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Fluorophlogopite from Piano delle Concazze (Mt. Etna, Italy): Crystal chemistry and implications for the crystallization conditions

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Abstract

Fluorine is an important proxy for magmatic differentiation processes in the shallow parts of volcanic plumbing systems. Fluorphlogopite is one of the more important fluorine carriers in magmatic rocks. In the present study, a full crystal chemical investigation of fluorophlogopite 1*M* from Piano delle Concazze, Mt. Etna volcano, Italy, is carried out. The fluorophlogopite occurs in a benmoreitic lava from prehistoric volcanic activity at Mt. Etna (post-caldera forming phase of the "Ellittico" eruptive center; ~15 ka BP). It is primarily associated with fluorapatite covered with amorphous SiO_2 and crystallized during syn/post-eruption pneumatolytic stages. The mica sample studied here is among the most Fe- and Ti-rich fluorophlogopite found in nature. EPMA data yielded the following mean chemical formula for this mineral

$$(K_{0.83}Na_{0.13})(Fe^{2+}_{0.44}Fe^{3+}_{0.09}Mg_{2.18}Al_{0.05}Ti_{0.23}Mn_{0.01})(Al_{0.92}Si_{3.08})O_{10.64}(Cl_{0.01}F_{1.35}).$$

Structure refinements on four fluorophlogopite crystals, performed in space group *C*2/*m*, converged at $R = 0.03–0.04$, with cell parameters in the ranges $a = 5.323–5.324$, $b = 9.219–9.222$, $c = 10.116–10.119$ Å, $\beta = 100.1–100.3°$. Major substitutions are $OH^- \leftrightarrow F^-$,M^{3+}-oxy ($^{VI}M^{2+}+OH^- \leftrightarrow {}^{VI}M^{3+}+O^{2-}$) and Ti-oxy substitution: $^{VI}M^{2+}+2(OH)^- \leftrightarrow {}^{VI}Ti^{4+}+2O^{2-}$. The fluorophlogopite from Piano delle Concazze exhibits the shortest *c*-parameter with respect to other fluorophlogopites found in nature. The short *c* parameter is essentially due to the absence of the hydroxyl group in favor of F^- and especially of O^{2-} and to the thus increased attractive interaction between the interlayer cation and the anion content (F^-, O^{2-}) located at the O4 site. A comparison with other natural fluorophlogopites (namely from Biancavilla, Etna and Presidente Olegario, Brazil) show intermediate crystal-chemical features for the Piano delle Concazze fluorophlogopite. Particularly at Etna, differences in the chemical composition of the crystallized fluorophlogopites could be related to the various extent of enrichments by transfer of a gas phase achieved in specific parts of the volcanic plumbing system.

Keywords: Fluorophlogopite, crystal chemistry, substitution mechanisms, IR spectroscopy, volatile transfer

Introduction

Fluorine-rich minerals are important indicators for the halogen activity in magmatic systems. From their investigation relevant inferences for the processes of magma differentiation acting in the shallow portions of the volcano plumbing system can be drawn. The intake of fluorine in micas is mainly controlled by the temperature, the hydrofluoric acid activity during the crystallization and post-crystallization phase, the mica chemical composition and the entropy variation involved in the reactions between mica and hydrothermal solutions (Boukili et al. 2002 and reference therein). Fluorine can be easily incorporated in trioctahedral micas as a substitute for hydroxyl at the O4 anion site. The proton H^+ points almost directly toward the interlayer cation and, consequently, has a negligible interaction with the tetrahedral basal O atoms. The effects of F^--OH^- substitution on the structure stability have attracted the attention of many researchers (Munoz 1984; Robert et al. 1993; Papin et al. 1997; Mason 1992; Boukili et al. 2001; Fechtelkord et al. 2003). The occurrences of F-rich natural micas are rare (Joswig 1972; Hazen and Burnham 1973; Russell and Guggenheim 1999) and several investigations concern synthetic fluorophlogopites (Takeda and Donnay 1966; McCauley et al. 1973; Takeda and Morosin 1975; Toraya et al. 1978, 1983), but only a small number of studies concentrated on natural F-bearing micas. However, recently, Gianfagna et al. (2007) performed a crystal-structure analysis of

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the end-member F-bearing phlogopite from Biancavilla (Etna) that had been approved by the IMA-CNMMN as a new mineral with the name fluorophlogopite, whereas Brigatti et al. (2007) carried out a structural study of a fluorannite emphasizing the structural changes due to F content with respect to annite. Schingaro et al. (2011) studying various F-rich phlogopite from kamafugitic lavas of Presidente Olegario (Brazil) with the highest Ti content in nature and with a minor tetraferriphlogopite component, discuss the mechanisms of Ti and Fe incorporation in the phlogopite structure. On the other hand several phlogopites of volcanic origin with variable oxy component have been investigated recently (Schingaro et al. 2007; Matarrese et al. 2008; Scordari et al. 2010; Lacalamita et al. 2011) to understand how oxy substitutions affect the mica structure.

In the present work we aim at the full characterization of an oxy-fluorophlogopite with the maximum ($F^{-}+O^{2-}$) content (2 apfu) found so far, occurring in benmoreitic lavas from the prehistoric volcanic activity of Mount Etna (~15 ka BP) (Nicotra et al. 2010). This mica is characterized by an F content (F = 1.35 apfu) intermediate between those from Biancavilla (F = 1.90 apfu; Gianfagna et al. 2007) and the Ti-rich fluorophlogopite from Presidente Olegario (F = 0.90 apfu; Schingaro et al. 2011). The main goal is to gain a deeper understanding of the structural effects caused in trioctahedral micas by F^{-}-OH^{-} substitution as well as by O^{2-}-OH^{-} via deprotonation associated with the intake of M^{3+} and Ti^{4+} on the octahedral layer. Another objective of this study is to investigate the relationship between the mineral phase rich in fluorine (fluorophlogopite) in some lavas erupted (15 ka) at Mt. Etna (Sicily, Italy) and the process active into the plumbing system. Due to the known substitutional complexity of micas, a combination of electron probe microanalysis (EPMA), single-crystal X-ray diffraction (SCXRD), and Fourier transform infrared (FTIR) spectroscopy was used. The relationships between the physical and chemical crystallization conditions of such minerals, the role played by volatiles and some geochemical and volcanological aspects are discussed.

