

data_WO3-Bpy-0Na

```
_audit_creation_method          SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common           ?
_chemical_melting_point         ?
_chemical_formula_moiety        'C5 H4 N1 O3 W1'
_chemical_formula_sum           'C5 H4 N1 O3 W1'
_chemical_formula_weight        309.94
```

```
loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
```

```
C C 0.0181 0.0091 '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 0.0311 0.0180 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
O O 0.0492 0.0322 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
W W -5.4734 5.5774 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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```
_symmetry_cell_setting          'Orthorhombic'
_symmetry_space_group_name_H-M  'P b c a'
```

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

```
_cell_length_a                  7.477709(13)
_cell_length_b                  7.38736(19)
_cell_length_c                  22.5625(4)
_cell_angle_alpha               90.00
_cell_angle_beta                90.00
_cell_angle_gamma               90.00
_cell_volume                    1245.97(5)
_cell_formula_units_Z           8
_cell_measurement_temperature    100(1)
_cell_measurement_reflns_used   4676
_cell_measurement_theta_min     14.21
_cell_measurement_theta_max     144.16
```

```
_exptl_crystal_description      plate
_exptl_crystal_colour           yellow
_exptl_crystal_size_max         0.08
```

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_exptl_crystal_size_mid      0.08
_exptl_crystal_size_min      0.01
_exptl_crystal_density_meas  ?
_exptl_crystal_density_diffn 3.305
_exptl_crystal_density_method 'not measured'
_exptl_crystal_F_000         1112
_exptl_absorpt_coefficient_mu 33.925
_exptl_absorpt_correction_type empirical
_exptl_absorpt_correction_T_min 0.379
_exptl_absorpt_correction_T_max 1.000
_exptl_absorpt_process_details

;
data scaling includes absorption [ABSCOR, Higashi (1995)]
_exptl_special_details
;

_diffn_ambient_temperature    100(1)
_diffn_radiation_wavelength    1.54178
_diffn_radiation_type          CuK\alpha
_diffn_radiation_source        'rotating anode'
_diffn_radiation_monochromator  'confocal optics'
_diffn_measurement_device_type  'Rigaku Spider'
_diffn_measurement_device_details 'cureved image plate detector'
_diffn_measurement_method      \w-scans
_diffn_detector_area_resol_mean 10
_diffn_standards_number        0
_diffn_standards_interval_count ?
_diffn_standards_interval_time ?
_diffn_standards_decay_%       0
_diffn_reflns_number           6395
_diffn_reflns_av_R_equivalents 0.0678
_diffn_reflns_av_sigmaI/netI   0.0551
_diffn_reflns_limit_h_min      -9
_diffn_reflns_limit_h_max      8
_diffn_reflns_limit_k_min      -7
_diffn_reflns_limit_k_max      8
_diffn_reflns_limit_l_min      -27
_diffn_reflns_limit_l_max      20
_diffn_reflns_theta_min        7.11
_diffn_reflns_theta_max        70.05
_reflns_number_total           1124
_reflns_number_gt              1000
_reflns_threshold_expression    >2sigma(I)

_computing_data_collection      'Crystal Clear (Rigaku, 2005)'
_computing_cell_refinement      'FS Process (Rigaku, 1998)'
_computing_data_reduction       'FS Process (Rigaku, 1998)'
_computing_structure_solution    'SHELXS-97 (Sheldrick, 2008)'
_computing_structure_refinement  'SHELXTL-6.10 (Sheldrick, 2008)'
_computing_molecular_graphics    'Mercury (Macrae et. al., 2006) and DS Viewpro (Accelrys Inc., 2002)'
_computing_publication_material 'SHELXL-97 (Sheldrick, 2008)'

_refine_special_details
;
Refinement of F2 against ALL reflections. The weighted R-factor wR
and goodness of fit S are based on F2, conventional R-factors R are

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based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ 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.

There is clear evidence of substantial disorder on the equatorial oxygen atoms of the W-O layer. This disorder appears also to extend, but to a lesser extent to the W atoms. Disorder of the W atoms has not been modelled. The pyridyl ring is well ordered. Despite the small, although highly significant, difference in the a and b cell lengths, there is no evidence for significant twinning.

A small number (20) of reflections with exceptionally poor agreement between F_{calc} and F_{obs} were eliminated from refinement, possibly because the small crystal was mounted against a 0.15 mm glass fibre.

