

```

data_p10_totale
_publ_requested_journal      Am.Miner.
_publ_section_title 'High-pressure phase transition of a natural pigeonite'
_publ_contact_author_name    'MATTEO ALVARO'
_publ_contact_author_address
;
'Dipartimento di Scienze della Terra,
Universit\`a di Pavia, Via Ferrata 1, I-27100 Pavia (Italy)'
;
loop_
_publ_author_name
_publ_author_address
'ALVARO, MATTEO'
;Dipartimento di Scienze della Terra,
Universit\`a di Pavia, Via Ferrata 1, I-27100 Pavia (Italy)'
;
'Nestola, Fabrizio'
;Dipartimento di Geoscienze, Universit\`a di Padova,
Via Giotto 1, I-35137 Padova (Italy)'
;
'BOFFA BALLARAN, TIZIANA'
;Bayerisches Geoinstitut, Universit\`at Bayreuth,
Universit\`atstrasse 37, D-95440 Bayreuth (Germany)'
;
'C\`AMARA, FERNANDO'
;C.N.R. - Istituto di Geoscienze e Georisorse - Unit\`a di Pavia,
Via Ferrata 1, I-27100 Pavia (Italy)'
;
'DOMENEGHETTI, CHIARA'
;Dipartimento di Scienze della Terra, Universit\`a di Pavia,
Via Ferrata 1, I-27100 Pavia (Italy)'
;
'TAZZOLI, VITTORIO'
;Dipartimento di Scienze della Terra, Universit\`a di Pavia,
Via Ferrata 1, I-27100 Pavia (Italy)'
;
_audit_creation_method      SHELXL-97
_chemical_name_systematic    Pigeonite
_chemical_name_common        ?
_chemical_melting_point      ?
_chemical_formula_sum         'Ca0.48 Fe4 Mg3.48 O24 Si8'
_chemical_formula_weight      935.96

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scatter_dispersion_real
_atom_type_scatter_dispersion_imag
_atom_type_scatter_source
O O 0.0106 0.0060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
O O-- 0.0080 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'
Si SiP4 0.0720 0.0710 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

```

Fe FeP2 0.3010 0.8450 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Mg MgP2 0.0420 0.0360 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Ca CaP2 0.2030 0.3060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting monoclinic
_symmetry_space_group_name_H-M 'C 2/c'
_symmetry_int_tables_number 15

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

_cell_length_a 9.402(6)
_cell_length_b 8.716(6)
_cell_length_c 4.996(3)
_cell_angle_alpha 90.00
_cell_angle_beta 103.28(6)
_cell_angle_gamma 90.00
_cell_volume 398.5(4)
_cell_formula_units_Z 4
_cell_measurement_temperature 293(2)
_cell_measurement_reflns_used ?
_cell_measurement_theta_min ?
_cell_measurement_theta_max ?

_exptl_crystal_description Irregular
_exptl_crystal_colour Green
_exptl_crystal_size_max 0.120
_exptl_crystal_size_mid 0.110
_exptl_crystal_size_min 0.100
_exptl_crystal_density_meas ?
_exptl_crystal_density_diffn 3.585
_exptl_crystal_density_method 'not measured'
_exptl_crystal_F_000 460.0
_exptl_absorpt_coefficient_mu 4.248
_exptl_absorpt_correction_type numerical
_exptl_absorpt_correction_T_min ?
_exptl_absorpt_correction_T_max ?
_exptl_absorpt_process_details ABSORB

_exptl_special_details
;
Angel, R.J. (2004) Absorption corrections for diamond-anvil pressure cells
implemented in the software package -- Absorb 6.0. Journal of Applied
Crystallography, 37, 486-492.
;

```

_diffrn_ambient_temperature    293(2)
_diffrn_radiation_probe        x-ray
_diffrn_radiation_type          MoK\alpha
_diffrn_radiation_wavelength    0.71073
_diffrn_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           459
_diffrn_reflns_av_R_equivalents 0.0000
_diffrn_reflns_av_sigmaI/netI   0.0489
_diffrn_reflns_limit_h_min      -11
_diffrn_reflns_limit_h_max      10
_diffrn_reflns_limit_k_min      0
_diffrn_reflns_limit_k_max      10
_diffrn_reflns_limit_l_min      0
_diffrn_reflns_limit_l_max      6
_diffrn_reflns_theta_min        3.23
_diffrn_reflns_theta_max        29.67
_reflns_number_total            459
_reflns_number_gt               355
_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/[ $s^2(F_o^2)+(0.1334P)^2+0.2187P$ ] where  $P=(F_o^2+2F_c^2)/3$ '
_atom_sites_solution_primary     direct

