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High-pressure behavior of zoisite

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Abstract

A high-pressure single-crystal X-ray diffraction (XRD) study has been carried out on two natural zoisite samples $Ca_2Al_{3-x}Fe_xSi_3O_{12}OH$, one Fe-free ($x = 0$) and one Fe-rich ($x = 0.12$). The unit-cell parameters were determined for the Fe-free sample at 18 different pressures up to 7.76 GPa and for the Fe-rich sample at 13 different pressures up to 7.63 GPa. The $P(V)$ data for both of the samples were fitted by a third-order Birch-Murnaghan equation of state (BM3 EoS). The equation of state coefficients are: $V_0 = 903.39(5)$ Å^3, $K_{T0} = 122.1(7)$ GPa, and $K_0' = 6.8(2)$ for the Fe-free sample and $V_0 = 906.95(5)$ Å^3, $K_{T0} = 119.1(7)$ GPa, and $K_0' = 7.3(2)$ for the Fe-rich sample. This shows that the addition of Fe in to the crystal structure of zoisite leads to a slight softening of the structure.

Both compositions exhibit axial compressibilities $\beta_c > \beta_a >> \beta_b$, with the compressibilities of the *a* and *b* axes of the two samples being indistinguishable. The softening of the bulk modulus of zoisite with Fe content follows from softening of the *c*-axis of the structure. A high-pressure structural study of the Fe-free sample showed that the main compression mechanisms in the structure are the compression of soft inter-octahedral distance along [001] and soft intra-octahedral distances along [010] directions, while along [100] the main compression occurs because of the compression of stiff intra-octahedral distances. The substitution of Fe on to the M3 octahedral site of the structure leads to an increase of the intra-octahedral distance of the M3 that triggers the rotation of M12 and therefore leads to the softening of the M12 inter-octahedral distances that accounts for the softening of the *c*-axis of the structure.

Keywords: Zoisite, high pressure, X-ray diffraction, compressibility, OH, water, hydrous minerals

Introduction

Several authors (e.g., Forneris and Holloway 2003; Kirby et al. 1996) have argued that the correct model for explaining the generation of much volcanism and many intermediate earthquakes in subduction zones involves the progressive dehydration of the subducting slab through a series of reactions that release H_2O into the mantle wedge. These fluids released from subducting slabs could trigger hydration-driven partial melting reactions, inducing partial melting of the mantle wedge above the slab, which in turn is considered responsible for the intermediate earthquakes (e.g., Peacock 2001; Kirby et al. 1996), as well as for arc volcanism (e.g., Forneris and Holloway 2003) in subduction zones. A key role in these dehydration reactions is played by the hydrous mineral phases that are mainly contained in the subducting slab. Therefore a detailed study of the properties of the hydrous phases, and especially their thermo-elastic behavior, will be an important requirement for understanding properties and processes within subduction zones, in particular for constraining their stability and the related dehydration reactions in which they are involved as a function of pressure and temperature (e.g., Hacker et al. 2003; Mao et al. 2007). Minerals of the epidote group, for example, participate in numerous relevant phase equilibria, which need to be accurately evaluated to understand these geological processes. Zoisite belongs to the epidote group and occurs in high- and ultrahigh-pressure metamorphic rocks from a wide variety of geological settings, including continental collisions and subduction zones (e.g., Hacker et al. 2003; Mao et al. 2007; Enami et al. 2004).

Zoisite is the only member of the epidote group that is orthorhombic, instead of monoclinic. The structure of zoisite (Fig. 1) was first determined by Fesenko et al. (1955, 1956), and was later refined by Dollase (1968). Monoclinic epidotes, including clinozoisite, have two distinct edge-sharing octahedral chains that run along [010]. Zoisite has only one type of octahedral chain parallel to [010] built up of edge-sharing octahedra designated M12 and occupied by Al^{3+} (Ghose and Tsang 1971). That chain of octahedra is decorated by a second octahedral site, M3, which can be occupied by both Al^{3+} and Fe^{3+} (Ghose and Tsang 1971). This site is more distorted and is attached to M12 octahedra by edge sharing. The octahedral chains are linked by isolated SiO_4 tetrahedra (T3) in the **c** direction and by Si_2O_7 groups (T1 and T2) in the **a** and **c** directions (see Fig. 1). In this framework of interconnected octahedral chains and bridging tetrahedra, there are two distinct, irregularly shaped, sevenfold-coordinated cavities (Ca1 and Ca2) occupied by Ca. Hydrogen is bonded to oxygen O10, which coordinates cations in the octahedral chains (Franz and Liebscher 2004).

Several authors have measured the equation of state of zoisite

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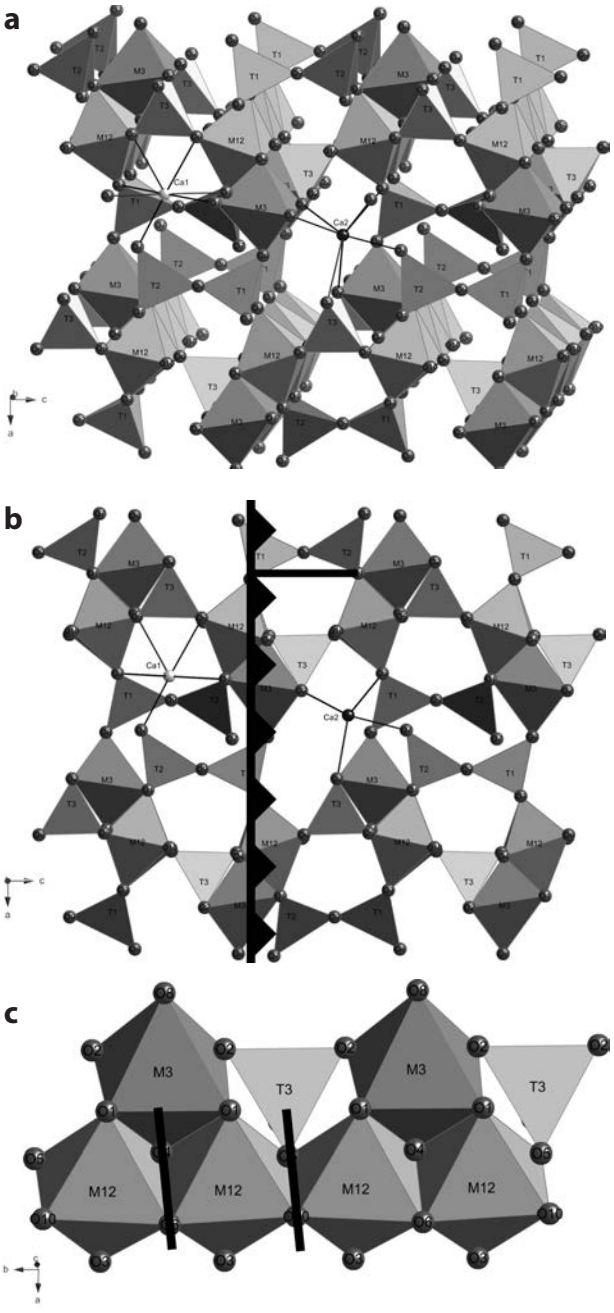

FIGURE 1. Schematic view of the structure along different crystallographic directions: (**a**) along **b** lattice tilted to show the direction of the octahedral chain going along **b** direction.; (**b**) along **b** lattice. Superimposed are a 2_1 screw axis running along [100] and a vector joining a pair of M12 sites related by a 2_1 screw axis running along [010]; (**c**) inclined from the **c** lattice direction showing the mirror plane (black line) relating the M12 sites.

by means of X-ray diffraction on samples with different composition and have obtained several different values for the bulk moduli; 125.1(2.1) GPa was measured by Grevel et al. (2000) on a synthetic zoisite sample with composition $Ca_2Al_3Si_3O_{12}OH$; 127(4) GPa was measured by Pawley et al. (1998) on a natural zoisite sample containing 2.3–4.3% of ferric end-member; and 102(7) GPa was obtained by Comodi and Zanazzi (1997) on a zoisite sample with composition $Ca_2Al_2(Al_{0.9}Fe_{0.1})Si_3O_{12}OH$. Recently Mao et al. (2007) measured the elastic constants at ambient conditions by Brillouin scattering on a Fe-free crystal of zoisite from Merelani Hills, to obtain a value of K_{S0} = 125.3(4) GPa, which, however, is not in agreement with the compression studies, as we discuss below.

To clarify the discrepancies among the literature data, and to determine the effect of the substitution of Fe on the bulk modulus, we investigated the high-pressure behavior of two samples with different Fe contents, Fe-free (X_{ps} = 0) and Fe-rich (X_{ps} = 0.12) up to 7.76 and 7.63 GPa, respectively, by means of single-crystal X-ray diffraction. We determined the unit-cell parameter evolution on both samples to calculate the equation of state and we measured intensity data on the Fe-free sample to follow the evolution of the structure with pressure.

EXPERIMENTAL METHODS

Samples

Two single crystals of natural zoisite with general formula $Ca_2(Al,Fe)_3Si_3O_{12}(OH)$, and different Fe contents were selected. The Fe-free (X_{ps} = 0) sample is from the Merelani Hills in the Arusha Region, United Republic of Tanzania (Barot and Boehm 1992), and was kindly provided by A. Zanetti. The Fe-rich sample (X_{ps} = 0.12) is from Carinthia, Austria, (sample number 8952 from the Museo di Mineralogia, Dipartimento di Scienze della Terra, Pavia, Italy).

Electron microprobe analysis

Chemical analyses were performed on different crystals from the same samples that X-ray data collection and structure refinement at ambient conditions showed had very similar structural parameters. Analyses of the Fe-free sample (X_{ps} = 0) sample are from Cámara et al. (2012). A Cameca-Camebax electron microprobe with a fine-focused beam (1 µm diameter) operating in the wavelength-dispersive (WDS) mode was used. Operating conditions were 20 kV accelerating voltage and 20 nA beam current for for Al, Si, K, Ca, and Fe and 20 kV and 40 nA for Na, Mg, Ti, V, Mn, Sr, La, and Ce, with a beam diameter of 20 µm. The following standards were used (spectral line, analyzing crystal): Amelia albite (Na*K*α, TAP), wollastonite (Ca*K*α, PET), diopside (Si*K*α, TAP), MgO (Mg*K*α, TAP), orthoclase (K*K*α, PET), $MnTiO_3$ (Mn*K*α and Ti*K*α, PET), vanadinite (V*K*α, LIF), Fe_2O_3 (Fe*K*α, LiF), corundum (Al*K*α, TAP), celestine (Sr*L*α, PET), and REE glass 3 (La*L*α and Ce*L*α, LIF). WDS scans showed the absence of fluorine. Data were corrected using the PAP method of Pouchou and Pichoir (1984). The empirical formulas of our zoisite samples are $(Ca_{1.99}Sr_{0.01})(Al_{2.99}V_{0.01})[Si_2O_7][SiO_4]O(OH)$ and $(Ca_{1.99}Sr_{0.01})(Al_{2.88}Fe_{0.12})[Si_2O_7][SiO_4]O(OH)$, with the OH content being assumed by stoichiometry as the crystals are fluorine free (Table 1).