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_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^)+(0.0414P)^2^+8.8141P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method       SHELXL
_refine_ls_extinction_coef         0.00026(3)
_refine_ls_extinction_expression
'Fc^*=kFc[1+0.001xFc^2^l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          1124
_refine_ls_number_parameters       111
_refine_ls_number_restraints       120
_refine_ls_R_factor_all            0.0379
_refine_ls_R_factor_gt             0.0338
_refine_ls_wR_factor_ref           0.0914
_refine_ls_wR_factor_gt            0.0856
_refine_ls_goodness_of_fit_ref     1.095
_refine_ls_restrained_S_all        1.043
_refine_ls_shift/su_max            0.001
_refine_ls_shift/su_mean           0.000
```

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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
```

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W1 W 0.52165(5) 1.05282(5) 0.263733(13) 0.0068(2) Uani 1 1 d . . .
N1 N 0.5181(8) 1.0385(10) 0.1557(4) 0.0151(11) Uani 1 1 d U . .
O3 O 0.5098(6) 1.0566(7) 0.3394(3) 0.0072(12) Uani 1 1 d U . .
C2 C 0.6245(10) 0.9283(10) 0.1276(3) 0.0148(11) Uani 1 1 d U . .
H2 H 0.7053 0.8599 0.1495 0.018 Uiso 1 1 calc R . .
C3 C 0.4039(9) 1.1431(11) 0.1252(3) 0.0152(11) Uani 1 1 d U . .
H3 H 0.3321 1.2258 0.1452 0.018 Uiso 1 1 calc R . .
C4 C 0.6227(10) 0.9085(10) 0.0669(3) 0.0148(11) Uani 1 1 d U . .
H4 H 0.7015 0.8286 0.0487 0.018 Uiso 1 1 calc R . .
C5 C 0.5038(9) 1.0075(15) 0.0333(4) 0.0144(12) Uani 1 1 d U . .
C6 C 0.3925(10) 1.1282(11) 0.0637(3) 0.0155(11) Uani 1 1 d U . .
H6 H 0.3107 1.1985 0.0428 0.019 Uiso 1 1 calc R . .
O1A O 0.4611(19) 0.8021(17) 0.2520(5) 0.010(2) Uani 0.493(16) 1 d PU . 1
O2A O 0.7588(16) 1.009(3) 0.2519(4) 0.012(2) Uani 0.493(16) 1 d PU . 1
O2B O 0.7585(15) 1.088(2) 0.2524(4) 0.0106(19) Uani 0.507(16) 1 d PU . 2
O1B O 0.5379(18) 0.7971(17) 0.2518(5) 0.012(2) Uani 0.507(16) 1 d PU . 2

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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
W1 0.0033(3) 0.0089(3) 0.0083(3) -0.00020(11) -0.00001(10) 0.00017(11)
N1 0.0142(18) 0.0175(19) 0.0136(18) -0.0021(16) 0.0003(15) 0.0002(15)
O3 0.0062(13) 0.0072(14) 0.0082(14) 0.0004(9) -0.0006(8) 0.0001(8)
C2 0.0134(18) 0.0158(19) 0.0150(18) -0.0021(16) -0.0002(16) 0.0009(16)
C3 0.0132(18) 0.0170(19) 0.0154(17) -0.0008(16) 0.0005(16) 0.0020(16)
C4 0.0134(18) 0.0154(19) 0.0157(18) -0.0014(16) 0.0015(16) 0.0012(16)
C5 0.0137(18) 0.0153(19) 0.0143(19) 0.0006(17) 0.0009(16) -0.0001(17)
C6 0.0135(18) 0.0174(19) 0.0156(18) -0.0007(16) 0.0004(16) 0.0019(16)
O1A 0.009(3) 0.012(3) 0.010(2) 0.000(2) -0.001(2) -0.001(2)
O2A 0.011(3) 0.011(3) 0.013(2) -0.001(2) 0.001(2) 0.000(2)
O2B 0.010(3) 0.010(3) 0.012(2) 0.001(2) 0.001(2) 0.000(2)
O1B 0.011(3) 0.013(3) 0.011(2) 0.000(2) -0.002(2) 0.001(2)

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_geom_special_details

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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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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

```

```

W1 O3 1.709(6) . ?
W1 O2B 1.808(11) . ?
W1 O2A 1.822(12) . ?
W1 O1A 1.880(13) 3_655 ?
W1 O1B 1.891(13) 3_655 ?
W1 O1B 1.912(13) . ?
W1 O1A 1.925(13) . ?
W1 O2B 2.018(11) 6_556 ?
W1 O2A 2.023(12) 6_556 ?
W1 N1 2.439(8) . ?
N1 C2 1.303(10) . ?
N1 C3 1.341(10) . ?
C2 C4 1.376(9) . ?
C3 C6 1.395(9) . ?
C4 C5 1.378(11) . ?
C5 C6 1.398(11) . ?
C5 C5 1.509(16) 5_675 ?
O1A W1 1.880(13) 3_645 ?
O2A W1 2.023(12) 6_656 ?
O2B W1 2.018(11) 6_656 ?
O1B W1 1.891(13) 3_645 ?

