

data\_WO3-bpy-2K

```
_audit_creation_method          SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common           ?
_chemical_melting_point         ?
_chemical_formula_moiety        'C5 H4 K0.03 N O3 W'
_chemical_formula_sum           'C5 H4 K0.03 N O3 W'
_chemical_formula_weight        311.11
```

```
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'
K K 0.3868 1.0657 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

```
_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                  7.4751(10)
_cell_length_b                  7.3943(10)
_cell_length_c                  22.5813(16)
_cell_angle_alpha               90.00
_cell_angle_beta                90.00
_cell_angle_gamma               90.00
_cell_volume                    1248.1(3)
_cell_formula_units_Z           8
_cell_measurement_temperature   153(2)
_cell_measurement_reflns_used   13478
_cell_measurement_theta_min     14.32
_cell_measurement_theta_max     144.38
```

```
_exptl_crystal_description      plate
_exptl_crystal_colour           yellow-green
```

```

_exptl_crystal_size_max      0.09
_exptl_crystal_size_mid     0.04
_exptl_crystal_size_min     0.01
_exptl_crystal_density_meas  ?
_exptl_crystal_density_diffn 3.311
_exptl_crystal_density_method 'not measured'
_exptl_crystal_F_000        1115.8
_exptl_absorpt_coefficient_mu 34.048
_exptl_absorpt_correction_type empirical
_exptl_absorpt_correction_T_min 0.405
_exptl_absorpt_correction_T_max 1.000
_exptl_absorpt_process_details

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

_diffn_ambient_temperature 153(2)
_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        18509
_diffn_reflns_av_R_equivalents 0.1107
_diffn_reflns_av_sigmaI/netI 0.0489
_diffn_reflns_limit_h_min   -7
_diffn_reflns_limit_h_max    7
_diffn_reflns_limit_k_min   -7
_diffn_reflns_limit_k_max    7
_diffn_reflns_limit_l_min   -23
_diffn_reflns_limit_l_max    23
_diffn_reflns_theta_min     7.10
_diffn_reflns_theta_max     54.23
_reflns_number_total        756
_reflns_number_gt           631
_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  $F^2$  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  $F^2 > 2\sigma(F^2)$  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.

;

```
_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^)+(0.0736P)^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     mixed
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          756
_refine_ls_number_parameters       89
_refine_ls_number_restraints       12
_refine_ls_R_factor_all            0.0465
_refine_ls_R_factor_gt             0.0423
_refine_ls_wR_factor_ref           0.1070
_refine_ls_wR_factor_gt            0.1039
_refine_ls_goodness_of_fit_ref     1.029
_refine_ls_restrained_S_all        1.042
_refine_ls_shift/su_max            0.558
_refine_ls_shift/su_mean           0.010
```

```
loop_
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  _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
K1 K 0.0000 0.0000 0.0000 0.12(6) Uiso 0.06 2 d SP . .
O1 O 0.0095(9) 0.0521(11) 0.1620(5) 0.024(3) Uiso 1 1 d . . .
O3 O -0.0031(10) 0.3066(18) 0.2532(4) 0.046(4) Uani 1 1 d U . .
O2 O 0.2597(13) 0.0510(15) 0.2486(2) 0.038(3) Uani 1 1 d U . .
W1 W 0.02077(8) 0.04938(9) 0.23627(3) 0.0145(4) Uani 1 1 d . . .
N1 N 0.0173(13) 0.0368(17) 0.3447(6) 0.030(4) Uani 1 1 d . . .
C2 C 0.1243(14) -0.0775(14) 0.3726(5) 0.020(3) Uani 1 1 d . . .
H2 H 0.2028 -0.1480 0.3505 0.024 Uiso 1 1 calc R . .
C5 C 0.0035(14) 0.007(3) 0.4674(7) 0.025(4) Uani 1 1 d . . .
C1 C -0.0986(13) 0.1402(16) 0.3762(5) 0.022(3) Uani 1 1 d . . .
H1 H -0.1738 0.2199 0.3563 0.027 Uiso 1 1 calc R . .
C3 C -0.1072(14) 0.1297(15) 0.4364(5) 0.025(3) Uani 1 1 d . . .
```

```

H3 H -0.1863 0.2034 0.4571 0.030 Uiso 1 1 calc R . .
C4 C 0.1229(14) -0.0954(13) 0.4327(5) 0.024(3) Uani 1 1 d . . .
H4 H 0.2012 -0.1757 0.4509 0.029 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
O3 0.058(6) 0.053(6) 0.029(4) 0.003(5) -0.004(3) -0.003(4)
O2 0.031(5) 0.052(6) 0.032(4) 0.003(3) -0.002(4) 0.000(4)
W1 0.0073(5) 0.0133(7) 0.0230(6) 0.0003(3) 0.0002(2) 0.0001(2)
N1 0.030(7) 0.044(11) 0.018(7) 0.017(6) -0.001(5) -0.005(5)
C2 0.012(6) 0.015(8) 0.033(7) 0.005(6) -0.004(6) 0.000(5)
C5 0.016(8) 0.040(10) 0.020(8) 0.005(8) 0.005(5) 0.003(6)
C1 0.009(6) 0.025(8) 0.033(7) -0.006(6) 0.005(6) 0.009(6)
C3 0.015(6) 0.023(8) 0.038(7) -0.002(6) 0.001(6) 0.013(6)
C4 0.014(6) 0.026(8) 0.033(7) 0.001(6) 0.000(6) 0.007(6)

