ?
C16 C23 1.521(13) . ?
C16 Ir1 2.143(9) . ?
C16 H16 0.9500 . ?
C17 C18 1.512(14) . ?
C17 Ir1 2.120(9) . ?
C17 H17 0.9500 . ?
C18 C19 1.485(16) . ?
C18 H18A 0.9900 . ?
C18 H18B 0.9900 . ?
C19 C20 1.507(14) . ?
C19 H19A 0.9900 . ?

C19 H19B 0.9900 . ?
C20 C21 1.372(16) . ?
C20 Ir1 2.173(8) . ?
C20 H20 0.9500 . ?
C21 C22 1.489(15) . ?
C21 Ir1 2.162(9) . ?
C21 H21 0.9500 . ?
C22 C23 1.509(17) . ?
C22 H22A 0.9900 . ?
C22 H22B 0.9900 . ?
C23 H23A 0.9900 . ?
C23 H23B 0.9900 . ?
Cl1 Ir1 2.381(2) . ?

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N2 C1 N1 114.6(7) . . ?
N2 C1 Ir1 122.8(6) . . ?
N1 C1 Ir1 122.5(6) . . ?
N1 C2 C3 115.7(8) . . ?
N1 C2 H2A 108.4 . . ?
C3 C2 H2A 108.3 . . ?
N1 C2 H2B 108.4 . . ?
C3 C2 H2B 108.4 . . ?
H2A C2 H2B 107.4 . . ?
C4 C3 C2 112.1(8) . . ?
C4 C3 H3A 109.2 . . ?
C2 C3 H3A 109.2 . . ?
C4 C3 H3B 109.2 . . ?
C2 C3 H3B 109.2 . . ?
H3A C3 H3B 107.9 . . ?
C3 C4 C5 110.7(8) . . ?
C3 C4 H4A 109.5 . . ?
C5 C4 H4A 109.5 . . ?
C3 C4 H4B 109.5 . . ?
C5 C4 H4B 109.5 . . ?
H4A C4 H4B 108.1 . . ?
N2 C5 C4 115.8(8) . . ?
N2 C5 H5A 108.3 . . ?
C4 C5 H5A 108.3 . . ?
N2 C5 H5B 108.3 . . ?
C4 C5 H5B 108.3 . . ?
H5A C5 H5B 107.4 . . ?
N1 C6 C7 115.8(7) . . ?
N1 C6 H6A 108.3 . . ?
C7 C6 H6A 108.3 . . ?
N1 C6 H6B 108.3 . . ?
C7 C6 H6B 108.3 . . ?

H6A C6 H6B 107.4 . . ?
C8 C7 C10 109.0(8) . . ?
C8 C7 C9 110.2(8) . . ?
C10 C7 C9 106.9(8) . . ?
C8 C7 C6 112.1(8) . . ?
C10 C7 C6 105.8(7) . . ?
C9 C7 C6 112.6(8) . . ?
C7 C8 H8A 109.5 . . ?
C7 C8 H8B 109.5 . . ?
H8A C8 H8B 109.5 . . ?
C7 C8 H8C 109.5 . . ?
H8A C8 H8C 109.5 . . ?
H8B C8 H8C 109.5 . . ?
C7 C9 H9A 109.5 . . ?
C7 C9 H9B 109.5 . . ?
H9A C9 H9B 109.5 . . ?
C7 C9 H9C 109.5 . . ?
H9A C9 H9C 109.5 . . ?
H9B C9 H9C 109.5 . . ?
C7 C10 H10A 109.5 . . ?
C7 C10 H10B 109.5 . . ?
H10A C10 H10B 109.5 . . ?
C7 C10 H10C 109.5 . . ?
H10A C10 H10C 109.5 . . ?
H10B C10 H10C 109.5 . . ?
N2 C11 C12 118.0(7) . . ?
N2 C11 H11A 107.8 . . ?
C12 C11 H11A 107.8 . . ?
N2 C11 H11B 107.8 . . ?
C12 C11 H11B 107.8 . . ?
H11A C11 H11B 107.2 . . ?
C14 C12 C13 111.1(8) . . ?
C14 C12 C15 107.8(8) . . ?
C13 C12 C15 110.7(8) . . ?
C14 C12 C11 111.5(8) . . ?
C13 C12 C11 110.2(8) . . ?
C15 C12 C11 105.3(8) . . ?
C12 C13 H13A 109.5 . . ?
C12 C13 H13B 109.5 . . ?
H13A C13 H13B 109.5 . . ?
C12 C13 H13C 109.5 . . ?
H13A C13 H13C 109.5 . . ?
H13B C13 H13C 109.5 . . ?
C12 C14 H14A 109.5 . . ?
C12 C14 H14B 109.5 . . ?
H14A C14 H14B 109.5 . . ?
C12 C14 H14C 109.5 . . ?
H14A C14 H14C 109.5 . . ?
H14B C14 H14C 109.5 . . ?
C12 C15 H15A 109.5 . . ?
C12 C15 H15B 109.5 . . ?
H15A C15 H15B 109.5 . . ?
C12 C15 H15C 109.5 . . ?
H15A C15 H15C 109.5 . . ?