High-pressure experiments

Two crystals, one Fe-free (size 0.150 × 0.100 × 0.080 mm) and one Fe-rich (size 0.110 × 0.090 × 0.060 mm) were selected for the high-pressure study on the basis of their size, optical sharp extinction, absence of twinning, and X-ray diffraction profiles. The Fe-free and Fe-rich crystals were loaded in two different ETH-type diamond-anvil cells (DACs, Miletich et al. 2000) using a steel gasket (T301) pre-indented to a thickness of 100 µm and with a hole of 250 µm in diameter for both the experiments. A single crystal of quartz was used as an internal diffraction pressure standard (Angel et al. 1997) and a 4:1 mixture of methanol:ethanol was used as pressure medium, which remains hydrostatic throughout the pressure range investigated in this work (Angel et al. 2007). The unit-cell parameters were determined by single-crystal X-ray diffraction using a Huber four-circle diffractometer (non-monochromatized Mo*K*α radiation) operating at 50 kV and 40 mA, automated by the SINGLE software (Angel and Finger 2011). The unit-cell parameters were measured at 18 different pressures up to ~7.76 GPa for the Fe-free sample and at 13 different pressures up to ~7.63 GPa for the Fe-rich sample by centering not less than 20 reflections in the 2θ range between 10° and 30° for each high-pressure data point. Typical half-widths of

reflections were between 0.05° and 0.08° in ω and no broadening was detected at any pressure. Full details of the instrument and the peak-centering algorithms are provided by Angel et al. (1997). During the centering procedure the effects of crystal offsets and diffractometer aberrations were eliminated from refined peak positions by the eight-position centering method of King and Finger (1979). Unconstrained unit-cell parameters, obtained by vector least-squares (Ralph and Finger 1982), were found to be similar, within one estimated standard deviation, to the symmetry-constrained ones, which are reported in Table 2.

Intensity data for structural refinements were collected simultaneously for the Fe-free crystal on an Xcalibur-1 Oxford Diffraction diffractometer equipped with a point detector (κ-geometry, graphite-monochromatized Mo$K\alpha$ radiation). Integrated intensity data were then corrected for absorption effects due to the crystal and the DAC using the ABSORB computer program (Burnham 1966; Angel 2004).

All of the structure refinements were performed with the SHELX-97 software (Sheldrick 2008), starting from the atomic coordinates of Cámara et al. (2012). Because of the complexity of the structure and the limited number of observations, all of the refinements were performed with isotropic displacement parameters for all atoms. Details of the structure refinement, bond distances and angles are reported in Tables 3 and 4. Atomic coordinates and displacement parameters are listed in Table 5[1] (deposited), and CIFs[1] are deposited.

[1] Deposit item AM-12-041, Table 5 and CIFs. Deposit items are available two ways: For a paper copy contact the Business Office of the Mineralogical Society of America (see inside front cover of recent issue) for price information. For an electronic copy visit the MSA web site at http://www.minsocam.org, go to the *American Mineralogist* Contents, find the table of contents for the specific volume/issue wanted, and then click on the deposit link there.

Table 1. Mean zoisite composition of the Fe-free and Fe-rich samples based on the average of 10 point analyses

Na_2O	0.01(1)	Na	0.001	Na_2O	0.06(12)	Na	0.009
MgO	0.01(1)	Mg	0.001	MgO	0.04(3)	Mg	0.005
Al_2O_3	33.56(15)	$Al^{(VI)}$	2.986	Al_2O_3	31.83(21)	$Al^{(VI)}$	2.876
SiO_2	39.91(18)	Si	3.014	SiO_2	38.96(15)	Si	2.987
K_2O	0.00(1)	K	0.000	K_2O	0.01(1)	K	0.001
CaO	24.25(16)	Ca	1.962	CaO	24.23(15)	Ca	1.990
TiO_2	0.05(1)	Ti	0.003	TiO_2	0.08(1)	Ti	0.005
V_2O_3	0.15(1)	V	0.009	V_2O_3	0.01(2)	V	0.001
Cr_2O_3	0.00(0)	Cr	0.000	Cr_2O_3	0.00(0)	Cr	0.000
MnO	0.01(1)	Mn	0.000	MnO	0.02(1)	Mn	0.005
Fe_2O_3	0.02(2)	Fe^{3+}	0.001	Fe_2O_3	2.06(22)	Fe^{3+}	0.119
SrO	0.17(5)	Sr	0.008	SrO	0.45(4)	Sr	0.020
La_2O_3	0.02(2)	La	0.000	La_2O3	0.02(2)	La	0.000
Ce_2O_3	0.02(3)	Ce	0.001	Ce_2O3	0.03(2)	Ce	0.001
H_2O	1.99(1)	H	1.000	H_2O	1.96(1)	H	1.000
Total	100.16	Total	8.985	Total	99.75	Total	9.015

Notes: Formulas are in atoms per formula unit (apfu) based on 13 anions. Analysis for the Fe-free sample is from Cámara et al. (2012). Calculated assuming 1 apfu of H. Fluorine was below the detection limit.

Results and discussion

Elastic behavior

The evolution of the unit-cell parameters and unit-cell volume of the two crystals measured in this study as a function of pressure is shown in Figure 2, along with the previously published data. All of the unit-cell parameters undergo a strong non-linear

Table 2a. Unit-cell parameters at different pressure values, measured on the Fe-free crystal

P (GPa)	*a* (Å)	*b* (Å)	*c* (Å)	*V* (Å^3)
0.0001	16.2004(5)	5.5529(4)	10.0423(5)	903.39(6)
0.697(5)	16.1869(5)	5.5411(4)	10.0169(6)	898.45(7)
1.144(7)	16.1760(4)	5.5342(4)	10.0007(6)	895.27(6)
1.926(8)	16.1587(4)	5.5215(3)	9.9746(5)	889.93(6)
2.945(7)	16.1353(4)	5.5063(3)	9.9432(5)	883.41(6)
3.426(6)	16.1239(4)	5.5001(3)	9.9288(5)	880.51(6)
4.025(11)	16.1124(5)	5.4917(4)	9.9110(6)	876.96(6)
4.685(12)	16.0988(5)	5.4827(3)	9.8921(6)	873.12(6)
5.533(11)	16.0796(5)	5.4721(4)	9.8690(6)	868.36(6)
6.515(12)	16.0583(4)	5.4602(3)	9.8439(5)	863.12(5)
6.511(8)	16.0540(4)	5.4609(4)	9.8437(6)	863.00(6)
7.766(9)	16.0271(4)	5.4473(3)	9.8119(5)	856.63(6)
5.145(6)*	16.0845(4)	5.4777(3)	9.8785(6)	870.36(6)
4.325(9)*	16.1038(5)	5.4886(3)	9.9011(6)	875.13(6)
4.243(8)*	16.1063(5)	5.4896(4)	9.9026(6)	875.56(7)
4.243(8)*	16.1054(5)	5.4897(3)	9.9020(5)	875.47(6)
4.239(6)*	16.1067(6)	5.4893(5)	9.9036(8)	875.61(9)
2.496(5)*	16.1468(4)	5.5130(3)	9.9560(5)	886.26(5)
1.680(5)*	16.1623(5)	5.5253(4)	9.9833(7)	891.52(7)
0.722(5)*	16.1831(5)	5.5409(4)	10.0150(6)	898.03(7)
0.0001	16.1992(4)	5.5529(4)	10.0420(6)	903.29(7)

Note: Standard deviations are given in parentheses.
* Data measured during decompression.

Table 2b. Unit-cell parameters at different pressure values, measured on the Fe-rich crystal

P (GPa)	*a* (Å)	*b* (Å)	*c* (Å)	*V* (Å^3)
0.0001(0)	16.2095(6)	5.5654(2)	10.0536(2)	906.95(5)
3.377(7)	16.1363(7)	5.5133(2)	9.9353(4)	883.88(6)
4.398(9)	16.1132(8)	5.4994(3)	9.9044(3)	877.66(7)
5.375(7)	16.0916(5)	5.4868(2)	9.8776(3)	872.10(5)
6.709(9)	16.0623(7)	5.4712(2)	9.8428(4)	864.98(6)
7.139(9)	16.0535(5)	5.4659(3)	9.8321(5)	862.72(7)
7.626(10)	16.0430(7)	5.4603(2)	9.8196(4)	860.19(6)
5.875(9)*	16.0801(6)	5.4809(2)	9.8644(4)	869.38(5)
2.785(5)*	16.1503(5)	5.5211(2)	9.9538(3)	887.55(5)
1.542(7)*	16.1753(8)	5.5402(4)	9.9958(2)	895.77(8)
1.124(6)*	16.1850(7)	5.5469(2)	10.0103(3)	898.70(6)
0.0001(0)*	16.2084(11)	5.5658(3)	10.0539(4)	906.99(9)

Note: Standard deviations are given in parentheses.
* Data measured during decompression.

Table 3. Structure refinement results for the Fe-free crystal

P (GPa)	0.0001	1.144(7)	2.945(7)	4.025(11)	5.533(11)	6.511(12)	7.766(9)	5.145(6)*
Label	P0	P2	P4	P6	P8	P9b	P10b	P11d
space group	*Pnma*	*Pnma*	*Pnma*	*Pnma*	*Pnma*	*Pnma*	*Pnma*	*Pnma*
Z	4	4	4	4	4	4	4	4
Density (calc) (g/cm^3)	3.223	3.252	3.296	3.32	3.353	3.374	3.399	3.345
Absorption coeff. (mm^{-1})	2.03	2.05	2.08	2.11	2.11	2.13	2.14	2.12
Resolution (θ)	2.51 to 29.99°	2.39 to 29.99°	2.41 to 29.96°	2.42 to 29.99°	2.42 to 29.93°	2.43 to 29.99°	2.43 to 29.99°	2.42 to 29.99°
Completeness (%)	66.2	72.3	73.1	72.7	72.4	71.0	71.9	70.8
Extinction coeff.	0.015(2)	0.015(2)	0.029(2)	0.035(2)	0.034(2)	0.038(5)	0.038(3)	0.021(2)
no. of $I/\sigma > 4$	591	493	539	459	459	552	421	439
n all	952	1028	1024	1014	1003	1000	990	998
GooF	1.06	0.937	0.923	0.903	0.905	1.023	0.929	0.909
R_{int} (%)	5.9	6.9	6.4	8.6	9.9	1.2	10.1	8.6
$R_{4\sigma}$ (%)	6.20	6.21	5.22	5.40	5.65	7.84	6.58	5.92
R_W (%)	9.25	9.49	10.62	10.51	11.07	14.56	11.14	10.11
$R_W$all (%)	13.7	18.2	14.36	18.79	18.35	16.80	13.61	12.52
*R*all (%)	10.9	11.74	12.25	12.63	13.20	17.04	21.06	20.10
NP†	57	57	57	57	57	57	57	57

* Data measured during decompression; † NP = number of refined parameters.