Geochemical and volcanological background

Mount Etna is a Quaternary composite volcano characterized by intra-plate magmatism (Viccaro and Cristofolini 2008 and references therein), which has grown up to its present elevation (<3340 m a.s.l.) by accumulation of lavas and tephra throughout the last <500 ka (Gillot et al. 1994). The first eruptive phases at Mt. Etna were characterized by the emission of tholeiitic pillow lavas and hyaloclastites together with transitional subaerial lavas (500–250 ka; Gillot et al. 1994). Most of the volcanic edifice is constituted, however, by Na-alkaline lavas and pyroclastics, emitted during the last 220 ka of activity at Etna (Romano 1982; Branca et al. 2008; Ferlito and Nicotra 2010; Nicotra et al. 2011; Viccaro et al. 2011 and references therein). The relevant volcano-stratigraphic succession is divided into four major phases (Monaco et al. 2011 and references therein), namely: (1) Timpe volcanics (220–100 ka); (2) Valle del Bove volcanics (100–60 ka); (3) Ellittico (60–15 ka); and (4) Recent Mongibello (15 ka to present). Fluorophlogopite crystals studied in this work were found by Nicotra et al. (2010) in a benmoreitic lava (EL85 sample) that belongs to the Ellittico volcanic center. The EL85 lava flow has a thickness of 2 m and lies at the base of a 40 m thick sub-horizontal volcanic sequence in the Piano delle Concazze area (~2790 m a.s.l.; Fig. 1). The EL85 benmoreite, which is one of the most differentiated of the entire Etnean volcanic succession (Corsaro and Cristofolini 1996; Viccaro and Cristofolini 2008; Nicotra et al. 2010, 2011), is characterized by the occurrence of fluorine-rich phases such as fluorophlogopite (grown within lava vesicles) and fluorapatite. Apart from these peculiar accessory phases, phenocrysts of the EL85 volcanic rock are those of the classical mineralogical assemblages at Etna, namely: plagioclase (~10 vol%), augitic clinopyroxene (~5 vol%), Fo_{57-69} olivine (~2 vol%), titaniferous magnetite (~2 vol%). Simulations of crystal fractionation demonstrated that the EL85 compositions have anomalous enrichments in some major and trace elements (e.g., Ti, Fe, K, Ba, and, to a minor extent, Rb and REEs). Furthermore, chlorine and fluorine concentrations (0.20 and 0.34 wt%, respectively) are significantly higher than those of other Etnean prehistoric mugearites and benmoreites. This selective enrichment was interpreted by Nicotra et al. (2010) as due to flushing of volatiles released by more primitive and volatile-rich magmas during their ascent toward shallower levels of the feeding system.

In the present work, four crystals of fluorophlogopite from EL85 were selected to be investigated using a multiple analytical approach.

Experimental methods

Chemical composition

EPMA data of the same crystals used for crystal structure refinement were obtained with a Cameca SX-50 apparatus installed at Istituto di Geologia Ambientale e Geoingegneria (IGAG), Università di Roma "La Sapienza." The analyses

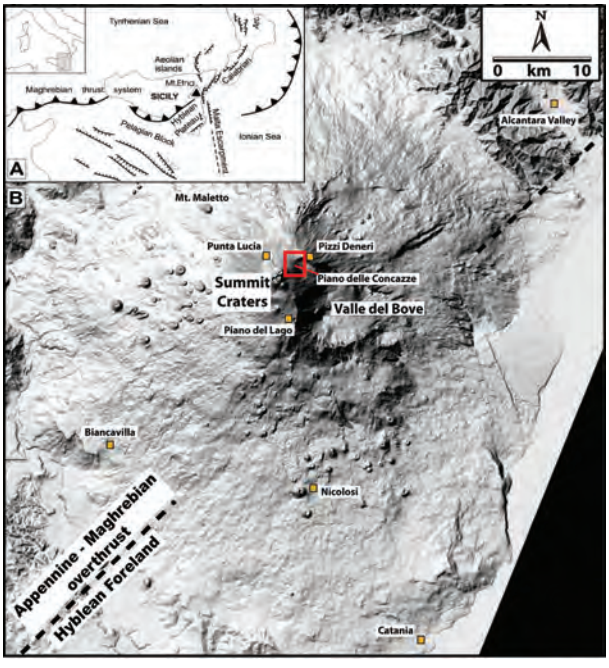

Figure 1. (**a**) Geodynamic framework of Southern Italy with the main domains involved in active tectonics; (**b**) digital elevation model of Mt. Etna volcano (from Monaco et al. 2011) with location of the North-East and South Rift zones. (Color online.)

were performed in wavelength-dispersive spectroscopic (WDS) mode with a 15 kV accelerating voltage, 15 nA beam current, and a 10 μm beam-spot size. The following standards were employed: jadeite (Na), periclase (Mg), wollastonite (Si and Ca), rutile (Ti), corundum (Al), magnetite (Fe), orthoclase (K), barite (Ba), fluorophlogopite (F), and sylvite (Cl). Conversion from X-ray counts to oxide weight percentages (wt%) was obtained with the PAP data reduction method (Pouchou and Pichoir 1985). Chemical composition results (Table 1), obtained from at least eight-point analyses, show that the samples examined are relatively homogeneous, particularly regarding Al_2O_3 (11.0–11.2 wt%), MgO (19.6–20.1 wt%), FeO (8.1–9.1 wt%), TiO_2 (3.8–4.3 wt%), K_2O (8.5–9.1 wt%), Na_2O (0.7–1.1 wt%), and fluorine (5.6–6.0 wt%) contents, in agreement with previous chemical determinations (Nicotra et al. 2010).

Infrared spectroscopy

Room-temperature FTIR measurements were carried out by means of a Nicolet Avatar FTIR spectrometer, equipped with a Continuum microscope, a MCT nitrogen-cooled detector, and a KBr beam splitter. Unpolarized spectra have been acquired in transmission mode on flakes of single crystals mounted on glass capillaries and oriented with the (001) cleavage plane normal to the incident infrared radiation. A final spectrum was collected in the 4000–1400 cm^{-1} range by adding 128 scans with a 4 cm^{-1} nominal resolution.

Single-crystal X-ray diffraction

Four selected crystals (labeled E0, E1, E2, E3 samples) were examined by single-crystal diffraction at room temperature using a Bruker AXS APEX2 diffractometer equipped with a CCD detector and graphite-monochromatized Mo$K\alpha$ radiation (λ = 0.71073 Å). Operating conditions were: 50 kV, 30 mA. For each measurement, three sets of 12 frames were acquired with 0.5° φ rotation and the results were used for the initial unit-cell determinations. The collection strategy was optimized by the Apex program suite (Bruker 2008a); the intensities of reflections in the entire Ewald sphere ($\pm h$, $\pm k$, $\pm l$) were recorded by a combination of ω and φ rotation sets with a 0.5° scan width and exposure times from 10 to 30 s/frame. Only one (E2) out of the four crystals selected for data collection resulted to be a simple 1*M* polytype, whereas the remaining three were found to be 1*M* polytypes twinned by pseudo-merohedry, each twin being composed of three individual components rotated by ±120° around **c***. In the latter case, the orientation matrices of the three components were identified using the program Cell_Now (Bruker 2008b) and the relevant twin laws employed in the structure refinement. The package SAINT-IRIX (Bruker 2008a) was used for data reduction, including intensity integration; the data were corrected for Lorentz-polarization, background effects and scale variation. The final unit-cell parameters were obtained from the *xyz* centroids of the measured reflections after integration and are reported in Table 2, together with details on data collection. A semi-empirical absorption correction based on the determination of transmission factors for equivalent reflections (Blessing 1995) was applied using TWINABS software (Bruker 2008a).

