

data_WO3-bpy-0Ca

```
_audit_creation_method      SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    'C5 H4 N O3 W'
_chemical_formula_sum       'C5 H4 N O3 W'
_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'
```

```
_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              7.4777(5)
_cell_length_b              7.3858(6)
_cell_length_c              22.5668(16)
_cell_angle_alpha           90.00
_cell_angle_beta            90.00
_cell_angle_gamma           90.00
_cell_volume                1246.34(16)
_cell_formula_units_Z       8
_cell_measurement_temperature 143(1)
_cell_measurement_reflns_used 992
_cell_measurement_theta_min  14.33
_cell_measurement_theta_max  141.70
```

```

_exptl_crystal_description      plate
_exptl_crystal_colour          green
_exptl_crystal_size_max        0.10
_exptl_crystal_size_mid        0.05
_exptl_crystal_size_min        0.01
_exptl_crystal_density_meas    ?
_exptl_crystal_density_diffn   3.304
_exptl_crystal_density_method  'not measured'
_exptl_crystal_F_000           1112
_exptl_absorpt_coefficient_mu  33.915
_exptl_absorpt_correction_type  empirical
_exptl_absorpt_correction_T_min 0.369
_exptl_absorpt_correction_T_max 1.000
_exptl_absorpt_process_details

;
data scaling includes absorption [ABSCOR, Higashi (1995)]
_exptl_special_details
;
_diffn_ambient_temperature      143(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            18100
_diffn_reflns_av_R_equivalents  0.1108
_diffn_reflns_av_sigmaI/netI    0.0738
_diffn_reflns_limit_h_min       -9
_diffn_reflns_limit_h_max       8
_diffn_reflns_limit_k_min       -8
_diffn_reflns_limit_k_max       9
_diffn_reflns_limit_l_min       -27
_diffn_reflns_limit_l_max       27
_diffn_reflns_theta_min         7.10
_diffn_reflns_theta_max         72.11
_reflns_number_total            1213
_reflns_number_gt               1010
_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^)+(0.0566P)^2^+72.6287P] 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          1213
_refine_ls_number_parameters       92
_refine_ls_number_restraints       54
_refine_ls_R_factor_all            0.0853
_refine_ls_R_factor_gt            0.0718
_refine_ls_wR_factor_ref           0.2303
_refine_ls_wR_factor_gt           0.1958
_refine_ls_goodness_of_fit_ref     1.156
_refine_ls_restrained_S_all        1.142
_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
O2 O 0.762(2) 1.048(2) 0.2518(4) 0.036(3) Uani 1 1 d U . .
W1 W 1.02169(12) 1.05125(12) 0.23623(3) 0.0185(5) Uani 1 1 d . . .
N1 N 1.0170(18) 1.041(2) 0.3443(8) 0.022(3) Uani 1 1 d U . .
O1 O 1.0098(15) 1.0538(18) 0.1598(8) 0.029(3) Uani 1 1 d U . .
C1 C 0.9038(19) 1.140(2) 0.3753(6) 0.024(3) Uani 1 1 d U . .
H1 H 0.8276 1.2193 0.3556 0.029 Uiso 1 1 calc R . .
C3 C 1.003(2) 1.013(4) 0.4677(8) 0.028(4) Uani 1 1 d U . .
C5 C 1.124(2) 0.925(2) 0.3725(7) 0.031(4) Uani 1 1 d U . .
H5 H 1.2029 0.8551 0.3504 0.037 Uiso 1 1 calc R . .
C2 C 0.895(2) 1.130(2) 0.4368(6) 0.028(3) Uani 1 1 d U . .
H2 H 0.8153 1.2032 0.4573 0.033 Uiso 1 1 calc R . .
C4 C 1.121(2) 0.904(2) 0.4329(6) 0.031(4) Uani 1 1 d U . .
H4 H 1.1951 0.8201 0.4510 0.037 Uiso 1 1 calc R . .
```

O3 O 1.0038(18) 0.813(3) 0.2457(7) 0.046(4) Uani 1 1 d U . .

```
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
O2 0.032(5) 0.044(6) 0.032(4) -0.001(3) -0.004(4) -0.002(4)
W1 0.0102(6) 0.0200(8) 0.0252(7) -0.0003(2) -0.0004(2) 0.0001(2)
N1 0.024(5) 0.023(5) 0.021(5) -0.001(4) -0.004(4) 0.000(4)
O1 0.023(4) 0.032(5) 0.034(5) -0.001(4) 0.000(3) -0.001(3)
C1 0.022(5) 0.022(5) 0.028(4) -0.001(4) 0.000(4) 0.000(4)
C3 0.025(5) 0.031(6) 0.027(5) 0.006(4) -0.001(4) -0.001(4)
C5 0.028(5) 0.033(6) 0.032(5) -0.002(4) 0.001(4) 0.001(4)
C2 0.025(5) 0.027(5) 0.031(4) -0.002(4) 0.001(4) 0.003(4)
C4 0.029(5) 0.033(6) 0.031(5) 0.001(4) -0.002(4) 0.005(4)
O3 0.051(6) 0.052(6) 0.034(4) -0.007(5) -0.004(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.

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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
O2 W1 1.820(15) 6_556 ?
O2 W1 1.971(15) . ?
W1 O1 1.727(18) . ?
W1 O3 1.78(2) . ?
W1 O2 1.820(15) 6_656 ?
W1 O3 1.98(2) 3_755 ?
W1 N1 2.439(17) . ?
N1 C1 1.32(2) . ?
N1 C5 1.33(2) . ?
C1 C2 1.391(18) . ?
C3 C2 1.37(3) . ?
C3 C4 1.43(3) . ?
C3 C3 1.47(3) 5_776 ?
C5 C4 1.37(2) . ?
O3 W1 1.98(2) 3_745 ?
```

```
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

W1 O2 W1 177.6(8) 6_556 . ?
 O1 W1 O3 97.3(7) . . ?
 O1 W1 O2 101.5(5) . 6_656 ?
 O3 W1 O2 92.4(6) . 6_656 ?
 O1 W1 O2 97.3(5) . . ?
 O3 W1 O2 83.7(6) . . ?
 O2 W1 O2 161.12(9) 6_656 . ?
 O1 W1 O3 100.9(6) . 3_755 ?
 O3 W1 O3 158.77(9) . 3_755 ?
 O2 W1 O3 94.6(6) 6_656 3_755 ?
 O2 W1 O3 83.2(6) . 3_755 ?
 O1 W1 N1 176.1(6) . . ?
 O3 W1 N1 81.3(6) . . ?
 O2 W1 N1 82.3(4) 6_656 . ?
 O2 W1 N1 78.9(4) . . ?
 O3 W1 N1 79.8(6) 3_755 . ?
 C1 N1 C5 119.1(16) . . ?
 C1 N1 W1 121.5(11) . . ?
 C5 N1 W1 119.2(12) . . ?
 N1 C1 C2 121.9(16) . . ?
 C2 C3 C4 115.9(15) . . ?
 C2 C3 C3 125(2) . 5_776 ?
 C4 C3 C3 119(2) . 5_776 ?
 N1 C5 C4 122.4(17) . . ?
 C3 C2 C1 121.0(16) . . ?
 C5 C4 C3 119.7(16) . . ?
 W1 O3 W1 174.9(9) . 3_745 ?

_diffrn_measured_fraction_theta_max	0.992
_diffrn_reflns_theta_full	72.11
_diffrn_measured_fraction_theta_full	0.992
_refine_diff_density_max	4.927
_refine_diff_density_min	-4.550
_refine_diff_density_rms	0.727