

data\_WO3-bpy-2Ca

```
_audit_creation_method      SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    'C5 H4 Ca0.05 N O3 W'
_chemical_formula_sum       'C5 H4 Ca0.05 N O3 W'
_chemical_formula_weight    311.94
```

```
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'
Ca Ca 0.3641 1.2855 '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              7.4775(10)
_cell_length_b              7.3925(10)
_cell_length_c              22.5829(16)
_cell_angle_alpha           90.00
_cell_angle_beta            90.00
_cell_angle_gamma           90.00
_cell_volume                 1248.3(3)
_cell_formula_units_Z       8
_cell_measurement_temperature 163(2)
_cell_measurement_reflns_used 8965
_cell_measurement_theta_min  14.21
_cell_measurement_theta_max  143.69
```

```
_exptl_crystal_description  plate
_exptl_crystal_colour        yellow-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 3.298
_exptl_crystal_density_method 'not measured'
_exptl_crystal_F_000         1112
_exptl_absorpt_coefficient_mu 33.861
_exptl_absorpt_correction_type empirical
_exptl_absorpt_correction_T_min 0.2934
_exptl_absorpt_correction_T_max 1.000
_exptl_absorpt_process_details
;
data scaling includes absorption [ABSCOR, Higashi (1995)]
_exptl_special_details
;
_diffn_ambient_temperature    163(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           14507
_diffn_reflns_av_R_equivalents 0.1163
_diffn_reflns_av_sigmaI/netI   0.0756
_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               693
_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)'

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```
_refine_special_details
```

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;
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
based on F, with F set to zero for negative F2. 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.

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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^)+(0.0581P)^2^+49.0621P] 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_expression
'Fc^*=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          756
_refine_ls_number_parameters       104
_refine_ls_number_restraints       54
_refine_ls_R_factor_all            0.0685
_refine_ls_R_factor_gt             0.0651
_refine_ls_wR_factor_ref           0.1319
_refine_ls_wR_factor_gt            0.1300
_refine_ls_goodness_of_fit_ref     1.120
_refine_ls_restrained_S_all        1.094
_refine_ls_shift/su_max            0.340
_refine_ls_shift/su_mean           0.003
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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
Ca1 Ca 0.495(19) 0.55(2) 0.464(9) 0.10(6) Uiso 0.05 1 d P . .
C5 C 0.504(2) 0.507(3) 0.0323(9) 0.024(4) Uani 1 1 d U . .
W1 W 0.52073(11) 0.54850(11) 0.26376(3) 0.0090(5) Uani 1 1 d . . .
O1 O 0.5034(16) 0.2948(19) 0.2536(5) 0.032(4) Uani 1 1 d U . .
O2 O 0.2596(19) 0.547(2) 0.2482(4) 0.039(4) Uani 1 1 d U . .
O3 O 0.5104(13) 0.5506(14) 0.3401(6) 0.016(3) Uani 1 1 d U . .
N1 N 0.5175(16) 0.5367(18) 0.1581(7) 0.013(3) Uani 1 1 d U . .
C2 C 0.624(2) 0.424(2) 0.1275(7) 0.019(4) Uani 1 1 d U . .
H2 H 0.7053 0.3543 0.1488 0.023 Uiso 1 1 calc R . .
C3 C 0.622(2) 0.404(2) 0.0671(7) 0.023(4) Uani 1 1 d U . .
H3 H 0.6995 0.3228 0.0490 0.028 Uiso 1 1 calc R . .
C4 C 0.394(2) 0.628(2) 0.0630(7) 0.022(4) Uani 1 1 d U . .
H4 H 0.31(2) 0.71(2) 0.039(6) 0.02(4) Uiso 1 1 d . . .
C1 C 0.403(2) 0.637(2) 0.1247(6) 0.020(4) Uani 1 1 d U . .
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H1 H 0.319(19) 0.74(2) 0.144(5) 0.01(4) Uiso 1 1 d . . .

```
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
C5 0.023(6) 0.024(6) 0.026(6) -0.002(5) -0.004(4) 0.000(4)
W1 0.0037(7) 0.0068(8) 0.0166(7) -0.0006(3) -0.0002(3) 0.0005(3)
O1 0.039(6) 0.031(6) 0.026(4) 0.003(4) 0.004(4) 0.002(4)
O2 0.036(6) 0.047(6) 0.035(4) 0.001(4) 0.000(4) 0.001(4)
O3 0.010(4) 0.011(5) 0.028(5) -0.001(3) 0.001(3) 0.000(3)
N1 0.011(5) 0.013(5) 0.014(5) -0.005(4) -0.002(4) 0.002(4)
C2 0.018(6) 0.018(6) 0.021(5) -0.001(4) 0.000(4) -0.001(4)
C3 0.021(6) 0.022(6) 0.027(5) -0.001(4) 0.001(4) 0.003(4)
C4 0.018(5) 0.019(5) 0.028(5) 0.001(4) 0.000(4) 0.004(5)
C1 0.018(5) 0.019(5) 0.022(5) 0.000(4) -0.001(4) 0.003(5)
```

