

data\_vmxl\_220k\_up\_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 4.83280(10)

\_cell\_length\_b 7.60780(10)

\_cell\_length\_c 9.63130(10)

\_cell\_angle\_alpha 90.00

\_cell\_angle\_beta 90.173(2)

\_cell\_angle\_gamma 90.00

\_cell\_volume 354.112(9)

\_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      ?
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_exptl_crystal_size_mid         ?
_exptl_crystal_size_min         ?
_exptl_crystal_density_meas     ?
_exptl_crystal_density_diffn    3.001
_exptl_crystal_density_method   'not measured'
_exptl_crystal_F_000            320
_exptl_absorpt_coefficient_mu    1.999
_exptl_absorpt_correction_type  ?
_exptl_absorpt_correction_T_min ?
_exptl_absorpt_correction_T_max ?
_exptl_absorpt_process_details  ?

_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
_diffrn_measurement_device_type ?
_diffrn_measurement_method      ?
_diffrn_detector_area_resol_mean ?
_diffrn_standards_number        ?
_diffrn_standards_interval_count ?
_diffrn_standards_interval_time ?
_diffrn_standards_decay_%       ?
_diffrn_reflns_number           8566
_diffrn_reflns_av_R_equivalents 0.0332
_diffrn_reflns_av_sigmaI/netI   0.0261
_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        40.07
_reflns_number_total             2204
_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)'

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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 0.086(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 0.0213  
\_refine\_ls\_wR\_factor\_ref 0.0472  
\_refine\_ls\_wR\_factor\_gt 0.0465  
\_refine\_ls\_goodness\_of\_fit\_ref 1.526  
\_refine\_ls\_restrained\_S\_all 1.526  
\_refine\_ls\_shift/su\_max 0.001  
\_refine\_ls\_shift/su\_mean 0.000

loop\_

\_atom\_site\_label  
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\_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(3) 0.10622(2) 0.336076(17) 0.00503(4) Uani 1 1 d . . .

Si Si 0.46923(5) 0.26636(3) 0.08431(2) 0.00350(5) Uani 1 1 d . . .  
 B B 0.56861(19) 0.41070(11) 0.34077(10) 0.00437(14) Uani 1 1 d . . .  
 O1 O 0.24094(13) 0.40154(8) 0.03724(7) 0.00609(10) Uani 1 1 d . . .  
 O2 O 0.67052(12) 0.30091(8) 0.45754(6) 0.00530(10) Uani 1 1 d . . .  
 O3 O 0.67570(12) 0.33461(8) 0.21059(6) 0.00513(10) Uani 1 1 d . . .  
 O4 O 0.31487(13) 0.08806(7) 0.14589(7) 0.00558(10) Uani 1 1 d . . .  
 O5 O 0.25888(13) 0.41333(8) 0.33645(7) 0.00602(10) Uani 1 1 d . . .  
 H H 0.213(3) 0.448(2) 0.4034(18) 0.030(5) Uiso 1 1 d . . .

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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 0.00532(7) 0.00561(6) 0.00415(7) -0.00006(5) 0.00047(5) -0.00031(5)  
 Si 0.00430(9) 0.00358(9) 0.00263(9) -0.00023(7) 0.00020(7) 0.00025(7)  
 B 0.0049(3) 0.0051(3) 0.0031(3) -0.0004(2) 0.0006(3) -0.0001(3)  
 O1 0.0065(2) 0.0063(2) 0.0054(2) -0.00026(19) -0.0005(2) 0.00191(18)  
 O2 0.0058(2) 0.0062(2) 0.0039(2) 0.00123(18) 0.00069(19) 0.00073(18)  
 O3 0.0051(2) 0.0069(2) 0.0034(2) -0.00095(18) 0.00037(18) 0.00019(18)  
 O4 0.0063(2) 0.0041(2) 0.0063(3) 0.00072(18) 0.00191(19) 0.00007(17)  
 O5 0.0044(2) 0.0086(2) 0.0050(3) -0.00111(19) 0.00061(19) 0.00078(19)

\_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.  
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loop\_  
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 \_geom\_bond\_site\_symmetry\_2  
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 Ca O1 2.2771(6) 2\_645 ?  
 Ca O1 2.2791(6) 4\_666 ?  
 Ca O4 2.4148(7) 1\_655 ?  
 Ca O2 2.4458(6) . ?  
 Ca O5 2.5236(7) 2\_645 ?  
 Ca O3 2.6077(6) . ?  
 Ca O3 2.6572(6) 2\_745 ?  
 Ca O5 2.6695(7) 1\_655 ?  
 Si O1 1.5740(6) . ?  
 Si O2 1.6451(7) 4\_565 ?  
 Si O3 1.6544(6) . ?  
 Si O4 1.6586(6) . ?