The structure refinements were performed in space group *C*2/*m* using the program CRYSTALS (Betteridge et al. 2003) starting from the atomic parameters reported in Scordari et al. (2006). Scattering curves for fully ionized chemical species were used for non-tetrahedral sites, whereas ionized vs. neutral scattering curves were employed for Si and O (Hawthorne et al. 1995). Reflections with $I > 3\sigma(I)$ were used for the structure refinements. Refined parameters were: atomic positions, cations occupancies, anisotropic atomic displacement parameters, overall scale factor and, whenever appropriate, twin component scale factors. The latter refined to the following values: 0.861, 0.138, 0.001 for sample E0; 0.983, 0.016, 0.001 for sample E1; 0.521, 0.467, 0.012 for sample E3. Accordingly, only sample E3 consisted of two twin components of comparable volume, whereas sample E0 can be described by one predominant twin component one subordinate in volume and E1 has only one predominant twin component. For each crystal the calculated difference-Fourier maps revealed no significant excess in electron density above the background. The slight asymmetry in $\Delta\rho_{min}$-$\Delta\rho_{max}$ values for E0 and E1 samples are likely due to the not optimal modeling of the subordinate twin component. The relevant details of each structure refinement are reported in Table 2. Final atomic coordinates, site occupancies and anisotropic and equivalent isotropic displacement parameters are given in Table 3[1]. Selected bond distances are listed in Table 4, whereas the distortion parameters for micas are reported in Table 5[1]. Mean atomic numbers and octahedral and tetrahedral bond distances are compared to those calculated from chemical analyses using radii from Shannon (1976) and mean anion radii from Kogarko et al. (2005) in Table 6[1].

TABLE 1. Chemical composition from EPMA and atomic proportion (apfu) determined by combining, EPMA, infrared, and structural data

Oxide (wt%)	E0	E1	E2	E3
SiO_2	41.54(43)	41.00(25)	41.58(34)	42.26(34)
TiO_2	4.09(9)	4.29(13)	3.84(9)	3.96(13)
Al_2O_3	10.97(16)	11.03(14)	11.10(15)	11.18(12)
FeO_{tot}	8.21(17)	8.21(17)	9.06(33)	8.12(37)
MnO	0.13(4)	0.13(6)	0.15(4)	0.15(3)
MgO	20.12(32)	19.81(33)	19.55(23)	19.62(58)
BaO	0.05(4)	0.06(5)	0.03(1)	0.04(3)
CaO	–	0.01(1)	0.02(1)	0.01(1)
Na_2O	0.72(8)	0.92(5)	1.11(3)	0.82(20)
K_2O	9.10(18)	8.84(15)	8.52(8)	8.83(38)
F	6.04(17)	5.72(37)	5.59(12)	5.75(38)
Cl	0.09(1)	0.09(1)	0.07(1)	0.08(3)
H_2O*	0.0	0.0	0.0	0.0
Total apfu	101.01	100.11	100.62	100.82
Si	3.08(2)	3.04(3)	3.08(2)	3.13(2)
Al	0.92(2)	0.96(3)	0.92(2)	0.87(2)
ΣT	4.00	4.00	4.00	4.00
Al	0.03(2)	0.01(1)	0.05(2)	0.10(2)
Mg	2.22(3)	2.19(3)	2.16(2)	2.17(5)
Fe^{2+}	0.44(7)	0.39(8)	0.40(4)	0.46(6)
Fe^{3+}	0.07(7)	0.16(8)	0.16(4)	0.04(6)
Ti	0.23(1)	0.24(1)	0.22(1)	0.22(1)
Mn	0.01(0)	0.01(0)	0.01(0)	0.01(0)
ΣOct	3.00	3.00	3.00	3.00
K	0.86(1)	0.84(1)	0.81(1)	0.83(3)
Na	0.10(1)	0.13(1)	0.16(1)	0.12(3)
Ca	–	–	–	–
Ba	–	–	–	–
ΣInt	0.96	0.97	0.97	0.95
OH*	0.0	0.0	0.0	0.0
F	1.41(4)	1.34(8)	1.31(3)	1.35(8)
Cl	0.01(0)	0.01(0)	0.01(0)	0.01(0)
ΣA	1.42	1.35	1.32	1.36

Notes: T = Tetrahedral; Oct = Octahedral; Int = Interlayer; A = Anion. Estimated standard deviations are given in parentheses.
* Estimated from FTIR analysis.

RESULTS AND DISCUSSION

Chemical features

Calculation of the structural formula can be complicated by the concomitant presence of elements with variable valence state (Fe, Ti) and possible occurrence of vacancies in the structure sites (Waters and Charnley 2002; Mesto et al. 2006; Scordari et al. 2008). The crystal-chemical formulas for the refined crystals shown in Table 1 were obtained by combining EPMA data and results of the structure refinement. The formulas were calculated on the basis of 12 (O, OH, F, Cl) total anions and assuming all Ti as Ti^{4+}. The H_2O content was set to zero according to the IR spectrum (Fig. 2), which did not show any typical OH-stretching absorption band near 3700 cm^{-1} [as shown in the inset from a biotite studied by Scordari et al. (2006)].

The Fe^{2+}/Fe^{3+} ratio was varied to obtain a good agreement between X-ref and EPMA derived mean atomic numbers, as well as between <M-O> observed and calculated distances from ionic radii of Shannon (1976).

