

data\_vmxl\_160k\_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 4.83110(10)

\_cell\_length\_b 7.60000(10)

\_cell\_length\_c 9.6311(2)

\_cell\_angle\_alpha 90.00

\_cell\_angle\_beta 90.169(2)

\_cell\_angle\_gamma 90.00

\_cell\_volume 353.617(11)

\_cell\_formula\_units\_Z 4

\_cell\_measurement\_temperature 433(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_min         ?
_exptl_crystal_density_meas     ?
_exptl_crystal_density_diffn    3.005
_exptl_crystal_density_method   'not measured'
_exptl_crystal_F_000            320
_exptl_absorpt_coefficient_mu    2.002
_exptl_absorpt_correction_type  ?
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_exptl_absorpt_process_details  ?

_exptl_special_details
;
?
;

_diffn_ambient_temperature      160(2)
_diffn_radiation_wavelength     0.71073
_diffn_radiation_type           MoK\alpha
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_diffn_radiation_monochromator  graphite
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_diffn_reflns_av_sigmaI/netI    0.0264
_diffn_reflns_limit_h_min       -7
_diffn_reflns_limit_h_max       8
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_diffn_reflns_limit_k_max       13
_diffn_reflns_limit_l_min       -16
_diffn_reflns_limit_l_max       17
_diffn_reflns_theta_min         4.22
_diffn_reflns_theta_max         40.10
_reflns_number_total             2203
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_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  
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'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 0.071(3)  
\_refine\_ls\_extinction\_expression  
'Fc^\*=kFc[1+0.001xFc^2\l^3/sin(2\q)]^-1/4^'  
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\_refine\_ls\_number\_parameters 78  
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\_refine\_ls\_R\_factor\_gt 0.0211  
\_refine\_ls\_wR\_factor\_ref 0.0467  
\_refine\_ls\_wR\_factor\_gt 0.0461  
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\_refine\_ls\_restrained\_S\_all 1.502  
\_refine\_ls\_shift/su\_max 0.001  
\_refine\_ls\_shift/su\_mean 0.000

loop\_

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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 0.99182(3) 0.10627(2) 0.336024(17) 0.00401(4) Uani 1 1 d . . .

Si Si 0.46951(5) 0.26650(3) 0.08425(2) 0.00292(5) Uani 1 1 d . . .  
 B B 0.56935(19) 0.41065(11) 0.34084(10) 0.00381(14) Uani 1 1 d . . .  
 O1 O 0.24099(13) 0.40164(7) 0.03714(7) 0.00505(10) Uani 1 1 d . . .  
 O2 O 0.67112(12) 0.30113(8) 0.45763(6) 0.00439(10) Uani 1 1 d . . .  
 O3 O 0.67624(12) 0.33458(8) 0.21051(6) 0.00421(10) Uani 1 1 d . . .  
 O4 O 0.31469(13) 0.08831(7) 0.14590(7) 0.00488(10) Uani 1 1 d . . .  
 O5 O 0.25885(13) 0.41339(8) 0.33644(7) 0.00493(10) Uani 1 1 d . . .  
 H H 0.207(3) 0.4462(18) 0.4126(17) 0.021(4) Uiso 1 1 d . . .

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 \_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  
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 Si 0.00348(9) 0.00309(9) 0.00217(9) -0.00008(7) 0.00021(7) 0.00020(7)  
 B 0.0039(3) 0.0042(3) 0.0033(3) 0.0000(2) 0.0001(3) 0.0000(2)  
 O1 0.0052(2) 0.0051(2) 0.0049(2) -0.00029(18) -0.00048(19) 0.00142(18)  
 O2 0.0049(2) 0.0052(2) 0.0031(2) 0.00105(18) 0.00061(18) 0.00051(18)  
 O3 0.0044(2) 0.0057(2) 0.0025(2) -0.00091(18) 0.00024(18) -0.00002(18)  
 O4 0.0053(2) 0.0036(2) 0.0057(2) 0.00064(18) 0.00162(19) 0.00017(17)  
 O5 0.0037(2) 0.0069(2) 0.0042(2) -0.00099(19) 0.00068(19) 0.00062(18)

\_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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 Ca O1 2.2768(6) 2\_645 ?  
 Ca O1 2.2783(6) 4\_666 ?  
 Ca O4 2.4128(7) 1\_655 ?  
 Ca O2 2.4446(6) . ?  
 Ca O5 2.5224(6) 2\_645 ?  
 Ca O3 2.6050(6) . ?  
 Ca O3 2.6534(6) 2\_745 ?  
 Ca O5 2.6669(6) 1\_655 ?  
 Si O1 1.5737(6) . ?  
 Si O2 1.6453(7) 4\_565 ?  
 Si O3 1.6542(6) . ?  
 Si O4 1.6578(6) . ?