H15B C15 H15C 109.5 . . ?
C17 C16 C23 123.2(10) . . ?
C17 C16 Ir1 69.6(5) . . ?
C23 C16 Ir1 112.0(7) . . ?
C17 C16 H16 118.4 . . ?
C23 C16 H16 118.4 . . ?
Ir1 C16 H16 88.4 . . ?
C16 C17 C18 125.5(9) . . ?
C16 C17 Ir1 71.4(5) . . ?
C18 C17 Ir1 110.7(7) . . ?
C16 C17 H17 117.3 . . ?
C18 C17 H17 117.3 . . ?
Ir1 C17 H17 87.9 . . ?
C19 C18 C17 115.5(9) . . ?
C19 C18 H18A 108.4 . . ?
C17 C18 H18A 108.4 . . ?
C19 C18 H18B 108.4 . . ?
C17 C18 H18B 108.4 . . ?
H18A C18 H18B 107.5 . . ?
C18 C19 C20 114.4(8) . . ?
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 . . ?
C21 C20 C19 124.3(9) . . ?
C21 C20 Ir1 71.1(5) . . ?
C19 C20 Ir1 111.9(6) . . ?
C21 C20 H20 117.9 . . ?
C19 C20 H20 117.9 . . ?
Ir1 C20 H20 87.0 . . ?
C20 C21 C22 126.6(11) . . ?
C20 C21 Ir1 72.0(5) . . ?
C22 C21 Ir1 110.7(7) . . ?
C20 C21 H21 116.7 . . ?
C22 C21 H21 116.7 . . ?
Ir1 C21 H21 87.2 . . ?
C21 C22 C23 114.5(10) . . ?
C21 C22 H22A 108.6 . . ?
C23 C22 H22A 108.6 . . ?
C21 C22 H22B 108.7 . . ?
C23 C22 H22B 108.6 . . ?
H22A C22 H22B 107.6 . . ?
C22 C23 C16 115.0(9) . . ?
C22 C23 H23A 108.5 . . ?
C16 C23 H23A 108.5 . . ?
C22 C23 H23B 108.5 . . ?
C16 C23 H23B 108.5 . . ?
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C16 Ir1 C20 91.4(4) . . ?
C21 Ir1 C20 36.9(4) . . ?
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H H 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2\s(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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C1 C 0.8087(8) 0.1013(5) 0.2978(3) 0.0395(16) Uani 1 1 d . . .
C2 C 0.7261(8) -0.0637(4) 0.2415(3) 0.0407(14) Uani 1 1 d . . .
H2A H 0.8329 -0.0879 0.2102 0.049 Uiso 1 1 calc R . .
H2B H 0.6054 -0.0890 0.2197 0.049 Uiso 1 1 calc R . .
C3 C 0.7484(8) -0.1050(4) 0.3188(3) 0.0382(13) Uani 1 1 d . . .
H3A H 0.7647 -0.1804 0.3171 0.046 Uiso 1 1 calc R . .
H3B H 0.6326 -0.0895 0.3481 0.046 Uiso 1 1 calc R . .
C4 C 0.9220(8) -0.0558(4) 0.3551(3) 0.0422(16) Uani 1 1 d . . .
H4A H 0.9309 -0.0780 0.4074 0.051 Uiso 1 1 calc R . .
H4B H 1.0395 -0.0780 0.3293 0.051 Uiso 1 1 calc R . .
C5 C 0.6236(7) 0.1074(4) 0.1855(3) 0.0422(15) Uani 1 1 d . . .
H5A H 0.5879 0.1753 0.2057 0.051 Uiso 1 1 calc R . .
H5B H 0.5040 0.0706 0.1734 0.051 Uiso 1 1 calc R . .
C6 C 0.7373(8) 0.1238(4) 0.1134(3) 0.0449(14) Uani 1 1 d . . .
C7 C 0.7635(14) 0.0224(4) 0.0726(3) 0.0591(18) Uani 1 1 d . . .
H7A H 0.8013 0.0359 0.0213 0.089 Uiso 1 1 calc R . .
H7B H 0.6432 -0.0159 0.0732 0.089 Uiso 1 1 calc R . .
H7C H 0.8629 -0.0180 0.0972 0.089 Uiso 1 1 calc R . .
C8 C 0.9300(9) 0.1726(5) 0.1310(4) 0.064(2) Uani 1 1 d . . .
H8A H 1.0048 0.1258 0.1619 0.096 Uiso 1 1 calc R . .
H8B H 0.9098 0.2373 0.1575 0.096 Uiso 1 1 calc R . .
H8C H 0.9989 0.1861 0.0848 0.096 Uiso 1 1 calc R . .
C9 C 0.6112(12) 0.1975(6) 0.0664(4) 0.082(3) Uani 1 1 d . . .
H9A H 0.5924 0.2620 0.0934 0.124 Uiso 1 1 calc R . .
H9B H 0.4872 0.1653 0.0570 0.124 Uiso 1 1 calc R . .
H9C H 0.6747 0.2117 0.0191 0.124 Uiso 1 1 calc R . .
C10 C 0.9965(7) 0.1207(5) 0.4078(3) 0.0391(14) Uani 1 1 d . . .
H10A H 0.9742 0.0896 0.4570 0.047 Uiso 1 1 calc R . .
H10B H 0.9330 0.1885 0.4075 0.047 Uiso 1 1 calc R . .
C11 C 1.2129(8) 0.1374(5) 0.3981(3) 0.0382(15) Uani 1 1 d . . .
C12 C 1.3277(9) 0.0423(6) 0.4174(4) 0.068(2) Uani 1 1 d . . .
H12A H 1.4638 0.0594 0.4183 0.102 Uiso 1 1 calc R . .
H12B H 1.3044 -0.0110 0.3803 0.102 Uiso 1 1 calc R . .
H12C H 1.2888 0.0171 0.4663 0.102 Uiso 1 1 calc R . .
C13 C 1.2652(10) 0.2223(5) 0.4527(3) 0.0550(17) Uani 1 1 d . . .
H13A H 1.4011 0.2387 0.4479 0.083 Uiso 1 1 calc R . .
H13B H 1.2390 0.1991 0.5033 0.083 Uiso 1 1 calc R . .
H13C H 1.1891 0.2836 0.4421 0.083 Uiso 1 1 calc R . .