The above considerations as well as charge-balance requirements lead to the following average structural for-

[1] Deposit item AM-13-046, Tables 3, 5, 6, and CIF. 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 2. Refined cell parameters and data-collection parameters for the samples studied by single-crystal XRD

Sample name	E0	E1	E2	E3
Crystal dimensions (mm)	0.320 x 0.310 x 0.040	0.630 x 0.340 x 0.030	0.400 x 0.270 x 0.050	0.450 x 0.410 x 0.040
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	*C2/m*	*C2/m*	*C2/m*	*C2/m*
Unit-cell dimension *a* (Å)	5.3192(3)	5.3239(3)	5.3200(2)	5.3237(2)
b (Å)	9.2181(5)	9.2204(4)	9.2138(4)	9.2221(3)
c (Å)	10.1096(6)	10.1192(5)	10.1130(4)	10.1159(3)
β (°)	100.168(4)	100.185(3)	100.228(2)	100.249(2)
Volume (Å^3)	487.92(5)	488.91(4)	487.84(3)	488.72(3)
θ range for data collection (°)	2 to 44.6	2 to 44.6	4 to 41.4	2 to 43.8
Index range	$-10 \le h \le 10$	$-10 \le h \le 10$	$-9 \le h \le 9$	$-10 \le h \le 10$
	$0 \le k \le 17$	$0 \le k \le 17$	$-17 \le k \le 17$	$0 \le k \le 17$
	$0 \le l \le 19$	$0 \le l \le 19$	$-17 \le l \le 18$	$0 \le l \le 19$
Reflections collected/ *R* merging [R_{int}] (%)	4265/6.93	4376/5.25	2919/2.43	4244/3.16
Reflections used	1686 with $I > 3\sigma(I)$	2072 with $I > 3\sigma(I)$	1250 with $I > 3\sigma(I)$	2127 with $I > 3\sigma(I)$
No. of refined parameters	71	71	68	71
Goof*	0.77	0.72	0.57	0.70
R_1† (on *F*)/wR_2‡ (on F^2)	0.0385/0.0415	0.0315/0.0324	0.0296/0.0304	0.0267/0.0258
$(\Delta/\sigma)_{max}$	0.016	0.014	0.014	0.014
$\Delta\rho_{min}/\Delta\rho_{max}$ (e$^-$/Å^3)	−1.81/2.39	−1.89/0.66	−0.62/0.71	−0.55/0.58

* Goodness-of-fit = $\{\Sigma[w(F_o^2 - F_c^2)^2]/(N - p)\}^{1/2}$, where *N* and *p* are the number of reflections and parameters, respectively.
† $R_1 = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$.
‡ $wR_2 = \{\Sigma[w(F_o - F_c)^2]/\Sigma[w(F_o)^2]\}^{1/2}$, *w* = Chebyshev optimized weights.

mula $(K_{0.83}Na_{0.13})(Fe^{2+}_{0.44}Fe^{3+}_{0.09}Mg_{2.18}Al_{0.05}Ti_{0.23}Mn_{0.01})(Al_{0.92}Si_{3.08})O_{10.64}(Cl_{0.01}F_{1.35})$. The latter formula as well as those reported in Table 1 evidence that in the analyzed crystals, the tetrahedral site is occupied by Si and Al in a ratio Si/(Si+Al) ~ 0.77; the octahedral sites are mainly occupied by Mg, Fe, and Ti with minor amounts of Al and Mn, and the interlayer site is almost full, with K as the dominant cation, minor Na, and negligible Ba (kinoshitalite component) and vacancy (~0.04 apfu) content. The other notable chemical features of the studied samples are the high-F content, almost constant around 1.35 apfu and the appreciable O^{2-} content (on average 0.64 apfu) that saturate the anionic site. With respect to fluorophlogopite from Biancavilla (Gianfagna et al. 2007), the Piano delle Concazze samples have lower F content and higher Ti and Fe contents, whereas if compared to Presidente Olegario micas (Schingaro et al. 2011) the same samples are oxy-fluorophlogopites with the highest Fe content investigated to date (e.g., Gianfagna et al. 2007 and references therein). The main substitutions in the studied micas are: fluorine hydroxyl substitution [$OH^- \leftrightarrow F^-$], Ti-oxy substitution [$^{VI}M^{2+}+2(OH)^- \leftrightarrow {}^{VI}Ti^{4+}+2(O^{2-})$], with minor M^{3+}-oxy substitution.

Structural features

The cell parameters (Table 2), bond distances (Table 4), and the main structural features (Table 5[1]) of the crystals investigated here display small variations, consistent with the chemical homogeneity of the sample analyzed (see Table 1). In particular, our fluorophlogopite sample is characterized by the smallest *c* parameter (see Fig. 3) recorded to date (Gianfagna et al. 2007; Brigatti and Guggenheim 2002). Neglecting the effect of the mean interlayer radius (see below), the reduction of the *c* parameter can be ascribed essentially to two different substitution mechanisms, both of which in different ways change the H^+-K^+ Coulombic repulsion into the electrostatic attractive interaction between the interlayer cation and the O4 anionic site: (1) the entrance of F in the mica structure through the $OH^- \leftrightarrow F^-$ substitution, and (2) the oxygen deprotonation at the O4 anionic site through $M^{3+,4+}$-oxy substitutions mechanism (Cesare et al. 2003; Scordari et al. 2006 and references therein). The decrease of the *c* parameter in phlogopites upon loss of H^+ is well documented in the literature (Redhammer et al. 2005; Ventruti et al. 2008). The structural effects of the two above substitutions cannot be easily solved, since usually they superimpose or enhance each other. All the selected samples plot in an area delimited by two straight lines: one relevant to the join end-member phlogopite (Redhammer and Roth 2002)—end-member fluorophlogopite (Takeda and Morosin 1975), the other to the join end-member phlogopite (Redhammer et al. 2005)—oxyphlogopite (Cesare et al. 2003). Note that the slope of the latter is steeper than that of the former due to increased K^+-O^{2-} attraction with respect to the K^+-F^- one. We observe that: (1) the investigated sample (F^- = 1.35 apfu; O^{2-} = 0.64 apfu) has a shorter *c* parameter compared to Biancavilla sample (F^- = 1.90 apfu; O^{2-} = 0.00 apfu) due to the enhanced attractive effect of the oxy component; (2) compared to Hazen and Burnham (1973), sample (F^- = 1.30 apfu; O^{2-} = 0.00 apfu), which has almost the same F content as Concazze

TABLE 4. Selected bond distances (Å) (standard deviations are given in parentheses)

	E0	E1	E2	E3
T-O1	1.650(2)	1.647(1)	1.647(1)	1.6482(9)
T-O1′	1.644(2)	1.649(1)	1.648(1)	1.6486(9)
T-O2	1.648(1)	1.6488(7)	1.6479(7)	1.6480(5)
T-O3	1.653(2)	1.655(1)	1.654(1)	1.6550(7)
<T-O>	1.649(2)	1.650(1)	1.649(1)	1.6500(8)
M1-O4 (x2)	2.044(3)	2.047(1)	2.042(1)	2.046(1)
M1-O3 (x4)	2.093(1)	2.0951(9)	2.092(1)	2.0944(7)
<M1-O>	2.077(2)	2.079(1)	2.075(1)	2.078(1)
M2-O4 (x2)	2.008(2)	2.002(1)	2.008(1)	2.0064(7)
M2-O3 (x2)	2.082(2)	2.0820(9)	2.079(1)	2.0795(7)
M2-O3′ (x2)	2.100(2)	2.103(1)	2.101(1)	2.1023(8)
<M2-O>	2.063(2)	2.062(1)	2.063(1)	2.0627(7)
<M-O>	2.068(2)	2.068(1)	2.067(1)	2.068(1)
K-O1 (x4)	3.020(2)	3.020(1)	3.020(1)	3.021(1)
K-O1′ (x4)	3.237(2)	3.239(1)	3.233(1)	3.237(1)
K-O2 (x2)	3.015(3)	3.018(2)	3.015(2)	3.018(1)
K-O2′ (x2)	3.244(3)	3.248(2)	3.244(2)	3.248(1)
$<K\text{-}O>_{inner}$	3.018(2)	3.019(1)	3.018(1)	3.020(1)
$<K\text{-}O>_{outer}$	3.239(2)	3.242(1)	3.237(1)	3.241(1)
<K-O>	3.129(2)	3.131(1)	3.128(1)	3.131(1)