```

```

_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.
;

```

```

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
K1 C3 3.194(11) 7_565 ?
K1 C3 3.194(11) 3_545 ?
K1 C4 3.279(10) 2_554 ?
K1 C4 3.279(10) 6_556 ?
K1 C3 3.406(10) 6_656 ?
K1 C3 3.406(10) 2_454 ?
K1 C4 3.479(10) 3 ?
K1 C4 3.479(10) 7 ?
K1 O1 3.679(12) . ?
O1 W1 1.680(12) . ?
O3 W1 1.816(13) 3 ?
O3 W1 1.948(13) . ?
O2 W1 1.808(10) . ?
O2 W1 1.981(10) 6_656 ?
W1 O3 1.816(13) 3_545 ?
W1 O2 1.981(10) 6_556 ?
W1 N1 2.451(14) . ?

```

N1 C2 1.323(15) . ?  
 N1 C1 1.356(16) . ?  
 C2 C4 1.364(14) . ?  
 C5 C4 1.408(18) . ?  
 C5 C3 1.413(19) . ?  
 C5 C5 1.48(3) 5\_556 ?  
 C1 C3 1.363(14) . ?  
 C3 K1 3.194(11) 3 ?  
 C3 K1 3.406(10) 2\_455 ?  
 C4 K1 3.279(10) 2 ?  
 C4 K1 3.479(10) 3\_545 ?