C14 C 1.2542(8) 0.1717(5) 0.3202(3) 0.0503(15) Uani 1 1 d . . .
H14A H 1.1721 0.2300 0.3076 0.075 Uiso 1 1 calc R . .
H14B H 1.2291 0.1150 0.2859 0.075 Uiso 1 1 calc R . .
H14C H 1.3883 0.1923 0.3162 0.075 Uiso 1 1 calc R . .
N1 N 0.7248(6) 0.0496(3) 0.2434(2) 0.0357(11) Uani 1 1 d . . .
N2 N 0.9050(6) 0.0556(4) 0.3516(3) 0.0373(12) Uani 1 1 d . . .
Br1 Br 1.23007(7) -0.12489(5) 0.18590(4) 0.0497(3) Uani 1 1 d . . .

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C2 0.042(3) 0.035(3) 0.045(4) -0.007(3) -0.005(3) -0.006(3)
C3 0.050(3) 0.027(3) 0.038(3) 0.006(3) 0.001(4) -0.001(3)
C4 0.057(4) 0.038(4) 0.032(4) -0.004(3) 0.004(3) 0.001(3)
C5 0.037(3) 0.049(4) 0.040(4) -0.005(4) -0.009(3) 0.001(3)
C6 0.061(4) 0.048(3) 0.026(3) -0.004(3) -0.011(3) 0.008(5)
C7 0.076(5) 0.069(4) 0.032(4) -0.006(3) -0.002(4) 0.011(5)
C8 0.081(5) 0.074(5) 0.037(5) 0.010(4) 0.007(4) -0.025(4)
C9 0.119(7) 0.076(6) 0.052(6) 0.019(5) 0.004(5) 0.037(5)
C10 0.055(3) 0.038(4) 0.024(3) -0.004(3) -0.006(3) -0.004(3)
C11 0.043(3) 0.040(4) 0.031(3) -0.001(3) 0.005(3) 0.004(3)
C12 0.058(5) 0.077(5) 0.069(5) 0.001(4) -0.014(4) 0.015(4)
C13 0.053(4) 0.060(4) 0.052(4) -0.014(3) 0.001(4) -0.009(4)
C14 0.038(3) 0.070(4) 0.043(4) -0.003(3) 0.008(4) -0.012(3)
N1 0.038(3) 0.043(3) 0.026(3) 0.003(2) 0.003(3) -0.002(2)
N2 0.046(3) 0.036(3) 0.030(3) -0.003(3) 0.002(3) -0.003(2)
Br1 0.0448(3) 0.0441(4) 0.0603(4) -0.0014(3) 0.0010(4) -0.0041(4)

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All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell s.u.'s are taken
into account individually in the estimation of s.u.'s in distances, angles
and torsion angles; correlations between s.u.'s in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.
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C2 C3 1.505(7) . ?

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C3 C4 1.524(8) . ?
C3 H3A 0.9900 . ?
C3 H3B 0.9900 . ?
C4 N2 1.458(7) . ?
C4 H4A 0.9900 . ?
C4 H4B 0.9900 . ?
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C5 C6 1.543(8) . ?
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C6 C7 1.524(8) . ?
C6 C8 1.526(8) . ?
C6 C9 1.557(8) . ?
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C7 H7B 0.9800 . ?
C7 H7C 0.9800 . ?
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C8 H8C 0.9800 . ?
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N1 C2 H2B 109.7 . . ?

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C2 C3 H3B 109.8 . . ?
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C3 C4 H4B 109.8 . . ?
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N1 C5 H5B 108.5 . . ?
C6 C5 H5B 108.5 . . ?
H5A C5 H5B 107.5 . . ?
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C7 C6 C5 110.5(5) . . ?
C8 C6 C5 109.8(4) . . ?
C7 C6 C9 109.9(5) . . ?
C8 C6 C9 111.0(6) . . ?
C5 C6 C9 104.7(5) . . ?
C6 C7 H7A 109.5 . . ?
C6 C7 H7B 109.5 . . ?
H7A C7 H7B 109.5 . . ?
C6 C7 H7C 109.5 . . ?
H7A C7 H7C 109.5 . . ?
H7B C7 H7C 109.5 . . ?
C6 C8 H8A 109.5 . . ?
C6 C8 H8B 109.5 . . ?
H8A C8 H8B 109.5 . . ?
C6 C8 H8C 109.5 . . ?
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H8B C8 H8C 109.5 . . ?
C6 C9 H9A 109.5 . . ?
C6 C9 H9B 109.5 . . ?
H9A C9 H9B 109.5 . . ?
C6 C9 H9C 109.5 . . ?
H9A C9 H9C 109.5 . . ?
H9B C9 H9C 109.5 . . ?
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N2 C10 H10B 108.4 . . ?
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H12A C12 H12B 109.5 . . ?
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H13A C13 H13C 109.5 . . ?
H13B C13 H13C 109.5 . . ?
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H14A C14 H14B 109.5 . . ?
C11 C14 H14C 109.5 . . ?
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H14B C14 H14C 109.5 . . ?
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C1 N1 C2 121.5(5) . . ?
C5 N1 C2 119.8(4) . . ?
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C4 N2 C10 120.6(5) . . ?