TABLE 4. Selected bond lengths (Å) and angles (°) in *Pnma* structures refined with the data from the Fe-free crystal

P (GPa)	0.0001	1.144(7)	2.945(7)	4.025(11)	5.145(6)*	5.533(11)	6.511(12)	7.766(9)
T1-O3 ×2	1.626(4)	1.625(4)	1.610(4)	1.610(5)	1.619(5)	1.625(5)	1.616(5)	1.624(5)
T1-O8	1.593(6)	1.595(7)	1.593(6)	1.591(7)	1.588(7)	1.596(8)	1.587(8)	1.581(9)
T1-O9	1.616(8)	1.631(9)	1.622(7)	1.619(9)	1.619(10)	1.611(10)	1.618(10)	1.609(11)
<T1-O>	1.62(1)	1.62(1)	1.61(1)	1.61(1)	1.61(0)	1.61(1)	1.61(1)	1.61(1)
V_{T1} (Å^3)	2.16(2)	2.17(2)	2.13(2)	2.13(2)	2.14(2)	2.16(2)	2.13(2)	2.13(2)
TQE_{T1}	1.0018	1.0023	1.0016	1.0019	1.0021	1.0022	1.0023	1.0029
TAV_{T1}	7.474	9.586	6.544	7.729	8.34	8.705	9.095	11.528
$T1V_{distortion}$	0.0024	0.0026	0.0021	0.0024	0.027	0.026	0.028	0.036
T2-O1 ×2	1.651(4)	1.661(5)	1.659(4)	1.655(5)	1.655(5)	1.657(5)	1.649(5)	1.647(5)
T2-O7	1.579(6)	1.573(7)	1.578(6)	1.565(7)	1.575(8)	1.582(8)	1.574(9)	1.583(8)
T2-O9	1.638(9)	1.613(10)	1.619(8)	1.623(9)	1.610(10)	1.620(10)	1.602(10)	1.597(12)
<T2-O>	1.63(4)	1.63(5)	1.63(4)	1.62(4)	1.62(4)	1.63(4)	1.62(5)	1.62(4)
V_{T2} (Å^3)	2.21(2)	2.20(2)	2.20(2)	2.18(2)	2.18(2)	2.20(2)	2.16(2)	2.16(2)
TQE_{T2}	1.0049	1.0056	1.0049	1.0053	1.0061	1.0065	1.0062	1.0069
TAV_{T2}	16.397	17.543	15.617	16.938	19.703	22.103	20.176	23.462
$T2V_{distortion}$	0.006	0.0093	0.0087	0.0087	0.0107	0.0106	0.0109	0.0113
T3-O2 ×2	1.625(4)	1.619(5)	1.605(4)	1.606(5)	1.602(5)	1.608(5)	1.605(5)	1.602(5)
T3-O5	1.650(7)	1.644(7)	1.644(6)	1.629(7)	1.632(7)	1.634(7)	1.629(8)	1.628(8)
T1-O6	1.665(7)	1.665(7)	1.663(6)	1.674(7)	1.646(7)	1.668(7)	1.643(8)	1.654(8)
<T3-O>	1.64(4)	1.64(4)	1.63(4)	1.63(4)	1.62(4)	1.63(4)	1.62(4)	1.62(5)
V_{T3} (Å^3)	2.24(2)	2.21(2)	2.18(2)	2.18(2)	2.14(2)	2.18(2)	2.14(2)	2.14(2)
TQE_{T3}	1.0104	1.0111	1.0131	1.0136	1.0133	1.0135	1.0133	1.0144
TAV_{T3}	40.28	42.851	50.675	51.937	51.788	51.615	51.77	55.568
$T3V_{distortion}$	0.0143	0.0151	0.0171	0.0187	0.0177	0.0182	0.0182	0.0194
M12-O4	1.841(5)	1.838(5)	1.839(4)	1.845(6)	1.841(6)	1.831(5)	1.837(6)	1.838(6)
M12-O10	1.843(5)	1.843(5)	1.833(5)	1.843(5)	1.834(6)	1.834(6)	1.830(6)	1.829(6)
M12-O3	1.862(4)	1.853(4)	1.852(4)	1.855(4)	1.842(5)	1.829(5)	1.835(5)	1.837(5)
M12-O5	1.901(5)	1.888(5)	1.882(4)	1.887(5)	1.879(6)	1.868(5)	1.868(6)	1.868(6)
M12-O6	1.925(5)	1.923(5)	1.911(5)	1.895(5)	1.900(6)	1.892(6)	1.896(7)	1.881(6)
M12-O1	1.976(4)	1.976(4)	1.983(4)	1.979(4)	1.986(5)	1.980(4)	1.983(5)	1.974(5)
<M12-O>	1.89(4)	1.89(4)	1.88(4)	1.88(4)	1.88(4)	1.87(4)	1.88(4)	1.87(4)
V_{M12} (Å^3)	8.94(4)	8.87(4)	8.83(4)	8.84(4)	8.79(5)	8.67(4)	8.71(5)	8.67(5)
OQE_{M12}	1.007	1.0068	1.0067	1.0062	1.0065	1.0069	1.0065	1.0062
OAV_{M12}	20.557	20.218	19.68	18.565	18.98	20.339	18.875	18.479
$M12V_{distortion}$	0.0083	0.0084	0.0078	0.0069	0.0073	0.0075	0.007	0.0067
M3-O8	1.768(6)	1.763(7)	1.765(6)	1.765(7)	1.764(8)	1.759(8)	1.766(8)	1.773(9)
M3-O4	1.831(6)	1.821(7)	1.831(6)	1.820(7)	1.845(7)	1.825(7)	1.832(8)	1.826(8)
M3-O2 ×2	1.959(5)	1.954(6)	1.961(5)	1.951(5)	1.949(6)	1.942(5)	1.942(6)	1.933(6)
M3-O1 ×2	2.115(5)	2.093(5)	2.060(5)	2.062(5)	2.042(6)	2.038(5)	2.035(6)	2.037(6)
<M3-O>	1.96(12)	1.95(12)	1.94(12)	1.94(12)	1.93(11)	1.92(11)	1.93(11)	1.92(11)
V_{M3} (Å^3)	9.77(4)	9.59(5)	9.51(4)	9.44(5)	9.39(5)	9.27(5)	9.30(5)	9.25(5)
OQE_{M3}	1.0212	1.0214	1.0191	1.0193	1.0182	1.0192	1.0183	1.0187
OAV_{M3}	48.207	50.718	47.591	47.527	48.284	50.723	48.94	50.78
$M3V_{distortion}$	0.0196	0.0213	0.0197	0.0203	0.019	0.0209	0.0201	0.0214
Ca1-O7	2.261(6)	2.261(7)	2.251(6)	2.255(7)	2.262(8)	2.236(8)	2.230(8)	2.257(8)
Ca1-O3 ×2	2.408(5)	2.405(5)	2.399(4)	2.381(5)	2.386(6)	2.385(5)	2.376(6)	2.374(6)
Ca1-O1 ×2	2.516(5)	2.502(5)	2.495(5)	2.489(5)	2.479(6)	2.472(5)	2.471(6)	2.461(8)
Ca1-O6	2.558(6)	2.541(7)	2.507(6)	2.509(7)	2.499(7)	2.483(7)	2.484(8)	2.464(6)
Ca1-O5	2.579(6)	2.573(7)	2.557(6)	2.551(7)	2.534(7)	2.542(7)	2.545(7)	2.518(8)
<Ca1-O>	2.46(17)	2.46(17)	2.44(17)	2.44(17)	2.43(18)	2.43(17)	2.42(17)	2.42(18)
V_{Ca1} (Å^3)	19.03(7)	18.81(8)	18.52(7)	18.36(8)	18.20(9)	18.01(9)	17.99(9)	17.80(9)
Ca2-O7	2.301(7)	2.293(8)	2.278(7)	2.275(8)	2.254(8)	2.256(8)	2.265(9)	2.222(9)
Ca2-O3 ×2	2.454(4)	2.461(5)	2.453(4)	2.454(5)	2.440(5)	2.452(6)	2.436(6)	2.421(6)
Ca2-O2 ×2	2.515(4)	2.507(5)	2.483(5)	2.465(5)	2.454(6)	2.452(5)	2.451(6)	2.438(6)
Ca2-O2 ×2	2.794(4)	2.770(5)	2.753(4)	2.726(5)	2.727(5)	2.724(5)	2.711(5)	2.709(5)
<Ca2-O>	2.55(10)	2.54(16)	2.52(16)	2.51(16)	2.50(17)	2.50(17)	2.49(16)	2.48(18)
V_{Ca2} (Å^3)	22.55(8)	22.33(9)	21.88(7)	21.54(9)	21.27(9)	21.34(9)	21.16(10)	20.78(10)
O4···O10	2.749(10)	2.725(10)	2.679(9)	2.632(11)	2.616(11)	2.630(11)	2.603(13)	2.580(12)
O2···O10	2.954(7)	2.954(8)	2.957(7)	2.959(8)	2.950(8)	2.946(8)	2.938(8)	2.932(8)
O5-M12-O10	82.7(2)	83.1(2)	83.1(2)	83.8(2)	83.8(2)	83.9(2)	84.1(2)	84.2(2)
O6-M12-O4	86.1(2)	85.9(2)	86.3(2)	86.4(2)	85.7(2)	85.7(2)	86.4(2)	86.3(2)

Note: TQE, TAV, OQE, and OAV are quadratic elongation and angle variance for tetrahedra and octahedra (Robinson et al. 1971); polyhedral volumes calculated with IVTON (Balić-Žunić and Vicković 1996).
* Data measured during decompression. Volume distortions ($V_{distortion}$) are calculated as defined by Makovicky and Balić-Žunić (1998).

