

data_WO3_phen

```
_audit_creation_method          SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common           ?
_chemical_melting_point         ?
_chemical_formula_moiety        'C6 H4 N1 O3 W1'
_chemical_formula_sum           'C6 H4 N1 O3 W1'
_chemical_formula_weight        321.94
```

```
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  'C m c a'
```

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

```
_cell_length_a                  22.6294(4)
_cell_length_b                  7.57790(10)
_cell_length_c                  14.7923(10)
_cell_angle_alpha               90.00
_cell_angle_beta                90.00
_cell_angle_gamma               90.00
_cell_volume                    2536.63(18)
_cell_formula_units_Z           16
```

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_cell_measurement_temperature      293(1)
_cell_measurement_reflns_used      6834
_cell_measurement_theta_min        13.70
_cell_measurement_theta_max        144.27

_exptl_crystal_description         plate
_exptl_crystal_colour              yellow
_exptl_crystal_size_max            0.05
_exptl_crystal_size_mid            0.05
_exptl_crystal_size_min            0.01
_exptl_crystal_density_meas        ?
_exptl_crystal_density_diffn       3.372
_exptl_crystal_density_method      'not measured'
_exptl_crystal_F_000               2320
_exptl_absorpt_coefficient_mu       33.384
_exptl_absorpt_correction_type      empirical
_exptl_absorpt_correction_T_min     0.537
_exptl_absorpt_correction_T_max     1.0000
_exptl_absorpt_process_details
;
data scaling includes absorption [ABSCOR, Higashi (1995)]
_exptl_special_details
;
_diffn_ambient_temperature         293(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                10463
_diffn_reflns_av_R_equivalents      0.0528
_diffn_reflns_av_sigmaI/netI        0.0395
_diffn_reflns_limit_h_min           -26
_diffn_reflns_limit_h_max           26
_diffn_reflns_limit_k_min           -8
_diffn_reflns_limit_k_max           8
_diffn_reflns_limit_l_min           -14
_diffn_reflns_limit_l_max           17
_diffn_reflns_theta_min             6.85
_diffn_reflns_theta_max             62.34
_reflns_number_total                1039
_reflns_number_gt                   856
_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)'

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_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 based
on F, with F set to zero for negative F2. The threshold expression of
F2 > 2sigma(F2) is used only for calculating R-factors(gt) etc. and
is not relevant to the choice of reflections for refinement. R-factors
based on F2 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/[\s2(Fo2)+(0.0290P)2+86.5338P] where P=(Fo2+2Fc2)/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 1039
_refine_ls_number_parameters 101
_refine_ls_number_restraints 66
_refine_ls_R_factor_all 0.0446
_refine_ls_R_factor_gt 0.0316
_refine_ls_wR_factor_ref 0.0810
_refine_ls_wR_factor_gt 0.0760
_refine_ls_goodness_of_fit_ref 1.101
_refine_ls_restrained_S_all 1.085
_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
O4 O 0.2500(3) 0.2656(9) 0.1253(4) 0.0289(17) Uani 1 1 d U . .
W1 W 0.262374(15) 0.02884(5) 0.12406(3) 0.0136(2) Uani 1 1 d . . .
O1 O 0.3379(3) 0.0215(8) 0.1195(4) 0.0265(17) Uani 1 1 d U . .
N1 N 0.1548(3) 0.0215(9) 0.1307(5) 0.0178(17) Uani 1 1 d U . .
O2 O 0.2452(5) 0.0000 0.0000 0.037(3) Uani 1 2 d SU . .
O3 O 0.2500 0.0063(15) 0.2500 0.039(3) Uani 1 2 d SU . .
C3 C 0.0322(4) 0.0218(12) 0.1420(7) 0.027(2) Uani 1 1 d U . .
C6 C 0.1239(4) 0.1383(13) 0.0823(7) 0.023(2) Uani 1 1 d U . .

```

```

H6 H 0.1441 0.2179 0.0457 0.028 Uiso 1 1 calc R . .
C1 C 0.1263(4) -0.0886(13) 0.1850(7) 0.027(2) Uani 1 1 d U . .
H1 H 0.1484 -0.1658 0.2204 0.033 Uiso 1 1 calc R . .
C4 C 0.0625(4) 0.1445(13) 0.0848(7) 0.025(2) Uani 1 1 d U . .
C2 C 0.0662(4) -0.0947(14) 0.1919(7) 0.032(2) Uani 1 1 d U . .
H2 H 0.0482 -0.1766 0.2297 0.039 Uiso 1 1 calc R . .
C5 C 0.0303(4) 0.2657(13) 0.0313(7) 0.030(2) Uani 1 1 d U . .
H5 H 0.0505 0.3467 -0.0044 0.035 Uiso 1 1 calc R . .