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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'
Cl Cl 0.1484 0.1585 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
I I -0.4742 1.8119 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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_exptl_absorpt_correction_T_max 0.7704
_exptl_absorpt_process_details Denzo/Scalepack


_exptl_special_details
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?
;

_diffrn_ambient_temperature 150(2)

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_diffrn_radiation_wavelength     0.71073
_diffrn_radiation_type           MoK\a
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_diffrn_radiation_monochromator  graphite
_diffrn_measurement_device_type  'Nonius KappaCCD'
_diffrn_measurement_method       'CCD scans'
_diffrn_detector_area_resol_mean ?
_diffrn_standards_number         ?
_diffrn_standards_interval_count ?
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_diffrn_reflns_number            19185
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_diffrn_reflns_av_sigmaI/netI    0.0596
_diffrn_reflns_limit_h_min       -32
_diffrn_reflns_limit_h_max       35
_diffrn_reflns_limit_k_min       -14
_diffrn_reflns_limit_k_max       12
_diffrn_reflns_limit_l_min       -17
_diffrn_reflns_limit_l_max       19
_diffrn_reflns_theta_min         1.62
_diffrn_reflns_theta_max         30.48
_reflns_number_total             8832
_reflns_number_gt                5458
_reflns_threshold_expression     >2\s(I)

_computing_data_collection       'cOLLECT,Nonius BV, 1997-2001'
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_computing_data_reduction        DENZO/SCALEPACK
_computing_structure_solution    SHELXs-97
_computing_structure_refinement  SHELXL-97

_refine_special_details
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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2\s(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type           full
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
'calc w=1/[\s^2^(Fo^2^)+(0.1186P)^2^+9.8045P] 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     SHELXL
_refine_ls_extinction_coef       0.0036(4)
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_refine_ls_extinction_expression Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^
_refine_ls_abs_structure_details 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack 0.52(7)
_refine_ls_number_reflns 8832
_refine_ls_number_parameters 317
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_refine_ls_R_factor_all 0.1184
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C1 C -0.7898(5) 0.2435(9) -0.296(2) 0.031(4) Uani 1 1 d . . .
H1 H -0.7824 0.2423 -0.2281 0.037 Uiso 1 1 calc R . .
C2 C -0.8135(5) 0.3840(16) -0.4333(11) 0.038(3) Uani 1 1 d . . .
H2A H -0.8160 0.4799 -0.4448 0.045 Uiso 1 1 calc R . .
H2B H -0.8502 0.3479 -0.4344 0.045 Uiso 1 1 calc R . .
C3 C -0.7816(6) 0.3223(16) -0.5145(12) 0.052(4) Uani 1 1 d . . .
H3A H -0.8017 0.3335 -0.5760 0.063 Uiso 1 1 calc R . .
H3B H -0.7477 0.3712 -0.5212 0.063 Uiso 1 1 calc R . .
C4 C -0.7688(5) 0.1793(16) -0.5027(11) 0.053(4) Uani 1 1 d . . .
H4A H -0.7327 0.1707 -0.4744 0.063 Uiso 1 1 calc R . .
H4B H -0.7684 0.1372 -0.5677 0.063 Uiso 1 1 calc R . .
C5 C -0.8096(5) 0.1052(16) -0.4362(11) 0.039(4) Uani 1 1 d . . .
H5A H -0.8462 0.1358 -0.4511 0.047 Uiso 1 1 calc R . .
H5B H -0.8079 0.0098 -0.4498 0.047 Uiso 1 1 calc R . .
C6 C -0.7782(5) 0.4757(12) -0.2749(10) 0.051(3) Uani 1 1 d . . .
H6 H -0.7657 0.4419 -0.2105 0.062 Uiso 1 1 calc R . .
C7 C -0.8257(5) 0.5587(12) -0.2567(12) 0.071(4) Uani 1 1 d . . .
H7A H -0.8342 0.6092 -0.3155 0.107 Uiso 1 1 calc R . .
H7B H -0.8184 0.6192 -0.2029 0.107 Uiso 1 1 calc R . .
H7C H -0.8561 0.5026 -0.2398 0.107 Uiso 1 1 calc R . .
C8 C -0.7313(4) 0.5534(11) -0.3208(12) 0.060(4) Uani 1 1 d . . .
H8A H -0.7426 0.5908 -0.3831 0.090 Uiso 1 1 calc R . .
H8B H -0.7010 0.4940 -0.3314 0.090 Uiso 1 1 calc R . .
H8C H -0.7205 0.6244 -0.2767 0.090 Uiso 1 1 calc R . .
C9 C -0.7986(4) 0.0132(11) -0.2688(9) 0.037(2) Uani 1 1 d . . .