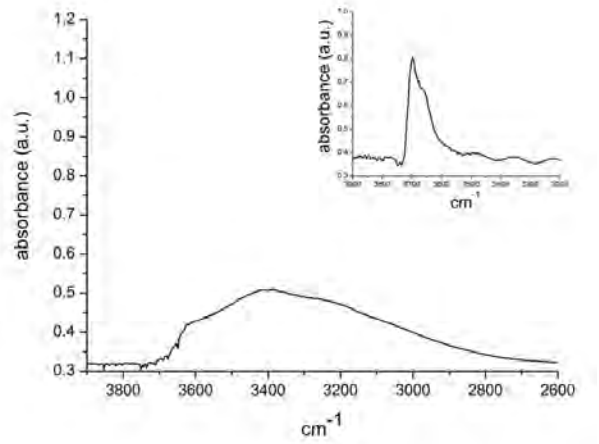

FIGURE 2. FTIR spectrum of a single crystal of Concazze fluorophlogopite recorded in the 3900–2600 cm^{-1} by transmission method. In the inset a typical OH-absorption band of a phlogopite is shown for comparison.

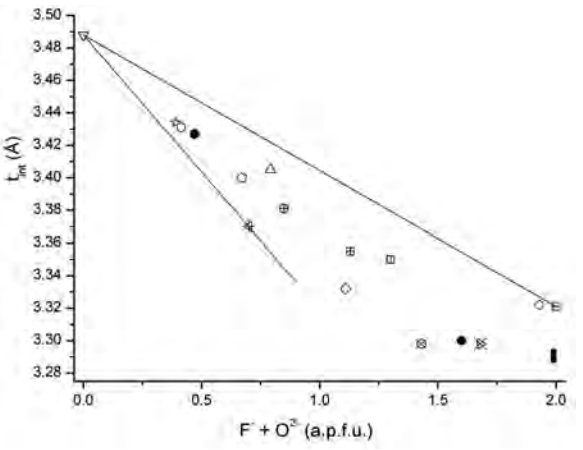

FIGURE 4. Relationship between the interlayer separation (t_{int}) and the $F^{-}+O^{2-}$ content. Solid lines, symbols, and average e.s.d. values as in Figure 3.

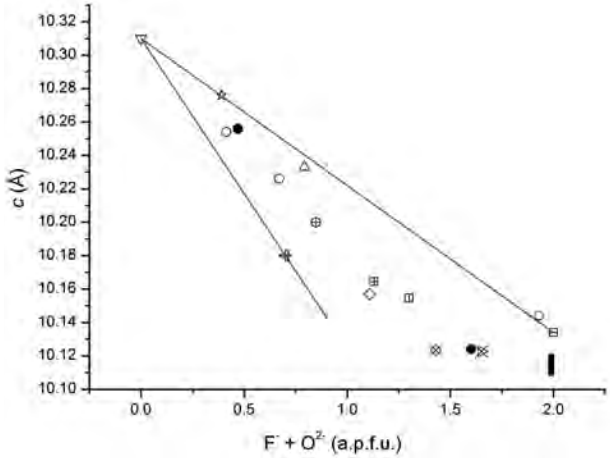

FIGURE 3. Relationship between the *c* parameter and the $F^{-}+O^{2-}$ content. Symbols: solid squares = fluorophlogopites of this study; open circle = fluorophlogopite from Biancavilla (Gianfagna et al. 2007); triangle pointing upward = SA phlogopite characterized by Ti and Fe^{3+}-oxy substitutions (Ventruti et al. 2009); triangle pointing downward = synthetic near end-member phlogopite (Redhammer and Roth 2002); triangle with plus = HO20 F-free, Ti-rich oxyphlogopite (Cesare et al. 2003); triangle with cross = MA1_14 Ti-rich fluorophlogopites from Olegario (Schingaro et al. 2011); diamond = VUT 191_19 phlogopite (Matarrese et al. 2008); pentagon = VUT 187_1 phlogopite (Schingaro et al. 2007); hexagon with plus = VUT 187_18 phlogopite (Schingaro et al. 2007); solid hexagon = PG5_1 phlogopite (Scordari et al. 2010); star = IGNA_24 phlogopite (Lacalamita et al. 2011); solid circle = VUT215_1 phlogopite (Lacalamita et al. 2011); square with plus = fluorophlogopite (Joswig 1972); hexagon with cross = VUT215_8 phlogopite (Lacalamita et al. 2011); square with vertical line = fluorophlogopite (Hazen and Burnham 1973); square with horizontal line = synthetic fluorophlogopite (Takeda and Morosin 1975); hexagon = VUT001_2 phlogopite (Scordari et al. 2010). Solid lines: join end-member phlogopite (Redhammer and Roth 2002)—end-member fluorophlogopite (Takeda and Morosin 1975); join end-member phlogopite (Redhammer et al. 2005)—oxyphlogopite (Cesare et al. 2003).

fluorophlogopite, the further contraction of the *c* parameter may be ascribed to the oxy component which is missing or negligible in the Hazen and Burnham (1973) sample; (3) compared to sample SA (F^{-} = 0.10 apfu; O^{2-} = 0.62 apfu), which has the same oxy concentration as our sample but a low-F content, the extra shortening detected in the Concazze fluorophlogopite is the result of the increased F content.

We also observe that the shortening effect caused by the oxy-substitution is not linear over the whole *c*-range. Indeed the comparison of the samples SA and HO20 (F^{-} = 0.00 apfu; O^{2-} = 0.71 apfu)—at high-*c* values in the figure—shows that, despite the similarity of the F^{-} + O^{2-} content (ΔO^{2-} = 0.09 apfu,) the slight difference in the oxy component leads to a strong reduction of the *c* parameter (Δc = 0.05 Å). On the other hand considering the sample from Takeda and Morosin (1975) (F^{-} = 2.00 apfu; O^{2-} = 0.00 apfu) and from Concazze (F^{-} = 1.35 apfu; O^{2-} = 0.64 apfu, this work)—at low-*c* values in the figure—it is apparent that a large difference in the oxy component (ΔO^{2-} = 0.64 apfu) results in a much smaller reduction of the *c* parameter (Δc = 0.02Å) than the previous case.