decrease up to 7.77 and 7.63 GPa for the Fe-free and Fe-rich samples, respectively (Figs. 2a–2d; Table 2). Over this pressure range, the *a*, *b*, and *c* lattice parameters decrease by 1.07, 1.90, and 2.29%, respectively, for the Fe-free sample and by 1.03, 1.89, and 2.33%, respectively, for the Fe-rich sample. The unit-cell volume decreases non-linearly at an average rate of 6.06 Å^3/GPa for the Fe-free sample and 6.13 Å^3/GPa for the Fe-rich sample. The compression of all of the unit-cell parameters is accompanied by significant stiffening of the *b* and *c* axes while the *a* axis shows marginal softening. The lattice parameter variation of both of the

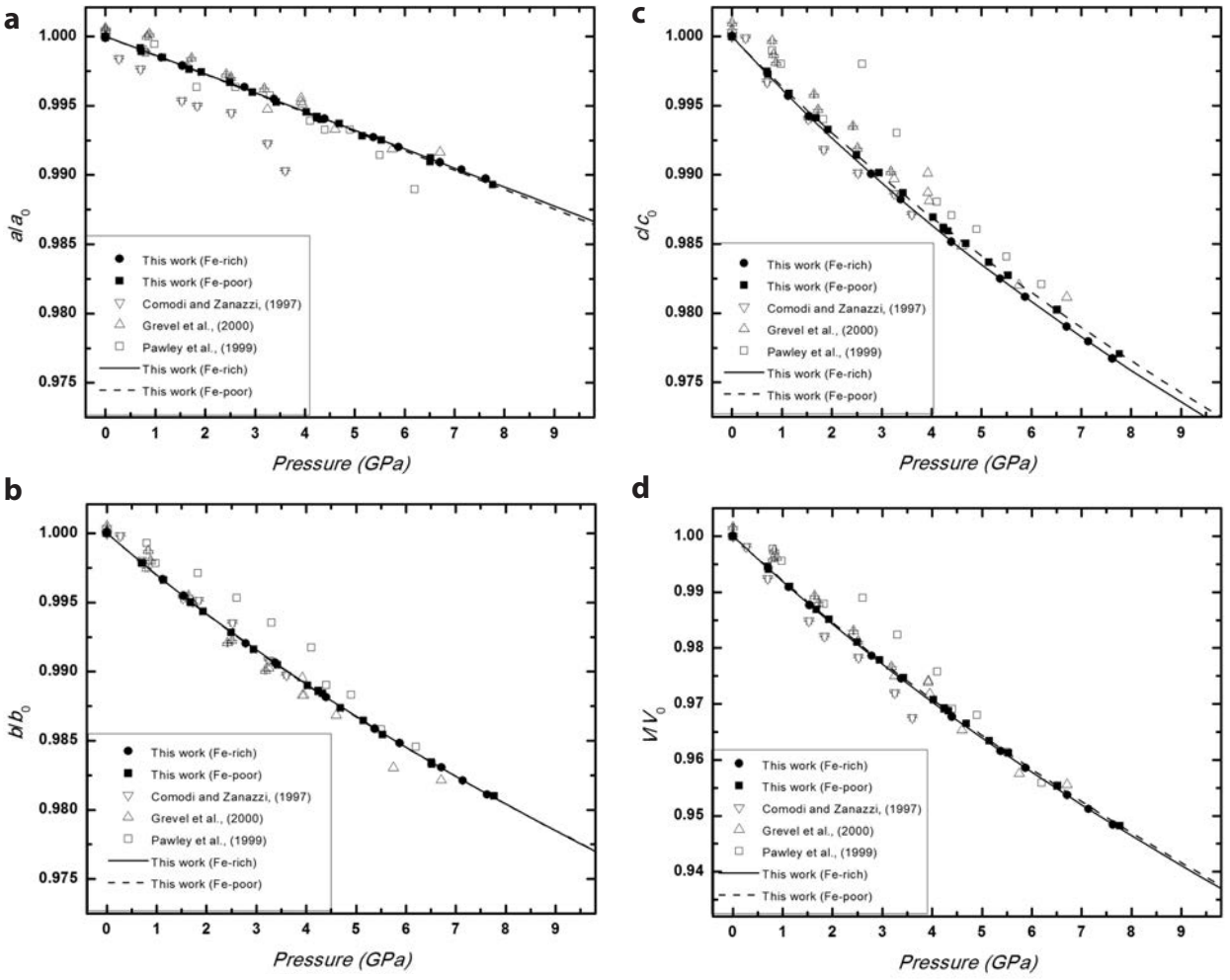

FIGURE 2. Evolution of the unit-cell parameters as a function of pressure: (**a**) a/a_0, (**b**) b/b_0, (**c**) c/c_0, and (**d**) V/V_0 unit-cell parameters as a function of pressure for the samples Fe-free (filled squares), Fe-rich (filled circles), data by Comodi and Zanazzi (1997) (open inverted triangle), Pawley et al. (1998) (open square), and Grevel et al. (2000) (open triangle).

samples follows the same compressibility scheme, and for both the compression is completely reversible, with no indication of any change in elastic behavior upon decompression, but for the Fe-free sample there is significant scatter in the *f-F* plots of the unit-cell parameters in the pressure range 4–4.5 GPa (Fig. 3). This appears to be associated with a subtle change in the structural evolution with pressure, as there is no discontinuity in the *P-V* curve nor is there a change in the slope of the *f-F* plot of the volume compression (Fig. 3d) at this pressure (see discussion).

The $P(V)$ data were fitted, for both Fe-free and the Fe-rich sample, by a third-order (BM3-EoS) Birch-Murnaghan equation of state (Birch 1947), using the EOSFIT-5.2 software (Angel 2000). The EoS coefficients of the two samples are reported together in Table 6. The values of the bulk modulus [$K_{T0Fe\text{-}free}$ = 122.1(7) GPa and $K_{T0Fe\text{-}rich}$ = 119.1(7) GPa], and its first pressure derivative [$K'_{Fe\text{-}free}$ = 6.8(2) and $K'_{Fe\text{-}rich}$ = 7.3(2)] are both in excellent agreement with those indicated by the normalized stress F_E – Eulerian strain f_E plot as shown in Figure 3 {Angel 2000; $F_E = P/3f_E(1 + 2f_E)^{5/2}$, $f_E = [(V_0/V)^{2/3} - 1]/2$}. The bulk moduli obtained from isothermal hydrostatic compression measurements such as those reported here correspond to the Reuss bound on the isothermal bulk modulus. The values of the components of the elastic modulus tensor of end-member zoisite (Mao et al. 2007) yield a value of the Reuss bound of the adiabatic modulus of 121.6(4) GPa, which, using their value for the factor $(1 + \alpha\gamma T)$ of 1.016, corresponds to an isothermal bulk modulus of 119.7(4) GPa, which is in between our values. The apparent agreement reported by Mao et al. (2007) between their Brillouin data and the previous compression studies of Grevel et al. (2000) and Pawley et al. (1998) was an artifact of Mao et al. (2007) incorrectly using the VRH average modulus instead of the Reuss bound as a basis for comparison with hydrostatic compression experiments. The slightly higher value of K_{T0} = 125(2) for end-member zoisite obtained by Grevel et al. (2000) is probably a consequence of their fixing the value of K' to 4. Considering the data scatter in the other published studies (Fig. 2) agreement with any EoS parameters derived from them is fortuitous, and depends on the assumptions made about constraining other EoS parameters such as V_0 and K'.

The axial compressibilities at room pressure for *a*, *b*, and *c* (Table 7) were obtained using a parameterized form of the

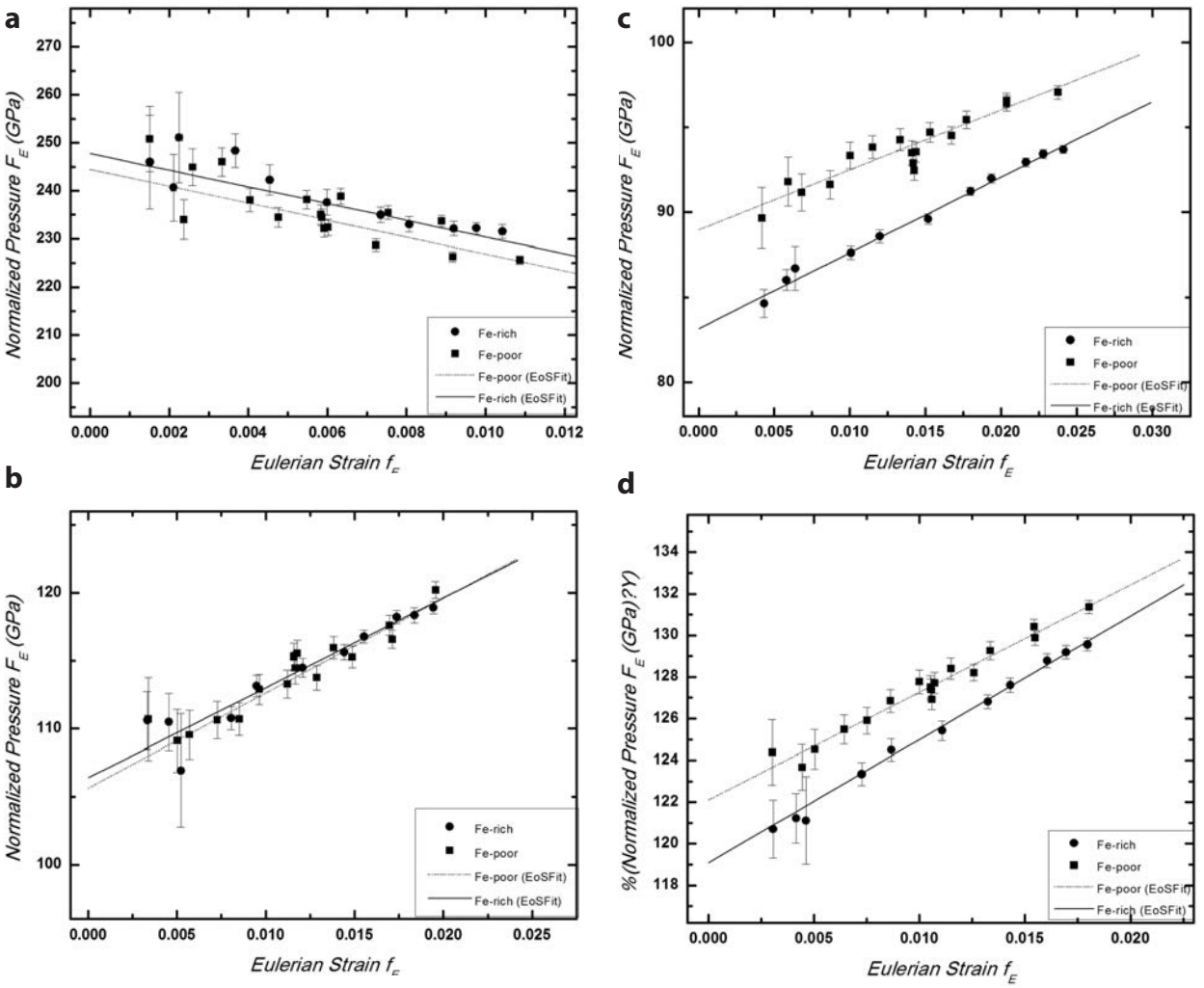

FIGURE 3. F_E-f_E plot for the (**a**) *a* lattice parameter, (**b**) *b* lattice parameter, (**c**) *c* lattice parameter, and (**d**) unit-cell volume.

