

data_vmxl_280k_abs

_audit_creation_method SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common ?
_chemical_melting_point ?
_chemical_formula_moiety ?
_chemical_formula_sum
'H B Ca O5 Si'
_chemical_formula_weight 159.99

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scatter_dispersion_real
_atom_type_scatter_dispersion_imag
_atom_type_scatter_source
'H' 'H' 0.0000 0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'B' 'B' 0.0013 0.0007
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'O' 'O' 0.0106 0.0060
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'Si' 'Si' 0.0817 0.0704
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'Ca' 'Ca' 0.2262 0.3064
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting ?
_symmetry_space_group_name_H-M ?

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

_cell_length_a 4.83550(10)
_cell_length_b 7.60850(10)
_cell_length_c 9.6326(2)
_cell_angle_alpha 90.00
_cell_angle_beta 90.159(2)
_cell_angle_gamma 90.00
_cell_volume 354.391(11)
_cell_formula_units_Z 4
_cell_measurement_temperature 553(2)
_cell_measurement_reflns_used ?
_cell_measurement_theta_min ?

```

_cell_measurement_theta_max      ?

_exptl_crystal_description      ?
_exptl_crystal_colour           ?
_exptl_crystal_size_max         ?
_exptl_crystal_size_mid         ?
_exptl_crystal_size_min         ?
_exptl_crystal_density_meas     ?
_exptl_crystal_density_diffn    2.999
_exptl_crystal_density_method   'not measured'
_exptl_crystal_F_000            320
_exptl_absorpt_coefficient_mu    1.997
_exptl_absorpt_correction_type  ?
_exptl_absorpt_correction_T_min ?
_exptl_absorpt_correction_T_max ?
_exptl_absorpt_process_details  ?

_exptl_special_details
;
?
;

_diffn_ambient_temperature      280(2)
_diffn_radiation_wavelength     0.71073
_diffn_radiation_type           MoK\alpha
_diffn_radiation_source         'fine-focus sealed tube'
_diffn_radiation_monochromator  graphite
_diffn_measurement_device_type  ?
_diffn_measurement_method       ?
_diffn_detector_area_resol_mean ?
_diffn_standards_number         ?
_diffn_standards_interval_count ?
_diffn_standards_interval_time  ?
_diffn_standards_decay_%        ?
_diffn_reflns_number            8553
_diffn_reflns_av_R_equivalents  0.0329
_diffn_reflns_av_sigmaI/netI    0.0262
_diffn_reflns_limit_h_min       -7
_diffn_reflns_limit_h_max       8
_diffn_reflns_limit_k_min       -13
_diffn_reflns_limit_k_max       13
_diffn_reflns_limit_l_min       -16
_diffn_reflns_limit_l_max       17
_diffn_reflns_theta_min         4.21
_diffn_reflns_theta_max         40.09
_reflns_number_total            2205
_reflns_number_gt               1921
_reflns_threshold_expression     >2sigma(I)

_computing_data_collection      ?
_computing_cell_refinement      ?
_computing_data_reduction       ?
_computing_structure_solution   ?
_computing_structure_refinement 'SHELXL-97 (Sheldrick, 1997)'

```

_computing_molecular_graphics ?
_computing_publication_material ?

_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^)+(0.0100P)^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 SHELXL
_refine_ls_extinction_coef 0.086(3)
_refine_ls_extinction_expression
'Fc^*=kFc[1+0.001xFc^2^/l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns 2205
_refine_ls_number_parameters 83
_refine_ls_number_restraints 0
_refine_ls_R_factor_all 0.0280
_refine_ls_R_factor_gt 0.0223
_refine_ls_wR_factor_ref 0.0481
_refine_ls_wR_factor_gt 0.0475
_refine_ls_goodness_of_fit_ref 1.542
_refine_ls_restrained_S_all 1.542
_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

Ca Ca 0.99167(4) 0.10616(2) 0.336116(18) 0.00615(4) Uani 1 1 d . . .

Si Si 0.46899(5) 0.26620(3) 0.08436(2) 0.00409(5) Uani 1 1 d . . .
 B B 0.56856(19) 0.41067(12) 0.34086(10) 0.00513(14) Uani 1 1 d . . .
 O1 O 0.24063(13) 0.40112(8) 0.03738(7) 0.00724(11) Uani 1 1 d . . .
 O2 O 0.66991(12) 0.30097(8) 0.45747(7) 0.00605(10) Uani 1 1 d . . .
 O3 O 0.67520(12) 0.33451(8) 0.21047(6) 0.00607(11) Uani 1 1 d . . .
 O4 O 0.31489(13) 0.08791(8) 0.14591(7) 0.00673(11) Uani 1 1 d . . .
 O5 O 0.25889(14) 0.41349(9) 0.33656(7) 0.00712(11) Uani 1 1 d . . .
 H H 0.206(3) 0.452(2) 0.408(2) 0.030(5) Uani 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

Ca 0.00645(7) 0.00675(7) 0.00525(7) -0.00003(5) 0.00055(5) -0.00039(5)
 Si 0.00491(10) 0.00428(9) 0.00308(10) -0.00016(7) 0.00023(7) 0.00028(7)
 B 0.0059(3) 0.0056(3) 0.0040(3) -0.0003(3) 0.0001(3) -0.0002(3)
 O1 0.0076(3) 0.0073(2) 0.0068(3) -0.0003(2) -0.0009(2) 0.00222(19)
 O2 0.0066(2) 0.0071(2) 0.0045(2) 0.00175(19) 0.00099(19) 0.00084(18)
 O3 0.0061(2) 0.0083(2) 0.0038(2) -0.00146(19) 0.00042(19) 0.00038(19)
 O4 0.0074(3) 0.0046(2) 0.0082(3) 0.00088(19) 0.0024(2) 0.00038(18)
 O5 0.0054(3) 0.0099(3) 0.0061(3) -0.0011(2) 0.0006(2) 0.0008(2)
 H 0.012(9) 0.032(11) 0.046(15) 0.003(10) -0.005(9) -0.006(8)