The relationship $c\sin\beta = 2\,t_{tet} + t_{oct} + t_{int}$ allows to discriminate the contribution of each sheet thickness on the whole *c* cell parameter. The reduction of the *c* cell parameter is mainly related to the shrinking of the interlayer thickness as illustrated in Figure 4. The effects of the $F^{-}+O^{2-}$ substitutions are better described in this figure, where the sample distribution is slightly different from that in Figure 3. This is due to the fact that the contributions of octahedral and tetrahedral sheets are missing. From the inspection of the Figure 4 the interlayer separation decreases from 3.32 and 3.30 Å, respectively in Biancavilla fluorophlogopite (Gianfagna et al. 2007) and Olegario fluorophlogopite (Schingaro et al. 2011) to about 3.29 Å in our samples. On the other hand, the tetrahedral thickness remains unchanged (2.24 Å) and the octahedral thickness increases slightly from 2.11 Å in the Biancavilla fluorophlogopite (Gianfagna et al. 2007) to 2.12 Å in the Olegario fluorophlogopite (Schingaro et al. 2011) and in the Piano delle Concazze fluorophlogopite of this study. The latter

effect reflects octahedral heterovalent substitutions, particularly those relevant to Fe and Ti contents. The *a* and *b* cell parameters are intermediate between those corresponding to the Biancavilla fluorophlogopite and the Olegario fluorophlogopite but closer to the latter in agreement with the presence of similar heterovalent substitutions. A detailed comparison of selected structural features of fluorophlogopites from Biancavilla and Olegario is given in Table 7. In the Piano delle Concazze fluorophlogopite, the tetrahedron is fairly regular for all of the four analyzed crystals with tetrahedral bond lengths T-O and the tetrahedral volume very close, within one standard deviation (Tables 4 and 5[1]), to the corresponding values found for the fluorophlogopite from Biancavilla (Gianfagna et al. 2007). This is due to the very similar composition of the tetrahedral site [Si/(Si+Al) = 0.8] of the Biancavilla sample. The slight stretching of the tetrahedra along the T-O apical bond distance (O_{basal}-T-O_{apical} ≈ 110.7°, O_{basal}-T-O_{basal} = 108.23°) of the Piano delle Concazze samples compared to that published in Brigatti and Guggenheim (2002) is also related to the high-Si content.

The tetrahedral sheet adapts its lateral dimension along the **a** and **b** axes through mutual rotation of the individual tetrahedra to compensate for the misfit with the octahedral sheet size (Mercier et al. 2005). This misfit, known as the in-plane rotation angle (α), is mainly a function of the octahedral sheet composition and secondarily of the tetrahedral sheet composition, and is the most effective mechanism to obtain congruence between tetrahedral and octahedral sheet sizes in micas. The Piano delle Concazze fluorophlogopite of this study showed a small in-plane rotation angle (α = 4.8°), which is similar to other natural fluorophlogopites and synthetic counterparts (e.g., Gianfagna et al. 2007). Taking into account that the fluorophlogopite from Piano Concazze and fluorophlogopite from Biancavilla are characterized by the same tetrahedral site size, the lower value of the ditrigonal angle α in our sample is due to an increased lateral dimension of the octahedral sheet. In addition to the in-plane rotation angle, α, the tetrahedral sheet corrugation (Δz) also reflects the slight mismatch between tetrahedral and octahedral layers. In regular tetrahedra, this corrugation is accomplished by cooperative tetrahedral tilting due to adjustments of the z-coordinates of the basal O1 atoms, keeping the O2 atoms fixed (Mercier et al. 2005). This parameter was observed to decrease with increasing F content (Brigatti et al. 2007). More generally, the basal corrugation parameter is also related to the geometric

TABLE 7. Selected structural parameters of Biancavilla and Olegario fluorophlogopites

	Biancavilla	Olegario	This study
a	5.3094(4)	5.3275(3)	5.3217(3)
b	9.1933(7)	9.2278(3)	9.2186(4)
c	10.1437(8)	10.1334(2)	10.1144(5)
β	100.062(5)	100.183(3)	100.208(3)
t_{tet} (Å)	2.239	2.242	2.236
<T-O>	1.648(2)	1.652(1)	1.650(1)
$Volume_T$ (Å^3)	2.30	2.31	2.30
τ (°)	110.89	110.8	110.7
α (°)	5.17	4.68	4.83
Δz (Å)	0.003	0.011	0.005
Ψ_{M1} (°)	59.20	59.33	59.29
Ψ_{M2} (°)	59.16	59.03	59.05
<M1-O>	2.064(2)	2.082(1)	2.077(1)
<M2-O>	2.061(2)	2.064(1)	2.063(1)
$Shift_{M2}$ (Å)	0.0016	0.062	0.0256
$Volume_{M1}$ (Å)	11.49	11.79	11.72
$Volume_{M2}$ (Å^3)	11.44	11.51	11.49
t_{oct} (Å)	2.113	2.124	2.122
t_{int} (Å)	3.322	3.298	3.291
$\Delta_{K\text{-}O}$ (Å)	0.235	0.214	0.221

Notes: Standard deviations are given in parentheses. t_{tet}: tetrahedral sheet thickness calculated from z coordinates of basal and apical O atoms; TQE: tetrahedral quadratic elongation calculated as TQE = $\sum_i(l_i/l_0)2/4$ where l_0 is the center to vertex distance for an undistorted tetrahedron (Robinson et al. 1971); TAV: tetrahedral angle variance defined as TAV = $\sum_i(\theta_i - 109.47°)2/5$ (Robinson et al. 1971); τ: tetrahedral flattening angle; α: tetrahedral rotation angle (Hazen and Burnham 1973); Δz: departure from coplanarity of the basal O atoms, calculated as $\Delta z = (z_{o2} - z_{o1})\csin\beta$ (Güven 1971); D.M.: dimensional misfit between tetrahedral and octahedral sheets defined as D.M. = $(2\sqrt{3}$ <O–O>$_{bas}$ – $3\sqrt{2}$ <M–O>) (Toraya 1981); Ψ: octahedral flattening angles (Donnay et al. 1964a, 1964b); BLD: bond-length distortions calculated as BLD = $100/n\cdot\sum_i|(M\text{–}O)_i - \text{<M–O>}|/\text{<M–O>}$ (Renner and Lehmann 1986); ELD: edge-length distortion defined as ELD = $100/n\cdot\sum_i|(O\text{–}O)_i - \text{<O–O>}|/\text{<O–O>}$ (Renner and Lehman 1986); $Shift_{M2}$: off-center shift of the M2 cation defined as the distance between the refined position of cation and the geometrical center of M2 site (coordinates: x/a = 0.0, y/b = 0.8333, z/c = 0.5); OQE: octahedral quadratic elongation calculated as OQE = $\sum_i(l_i/l_0)2/6$ where l_0 is the center to vertex distance for an undistorted octahedron (Robinson et al. 1971); OAV: octahedral angle variance defined as OAV = $\sum_i(\theta_i - 90°)2/11$ (Robinson et al. 1971); e_u,e_s: mean lengths of unshared and shared edges (Toraya 1981), respectively; t_{oct}: octahedral sheet thickness (Toraya 1981); t_{int} calculated from the z coordinates of basal O atoms; ΔK-O = <K-O>$_{outer}$ – <K-O>$_{inner}$; $t_{K\text{-}O4}$: projection of K-O4 distance along c*.