TABLE 6. Fitted unit-cell parameters, bulk moduli, and first derivative of the bulk modulus at room pressure obtained using a BM3 EoS

	Fe-free				Fe-rich			
	a (Å)	*b* (Å)	*c* (Å)	*V* (Å^3)	*a* (Å)	*b* (Å)	*c* (Å)	*V* (Å^3)
Val0	16.2008(8)	5.5530(3)	10.0421(5)	903.39(5)	16.2098(6)	5.5655(2)	10.0536(2)	906.95(5)
K_{To} (GPa)	244(5)	105.6(1.6)	89.1(9)	122.1(7)	248(4)	106(1)	83.2(5)	119.1(7)
K'	−0.7(1.2)	8.4(5)	6.6(3)	6.8(2)	−0.7(0.9)	8.2(3)	7.5(2)	7.3(2)

BM3-EoS, in which the individual axes are cubed, following the procedure implemented in the EoS-FIT5.2 software (Angel 2000). Note that the resulting moduli given by EosFit (Table 7) must be multiplied by a factor of 3 to obtain the linear moduli equal to the inverse of the linear compressibilities defined as

$$\beta_{l0} = \frac{-1}{l}\left(\frac{\delta l}{\delta P}\right)_{P=0},$$

which is in turn equal to the sum of the three compressional terms in the elastic compliance matrix for the axis. Thus, for example, the compressibility of the *a*-lattice parameter of zoisite is given by $\beta_1 = s_{11} + s_{12} + s_{13}$. The resulting linear compressibilities for the Fe-free sample in this study (Table 7) agree within the uncertainties with those determined from the individual values of the compliance matrix inverted from the c_{ij} values given by Mao et al. (2007). The compressibility scheme is $\beta_a << \beta_b < \beta_c$ for both samples, (β_a:β_b:β_c = 1.00:2.29:2.74 for the Fe-free sample and β_a:β_b:β_c = 1.00:2.34:2.99 for the Fe-rich sample) with **a** being the most rigid direction and **c** the softest. Although the compressibility scheme shows the same general pattern for both samples, and the compressibilities of the **a** and **b** directions do not change between the two, the increased Fe content leads to a significant softening of **c**, and it is this axial softening that results in the reduction of bulk modulus with the addition of Fe to the structure. The same general pattern of axial compressibilities is apparent in the previous experimental studies (Fig. 2), although the compressibility of **a** was significantly overestimated by Pawley et al. (1998) and no meaningful values of the linear compressibilities can be obtained from those data.

TABLE 7. Linear axial and volume compressibilities (GPa^{-1}) for Fe-free and Fe-rich crystals

	Fe-free			Fe-rich			
	a (Å)	*b* (Å)	*c* (Å)	*a* (Å)	*b* (Å)	*c* (Å)	
K_{T0} (GPa)	244(5)	105.6(1.6)	89.1(9)	248(4)	106(1)	83.2(5)	*
Linear modulus (GPa)	732(15)	317(5)	267(3)	744(12)	318(3)	249.60(15)	†
Isothermal linear compressibilities (GPa^{-1})	0.00137(3)	0.00314(6)	0.00375(4)	0.00134(2)	0.00314(3)	0.004006(2)	‡
Adiabatic linear compressibilities (GPa^{-1})	0.00132	0.00309	0.00381				§

* This work, *K* value obtained from EosFit 5.2 using the method described in the text.
† This work, linear modulus, calculated as described in the text.
‡ This work, isothermal linear compressibilities, calculated as described in the text.
§ Mao et al. (2007), adiabatic linear compressibilities calculated from the s_{ij} as described in the text.

Structure evolution with pressure

To better understand the reasons of the change in the compressibility of zoisites with Fe content, and why the biggest change in compressibility is along the **c**-axis, we describe in the following section briefly the structural evolution of the Fe-free crystal with pressure. The compression of the bond lengths and bond angles at the bridging oxygen atoms alone is not sufficient to explain either the compression mechanism in each direction, nor the strong anisotropy of the structure itself. Therefore, distortion of the polyhedra and their cooperative rotations are the key to explain the main mechanisms that control the average compression of the whole structure.

Framework compression. During the pressure increase from room pressure up to 7.77 GPa the *a*-axis undergoes a compression of about 0.17 Å. The [100] direction is the least compressible of the whole structure with a bulk modulus of 244(5) GPa for the Fe-free sample and 248(4) GPa for the Fe-rich one. The M3 octahedra share one oxygen atom (O8) with T1 tetrahedra on one side while on the other side they share one edge (O1-O4) with M12 octahedra, which also share an oxygen atom (O3) with T1 tetrahedra (see Figs. 1a–1c). These polyhedral linkages follow the path of the 2_1 screw axis running parallel to [100] (Fig. 1b) and therefore they span one half of the unit-cell length (*a*/2), which compresses by 0.085 Å over the pressure range studied. The contribution to the compression from the rotation and compression of M12 and M3 octahedra is equal to the shortening and rotation of the vector from O8 to O5 [O8 shared between T1 and M3 and O5 shared between T3 and M12 (0.008 Å)] and the one from O5 to O3 (0.028 Å). The rotation of the M3 and M12 octahedra relative to one another shortens the M3-M12 cation-cation distance by 0.033 Å, and results in significant compression of the T3 tetrahedra (from 2.236 to 2.142 $Å^3$, see Table 4 and Fig. 4) and increase of the distortion [the volume distortion parameter of Makovicky and Balić Žunić (1998) increases from 1.43 to 1.94%] of the T3 tetrahedron (see Table 4 and Fig. 5). The largest contribution from the framework to the shortening along [100] actually comes from the shortening and rotation of the O3-O8 edge of the T1 tetrahedron that contributes 0.051 Å, which also leads to a significant increase in distortion of the tetrahedra.

The lengths of the O-O edges shared between the consecutive M12 octahedra forming continuous chains along the [010] direction (see Fig. 1c) are considerably shorter (by 0.2–0.3 Å) than the unshared edges, indicating significant cation-cation repulsion between the octahedral cations. One might therefore expect that the [010] direction would be the stiffest direction in the structure. That this is not the case indicates that compression and/or deformation of the octahedra is important in the compression of the structure. Each octahedron is related to the next in the chain by a mirror plane perpendicular to **b** (shown in Fig. 1c with a solid line), and therefore the compression of *b*/2 along ½[010] must be accommodated within a single octahedron. Under compression to 7.6 GPa the opening of the O5-M12-O10 and O6-M12-O4 angles (by 1.4° and 0.25°, respectively), together with the shortening of the bond distances M12-O5 (by 0.033 Å), M12-O10 (by 0.015 Å), M12-O4 (by 0.0028 Å), and M12-O6 (by 0.043 Å) are the main mechanisms of compression and contribute the total of 0.053 Å to the shortening of each octahedron along [010] (see Table 4 and Fig. 6). The individual chains of M12 octahedra are linked to one another through parallel chains of alternating M3 octahedra and T3 tetrahedra (Fig. 1a), which also have to shorten under compression. With increasing pressure the distance between the O2 atoms linked to M3 decreases by 0.073 Å through a combination of bond shortening and closing of the O2-M3-O2 angle, while the O2-O2 distance forming the edge of the T3 tetrahedron shortens by 0.033 Å through bond compression alone.

The octahedral chains in zoisite are softer along their length, than the AlO_6 edge-sharing octahedral chains of the Al_2SiO_5 polymorphs. Direct compression of AlO_6 octahedra along the chain directions results in moduli in excess of 200 GPa in sillimanite and andalusite (Burt et al. 2006), while in kyanite (Yang et al. 1997; Comodi et al. 1997) the linear modulus is still 166 GPa, much greater than in zoisite. In all cases the chains are compressed by shortening Al-O bonds (see Table 8 and Fig. 8). The compression of the chains of octahedra must be accommodated by the rest of the structure; therefore the difference in compressibility must indicate that in andalusite and sillimanite the octahedral chains are better supported by the surrounding structure.

In zoisite, the [001] direction is the most compressible of the entire structure with a modulus of 89.1(9) GPa for the Fe-free sample and 83.2(5) GPa for the Fe-rich one. In Figure 1b, the horizontal line joining the M12 sites indicates a pair of sites related by the 2_1 screw axis running along [001]. Therefore, their z-coordinates differ by ½ and the compression of the framework along **c** must result in the shortening of this M12-M12 vector, which requires shortening of both the polyhedral links M12-T3-M12 and M12-T1-T2-M12. Within the pressure increase from room *P* to 7.77GPa the biggest contribution to the shortening of *c*/2 (0.115 Å) in the first link is from the shortening by 0.07 Å of the O5-O6 edge of the T3 tetrahedron. Most of the compression of this O5-O6 edge (see Fig. 9) comes from a decrease in the O5-T3-O6 angle of about 3°, and a small contribution comes from shortening of T3-O5 but not T3-O6 (Fig. 7e and Table 4). The rest of the required compression (0.04 Å) comes from shortening of the M12-O5 and M12-O6 bonds (Fig. 7a; Table 4). In the