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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
O3 W1 O2B 100.9(3) . . ?
O3 W1 O2A 101.5(3) . . ?
O2B W1 O2A 18.5(6) . . ?
O3 W1 O1A 100.1(4) . 3_655 ?
O2B W1 O1A 76.4(5) . 3_655 ?
O2A W1 O1A 94.6(6) . 3_655 ?
O3 W1 O1B 99.0(4) . 3_655 ?
O2B W1 O1B 93.9(5) . 3_655 ?
O2A W1 O1B 111.8(6) . 3_655 ?
O1A W1 O1B 17.5(4) 3_655 3_655 ?
O3 W1 O1B 99.2(4) . . ?
O2B W1 O1B 93.4(5) . . ?
O2A W1 O1B 75.0(6) . . ?
O1A W1 O1B 159.6(6) 3_655 . ?
O1B W1 O1B 158.67(8) 3_655 . ?
O3 W1 O1A 98.1(4) . . ?
O2B W1 O1A 110.4(5) . . ?
O2A W1 O1A 92.1(6) . . ?
O1A W1 O1A 158.87(5) 3_655 . ?
O1B W1 O1A 146.8(5) 3_655 . ?
O1B W1 O1A 17.2(4) . . ?

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O3 W1 O2B 97.4(3) . 6_556 ?
 O2B W1 O2B 155.7(7) . 6_556 ?
 O2A W1 O2B 160.9(5) . 6_556 ?
 O1A W1 O2B 84.6(8) 3_655 6_556 ?
 O1B W1 O2B 67.3(8) 3_655 6_556 ?
 O1B W1 O2B 99.4(8) . 6_556 ?
 O1A W1 O2B 82.5(8) . 6_556 ?
 O3 W1 O2A 97.2(3) . 6_556 ?
 O2B W1 O2A 161.9(5) . 6_556 ?
 O2A W1 O2A 153.0(8) . 6_556 ?
 O1A W1 O2A 101.0(8) 3_655 6_556 ?
 O1B W1 O2A 83.8(8) 3_655 6_556 ?
 O1B W1 O2A 83.0(8) . 6_556 ?
 O1A W1 O2A 66.0(8) . 6_556 ?
 O2B W1 O2A 16.6(6) 6_556 6_556 ?
 O3 W1 N1 176.1(2) . . ?
 O2B W1 N1 82.9(3) . . ?
 O2A W1 N1 81.7(3) . . ?
 O1A W1 N1 81.7(4) 3_655 . ?
 O1B W1 N1 81.6(4) 3_655 . ?
 O1B W1 N1 79.4(4) . . ?
 O1A W1 N1 79.5(4) . . ?
 O2B W1 N1 79.3(3) 6_556 . ?
 O2A W1 N1 79.0(3) 6_556 . ?
 C2 N1 C3 119.9(8) . . ?
 C2 N1 W1 120.5(5) . . ?
 C3 N1 W1 119.6(5) . . ?
 N1 C2 C4 123.0(8) . . ?
 N1 C3 C6 120.3(7) . . ?
 C2 C4 C5 119.8(7) . . ?
 C4 C5 C6 117.0(7) . . ?
 C4 C5 C5 122.2(10) . 5_675 ?
 C6 C5 C5 120.8(10) . 5_675 ?
 C3 C6 C5 120.0(7) . . ?
 W1 O1A W1 162.3(8) 3_645 . ?
 W1 O2A W1 160.5(12) . 6_656 ?
 W1 O2B W1 164.2(10) . 6_656 ?
 W1 O1B W1 162.5(8) 3_645 . ?

_diffirn_measured_fraction_theta_max	0.953
_diffirn_reflms_theta_full	70.05
_diffirn_measured_fraction_theta_full	0.953
_refine_diff_density_max	2.290
_refine_diff_density_min	-1.704
_refine_diff_density_rms	0.332