```

```

_atom_sites_solution_secondary difmap
_refine_ls_extinction_method SHELXL
_refine_ls_extinction_coef 0.006(5)
_refine_ls_extinction_expression  $F_c^{*^2} = k F_c [1 + 0.001 x F_c^{*^2} | \sin(2\theta) |^3 / \sin(2\theta)]^{-1/4}$ 
_refine_ls_number_reflns 459
_refine_ls_number_parameters 34
_refine_ls_number_restraints 0
_refine_ls_R_factor_all 0.1020
_refine_ls_R_factor_gt 0.0764
_refine_ls_wR_factor_ref 0.2186
_refine_ls_wR_factor_gt 0.1971
_refine_ls_goodness_of_fit_ref 1.183
_refine_ls_restrained_S_all 1.183
_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
O1 O 0.1218(9) 0.0889(6) 0.1417(12) 0.0085(13) Uiso 1 1 d ...
O2 O 0.3739(8) 0.2411(7) 0.3601(12) 0.0128(14) Uiso 1 1 d ...
O3 O 0.3517(8) 0.0523(7) 0.9359(11) 0.0148(15) Uiso 1 1 d ...
Si Si 0.2959(3) 0.0911(2) 0.2203(4) 0.0090(8) Uiso 1 1 d ...
Fe1A FeP2 0.0000 0.9077(3) 0.2500 0.0113(9) Uani 0.25 2 d SP ..
FE1B MgP2 0.0000 0.9077(3) 0.2500 0.0113(9) Uani 0.75 2 d SP ..
Fe2A FeP2 0.0000 0.2797(2) 0.2500 0.0148(8) Uani 0.75 2 d SP ..
FE2B MgP2 0.0000 0.2797(2) 0.2500 0.0148(8) Uani 0.12 2 d SP ..
FE2C CaP2 0.0000 0.2797(2) 0.2500 0.0148(8) Uani 0.12 2 d SP ..