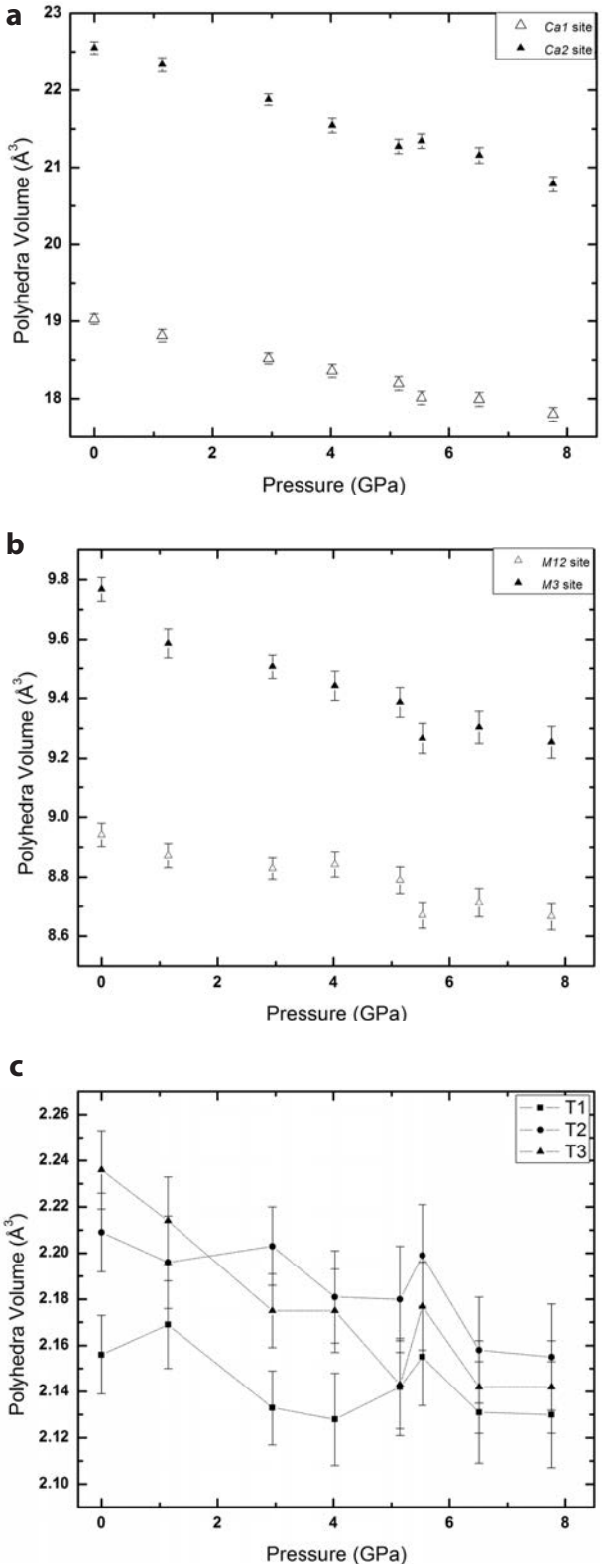

Figure 4. Evolution of the volumes of the polyhedra with increasing pressure: (**a**) Ca1 and Ca2 sites (open and filled triangles, respectively); (**b**) M12 and M3 sites (open and filled triangles, respectively); (**c**) T1, T2, and T3 (squares, circles and triangles, respectively).

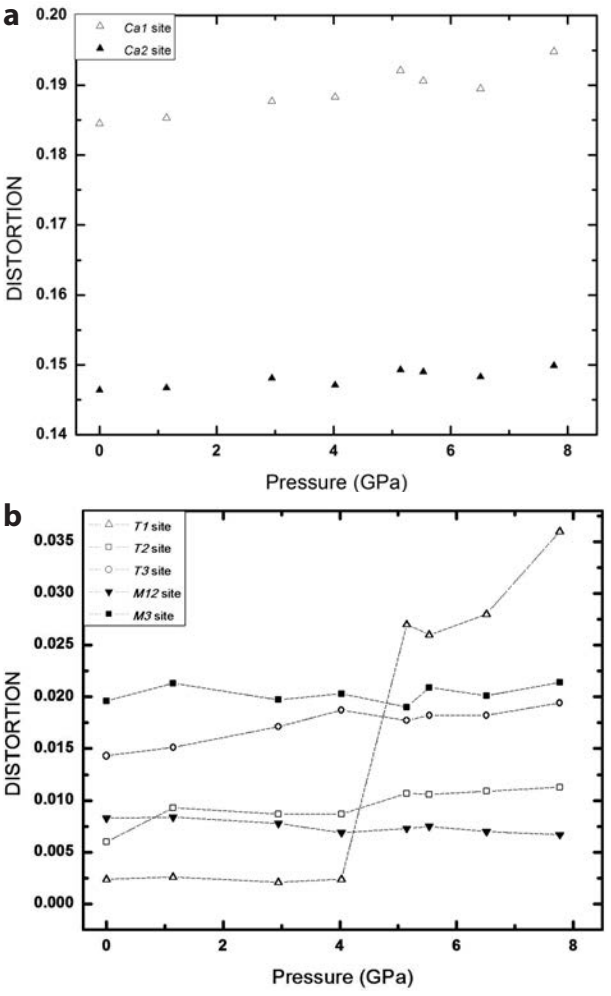

Figure 5. Variation of the polyhedra distortions with pressure. (**a**) Ca1 and Ca2 sites (open triangle and filled triangle, respectively); (**b**) T1, T2, T3, M12, M3 (open triangle, filled triangle, open inverse triangle, filled inverse triangle, and open square, respectively).

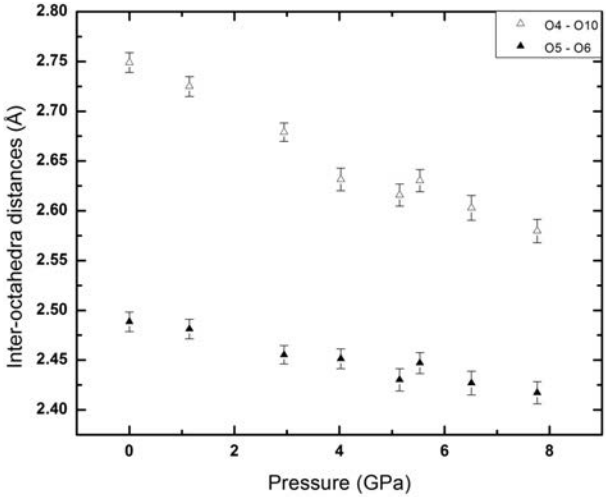

Figure 6. Inter-octahedra distances O4-O10 (open triangle) and O5-O6 (filled triangle).

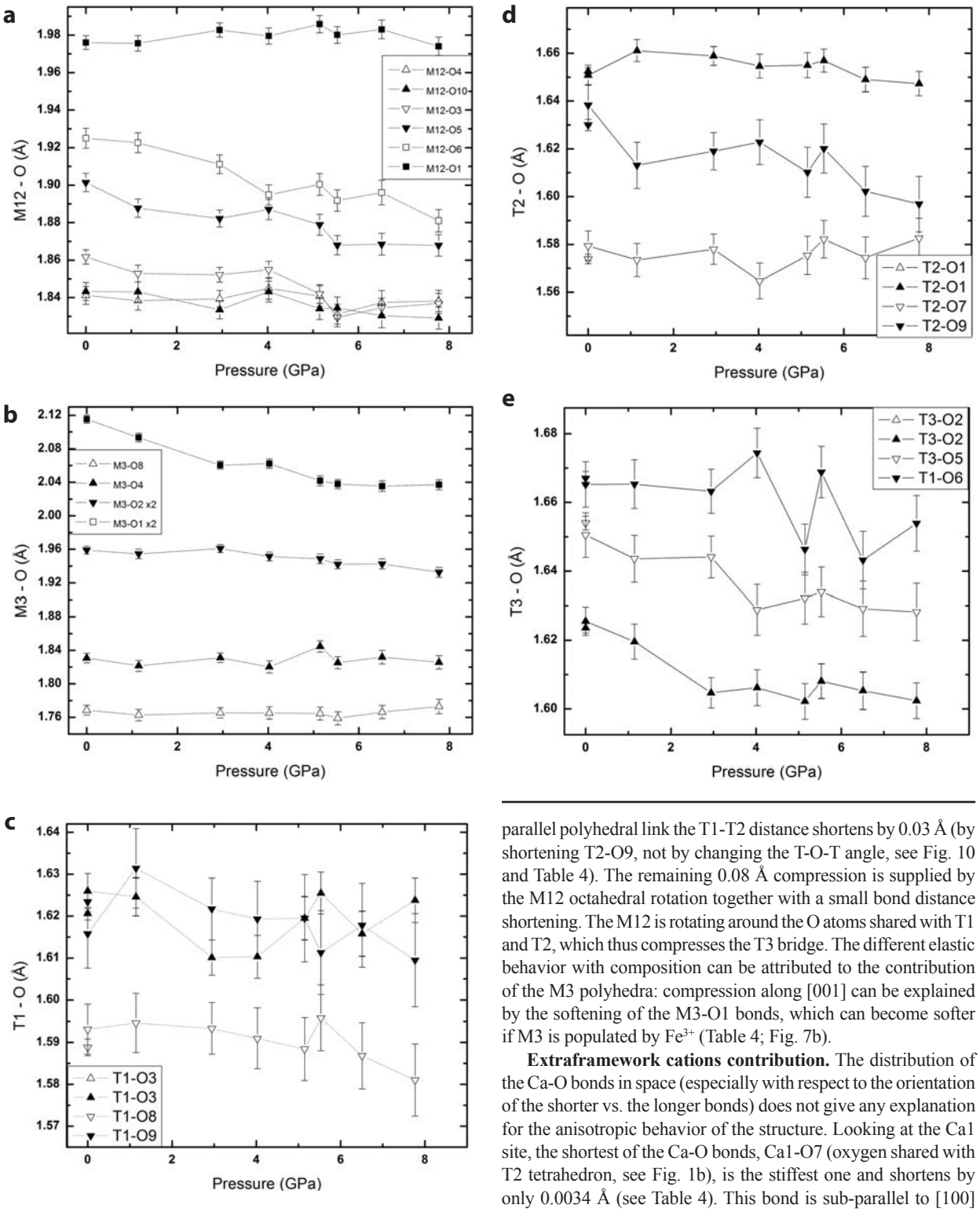

Figure 7. Evolution of the M12-O (**a**), M3-O (**b**), T1 (**c**), T2 (**d**), and T3 (**e**) bond distances with increasing pressure.

parallel polyhedral link the T1-T2 distance shortens by 0.03 Å (by shortening T2-O9, not by changing the T-O-T angle, see Fig. 10 and Table 4). The remaining 0.08 Å compression is supplied by the M12 octahedral rotation together with a small bond distance shortening. The M12 is rotating around the O atoms shared with T1 and T2, which thus compresses the T3 bridge. The different elastic behavior with composition can be attributed to the contribution of the M3 polyhedra: compression along [001] can be explained by the softening of the M3-O1 bonds, which can become softer if M3 is populated by Fe^{3+} (Table 4; Fig. 7b).