H9 H -0.7968 0.0476 -0.2007 0.044 Uiso 1 1 calc R . .
C10 C -0.8503(4) -0.0657(11) -0.2781(10) 0.054(3) Uani 1 1 d . . .
H10A H -0.8806 -0.0103 -0.2594 0.081 Uiso 1 1 calc R . .
H10B H -0.8487 -0.1426 -0.2351 0.081 Uiso 1 1 calc R . .
H10C H -0.8547 -0.0948 -0.3456 0.081 Uiso 1 1 calc R . .
C11 C -0.7493(4) -0.0702(11) -0.2854(10) 0.051(3) Uani 1 1 d . . .
H11A H -0.7467 -0.0928 -0.3546 0.076 Uiso 1 1 calc R . .
H11B H -0.7519 -0.1508 -0.2467 0.076 Uiso 1 1 calc R . .
H11C H -0.7176 -0.0207 -0.2657 0.076 Uiso 1 1 calc R . .
C12 C -1.0369(5) 0.2507(6) -0.1370(17) 0.024(4) Uani 1 1 d . . .
H12 H -1.0245 0.2524 -0.2023 0.029 Uiso 1 1 calc R . .
C13 C -1.0596(5) 0.3884(16) 0.0069(10) 0.037(3) Uani 1 1 d . . .
H13A H -1.0590 0.4840 0.0201 0.044 Uiso 1 1 calc R . .
H13B H -1.0972 0.3590 0.0103 0.044 Uiso 1 1 calc R . .
C14 C -1.0289(5) 0.3203(14) 0.0838(11) 0.045(3) Uani 1 1 d . . .
H14A H -1.0477 0.3323 0.1465 0.054 Uiso 1 1 calc R . .
H14B H -0.9937 0.3634 0.0897 0.054 Uiso 1 1 calc R . .
C15 C -1.0200(5) 0.1721(14) 0.0678(12) 0.046(3) Uani 1 1 d . . .
H15A H -0.9839 0.1597 0.0401 0.055 Uiso 1 1 calc R . .
H15B H -1.0206 0.1280 0.1320 0.055 Uiso 1 1 calc R . .
C16 C -1.0588(5) 0.1052(17) 0.0034(10) 0.038(4) Uani 1 1 d . . .
H16A H -1.0953 0.1337 0.0212 0.045 Uiso 1 1 calc R . .
H16B H -1.0565 0.0092 0.0142 0.045 Uiso 1 1 calc R . .
C17 C -1.0229(5) 0.4804(11) -0.1543(10) 0.047(3) Uani 1 1 d . . .
H17 H -1.0113 0.4484 -0.2198 0.056 Uiso 1 1 calc R . .
C18 C -1.0712(5) 0.5700(12) -0.1670(12) 0.070(4) Uani 1 1 d . . .
H18A H -1.1032 0.5163 -0.1771 0.105 Uiso 1 1 calc R . .
H18B H -1.0657 0.6271 -0.2236 0.105 Uiso 1 1 calc R . .
H18C H -1.0757 0.6241 -0.1086 0.105 Uiso 1 1 calc R . .
C19 C -0.9768(4) 0.5522(12) -0.1070(11) 0.056(3) Uani 1 1 d . . .
H19A H -0.9869 0.5786 -0.0409 0.084 Uiso 1 1 calc R . .
H19B H -0.9680 0.6305 -0.1453 0.084 Uiso 1 1 calc R . .
H19C H -0.9458 0.4940 -0.1041 0.084 Uiso 1 1 calc R . .
C20 C -1.0485(4) 0.0201(12) -0.1730(10) 0.046(3) Uani 1 1 d . . .
H20 H -1.0446 0.0559 -0.2404 0.055 Uiso 1 1 calc R . .
C21 C -1.1014(4) -0.0548(12) -0.1680(12) 0.058(3) Uani 1 1 d . . .
H21A H -1.1028 -0.1061 -0.1077 0.088 Uiso 1 1 calc R . .
H21B H -1.1042 -0.1141 -0.2240 0.088 Uiso 1 1 calc R . .
H21C H -1.1311 0.0079 -0.1691 0.088 Uiso 1 1 calc R . .
C22 C -1.0000(4) -0.0671(11) -0.1516(10) 0.051(3) Uani 1 1 d . . .
H22A H -0.9674 -0.0142 -0.1558 0.077 Uiso 1 1 calc R . .
H22B H -0.9984 -0.1385 -0.1994 0.077 Uiso 1 1 calc R . .
H22C H -1.0032 -0.1041 -0.0861 0.077 Uiso 1 1 calc R . .
N1 N -0.7900(4) 0.3608(14) -0.3363(9) 0.037(3) Uani 1 1 d . . .
N2 N -0.7983(4) 0.1281(13) -0.3353(8) 0.032(3) Uani 1 1 d . . .
N3 N -1.0391(4) 0.3647(14) -0.0939(9) 0.038(3) Uani 1 1 d . . .
N4 N -1.0493(4) 0.1340(14) -0.1036(9) 0.032(2) Uani 1 1 d . . .
I1 I -1.20604(4) 0.25541(6) -0.06359(4) 0.0363(3) Uani 1 1 d . . .
I2 I -0.95620(3) 0.24558(6) -0.36897(4) 0.0368(3) Uani 1 1 d . . .
C23 C -0.8563(5) 0.2126(17) 0.0041(12) 0.064(3) Uani 1 1 d . A 1
H23A H -0.8645 0.1246 0.0310 0.076 Uiso 1 1 calc R A 1
H23B H -0.8173 0.2179 -0.0058 0.076 Uiso 1 1 calc R A 1
Cl1 Cl -0.87507(12) 0.3296(4) 0.0865(5) 0.0852(12) Uani 1 1 d . A 1
Cl2 Cl -0.88866(17) 0.2299(4) -0.1102(6) 0.0577(11) Uani 1 1 d . A 1