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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
O4 0.027(3) 0.023(3) 0.037(4) 0.001(3) 0.000(3) -0.003(3)
W1 0.0150(3) 0.0122(3) 0.0137(3) 0.00015(17) 0.00020(17) 0.00028(14)
O1 0.032(3) 0.021(3) 0.027(4) -0.001(3) -0.001(3) 0.001(2)
N1 0.017(3) 0.020(3) 0.016(4) -0.003(3) 0.002(3) -0.004(3)
O2 0.032(4) 0.051(5) 0.030(4) 0.003(4) 0.000 0.000
O3 0.028(4) 0.052(5) 0.038(5) 0.000 -0.001(4) 0.000
C3 0.026(4) 0.027(4) 0.027(4) 0.000(3) 0.000(4) 0.001(3)
C6 0.025(4) 0.022(4) 0.022(4) -0.004(3) -0.002(3) -0.001(3)
C1 0.029(4) 0.024(4) 0.029(4) -0.003(3) -0.002(3) 0.002(3)
C4 0.029(4) 0.022(4) 0.025(4) -0.003(3) -0.003(3) 0.001(3)
C2 0.029(4) 0.030(4) 0.037(4) 0.001(4) -0.002(4) -0.003(3)
C5 0.030(4) 0.023(4) 0.036(4) 0.003(3) 0.000(4) -0.002(3)

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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
O4 W1 1.816(7) . ?
O4 W1 2.014(7) 16 ?
W1 O1 1.712(7) . ?
W1 O2 1.889(2) . ?
W1 O3 1.8916(11) . ?
W1 O4 2.014(7) 16_545 ?
W1 N1 2.436(7) . ?
N1 C1 1.327(12) . ?
N1 C6 1.336(12) . ?
O2 W1 1.889(2) 4 ?
O3 W1 1.8916(11) 7_545 ?

```

C3 C2 1.383(14) . ?
 C3 C4 1.432(14) . ?
 C3 C3 1.46(2) 12 ?
 C6 C4 1.392(13) . ?
 C1 C2 1.365(14) . ?
 C4 C5 1.414(13) . ?
 C5 C5 1.37(2) 12 ?

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 O4 W1 178.6(4) . 16 ?
 O1 W1 O4 100.7(3) . . ?
 O1 W1 O2 99.4(4) . . ?
 O4 W1 O2 95.3(2) . . ?
 O1 W1 O3 100.6(2) . . ?
 O4 W1 O3 93.2(4) . . ?
 O2 W1 O3 156.4(3) . . ?
 O1 W1 O4 96.2(3) . 16_545 ?
 O4 W1 O4 163.11(4) . 16_545 ?
 O2 W1 O4 82.26(18) . 16_545 ?
 O3 W1 O4 83.2(4) . 16_545 ?
 O1 W1 N1 176.8(3) . . ?
 O4 W1 N1 82.5(3) . . ?
 O2 W1 N1 80.3(4) . . ?
 O3 W1 N1 79.08(17) . . ?
 O4 W1 N1 80.7(2) 16_545 . ?
 C1 N1 C6 119.1(8) . . ?
 C1 N1 W1 121.7(6) . . ?
 C6 N1 W1 119.1(6) . . ?
 W1 O2 W1 156.2(7) . 4 ?
 W1 O3 W1 169.7(7) 7_545 . ?
 C2 C3 C4 117.7(9) . . ?
 C2 C3 C3 123.7(6) . 12 ?
 C4 C3 C3 118.5(6) . 12 ?
 N1 C6 C4 122.1(9) . . ?
 N1 C1 C2 123.5(10) . . ?
 C6 C4 C5 121.5(9) . . ?
 C6 C4 C3 118.1(9) . . ?
 C5 C4 C3 120.4(9) . . ?
 C1 C2 C3 119.5(10) . . ?
 C5 C5 C4 121.0(6) 12 . ?

_diffraction_measured_fraction_theta_max	0.991
_diffraction_reflns_theta_full	62.34
_diffraction_measured_fraction_theta_full	0.991
_refine_diff_density_max	2.478
_refine_diff_density_min	-1.707
_refine_diff_density_rms	0.319