Extraframework cations contribution. The distribution of the Ca-O bonds in space (especially with respect to the orientation of the shorter vs. the longer bonds) does not give any explanation for the anisotropic behavior of the structure. Looking at the Ca1 site, the shortest of the Ca-O bonds, Ca1-O7 (oxygen shared with T2 tetrahedron, see Fig. 1b), is the stiffest one and shortens by only 0.0034 Å (see Table 4). This bond is sub-parallel to [100] and is opposite to the Ca1-O6 and Ca1-O5 (see Fig. 1b) that are the longest and softest among the Ca1-O bonds, with a compression of 0.094 and 0.061 Å, respectively (see Table 4). Therefore the combined effect given by those bonds is that the Ca1 cavity shrinks isotropically. In the Ca2 site, the stiffest bonds are to the

TABLE 8. Average rate of change of the distances Al-O (to the shared octahedral edge), Al-Al and O-O (length of the shared edge) in zoisite, sillimanite, and andalusite

	Andalusite			Sillimanite			Zoisite		
	Label	Bond distances (room *P*)	Rate (Å/GPa)	Label	Bond distances (room *P*)	Rate (Å/GPa)	Label	Bond distances (room *P*)	Rate (Å/GPa)
Shared edge	Al1-OA	1.824(3)	–0.0016	Al1-OA	1.916(3)	–0.0074	M12-O4	1.841(5)	–0.0005
	Al1-OB	1.893(3)	–0.0013	Al1-OB	1.863(3)	–0.0039	M12-O10	1.843(5)	–0.0019
							M12-O5	1.901(5)	–0.0041
							M12-O6	1.925(5)	–0.0055
	OA-OA	2.468(7)	–0.0009	OA-OB	2.892(8)	–0.0032	O4-O6	2.5707(9)	–0.0035
	OA-OA	2.468(7)	–0.0009	OA-OB	2.433(8)	–0.0139	O10-O5	2.4754(9)	0.0007
Apical oxygen	Al1-OD	2.076(3)	–0.0077	Al1-OD	1.954(7)	–0.0097	M12-O1	1.977(4)	0.0004
							M12-O3	1.862(4)	–0.0037
Al - Al	Al1-Al1	2.686(3)	–0.0034	Al1-Al1	2.884(0)	–0.0034	M12-M12	2.744(4)	–0.0046
	Al1′-Al1′	2.686(3)	0.0070						

Notes: Data for sillimanite and andalusite taken from Burt et al. (2004). The rates of change of the distances are calculated with a linear fit from the length data over the whole pressure range and are recorded as Å/GPa. Atoms labeled as in Figure 1.

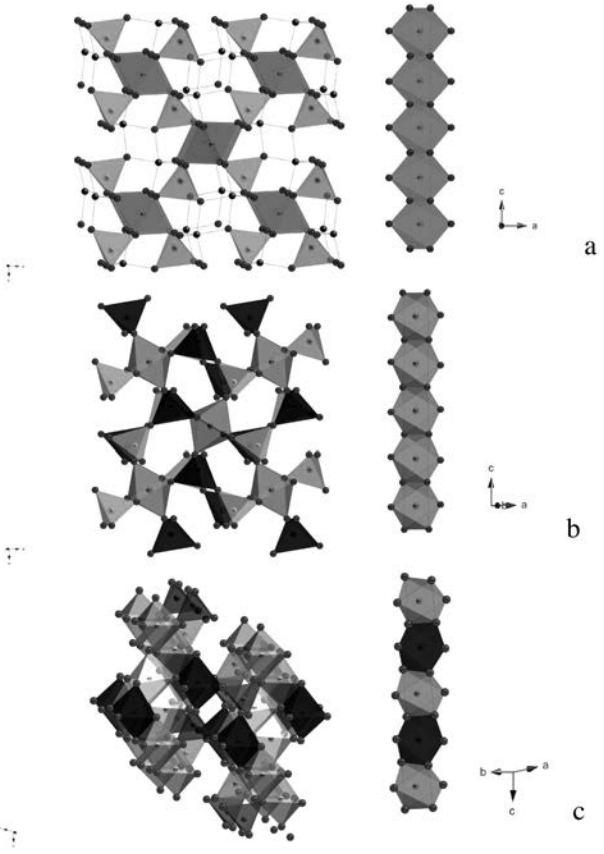

FIGURE 8. Comparison of the structure of andalusite (**a**), sillimanite (**b**), and kyanite (**c**) showing the difference in the linkage between the octahedra and the different orientation of the chain of octahedra.

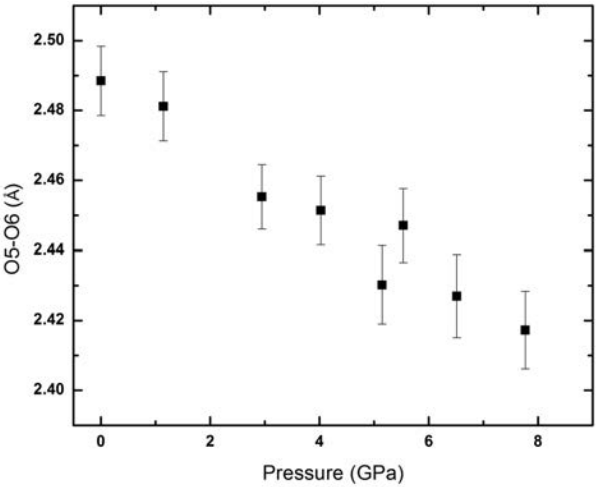

FIGURE 9. Variation of O5-O6 distance with *P*.

O3 oxygen, which is shared with T1 tetrahedron (Fig. 1b) although these are not the shortest bonds. These Ca-O3 bonds lie sub-parallel to [100] and would therefore stiffen *a*, were it not for the fact that the two Ca2-O2, which are the longest and softest among the Ca2-O bonds are also sub-parallel to [100]. As a consequence the combined effect is that the Ca2 cavity, as well as the Ca1 cavity, shrinks almost isotropically.

Hydrogen-bond geometry. As described above, the Fe-free sample shows significant scatter in the *f-F* plots of the unit-cell parameters in the pressure range 4–4.5 GPa (Fig. 3), which is not accompanied by a discontinuity in the *P-V* curve nor is there a change in the slope of the *f-F* plot of the volume compression (Fig. 3d). Therefore, this data scatter is unlikely to be associated with the isostructural phase transition that occurs with increasing iron contents (Liebscher et al. 2002) as that transition leads to an increase in volume compared to that of the end-member composition, and so is not expected to occur upon compression. Nevertheless, it is worth noting that there is a sharp increase of distortion of the T1 polyhedron (Fig. 5b) and a discontinuity in the O7-O9-O8 angle (Fig. 11) between 4 and 5 GPa. A close inspection of the O10···O4 distance (with the O4 anion being the acceptor of the hydrogen bond in zoisite structure) shows that there is a consistent reduction of this distance in the room-*P* to 4–5 GPa pressure range (up to 0.2 Å; Fig. 12a). The same is observed for the O10···O2 distance, which remains unchanged up to 4 GPa, and then begins to decrease. If we combine shortening of these distances with the behavior of the T1 tetrahedron, we can infer that something is happening to the hydrogen bonding topology. Quite likely, the H atom is going off of the O4···O10 junction and begins to form a bifurcated bond with O2. This change in bonding topology must be instantaneous and would explain the softening in the *c* lattice parameter and the hardening in the *b* lattice parameter (Figs. 3b and 3c).

In our study, we have shown that the addition of Fe to the structure of pure zoisite results in a slight decrease in the bulk

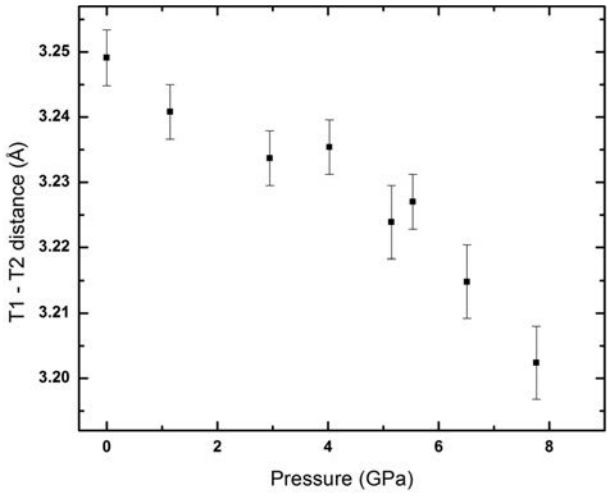

FIGURE 10. Variation of T1-T2 distance with *P*.

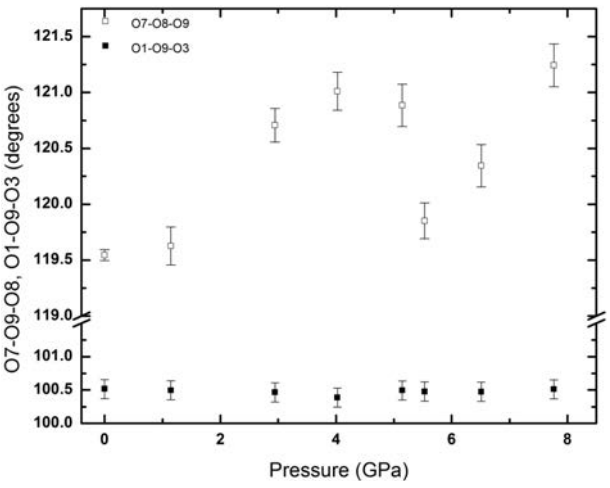

FIGURE 11. Evolution of the O7-O9-O8 and O1-O9-O3 angles with increasing pressure.