B O4 1.4673(10) 2\_655 ?  
 B O3 1.4766(11) . ?  
 B O2 1.4821(11) . ?  
 B O5 1.5007(11) . ?  
 O1 Ca 2.2768(6) 2\_655 ?  
 O1 Ca 2.2783(6) 4\_465 ?  
 O2 Si 1.6453(7) 4\_566 ?  
 O3 Ca 2.6534(6) 2\_755 ?  
 O4 B 1.4673(10) 2\_645 ?  
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 O5 Ca 2.5224(6) 2\_655 ?  
 O5 Ca 2.6669(6) 1\_455 ?

loop\_  
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 O1 Ca O4 133.63(2) 2\_645 1\_655 ?  
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 O1 Ca O2 80.91(2) 2\_645 . ?  
 O1 Ca O2 86.71(2) 4\_666 . ?  
 O4 Ca O2 144.09(2) 1\_655 . ?  
 O1 Ca O5 73.70(2) 2\_645 2\_645 ?  
 O1 Ca O5 142.74(2) 4\_666 2\_645 ?  
 O4 Ca O5 77.15(2) 1\_655 2\_645 ?  
 O2 Ca O5 111.33(2) . 2\_645 ?  
 O1 Ca O3 114.49(2) 2\_645 . ?  
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 O4 Ca O3 93.69(2) 1\_655 . ?  
 O2 Ca O3 56.49(2) . . ?  
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 O2 Ca O3 159.48(2) . 2\_745 ?  
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 O1 Ca O5 146.80(2) 2\_645 1\_655 ?  
 O1 Ca O5 76.55(2) 4\_666 1\_655 ?  
 O4 Ca O5 74.74(2) 1\_655 1\_655 ?  
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 O5 Ca O5 137.810(14) 2\_645 1\_655 ?  
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 O3 Ca O5 112.877(19) 2\_745 1\_655 ?  
 O1 Si O2 113.98(3) . 4\_565 ?  
 O1 Si O3 115.42(3) . . ?  
 O2 Si O3 106.56(3) 4\_565 . ?  
 O1 Si O4 108.63(3) . . ?  
 O2 Si O4 106.20(3) 4\_565 . ?

O3 Si O4 105.32(3) . . ?  
 O4 B O3 107.45(7) 2\_655 . ?  
 O4 B O2 108.97(6) 2\_655 . ?  
 O3 B O2 108.01(7) . . ?  
 O4 B O5 111.78(6) 2\_655 . ?  
 O3 B O5 109.48(6) . . ?  
 O2 B O5 111.01(7) . . ?  
 Si O1 Ca 129.67(4) . 2\_655 ?  
 Si O1 Ca 126.61(3) . 4\_465 ?  
 Ca O1 Ca 102.33(2) 2\_655 4\_465 ?  
 B O2 Si 122.83(5) . 4\_566 ?  
 B O2 Ca 100.71(5) . . ?  
 Si O2 Ca 122.91(3) 4\_566 . ?  
 B O3 Si 122.37(5) . . ?  
 B O3 Ca 94.13(4) . . ?  
 Si O3 Ca 118.95(3) . . ?  
 B O3 Ca 92.98(4) . 2\_755 ?  
 Si O3 Ca 118.89(3) . 2\_755 ?  
 Ca O3 Ca 104.10(2) . 2\_755 ?  
 B O4 Si 127.61(6) 2\_645 . ?  
 B O4 Ca 103.50(5) 2\_645 1\_455 ?  
 Si O4 Ca 121.26(3) . 1\_455 ?  
 B O5 Ca 120.30(5) . 2\_655 ?  
 B O5 Ca 118.13(5) . 1\_455 ?  
 Ca O5 Ca 106.04(2) 2\_655 1\_455 ?

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