_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
 Ca O1 2.2777(7) 2_645 ?
 Ca O1 2.2799(7) 4_666 ?
 Ca O4 2.4155(7) 1_655 ?
 Ca O2 2.4483(7) . ?
 Ca O5 2.5243(7) 2_645 ?
 Ca O3 2.6105(6) . ?
 Ca O3 2.6593(7) 2_745 ?
 Ca O5 2.6716(7) 1_655 ?
 Si O1 1.5733(6) . ?
 Si O2 1.6449(7) 4_565 ?
 Si O3 1.6533(6) . ?

Si O4 1.6581(6) . ?
 B O4 1.4670(11) 2_655 ?
 B O3 1.4774(11) . ?
 B O2 1.4817(11) . ?
 B O5 1.4980(12) . ?
 O1 Ca 2.2777(7) 2_655 ?
 O1 Ca 2.2799(7) 4_465 ?
 O2 Si 1.6449(7) 4_566 ?
 O3 Ca 2.6593(7) 2_755 ?
 O4 B 1.4670(11) 2_645 ?
 O4 Ca 2.4155(7) 1_455 ?
 O5 Ca 2.5243(7) 2_655 ?
 O5 Ca 2.6716(7) 1_455 ?

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
 O1 Ca O1 77.79(2) 2_645 4_666 ?
 O1 Ca O4 133.43(2) 2_645 1_655 ?
 O1 Ca O4 107.60(2) 4_666 1_655 ?
 O1 Ca O2 81.04(2) 2_645 . ?
 O1 Ca O2 86.76(2) 4_666 . ?
 O4 Ca O2 144.13(2) 1_655 . ?
 O1 Ca O5 73.64(2) 2_645 2_645 ?
 O1 Ca O5 142.89(2) 4_666 2_645 ?
 O4 Ca O5 77.13(2) 1_655 2_645 ?
 O2 Ca O5 111.20(2) . 2_645 ?
 O1 Ca O3 114.49(2) 2_645 . ?
 O1 Ca O3 135.88(2) 4_666 . ?
 O4 Ca O3 93.80(2) 1_655 . ?
 O2 Ca O3 56.35(2) . . ?
 O5 Ca O3 78.54(2) 2_645 . ?
 O1 Ca O3 81.83(2) 2_645 2_745 ?
 O1 Ca O3 78.81(2) 4_666 2_745 ?
 O4 Ca O3 55.51(2) 1_655 2_745 ?
 O2 Ca O3 159.58(2) . 2_745 ?
 O5 Ca O3 74.18(2) 2_645 2_745 ?
 O3 Ca O3 142.523(10) . 2_745 ?
 O1 Ca O5 146.92(2) 2_645 1_655 ?
 O1 Ca O5 76.45(2) 4_666 1_655 ?
 O4 Ca O5 74.78(2) 1_655 1_655 ?
 O2 Ca O5 77.14(2) . 1_655 ?
 O5 Ca O5 137.781(15) 2_645 1_655 ?
 O3 Ca O5 72.61(2) . 1_655 ?
 O3 Ca O5 112.79(2) 2_745 1_655 ?
 O1 Si O2 113.86(4) . 4_565 ?
 O1 Si O3 115.32(3) . . ?
 O2 Si O3 106.70(3) 4_565 . ?
 O1 Si O4 108.72(3) . . ?

O2 Si O4 106.17(3) 4_565 . ?
 O3 Si O4 105.38(3) . . ?
 O4 B O3 107.43(7) 2_655 . ?
 O4 B O2 109.01(7) 2_655 . ?
 O3 B O2 107.94(7) . . ?
 O4 B O5 111.90(7) 2_655 . ?
 O3 B O5 109.47(7) . . ?
 O2 B O5 110.95(7) . . ?
 Si O1 Ca 129.78(4) . 2_655 ?
 Si O1 Ca 126.68(4) . 4_465 ?
 Ca O1 Ca 102.21(2) 2_655 4_465 ?
 B O2 Si 122.92(6) . 4_566 ?
 B O2 Ca 100.86(5) . . ?
 Si O2 Ca 122.97(3) 4_566 . ?
 B O3 Si 122.46(5) . . ?
 B O3 Ca 94.15(4) . . ?
 Si O3 Ca 118.88(3) . . ?
 B O3 Ca 92.96(5) . 2_755 ?
 Si O3 Ca 118.99(3) . 2_755 ?
 Ca O3 Ca 103.94(2) . 2_755 ?
 B O4 Si 127.61(6) 2_645 . ?
 B O4 Ca 103.66(5) 2_645 1_455 ?
 Si O4 Ca 121.10(3) . 1_455 ?
 B O5 Ca 120.29(5) . 2_655 ?
 B O5 Ca 118.09(5) . 1_455 ?
 Ca O5 Ca 106.02(2) 2_655 1_455 ?

_diffn_measured_fraction_theta_max 0.995
 _diffn_reflns_theta_full 40.00
 _diffn_measured_fraction_theta_full 0.995
 _refine_diff_density_max 0.602
 _refine_diff_density_min -0.450
 _refine_diff_density_rms 0.111