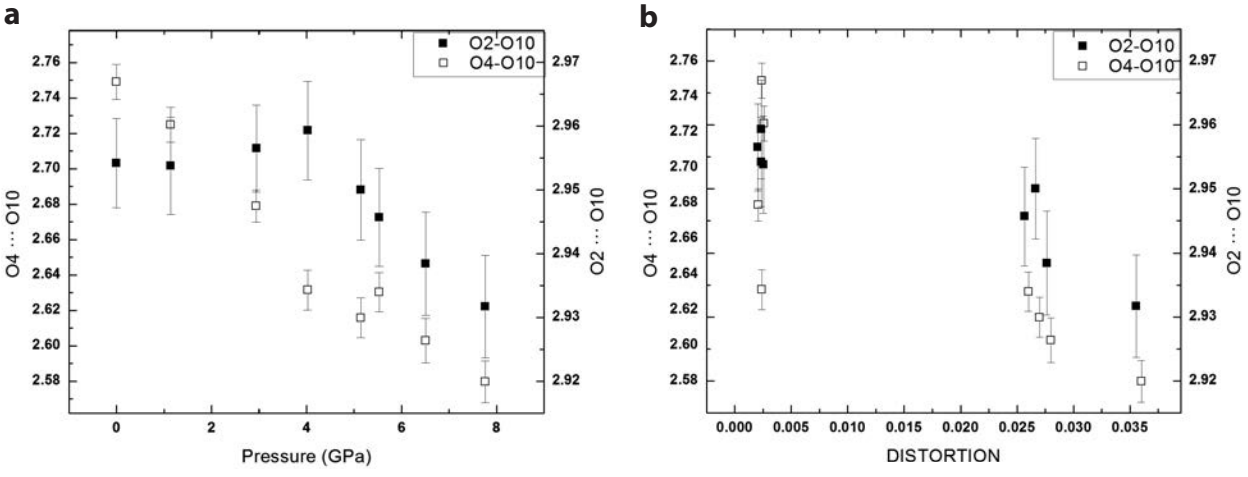

FIGURE 12. Evolution of the O4-O10 and O2-O10 bond distance increasing pressure (**a**) and with the variations of the T1 tetrahedron distortion parameter (**b**) as defined by Makovicky and Balić-Žunić (1998).

modulus, which is the result of a softening of the *c* axis. The pattern of pressure-induced strains and the structural evolution at high pressure is also similar to the high-temperature behavior measured by Cámara et al. (2012) on an Fe-free crystal from the same sample studied in this work. At high temperatures, in the *T* range from 303 to 770 K, the *a* lattice parameter shows negative thermal expansion (NTE) and shortens by 0.018 Å, while *b* and *c* lattice parameters expand during the temperature increase by 0.076 and 0.150 Å, respectively. The anisotropy of the elastic response of zoisite to pressure and temperature is therefore related. In both cases the *c* lattice parameter shows the biggest variation, being the softest direction at high-*P* [with a bulk modulus of $K_{T0cFe\text{-}free} = 89.1(9)$GPa], and at high-*T* it is the direction of greatest expansion, with a thermal expansion coefficient of $\alpha^{\circ}_{33(1bar,298\ K)} = 18.95(2)\cdot10^{-6}K^{-1}$ (Cámara et al. 2012). The *a* lattice parameter is the stiffest direction at high-*P* [with a bulk modulus of $K_{T0cFe\text{-}free} = 244(5)$GPa] and at high-*T* it shows negative thermal expansion [$\alpha^{\circ}_{11(1bar,298\ K)} = -1.18(3)\cdot10^{-6}K^{-1}$, Cámara et al. (2012)]. The *b* lattice parameter has a bulk modulus close to that of *c* at high-*P* [$K_{T0bFe\text{-}free} = 105(2)$GPa] and a thermal expansion coefficient also close to *c* [$\alpha^{\circ}_{22(1bar,298\ K)} = 17.31(2)\cdot10^{-6}K^{-1}$ Cámara et al. (2012)]. The reasons for this anisotropy can be found in the linkage system of the framework. Along the [001] direction, the compression occurs mainly by shortening of the inter-polyhedral distances (see Fig. 6), and is therefore certainly bigger with respect to the compression along [100] and [010] that is achieved by compression of the intra-polyhedral distances (see Fig. 7) and rotation of the polyhedra with respect one to another.

The compressibility behavior is also affected by the Fe contents at the M3 site. As reported by Cámara et al. (2012), the thermoelastic parameters of the unit-cell volume of zoisite obtained in their study compared with those of a natural epidote previously reported by Gatta et al. (2011) with higher Fe contents [i.e., $Ca_{1.925}Fe_{0.745}Al_{2.265}Ti_{0.004}Si_{3.037}O_{12}(OH)$] showed significant differences, being $\alpha_{V0} = 5.93(5)\cdot10^{-5}K^{-1}$ for zoisite and $5.1(2)\cdot10^{-5}K^{-1}$ for epidote. Therefore, comparing two slightly different structures (epidote and zoisite) it seems that at high-*T*, higher Fe contents reduce the volume thermal expansion coefficient (Cámara et al.

2012); however several high-temperature data on zoisite samples with different Fe contents (Cámara, unpublished data), clearly showed that the volume thermal expansion coefficient increases with increasing Fe content. This is clearly in agreement with the high-*P* behavior shown by our data for all the lattice parameters, where the higher the Fe contents the lower the bulk modulus, as shown in Figure 2d and Table 6.

The key to understanding this behavior can be found in the evolution of the structure with increasing Fe content. As reported by Liebscher et al. (2002), and confirmed by our data, increasing Fe content causes an increase in the volume of the M3 octahedron while the other polyhedral volumes remain practically constant (Liebscher et al. 2002). The increase in volume of M3 is accommodated by increasing the distance between the cation and the apical O atoms (O4-O8, see Fig. 1a) together with the M3-O1 bond lengths (see Figs. 1a and 1b). The increase of the O4-O8 distance causes both, a regularization and a rotation of the M3 octahedra together with a rotation (by 0.5°) and regularization of the M12 octahedra (Liebscher et al. 2002). The increase of the O4-O8 distance along **a** together with the rotation of M3 is compensated by rotation of the T2 tetrahedra around the relatively fixed apical O3 atom of the M12 octahedron. The rotation of T2 leads to rotation of the corner-linked T1 tetrahedron in the opposite direction, which is reflected by the increasing O8-O9-O7 angle of the Si_2O_7 group. Therefore, an increase in the Fe content has essentially the same effect on the tilt of T1 and T2 tetrahedra as does increasing pressure that opens the O8-O9-O7 (see Fig. 11). Therefore, this mechanism of rotation of T1 and T2 tetrahedra that takes place with increasing Fe content softens the [001] direction by making the relative rotation of T1 and T2 tetrahedra even easier, while the increase in the distance O4-O8 together with the rotation of M12 stiffens the [100] direction. In addition, the increase of the M3-O1 bond lengths with increasing Fe content is a further mechanism that softens the [001] direction.

Acknowledgments

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O10 O 0.2677(4) 0.2500 0.0743(7) 0.0099(13) Uiso 1 2 d S . .

_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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Please consider this CIF for publication. I certify that this contibution is the
original work of those listed as authors; that it has not been published
before (in any language or medium) and is not being considered for publication
elsewhere; that all authors concur with and are aware of the submission; that
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my knowledge the paper contains no statements which are libellous, unlawful or
in any way actionable. All coauthors have made significant scientific
contributions to the work reported, including the ideas and their execution,
and share responsibility and accountability for the results.
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# NB if using publCIF, the Author database tool might prove useful
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;
Cryst Lab, Dept of Geosciences, Virginia Tech, Blacksburg, VA 24060, USA
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Dipartimento di ScienzeMineralogiche e Petrologiche, via Valperga Caluso 35 -
10125 Torino (Italy)
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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The stucture of the title compound, (I), is shown below. Dimensions are
available in the archived CIF.

For related literature, see [type here to add references to related
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# Used for convenience to store draft or replaced versions
# of the abstract, comment etc.
# Its contents will not be output
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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^ > 2sigma(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-
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 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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O5 O 0.2269(4) 0.2500 0.3123(7) 0.0110(15) Uiso 1 2 d S . .
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O7 O 0.9907(4) 0.2500 0.1665(8) 0.0144(16) Uiso 1 2 d S . .
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O10 O 0.2633(4) 0.2500 0.0679(8) 0.0141(15) Uiso 1 2 d S . .

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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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Cryst Lab, Dept of Geosciences, Virginia Tech, Blacksburg, VA 24060, USA
;

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Please consider this CIF for publication. I certify that this contibution is the
original work of those listed as authors; that it has not been published
before (in any language or medium) and is not being considered for publication
elsewhere; that all authors concur with and are aware of the submission; that
all workers involved in the study are listed as authors or given proper credit
in the acknowledgements; that I have obtained permission for and acknowledged
the source of any excerpts from other copyright works; and that to the best of
my knowledge the paper contains no statements which are libellous, unlawful or
in any way actionable. All coauthors have made significant scientific
contributions to the work reported, including the ideas and their execution,
and share responsibility and accountability for the results.
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# NB if using publCIF, the Author database tool might prove useful
# (see the Tools menu in publCIF)

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'Angel, Ross J.'
;
Cryst Lab, Dept of Geosciences, Virginia Tech, Blacksburg, VA 24060, USA
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Dipartimento di ScienzeMineralogiche e Petrologiche, via Valperga Caluso 35 -
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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Si Si 0.0817 0.0704 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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Al Al 0.0645 0.0514 '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'
H H 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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'-x, -y, -z'
'x-1/2, y, -z-1/2'
'x, -y-1/2, z'
'-x-1/2, y-1/2, z-1/2'

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_cell_length_b                   5.4609(4)
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_cell_angle_gamma                90.00
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_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^ > 2sigma(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_matrix_type           full
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
'calc w=1/[\s^2^(Fo^2^)+(0.0795P)^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       0.040(4)
_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     57
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O5 O 0.2267(4) 0.2500 0.3110(8) 0.0072(15) Uiso 1 2 d S . .
O6 O 0.2702(4) 0.7500 0.0575(9) 0.0121(15) Uiso 1 2 d S . .
O7 O 0.9893(5) 0.2500 0.1666(9) 0.0185(19) Uiso 1 2 d S . .
O8 O 0.9953(4) 0.7500 0.3024(9) 0.0142(17) Uiso 1 2 d S . .
O9 O 0.4207(6) 0.7500 0.4431(10) 0.028(2) Uiso 1 2 d S . .
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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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T2 O9 1.602(10) 2_564 ?
T2 O1 1.649(5) 7_575 ?
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Dipartimento di ScienzeMineralogiche e Petrologiche, via Valperga Caluso 35 -
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_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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Cryst Lab, Dept of Geosciences, Virginia Tech, Blacksburg, VA 24060, USA
;

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Please consider this CIF for publication. I certify that this contibution is the
original work of those listed as authors; that it has not been published
before (in any language or medium) and is not being considered for publication
elsewhere; that all authors concur with and are aware of the submission; that
all workers involved in the study are listed as authors or given proper credit
in the acknowledgements; that I have obtained permission for and acknowledged
the source of any excerpts from other copyright works; and that to the best of
my knowledge the paper contains no statements which are libellous, unlawful or
in any way actionable. All coauthors have made significant scientific
contributions to the work reported, including the ideas and their execution,
and share responsibility and accountability for the results.
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# NB if using publCIF, the Author database tool might prove useful
# (see the Tools menu in publCIF)

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Cryst Lab, Dept of Geosciences, Virginia Tech, Blacksburg, VA 24060, USA
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Dipartimento di ScienzeMineralogiche e Petrologiche, via Valperga Caluso 35 -
10125 Torino (Italy)
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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