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  
 C3 K1 C3 180.0(5) 7\_565 3\_545 ?  
 C3 K1 C4 79.8(3) 7\_565 2\_554 ?  
 C3 K1 C4 100.2(3) 3\_545 2\_554 ?  
 C3 K1 C4 100.2(3) 7\_565 6\_556 ?  
 C3 K1 C4 79.8(3) 3\_545 6\_556 ?  
 C4 K1 C4 180.0(4) 2\_554 6\_556 ?  
 C3 K1 C3 99.5(2) 7\_565 6\_656 ?  
 C3 K1 C3 80.5(2) 3\_545 6\_656 ?  
 C4 K1 C3 52.7(3) 2\_554 6\_656 ?  
 C4 K1 C3 127.3(3) 6\_556 6\_656 ?  
 C3 K1 C3 80.5(2) 7\_565 2\_454 ?  
 C3 K1 C3 99.5(2) 3\_545 2\_454 ?  
 C4 K1 C3 127.3(3) 2\_554 2\_454 ?  
 C4 K1 C3 52.7(3) 6\_556 2\_454 ?  
 C3 K1 C3 180.0(3) 6\_656 2\_454 ?  
 C3 K1 C4 52.6(3) 7\_565 3 ?  
 C3 K1 C4 127.4(3) 3\_545 3 ?  
 C4 K1 C4 104.1(2) 2\_554 3 ?  
 C4 K1 C4 75.9(2) 6\_556 3 ?  
 C3 K1 C4 78.5(3) 6\_656 3 ?  
 C3 K1 C4 101.5(3) 2\_454 3 ?  
 C3 K1 C4 127.4(3) 7\_565 7 ?  
 C3 K1 C4 52.6(3) 3\_545 7 ?  
 C4 K1 C4 75.9(2) 2\_554 7 ?  
 C4 K1 C4 104.1(2) 6\_556 7 ?  
 C3 K1 C4 101.5(3) 6\_656 7 ?  
 C3 K1 C4 78.5(3) 2\_454 7 ?  
 C4 K1 C4 180.0(5) 3 7 ?  
 C3 K1 O1 111.2(2) 7\_565 . ?  
 C3 K1 O1 68.8(2) 3\_545 . ?  
 C4 K1 O1 114.9(2) 2\_554 . ?  
 C4 K1 O1 65.1(2) 6\_556 . ?  
 C3 K1 O1 62.2(2) 6\_656 . ?  
 C3 K1 O1 117.8(2) 2\_454 . ?  
 C4 K1 O1 58.7(2) 3 . ?  
 C4 K1 O1 121.3(2) 7 . ?  
 W1 O1 K1 173.1(4) . . ?  
 W1 O3 W1 176.0(6) 3 . ?

W1 O2 W1 178.7(5) . 6\_656 ?  
 O1 W1 O2 101.7(3) . . ?  
 O1 W1 O3 98.0(4) . 3\_545 ?  
 O2 W1 O3 93.3(4) . 3\_545 ?  
 O1 W1 O3 100.4(4) . . ?  
 O2 W1 O3 93.1(4) . . ?  
 O3 W1 O3 158.89(5) 3\_545 . ?  
 O1 W1 O2 97.0(3) . 6\_556 ?  
 O2 W1 O2 161.19(5) . 6\_556 ?  
 O3 W1 O2 85.0(4) 3\_545 6\_556 ?  
 O3 W1 O2 82.5(4) . 6\_556 ?  
 O1 W1 N1 176.2(4) . . ?  
 O2 W1 N1 81.7(3) . . ?  
 O3 W1 N1 80.3(4) 3\_545 . ?  
 O3 W1 N1 80.8(4) . . ?  
 O2 W1 N1 79.5(3) 6\_556 . ?  
 C2 N1 C1 119.8(12) . . ?  
 C2 N1 W1 119.5(9) . . ?  
 C1 N1 W1 120.6(8) . . ?  
 N1 C2 C4 122.0(11) . . ?  
 C4 C5 C3 116.2(13) . . ?  
 C4 C5 C5 122.5(16) . 5\_556 ?  
 C3 C5 C5 121.2(16) . 5\_556 ?  
 N1 C1 C3 121.3(11) . . ?  
 C1 C3 C5 120.2(11) . . ?  
 C1 C3 K1 112.8(8) . 3 ?  
 C5 C3 K1 100.4(9) . 3 ?  
 C1 C3 K1 118.5(7) . 2\_455 ?  
 C5 C3 K1 96.6(8) . 2\_455 ?  
 K1 C3 K1 105.5(3) 3 2\_455 ?  
 C2 C4 C5 120.4(11) . . ?  
 C2 C4 K1 115.7(7) . 2 ?  
 C5 C4 K1 99.9(8) . 2 ?  
 C2 C4 K1 121.3(7) . 3\_545 ?  
 C5 C4 K1 93.0(9) . 3\_545 ?  
 K1 C4 K1 102.1(3) 2 3\_545 ?

_diffraction_measured_fraction_theta_max	0.999
_diffraction_reflns_theta_full	54.23
_diffraction_measured_fraction_theta_full	0.999
_refine_diff_density_max	2.358
_refine_diff_density_min	-2.140
_refine_diff_density_rms	0.352