```

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
Fe1A 0.020(3) 0.0040(18) 0.0103(13) 0.000 0.0049(14) 0.000
FE1B 0.020(3) 0.0040(18) 0.0103(13) 0.000 0.0049(14) 0.000
Fe2A 0.0230(19) 0.0125(14) 0.0084(10) 0.000 0.0023(10) 0.000
FE2B 0.0230(19) 0.0125(14) 0.0084(10) 0.000 0.0023(10) 0.000
FE2C 0.0230(19) 0.0125(14) 0.0084(10) 0.000 0.0023(10) 0.000

```

_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

O1 Si 1.593(9) . ?

O1 Fe1A 2.028(7) 5_565 ?

O1 Fe1A 2.093(6) 1_545 ?

O1 Fe2A 2.157(6) . ?

O2 Si 1.581(7) . ?

O2 Fe1A 2.029(7) 3_545 ?

O2 FE2C 2.043(7) 7_556 ?

O2 Fe2A 2.043(7) 7_556 ?

O3 Si 1.655(6) 6_556 ?

O3 Si 1.659(6) 1_556 ?

O3 Fe2A 2.355(6) 7_556 ?

O3 O3 2.659(4) 6 ?

O3 O3 2.659(4) 6_556 ?

Si O3 1.655(6) 6 ?

Si O3 1.659(6) 1_554 ?

Fe1A O1 2.028(7) 6_566 ?

Fe1A O1 2.028(7) 5_565 ?

Fe1A O2 2.029(7) 3_455 ?

Fe1A O2 2.029(7) 4 ?

Fe1A O1 2.093(6) 1_565 ?

Fe1A O1 2.093(6) 2_565 ?

Fe1A Fe1A 2.971(3) 5_576 ?

Fe1A Fe1A 2.971(3) 5_575 ?

Fe2A O2 2.043(7) 8_455 ?

Fe2A O2 2.043(7) 7_556 ?

Fe2A O1 2.157(6) 2 ?

Fe2A O3 2.355(6) 7_556 ?

Fe2A O3 2.355(6) 8_455 ?

Fe2A O3 3.008(7) 4_556 ?

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
 Si O1 Fe1A 123.9(3) . 5_565 ?
 Si O1 Fe1A 122.5(3) . 1_545 ?
 Fe1A O1 Fe1A 92.2(3) 5_565 1_545 ?
 Si O1 Fe2A 120.2(3) . . ?
 Fe1A O1 Fe2A 90.9(3) 5_565 . ?
 Fe1A O1 Fe2A 99.4(3) 1_545 . ?
 Si O2 Fe1A 135.5(3) . 3_545 ?
 Si O2 FE2C 117.1(4) . 7_556 ?
 Fe1A O2 FE2C 94.3(3) 3_545 7_556 ?
 Si O2 Fe2A 117.1(4) . 7_556 ?
 Fe1A O2 Fe2A 94.3(3) 3_545 7_556 ?
 Si O3 Si 126.6(4) 6_556 1_556 ?
 Si O3 Fe2A 109.9(3) 6_556 7_556 ?
 Si O3 Fe2A 122.9(3) 1_556 7_556 ?
 Si O3 O3 160.5(3) 1_556 6 ?
 Fe2A O3 O3 73.44(17) 7_556 6 ?
 Si O3 O3 106.5(4) 6_556 6_556 ?
 Fe2A O3 O3 135.4(2) 7_556 6_556 ?
 O3 O3 O3 139.9(5) 6 6_556 ?
 O2 Si O1 117.7(4) . . ?
 O2 Si O3 107.0(4) . 6 ?
 O1 Si O3 107.8(4) . 6 ?
 O2 Si O3 109.9(3) . 1_554 ?
 O1 Si O3 107.2(3) . 1_554 ?
 O3 Si O3 106.7(3) 6 1_554 ?
 O1 Fe1A O1 178.3(3) 6_566 5_565 ?
 O1 Fe1A O2 89.1(3) 6_566 3_455 ?
 O1 Fe1A O2 92.1(3) 5_565 3_455 ?
 O1 Fe1A O2 92.1(3) 6_566 4 ?
 O1 Fe1A O2 89.1(3) 5_565 4 ?
 O2 Fe1A O2 88.6(4) 3_455 4 ?
 O1 Fe1A O1 91.0(3) 6_566 1_565 ?
 O1 Fe1A O1 87.8(3) 5_565 1_565 ?
 O2 Fe1A O1 176.7(3) 3_455 1_565 ?
 O2 Fe1A O1 94.7(3) 4 1_565 ?
 O1 Fe1A O1 87.8(3) 6_566 2_565 ?
 O1 Fe1A O1 91.0(3) 5_565 2_565 ?
 O2 Fe1A O1 94.7(3) 3_455 2_565 ?
 O2 Fe1A O1 176.7(3) 4 2_565 ?
 O1 Fe1A O1 82.1(4) 1_565 2_565 ?
 O1 Fe1A Fe1A 44.74(17) 6_566 5_576 ?
 O1 Fe1A Fe1A 134.0(2) 5_565 5_576 ?
 O2 Fe1A Fe1A 92.67(17) 3_455 5_576 ?
 O2 Fe1A Fe1A 136.7(2) 4 5_576 ?
 O1 Fe1A Fe1A 85.10(19) 1_565 5_576 ?
 O1 Fe1A Fe1A 43.0(2) 2_565 5_576 ?
 O1 Fe1A Fe1A 134.0(2) 6_566 5_575 ?
 O1 Fe1A Fe1A 44.74(17) 5_565 5_575 ?
 O2 Fe1A Fe1A 136.7(2) 3_455 5_575 ?
 O2 Fe1A Fe1A 92.67(17) 4 5_575 ?
 O1 Fe1A Fe1A 43.0(2) 1_565 5_575 ?
 O1 Fe1A Fe1A 85.10(19) 2_565 5_575 ?
 Fe1A Fe1A Fe1A 114.46(18) 5_576 5_575 ?

O2 Fe2A O2 169.8(4) 8_455 7_556 ?
 O2 Fe2A O1 86.9(3) 8_455 2 ?
 O2 Fe2A O1 85.3(3) 7_556 2 ?
 O2 Fe2A O1 85.3(3) 8_455 . ?
 O2 Fe2A O1 86.9(3) 7_556 . ?
 O1 Fe2A O1 79.1(3) 2 . ?
 O2 Fe2A O3 86.9(3) 8_455 7_556 ?
 O2 Fe2A O3 99.5(3) 7_556 7_556 ?
 O1 Fe2A O3 167.1(2) 2 7_556 ?
 O1 Fe2A O3 89.1(2) . 7_556 ?
 O2 Fe2A O3 99.5(3) 8_455 8_455 ?
 O2 Fe2A O3 86.9(3) 7_556 8_455 ?
 O1 Fe2A O3 89.1(2) 2 8_455 ?
 O1 Fe2A O3 167.1(2) . 8_455 ?
 O3 Fe2A O3 103.1(3) 7_556 8_455 ?
 O2 Fe2A O3 131.7(2) 8_455 4_556 ?
 O2 Fe2A O3 58.3(2) 7_556 4_556 ?
 O1 Fe2A O3 133.42(19) 2 4_556 ?
 O1 Fe2A O3 122.1(3) . 4_556 ?
 O3 Fe2A O3 57.94(11) 7_556 4_556 ?
 O3 Fe2A O3 63.2(3) 8_455 4_556 ?

_diffn_measured_fraction_theta_max 0.658
 _diffn_reflns_theta_full 29.67
 _diffn_measured_fraction_theta_full 0.658
 _refine_diff_density_max 1.395
 _refine_diff_density_min -1.090
 _refine_diff_density_rms 0.279
 _diffn_ambient_pressure 7762000
 _chemical_formula_moiety 'Ca0.48 Fe4 Mg3.48 O24 Si8'