

data_vmxl_220k_abs

_audit_creation_method SHELXL-97

_chemical_name_systematic

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?

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_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 4.83250(10)

_cell_length_b 7.60410(10)

_cell_length_c 9.6300(2)

_cell_angle_alpha 90.00

_cell_angle_beta 90.184(2)

_cell_angle_gamma 90.00

_cell_volume 353.870(11)

_cell_formula_units_Z 4

_cell_measurement_temperature 493(2)

_cell_measurement_reflns_used ?

_cell_measurement_theta_min ?

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_cell_measurement_theta_max      ?

_exptl_crystal_description      ?
_exptl_crystal_colour           ?
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_exptl_crystal_size_mid         ?
_exptl_crystal_size_min         ?
_exptl_crystal_density_meas     ?
_exptl_crystal_density_diffn    3.003
_exptl_crystal_density_method   'not measured'
_exptl_crystal_F_000            320
_exptl_absorpt_coefficient_mu    2.000
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_exptl_special_details
;
?
;

_diffrn_ambient_temperature     220(2)
_diffrn_radiation_wavelength    0.71073
_diffrn_radiation_type          MoK\alpha
_diffrn_radiation_source        'fine-focus sealed tube'
_diffrn_radiation_monochromator graphite
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_diffrn_reflns_av_sigmaI/netI   0.0265
_diffrn_reflns_limit_h_min      -7
_diffrn_reflns_limit_h_max      8
_diffrn_reflns_limit_k_min      -13
_diffrn_reflns_limit_k_max      13
_diffrn_reflns_limit_l_min      -16
_diffrn_reflns_limit_l_max      17
_diffrn_reflns_theta_min        4.22
_diffrn_reflns_theta_max        40.08
_reflns_number_total             2204
_reflns_number_gt               1934
_reflns_threshold_expression     >2sigma(I)

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

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_computing_molecular_graphics ?
_computing_publication_material ?

_refine_special_details

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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.

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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^)+(0.0100P)^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 0.088(3)
_refine_ls_extinction_expression
'Fc^*=kFc[1+0.001xFc^2^/l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns 2204
_refine_ls_number_parameters 78
_refine_ls_number_restraints 0
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_refine_ls_R_factor_gt 0.0216
_refine_ls_wR_factor_ref 0.0471
_refine_ls_wR_factor_gt 0.0464
_refine_ls_goodness_of_fit_ref 1.497
_refine_ls_restrained_S_all 1.497
_refine_ls_shift/su_max 0.001
_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 0.99172(3) 0.10625(2) 0.336071(17) 0.00500(4) Uani 1 1 d . . .

Si Si 0.46932(5) 0.26637(3) 0.08433(2) 0.00345(5) Uani 1 1 d . . .
 B B 0.56882(19) 0.41054(11) 0.34091(9) 0.00444(13) Uani 1 1 d . . .
 O1 O 0.24116(13) 0.40145(8) 0.03713(7) 0.00601(10) Uani 1 1 d . . .
 O2 O 0.67052(12) 0.30102(8) 0.45757(6) 0.00523(10) Uani 1 1 d . . .
 O3 O 0.67577(12) 0.33471(8) 0.21049(6) 0.00499(10) Uani 1 1 d . . .
 O4 O 0.31490(12) 0.08820(7) 0.14588(7) 0.00569(10) Uani 1 1 d . . .
 O5 O 0.25916(13) 0.41342(8) 0.33646(7) 0.00596(10) Uani 1 1 d . . .
 H H 0.207(3) 0.4508(19) 0.4027(19) 0.025(4) Uiso 1 1 d . . .

loop_

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 _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 0.00523(7) 0.00553(6) 0.00425(6) -0.00001(5) 0.00057(5) -0.00037(5)
 Si 0.00418(9) 0.00364(9) 0.00253(9) -0.00019(7) 0.00032(7) 0.00025(7)
 B 0.0049(3) 0.0050(3) 0.0034(3) -0.0003(2) 0.0006(3) -0.0001(3)
 O1 0.0062(2) 0.0063(2) 0.0055(2) -0.00022(18) -0.00034(19) 0.00172(18)
 O2 0.0057(2) 0.0062(2) 0.0039(2) 0.00103(18) 0.00078(18) 0.00083(18)
 O3 0.0051(2) 0.0068(2) 0.0031(2) -0.00113(18) 0.00041(18) 0.00035(18)
 O4 0.0061(2) 0.0041(2) 0.0069(3) 0.00082(18) 0.0022(2) 0.00024(17)
 O5 0.0046(2) 0.0083(2) 0.0049(2) -0.00118(19) 0.00048(19) 0.00072(18)

_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_

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 _geom_bond_atom_site_label_2
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 Ca O1 2.2785(6) 2_645 ?
 Ca O4 2.4147(7) 1_655 ?
 Ca O2 2.4462(6) . ?
 Ca O5 2.5234(6) 2_645 ?
 Ca O3 2.6077(6) . ?
 Ca O3 2.6554(6) 2_745 ?
 Ca O5 2.6695(7) 1_655 ?
 Si O1 1.5729(6) . ?
 Si O2 1.6449(7) 4_565 ?
 Si O3 1.6534(6) . ?
 Si O4 1.6573(6) . ?