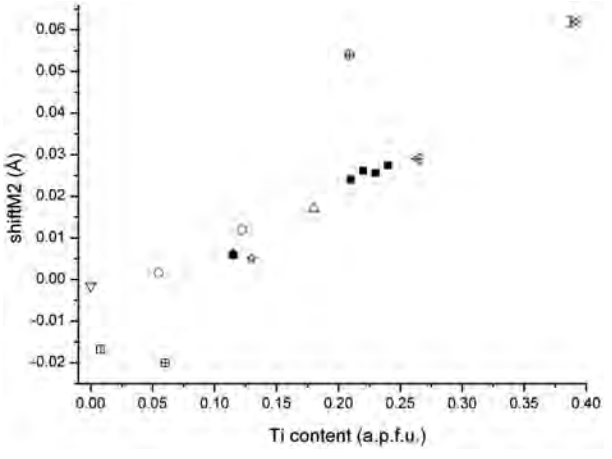

FIGURE 5. Off-center shift of the M2 cation ($shift_{M2}$) vs. Ti content. Symbols and average e.s.d. values as in Figure 3.

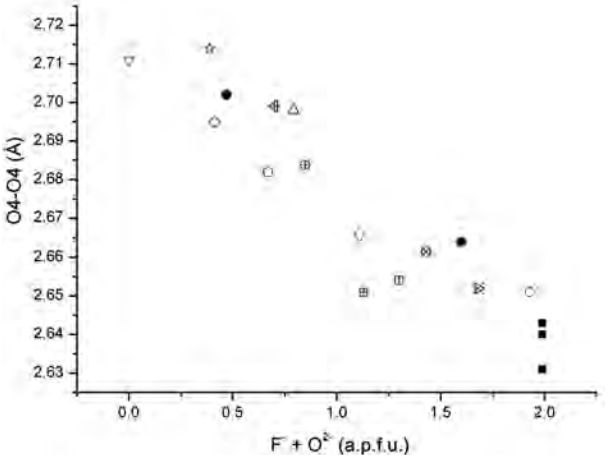

FIGURE 6. O4-O4 bond length vs. the O^{2-}+F^- content. Symbols as in Figure 3.

meso-octahedral character (Brigatti and Guggenheim 2002). This is the case for the Concazze phlogopite, which displays average <M1-O> distances systematically larger than <M2-O> distances (see Table 4) resulting in a meso-octahedral character from a geometric view (Weiss et al. 1985, 1992). Although the differences (see Table 6) in the refined mean atomic numbers (m.a.n.) between M1 and M2 site (see below) suggest that the samples analyzed here are chemically homo-octahedral (Ďurovíč 1994; Ferraris and Ivaldi 2002; Nespolo and Ďurovič 2002), the inspection of octahedral distortion parameters (individual mean bond distances, flattening angles, and off-center shift of the M2 site, Tables 4 and 5[1]) point to some degree of chemical ordering within the octahedral sites.

Specifically the H deficiency and the cationic displacement from the geometric center of the M2 octahedra (Table 5[1]) are clear clues for the occurrence of oxy type substitution mechanisms. This results in the alteration of several structural parameters that, in turn, are employed to identify the occurrence of $M^{3+,4+}$-oxy substitution mechanisms (Schingaro et al. 2005, 2007; Scordari et al. 2006, 2008, 2010; Cesare et al. 2008; Matarrese et al. 2008; Ventruti et al. 2008).

In particular, Figure 5 shows a good correlation between the off-center shift vs. Ti content. The Ti-oxy substitution implies the full partitioning of Ti into the M2 site. The most likely cation partition that can be derived from the above considerations and from the average chemical composition is: $Mg_{0.70}Fe^{2+}_{0.23}Fe^{3+}_{0.01}Al_{0.05}Mn_{0.01}$ for the M1 site, (m.a.n. = 15.54 e^-; <M1-O> = 2.075), and $Mg_{0.74}Fe^{2+}_{0.095}Fe^{3+}_{0.05}Ti_{0.115}$ for the M2 site (m.a.n. = 15.18 e^-; <M2-O> = 2.059).

Other effects associated with the deprotonation of the O4 anion sites are: a move to lower z-values of the O4 oxygen that is responsible for the non coplanarity of the O4 anionic site together with the O3 tetrahedral apical oxygen atom; the decrease of the O4-O4 distance to provide a better screening to the increased positive charge between adjacent M2 sites (Ventruti et al. 2008).

This effect is further enhanced by the F^--OH^- replacement in our sample, as also illustrated in Figures 3 and 4 above. In Figure 6, a clear correlation is apparent between O4-O4 shortening and the increase of the (O^{2-},F^-) content at the anionic site.

Finally, a more regular arrangement of the interlayer site is also observed as a consequence of an increase of the shorter cation-oxygen distances $<K\text{-}O>_{inner}$ and a concomitant decrease of the longer ones, $<K\text{-}O>_{outer}$, that is consistent with the small value of the ditrigonal rotation angle α. The small value of ΔK-O (Table 5[1]) is directly related to the tetrahedral site volume (Fig. 7). This effect is due to the increase of the Si content and the consequent decrease of the tetrahedral bond distances that provide a greater bond valence contribution to the charge balance of the basal oxygen atoms, thus stabilizing the structure. Finally Hawthorne et al. (1999) showed that a monotonic correlation occurs between the *c* parameter and the mean interlayer radius. Actually their correlation does not take into account structural distortion due to the interaction between interlayer and O4 sites. In our sample, the mean interlayer cation radius results into a *c* value considerably smaller than that expected from the analysis in Hawthorne et al. (1999) (see Fig. 8). This is due to the fact that the structural effects caused by the F^--OH^- substitution are enhanced by the concomitant occurrence of the OH^- group deprotonation.