C24 C -0.6089(5) 0.2098(16) -0.4300(13) 0.065(3) Uani 1 1 d . B 1
H24A H -0.6196 0.1244 -0.4585 0.078 Uiso 1 1 calc R B 1
H24B H -0.5697 0.2080 -0.4206 0.078 Uiso 1 1 calc R B 1
Cl3 Cl -0.62478(13) 0.3354(4) -0.5121(5) 0.0871(12) Uani 1 1 d . B 1
Cl4 Cl -0.63949(17) 0.2275(4) -0.3174(5) 0.0549(9) Uani 1 1 d . B 1

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C1 0.027(7) 0.033(9) 0.033(10) -0.001(4) -0.006(7) 0.000(3)
C2 0.031(6) 0.043(8) 0.040(8) 0.005(7) -0.001(6) -0.002(5)
C3 0.051(7) 0.063(9) 0.042(7) 0.014(6) 0.001(6) -0.010(6)
C4 0.055(7) 0.077(10) 0.027(6) -0.003(6) 0.004(5) 0.001(7)
C5 0.037(7) 0.043(9) 0.038(8) 0.002(7) -0.005(6) -0.004(6)
C6 0.080(8) 0.028(6) 0.045(6) 0.002(5) -0.027(6) 0.000(5)
C7 0.107(10) 0.033(6) 0.074(9) -0.003(6) 0.024(9) 0.014(7)
C8 0.055(7) 0.035(6) 0.090(10) 0.002(6) -0.028(6) -0.001(5)
C9 0.057(6) 0.030(5) 0.024(4) -0.004(4) -0.007(4) -0.002(4)
C10 0.065(7) 0.042(6) 0.054(7) 0.010(5) 0.000(6) 0.000(5)
C11 0.060(6) 0.037(6) 0.054(7) -0.004(5) -0.018(6) 0.007(5)
C12 0.041(8) 0.013(6) 0.019(7) -0.002(2) -0.003(7) 0.003(3)
C13 0.039(6) 0.044(8) 0.027(6) -0.014(6) 0.008(6) -0.002(6)
C14 0.049(6) 0.055(8) 0.032(6) -0.009(6) -0.003(5) -0.009(5)
C15 0.044(6) 0.047(7) 0.045(7) 0.002(6) -0.010(5) 0.005(5)
C16 0.040(6) 0.047(9) 0.026(6) 0.013(6) 0.006(5) 0.008(6)
C17 0.070(7) 0.028(6) 0.043(6) 0.004(4) 0.009(6) 0.002(6)
C18 0.094(9) 0.042(7) 0.074(9) 0.013(6) -0.026(9) 0.000(7)
C19 0.055(7) 0.043(6) 0.072(8) -0.007(6) 0.010(6) -0.004(5)
C20 0.067(8) 0.034(6) 0.035(6) -0.003(5) 0.002(5) 0.015(5)
C21 0.069(7) 0.042(6) 0.065(7) -0.012(6) 0.002(7) -0.005(6)
C22 0.057(7) 0.037(6) 0.060(8) -0.001(5) 0.012(6) 0.007(5)
N1 0.049(6) 0.032(6) 0.030(6) 0.004(4) -0.004(4) -0.002(4)
N2 0.045(5) 0.026(5) 0.025(5) 0.002(4) -0.003(4) 0.003(4)
N3 0.044(6) 0.035(6) 0.034(6) -0.005(5) 0.006(4) -0.002(4)
N4 0.033(4) 0.033(6) 0.028(5) 0.002(4) 0.007(4) 0.003(3)
I1 0.0345(5) 0.0398(7) 0.0346(6) -0.0030(4) -0.0029(6) 0.0015(2)
I2 0.0349(6) 0.0412(7) 0.0344(7) 0.0018(4) 0.0045(7) 0.0022(2)
C23 0.046(7) 0.095(9) 0.051(8) -0.006(9) -0.002(6) 0.016(7)
Cl1 0.0678(19) 0.109(3) 0.079(3) -0.032(2) -0.0086(18) 0.0050(19)
Cl2 0.0409(17) 0.073(2) 0.059(3) 0.000(2) 0.0036(17) 0.0051(14)
C24 0.059(8) 0.068(8) 0.068(10) -0.003(9) -0.008(7) 0.004(7)
Cl3 0.078(2) 0.107(3) 0.077(2) 0.029(2) 0.0123(18) 0.010(2)
Cl4 0.0459(18) 0.0685(19) 0.050(2) 0.003(2) -0.0002(16) 0.0030(15)

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;
All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell s.u.'s are taken
into account individually in the estimation of s.u.'s in distances, angles

and torsion angles; correlations between s.u.'s in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.
;