\_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
Ca1 Ca1 1.8(4) 5_666 ?
Ca1 O3 2.8(2) . ?
Ca1 C3 2.88(17) 3_655 ?
Ca1 C3 3.06(15) 6_556 ?
Ca1 C4 3.11(15) 6_656 ?
C5 C4 1.40(3) . ?
C5 C3 1.40(3) . ?
C5 C5 1.46(4) 5_665 ?
W1 O3 1.725(13) . ?
W1 O2 1.806(14) 6_656 ?
W1 O1 1.871(14) 3_655 ?
W1 O1 1.894(15) . ?
W1 O2 1.984(14) . ?
W1 N1 2.387(15) . ?
O1 W1 1.871(14) 3_645 ?
O2 W1 1.806(14) 6_556 ?
N1 C2 1.35(2) . ?
N1 C1 1.36(2) . ?
C2 C3 1.37(2) . ?
C3 Ca1 2.88(17) 3_645 ?
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C3 Ca1 3.06(15) 6_656 ?
C4 C1 1.40(2) . ?
C4 Ca1 3.11(15) 6_556 ?

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
Ca1 Ca1 O3 158(10) 5_666 . ?
Ca1 Ca1 C3 126(10) 5_666 3_655 ?
O3 Ca1 C3 76(5) . 3_655 ?
Ca1 Ca1 C3 97(10) 5_666 6_556 ?
O3 Ca1 C3 79(4) . 6_556 ?
C3 Ca1 C3 89(4) 3_655 6_556 ?
Ca1 Ca1 C4 102(10) 5_666 6_656 ?
O3 Ca1 C4 76(4) . 6_656 ?
C3 Ca1 C4 94(5) 3_655 6_656 ?
C3 Ca1 C4 154(7) 6_556 6_656 ?
C4 C5 C3 116.0(17) . . ?
C4 C5 C5 121(2) . 5_665 ?
C3 C5 C5 123(2) . 5_665 ?
O3 W1 O2 101.2(5) . 6_656 ?
O3 W1 O1 101.3(5) . 3_655 ?
O2 W1 O1 94.0(6) 6_656 3_655 ?
O3 W1 O1 97.3(5) . . ?
O2 W1 O1 92.5(6) 6_656 . ?
O1 W1 O1 158.69(6) 3_655 . ?
O3 W1 O2 97.6(4) . . ?
O2 W1 O2 161.19(6) 6_656 . ?
O1 W1 O2 82.7(6) 3_655 . ?
O1 W1 O2 84.6(6) . . ?
O3 W1 N1 176.5(5) . . ?
O2 W1 N1 82.0(4) 6_656 . ?
O1 W1 N1 80.0(5) 3_655 . ?
O1 W1 N1 80.9(5) . . ?
O2 W1 N1 79.2(4) . . ?
W1 O1 W1 174.6(7) 3_645 . ?
W1 O2 W1 178.2(7) 6_556 . ?
W1 O3 Ca1 179(3) . . ?
C2 N1 C1 115.1(15) . . ?
C2 N1 W1 122.0(11) . . ?
C1 N1 W1 122.8(10) . . ?
N1 C2 C3 124.7(16) . . ?
C2 C3 C5 120.4(16) . . ?
C2 C3 Ca1 110(4) . 3_645 ?
C5 C3 Ca1 100(4) . 3_645 ?
C2 C3 Ca1 100(4) . 6_656 ?
C5 C3 Ca1 105(4) . 6_656 ?
Ca1 C3 Ca1 122(5) 3_645 6_656 ?
C1 C4 C5 119.5(16) . . ?
C1 C4 Ca1 105(4) . 6_556 ?
C5 C4 Ca1 110(3) . 6_556 ?
N1 C1 C4 124.2(15) . . ?

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_diffraction_measured_fraction_theta_max	0.999
_diffraction_reflns_theta_full	54.23
_diffraction_measured_fraction_theta_full	0.999
_refine_diff_density_max	2.286
_refine_diff_density_min	-2.654
_refine_diff_density_rms	0.394