B O4 1.4676(11) 2\_655 ?  
B O3 1.4761(11) . ?  
B O2 1.4838(11) . ?  
B O5 1.4974(11) . ?  
O1 Ca 2.2771(6) 2\_655 ?  
O1 Ca 2.2791(6) 4\_465 ?  
O2 Si 1.6451(7) 4\_566 ?  
O3 Ca 2.6572(6) 2\_755 ?  
O4 B 1.4676(11) 2\_645 ?  
O4 Ca 2.4148(7) 1\_455 ?  
O5 Ca 2.5236(7) 2\_655 ?  
O5 Ca 2.6695(7) 1\_455 ?

loop\_

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O1 Ca O1 77.72(2) 2\_645 4\_666 ?  
O1 Ca O4 133.54(2) 2\_645 1\_655 ?  
O1 Ca O4 107.57(2) 4\_666 1\_655 ?  
O1 Ca O2 80.96(2) 2\_645 . ?  
O1 Ca O2 86.77(2) 4\_666 . ?  
O4 Ca O2 144.13(2) 1\_655 . ?  
O1 Ca O5 73.65(2) 2\_645 2\_645 ?  
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O4 Ca O5 77.14(2) 1\_655 2\_645 ?  
O2 Ca O5 111.29(2) . 2\_645 ?  
O1 Ca O3 114.50(2) 2\_645 . ?  
O1 Ca O3 135.93(2) 4\_666 . ?  
O4 Ca O3 93.77(2) 1\_655 . ?  
O2 Ca O3 56.40(2) . . ?  
O5 Ca O3 78.65(2) 2\_645 . ?  
O1 Ca O3 81.88(2) 2\_645 2\_745 ?  
O1 Ca O3 78.76(2) 4\_666 2\_745 ?  
O4 Ca O3 55.539(19) 1\_655 2\_745 ?  
O2 Ca O3 159.55(2) . 2\_745 ?  
O5 Ca O3 74.08(2) 2\_645 2\_745 ?  
O3 Ca O3 142.511(10) . 2\_745 ?  
O1 Ca O5 146.86(2) 2\_645 1\_655 ?  
O1 Ca O5 76.53(2) 4\_666 1\_655 ?  
O4 Ca O5 74.76(2) 1\_655 1\_655 ?  
O2 Ca O5 77.12(2) . 1\_655 ?  
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O3 Ca O5 112.83(2) 2\_745 1\_655 ?  
O1 Si O2 113.89(3) . 4\_565 ?  
O1 Si O3 115.30(3) . . ?  
O2 Si O3 106.65(3) 4\_565 . ?  
O1 Si O4 108.77(3) . . ?  
O2 Si O4 106.18(3) 4\_565 . ?

O3 Si O4 105.35(3) . . ?  
 O4 B O3 107.43(7) 2\_655 . ?  
 O4 B O2 108.96(6) 2\_655 . ?  
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 O4 B O5 111.93(7) 2\_655 . ?  
 O3 B O5 109.55(6) . . ?  
 O2 B O5 110.98(7) . . ?  
 Si O1 Ca 129.73(4) . 2\_655 ?  
 Si O1 Ca 126.60(3) . 4\_465 ?  
 Ca O1 Ca 102.28(2) 2\_655 4\_465 ?  
 B O2 Si 122.82(5) . 4\_566 ?  
 B O2 Ca 100.84(5) . . ?  
 Si O2 Ca 122.97(3) 4\_566 . ?  
 B O3 Si 122.36(5) . . ?  
 B O3 Ca 94.24(4) . . ?  
 Si O3 Ca 118.86(3) . . ?  
 B O3 Ca 93.01(4) . 2\_755 ?  
 Si O3 Ca 118.92(3) . 2\_755 ?  
 Ca O3 Ca 104.05(2) . 2\_755 ?  
 B O4 Si 127.62(6) 2\_645 . ?  
 B O4 Ca 103.60(5) 2\_645 1\_455 ?  
 Si O4 Ca 121.14(3) . 1\_455 ?  
 B O5 Ca 120.24(5) . 2\_655 ?  
 B O5 Ca 118.16(5) . 1\_455 ?  
 Ca O5 Ca 106.08(2) 2\_655 1\_455 ?

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