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C1 N1 1.317(19) . ?
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C2 N1 1.476(18) . ?
C2 C3 1.51(2) . ?
C2 H2A 0.9900 . ?
C2 H2B 0.9900 . ?
C3 C4 1.50(3) . ?
C3 H3A 0.9900 . ?
C3 H3B 0.9900 . ?
C4 C5 1.57(2) . ?
C4 H4A 0.9900 . ?
C4 H4B 0.9900 . ?
C5 N2 1.434(19) . ?
C5 H5A 0.9900 . ?
C5 H5B 0.9900 . ?
C6 N1 1.471(17) . ?
C6 C7 1.483(16) . ?
C6 C8 1.550(17) . ?
C6 H6 1.0000 . ?
C7 H7A 0.9800 . ?
C7 H7B 0.9800 . ?
C7 H7C 0.9800 . ?
C8 H8A 0.9800 . ?
C8 H8B 0.9800 . ?
C8 H8C 0.9800 . ?
C9 N2 1.484(15) . ?
C9 C11 1.516(14) . ?
C9 C10 1.530(13) . ?
C9 H9 1.0000 . ?
C10 H10A 0.9800 . ?
C10 H10B 0.9800 . ?
C10 H10C 0.9800 . ?
C11 H11A 0.9800 . ?
C11 H11B 0.9800 . ?
C11 H11C 0.9800 . ?
C12 N3 1.304(18) . ?
C12 N4 1.311(17) . ?
C12 H12 0.9500 . ?
C13 C14 1.48(2) . ?
C13 N3 1.498(17) . ?
C13 H13A 0.9900 . ?
C13 H13B 0.9900 . ?

C14 C15 1.54(2) . ?
C14 H14A 0.9900 . ?
C14 H14B 0.9900 . ?
C15 C16 1.48(2) . ?
C15 H15A 0.9900 . ?
C15 H15B 0.9900 . ?
C16 N4 1.518(17) . ?
C16 H16A 0.9900 . ?
C16 H16B 0.9900 . ?
C17 N3 1.496(17) . ?
C17 C19 1.513(15) . ?
C17 C18 1.527(15) . ?
C17 H17 1.0000 . ?
C18 H18A 0.9800 . ?
C18 H18B 0.9800 . ?
C18 H18C 0.9800 . ?
C19 H19A 0.9800 . ?
C19 H19B 0.9800 . ?
C19 H19C 0.9800 . ?
C20 N4 1.500(17) . ?
C20 C21 1.531(15) . ?
C20 C22 1.534(14) . ?
C20 H20 1.0000 . ?
C21 H21A 0.9800 . ?
C21 H21B 0.9800 . ?
C21 H21C 0.9800 . ?
C22 H22A 0.9800 . ?
C22 H22B 0.9800 . ?
C22 H22C 0.9800 . ?
C23 Cl1 1.709(15) . ?
C23 Cl2 1.776(16) . ?
C23 H23A 0.9900 . ?
C23 H23B 0.9900 . ?
C24 Cl4 1.737(17) . ?
C24 Cl3 1.751(16) . ?
C24 H24A 0.9900 . ?
C24 H24B 0.9900 . ?

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N2 C1 N1 130(2) . . ?
N2 C1 H1 115.2 . . ?
N1 C1 H1 115.2 . . ?
N1 C2 C3 112.9(10) . . ?
N1 C2 H2A 109.0 . . ?
C3 C2 H2A 109.0 . . ?
N1 C2 H2B 109.0 . . ?
C3 C2 H2B 109.0 . . ?

H2A C2 H2B 107.8 . . ?
C4 C3 C2 115.9(10) . . ?
C4 C3 H3A 108.3 . . ?
C2 C3 H3A 108.3 . . ?
C4 C3 H3B 108.3 . . ?
C2 C3 H3B 108.3 . . ?
H3A C3 H3B 107.4 . . ?
C3 C4 C5 113.0(11) . . ?
C3 C4 H4A 109.0 . . ?
C5 C4 H4A 109.0 . . ?
C3 C4 H4B 109.0 . . ?
C5 C4 H4B 109.0 . . ?
H4A C4 H4B 107.8 . . ?
N2 C5 C4 110.9(12) . . ?
N2 C5 H5A 109.5 . . ?
C4 C5 H5A 109.5 . . ?
N2 C5 H5B 109.5 . . ?
C4 C5 H5B 109.5 . . ?
H5A C5 H5B 108.1 . . ?
N1 C6 C7 112.7(11) . . ?
N1 C6 C8 109.0(11) . . ?
C7 C6 C8 112.9(10) . . ?
N1 C6 H6 107.3 . . ?
C7 C6 H6 107.3 . . ?
C8 C6 H6 107.3 . . ?
C6 C7 H7A 109.5 . . ?
C6 C7 H7B 109.5 . . ?
H7A C7 H7B 109.5 . . ?
C6 C7 H7C 109.5 . . ?
H7A C7 H7C 109.5 . . ?
H7B C7 H7C 109.5 . . ?
C6 C8 H8A 109.5 . . ?
C6 C8 H8B 109.5 . . ?
H8A C8 H8B 109.5 . . ?
C6 C8 H8C 109.5 . . ?
H8A C8 H8C 109.5 . . ?
H8B C8 H8C 109.5 . . ?
N2 C9 C11 110.1(10) . . ?
N2 C9 C10 111.5(9) . . ?
C11 C9 C10 112.6(9) . . ?
N2 C9 H9 107.5 . . ?
C11 C9 H9 107.5 . . ?
C10 C9 H9 107.5 . . ?
C9 C10 H10A 109.5 . . ?
C9 C10 H10B 109.5 . . ?
H10A C10 H10B 109.5 . . ?
C9 C10 H10C 109.5 . . ?
H10A C10 H10C 109.5 . . ?
H10B C10 H10C 109.5 . . ?
C9 C11 H11A 109.5 . . ?
C9 C11 H11B 109.5 . . ?
H11A C11 H11B 109.5 . . ?
C9 C11 H11C 109.5 . . ?
H11A C11 H11C 109.5 . . ?

