

data_MoO3-phen

```
_audit_creation_method      SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    'C6 H4 Mo1 N O3'
_chemical_formula_sum       'C6 H4 Mo1 N O3'
_chemical_formula_weight    234.04
```

```
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'
Mo Mo -0.0483 2.7339 '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              22.7233(4)
_cell_length_b              7.73550(10)
_cell_length_c              14.5903(10)
_cell_angle_alpha           90.00
_cell_angle_beta            90.00
_cell_angle_gamma           90.00
_cell_volume                 2564.63(18)
_cell_formula_units_Z       16
```

```

_cell_measurement_temperature      293(2)
_cell_measurement_reflns_used      9441
_cell_measurement_theta_min        13.53
_cell_measurement_theta_max        143.76

_exptl_crystal_description         plate
_exptl_crystal_colour              green
_exptl_crystal_size_max            0.13
_exptl_crystal_size_mid            0.09
_exptl_crystal_size_min            0.02
_exptl_crystal_density_meas        ?
_exptl_crystal_density_diffn       2.425
_exptl_crystal_density_method      'not measured'
_exptl_crystal_F_000               1808
_exptl_absorpt_coefficient_mu       16.362
_exptl_absorpt_correction_type      empirical
_exptl_absorpt_correction_T_min     0.612
_exptl_absorpt_correction_T_max     1.000
_exptl_absorpt_process_details
;
data scaling includes absorption [ABSCOR, Higashi (1995)]
_exptl_special_details
;

_diffn_ambient_temperature         293(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                13252
_diffn_reflns_av_R_equivalents      0.0989
_diffn_reflns_av_sigmaI/netI        0.0543
_diffn_reflns_limit_h_min           -23
_diffn_reflns_limit_h_max           23
_diffn_reflns_limit_k_min           -7
_diffn_reflns_limit_k_max           8
_diffn_reflns_limit_l_min           -15
_diffn_reflns_limit_l_max           15
_diffn_reflns_theta_min             6.76
_diffn_reflns_theta_max             54.18
_reflns_number_total                808
_reflns_number_gt                   655
_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^)+(0.0720P)^2^+126.9885P]

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 0.00002(2)

_refine_ls_extinction_expression

'Fc^*=kFc[1+0.001xFc^2^l^3^/sin(2\q)]^-1/4^'

_refine_ls_number_reflns 808

_refine_ls_number_parameters 97

_refine_ls_number_restraints 60

_refine_ls_R_factor_all 0.0650

_refine_ls_R_factor_gt 0.0580

_refine_ls_wR_factor_ref 0.1795

_refine_ls_wR_factor_gt 0.1753

_refine_ls_goodness_of_fit_ref 1.161

_refine_ls_restrained_S_all 1.118

_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 0.2508(5) 0.2678(16) 0.6247(6) 0.047(3) Uiso 1 1 d . . .

C10 C 0.0298(6) 0.2664(18) 0.5311(9) 0.041(4) Uani 1 1 d U . .

H10 H 0.0500 0.3462 0.4952 0.049 Uiso 1 1 calc R . .

Mo1 Mo 0.26191(5) 0.04228(15) 0.62357(7) 0.0272(7) Uani 1 1 d . . .