B O4 1.4685(11) 2_655 ?
 B O3 1.4769(11) . ?
 B O2 1.4811(10) . ?
 B O5 1.4971(11) . ?
 O1 Ca 2.2782(6) 4_465 ?
 O1 Ca 2.2785(6) 2_655 ?
 O2 Si 1.6449(7) 4_566 ?
 O3 Ca 2.6554(6) 2_755 ?
 O4 B 1.4685(11) 2_645 ?
 O4 Ca 2.4147(7) 1_455 ?
 O5 Ca 2.5234(6) 2_655 ?
 O5 Ca 2.6695(7) 1_455 ?

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 _geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
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 _geom_angle_site_symmetry_3
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 O1 Ca O4 107.54(2) 4_666 1_655 ?
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 O1 Ca O2 86.78(2) 4_666 . ?
 O1 Ca O2 80.92(2) 2_645 . ?
 O4 Ca O2 144.11(2) 1_655 . ?
 O1 Ca O5 142.79(2) 4_666 2_645 ?
 O1 Ca O5 73.66(2) 2_645 2_645 ?
 O4 Ca O5 77.18(2) 1_655 2_645 ?
 O2 Ca O5 111.26(2) . 2_645 ?
 O1 Ca O3 135.94(2) 4_666 . ?
 O1 Ca O3 114.48(2) 2_645 . ?
 O4 Ca O3 93.74(2) 1_655 . ?
 O2 Ca O3 56.424(19) . . ?
 O5 Ca O3 78.60(2) 2_645 . ?
 O1 Ca O3 78.74(2) 4_666 2_745 ?
 O1 Ca O3 81.91(2) 2_645 2_745 ?
 O4 Ca O3 55.564(19) 1_655 2_745 ?
 O2 Ca O3 159.53(2) . 2_745 ?
 O5 Ca O3 74.140(19) 2_645 2_745 ?
 O3 Ca O3 142.510(10) . 2_745 ?
 O1 Ca O5 76.49(2) 4_666 1_655 ?
 O1 Ca O5 146.85(2) 2_645 1_655 ?
 O4 Ca O5 74.72(2) 1_655 1_655 ?
 O2 Ca O5 77.14(2) . 1_655 ?
 O5 Ca O5 137.812(14) 2_645 1_655 ?
 O3 Ca O5 72.571(19) . 1_655 ?
 O3 Ca O5 112.791(19) 2_745 1_655 ?
 O1 Si O2 113.86(3) . 4_565 ?
 O1 Si O3 115.33(3) . . ?
 O2 Si O3 106.65(3) 4_565 . ?
 O1 Si O4 108.73(3) . . ?
 O2 Si O4 106.18(3) 4_565 . ?

O3 Si O4 105.39(3) . . ?
 O4 B O3 107.34(7) 2_655 . ?
 O4 B O2 109.00(6) 2_655 . ?
 O3 B O2 108.02(7) . . ?
 O4 B O5 111.79(7) 2_655 . ?
 O3 B O5 109.50(6) . . ?
 O2 B O5 111.05(7) . . ?
 Si O1 Ca 126.68(3) . 4_465 ?
 Si O1 Ca 129.67(4) . 2_655 ?
 Ca O1 Ca 102.25(2) 4_465 2_655 ?
 B O2 Si 122.86(5) . 4_566 ?
 B O2 Ca 100.79(5) . . ?
 Si O2 Ca 122.94(3) 4_566 . ?
 B O3 Si 122.39(5) . . ?
 B O3 Ca 94.11(4) . . ?
 Si O3 Ca 118.84(3) . . ?
 B O3 Ca 93.07(4) . 2_755 ?
 Si O3 Ca 118.97(3) . 2_755 ?
 Ca O3 Ca 104.04(2) . 2_755 ?
 B O4 Si 127.62(6) 2_645 . ?
 B O4 Ca 103.58(5) 2_645 1_455 ?
 Si O4 Ca 121.19(3) . 1_455 ?
 B O5 Ca 120.36(5) . 2_655 ?
 B O5 Ca 118.11(5) . 1_455 ?
 Ca O5 Ca 106.02(2) 2_655 1_455 ?

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