H11B C11 H11C 109.5 . . ?
N3 C12 N4 130(2) . . ?
N3 C12 H12 115.2 . . ?
N4 C12 H12 115.2 . . ?
C14 C13 N3 114.0(11) . . ?
C14 C13 H13A 108.8 . . ?
N3 C13 H13A 108.8 . . ?
C14 C13 H13B 108.8 . . ?
N3 C13 H13B 108.8 . . ?
H13A C13 H13B 107.6 . . ?
C13 C14 C15 115.6(10) . . ?
C13 C14 H14A 108.4 . . ?
C15 C14 H14A 108.4 . . ?
C13 C14 H14B 108.4 . . ?
C15 C14 H14B 108.4 . . ?
H14A C14 H14B 107.4 . . ?
C16 C15 C14 116.1(10) . . ?
C16 C15 H15A 108.3 . . ?
C14 C15 H15A 108.3 . . ?
C16 C15 H15B 108.3 . . ?
C14 C15 H15B 108.3 . . ?
H15A C15 H15B 107.4 . . ?
C15 C16 N4 112.8(12) . . ?
C15 C16 H16A 109.0 . . ?
N4 C16 H16A 109.0 . . ?
C15 C16 H16B 109.0 . . ?
N4 C16 H16B 109.0 . . ?
H16A C16 H16B 107.8 . . ?
N3 C17 C19 110.4(10) . . ?
N3 C17 C18 108.5(10) . . ?
C19 C17 C18 111.5(10) . . ?
N3 C17 H17 108.8 . . ?
C19 C17 H17 108.8 . . ?
C18 C17 H17 108.8 . . ?
C17 C18 H18A 109.5 . . ?
C17 C18 H18B 109.5 . . ?
H18A C18 H18B 109.5 . . ?
C17 C18 H18C 109.5 . . ?
H18A C18 H18C 109.5 . . ?
H18B C18 H18C 109.5 . . ?
C17 C19 H19A 109.5 . . ?
C17 C19 H19B 109.5 . . ?
H19A C19 H19B 109.5 . . ?
C17 C19 H19C 109.5 . . ?
H19A C19 H19C 109.5 . . ?
H19B C19 H19C 109.5 . . ?
N4 C20 C21 110.1(10) . . ?
N4 C20 C22 109.6(10) . . ?
C21 C20 C22 113.0(10) . . ?
N4 C20 H20 108.0 . . ?
C21 C20 H20 108.0 . . ?
C22 C20 H20 108.0 . . ?
C20 C21 H21A 109.5 . . ?
C20 C21 H21B 109.5 . . ?

H21A C21 H21B 109.5 . . ?
C20 C21 H21C 109.5 . . ?
H21A C21 H21C 109.5 . . ?
H21B C21 H21C 109.5 . . ?
C20 C22 H22A 109.5 . . ?
C20 C22 H22B 109.5 . . ?
H22A C22 H22B 109.5 . . ?
C20 C22 H22C 109.5 . . ?
H22A C22 H22C 109.5 . . ?
H22B C22 H22C 109.5 . . ?
C1 N1 C6 118.5(15) . . ?
C1 N1 C2 121.8(17) . . ?
C6 N1 C2 118.1(13) . . ?
C1 N2 C5 125.3(17) . . ?
C1 N2 C9 116.8(14) . . ?
C5 N2 C9 117.8(13) . . ?
C12 N3 C17 115.9(13) . . ?
C12 N3 C13 125.3(16) . . ?
C17 N3 C13 118.7(13) . . ?
C12 N4 C20 118.3(14) . . ?
C12 N4 C16 123.5(15) . . ?
C20 N4 C16 118.0(13) . . ?
Cl1 C23 Cl2 113.0(8) . . ?
Cl1 C23 H23A 109.0 . . ?
Cl2 C23 H23A 109.0 . . ?
Cl1 C23 H23B 109.0 . . ?
Cl2 C23 H23B 109.0 . . ?
H23A C23 H23B 107.8 . . ?
Cl4 C24 Cl3 113.4(8) . . ?
Cl4 C24 H24A 108.9 . . ?
Cl3 C24 H24A 108.9 . . ?
Cl4 C24 H24B 108.9 . . ?
Cl3 C24 H24B 108.9 . . ?
H24A C24 H24B 107.7 . . ?

_diffrn_measured_fraction_theta_max 0.995
_diffrn_reflns_theta_full 28.50
_diffrn_measured_fraction_theta_full 0.995
_refine_diff_density_max 1.019
_refine_diff_density_min -1.178
_refine_diff_density_rms 0.186