```

O2 O 0.2427(6) 0.0000 0.5000 0.053(5) Uani 1 2 d SU . .
N1 N 0.1556(5) 0.0266(15) 0.6321(7) 0.032(3) Uani 1 1 d U . .
O1 O 0.3364(5) 0.0283(12) 0.6169(6) 0.035(3) Uani 1 1 d U . .
O3 O 0.2500 0.006(3) 0.7500 0.054(5) Uani 1 2 d SU . .
C4 C 0.0319(7) 0.025(2) 0.6419(10) 0.040(4) Uani 1 1 d U . .
C2 C 0.1237(6) 0.1406(19) 0.5833(10) 0.035(4) Uani 1 1 d U . .
H2 H 0.1437 0.2193 0.5466 0.043 Uiso 1 1 calc R . .
C5 C 0.0668(7) -0.090(2) 0.6921(10) 0.043(4) Uani 1 1 d U . .
H5 H 0.0491 -0.1715 0.7301 0.051 Uiso 1 1 calc R . .
C3 C 0.0617(7) 0.1476(19) 0.5846(10) 0.037(4) Uani 1 1 d U . .
C6 C 0.1260(6) -0.085(2) 0.6858(10) 0.037(4) Uani 1 1 d U . .
H6 H 0.1476 -0.1624 0.7207 0.044 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
C10 0.055(8) 0.026(8) 0.041(8) 0.011(7) 0.004(7) -0.003(7)
Mo1 0.0332(10) 0.0226(10) 0.0259(9) -0.0002(5) -0.0001(5) 0.0007(5)
O2 0.034(8) 0.107(13) 0.017(8) 0.014(8) 0.000 0.000
N1 0.029(7) 0.032(8) 0.036(7) -0.002(6) 0.004(5) -0.001(6)
O1 0.052(7) 0.024(6) 0.029(5) 0.003(4) -0.001(4) 0.001(5)
O3 0.030(7) 0.100(13) 0.031(9) 0.000 -0.006(7) 0.000
C4 0.042(8) 0.042(10) 0.038(8) -0.005(8) 0.004(7) -0.005(8)
C2 0.039(9) 0.037(9) 0.030(8) -0.005(7) 0.006(7) -0.002(7)
C5 0.048(11) 0.042(10) 0.039(9) 0.002(8) 0.000(7) -0.003(8)
C3 0.054(10) 0.031(9) 0.027(8) -0.007(7) 0.001(7) 0.002(7)
C6 0.033(9) 0.040(9) 0.038(9) -0.010(8) 0.004(7) -0.004(7)

```

```

_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.
;

```

```

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 Mo1 1.763(12) . ?
O4 Mo1 2.143(12) 16 ?
C10 C10 1.36(3) 12 ?
C10 C3 1.41(2) . ?
Mo1 O1 1.699(11) . ?
Mo1 O2 1.884(4) . ?
Mo1 O3 1.885(3) . ?
Mo1 O4 2.143(12) 16_545 ?

```

Mo1 N1 2.422(12) . ?
 O2 Mo1 1.884(4) 4_556 ?
 N1 C6 1.344(18) . ?
 N1 C2 1.345(18) . ?
 O3 Mo1 1.885(3) 7_546 ?
 C4 C5 1.40(2) . ?
 C4 C3 1.44(2) . ?
 C4 C4 1.45(3) 12 ?
 C2 C3 1.41(2) . ?
 C5 C6 1.35(2) . ?

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
 Mo1 O4 Mo1 178.9(6) . 16 ?
 C10 C10 C3 121.0(9) 12 . ?
 O1 Mo1 O4 101.9(5) . . ?
 O1 Mo1 O2 99.5(5) . . ?
 O4 Mo1 O2 98.5(3) . . ?
 O1 Mo1 O3 100.9(3) . . ?
 O4 Mo1 O3 96.7(7) . . ?
 O2 Mo1 O3 151.4(5) . . ?
 O1 Mo1 O4 94.1(4) . 16_545 ?
 O4 Mo1 O4 163.99(10) . 16_545 ?
 O2 Mo1 O4 78.7(2) . 16_545 ?
 O3 Mo1 O4 80.0(6) . 16_545 ?
 O1 Mo1 N1 173.5(4) . . ?
 O4 Mo1 N1 84.6(4) . . ?
 O2 Mo1 N1 79.0(5) . . ?
 O3 Mo1 N1 78.4(3) . . ?
 O4 Mo1 N1 79.4(4) 16_545 . ?
 Mo1 O2 Mo1 153.2(9) . 4_556 ?
 C6 N1 C2 117.2(13) . . ?
 C6 N1 Mo1 124.2(10) . . ?
 C2 N1 Mo1 118.6(9) . . ?
 Mo1 O3 Mo1 163.0(12) 7_546 . ?
 C5 C4 C3 117.3(14) . . ?
 C5 C4 C4 124.5(9) . 12 ?
 C3 C4 C4 118.2(9) . 12 ?
 N1 C2 C3 123.9(14) . . ?
 C6 C5 C4 120.5(15) . . ?
 C10 C3 C2 122.3(14) . . ?
 C10 C3 C4 120.8(14) . . ?
 C2 C3 C4 117.0(14) . . ?
 N1 C6 C5 124.1(15) . . ?

_diffraction_measured_fraction_theta_max	0.998
_diffraction_reflns_theta_full	54.18
_diffraction_measured_fraction_theta_full	0.998
_refinement_diff_density_max	1.303
_refinement_diff_density_min	-1.659
_refinement_diff_density_rms	0.213