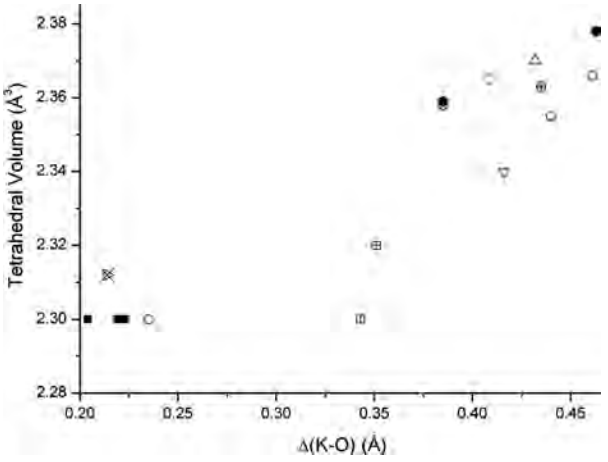

FIGURE 7. Tetrahedral volume vs. the ΔK-O distance. Symbols as in Figure 3.

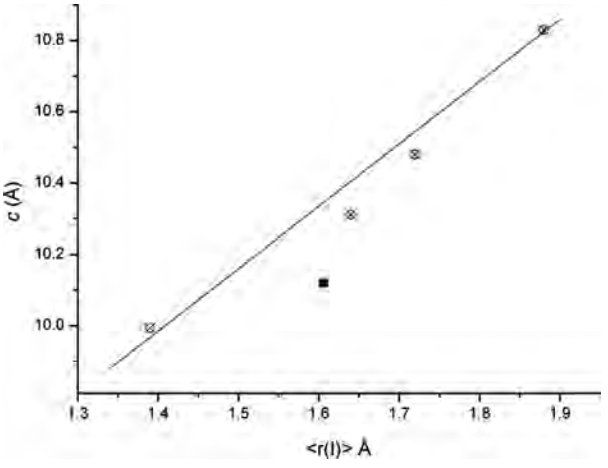

FIGURE 8. Solid line represents linear relation between *c* parameter and mean interlayer cation radius after Hawthorne et al. (1999); circles = phlogopites with different interlayer content from Hawthorne et al. (1999); solid square symbol = Concazze fluorophlogopite.

All the observed trends confirm the strong influence of the anionic site on the overall unit layer topology of the examined fluorophlogopite sample, with particular reference to the structural features measured along the K-O4 direction.

PETROLOGICAL AND VOLCANOLOGICAL IMPLICATIONS

The fluorophlogopite found in the Piano delle Concazze lavas differs from the fluorophlogopite of the Biancavilla lavas mainly by the higher Fe and Ti and the lower OH^- and F^-. These compositional differences should reflect distinct physical-chemical conditions at the time of crystallization, in relation primarily to the amount of available volatiles into the feeding system. As noted by some authors (Nicotra et al. 2010; Mazziotti-Tagliani et al. 2012a, 2012b), the occurrence of the F-rich mineralogical phases (particularly fluorophlogopite) is attributed to volatile flushing of a resident magma reservoir. This mechanism can produce selective transfer of some elements that are carried by

a gas phase, finally leading to anomalous concentrations of elements with great affinity with fluids or that can be complexed (Rittmann 1962; Caroff et al.1997; Greenough et al. 1999; de Hoog and van Bergen 2000; Ferlito et al. 2008; Nicotra et al. 2010). Experimental results confirm the combined action of water and halogens in complexing and carrying elements such as Fe, Ti, K, and P in magmatic systems (Frank et al. 2003; Stelling et al. 2008). The consequent influx of a gas phase enriched in metal-halogen-complexes into a volatile-undersaturated magma may then be viewed as the process leading to selective enrichments in some elements, and particularly halogens, in the residing magma.

In such a picture, differences in the chemical composition of the fluorophlogopites crystallized in products emitted at Piano delle Concazze and Biancavilla could be ascribed to variable degrees of differentiation induced by volatile transfer that was attained in distinct zones of the plumbing system. In this regard, the geometry of the Etnean plumbing system is thought to consist of a dense network of dykes and sills in the central portions (corresponding to the main open-conduit system), which can also coalesce to form shallow magma reservoirs (Ferlito and Nicotra 2010; Nicotra and Viccaro 2012). By contrast, peripheral zones of the volcano plumbing system are more tectonically-controlled, and minor magma intrusions occur. Thus, magma ascent and degassing dynamics in the central conduit and peripheral portions of the edifice are expected to be significantly different. A reason for the higher Fe and Ti and lower OH^- and F^- in the Piano delle Concazze fluorophlogopite (open-conduit system) than in the Biancavilla crystals (peripheral closed system) could be found in a more efficient gas flushing that took part into the central portion of the plumbing system. Furthermore, the lower amount of OH^- and F^- of fluorophlogopite crystals of Piano delle Concazze could be an indication that these lavas experienced, before the eruption, very shallow degassing in the summit portion of the central conduit (<10 MPa, i.e., the exsolution pressure of fluorine in basaltic magmas at Mt. Etna; see Alletti et al. 2009). Conversely, the peripheral closed system erupting at Biancavilla did not allow marked degassing, and the formed gas phase at depth was kept in contact with the magma until the eruption. This leads to a high concentration of H_2O and F in the system, and great availability of OH^- and F^- during the later syn/post eruptive stages of crystallization.

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Atom	x/a	y/b	z/c	Occupancy	U_{iso}	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sample	E0										
K	0	0	0	0.9049(9)	0.0318	0.0331(10)	0.0300(6)	0.0325(6)	0	0.0059(7)	
T[Si]	0.5745(1)	0.16681(5)	0.22369(5)	0.9861(10)	0.0092	0.0086(3)	0.0074(2)	0.0119(2)	0.0001(2)	0.0026(2)	0.0002(
M1[Mg]	0	0.5	0.5	0.7726(8)	0.0098	0.0088(6)	0.0060(3)	0.0154(4)	0	0.0042(4)	
M1[Fe]	0	0.5	0.5	0.2277(7)	0.0098	0.0088(6)	0.0060(3)	0.0154(4)	0	0.0042(4)	
M2[Mg]	0	0.83604(9)	0.5	0.8113(8)	0.0105	0.0074(4)	0.0119(3)	0.0125(3)	0	0.0025(3)	
M2[Fe]	0	0.83604(9)	0.5	0.1889(7)	0.0105	0.0074(4)	0.0119(3)	0.0125(3)	0	0.0025(3)	
O1	0.8176(4)	0.2381(2)	0.1652(2)	1.0000(8)	0.0183	0.0162(10)	0.0225(7)	0.0165(6)	-0.0011(5)	0.0038(7)	-0.0064(
O2	0.5302(6)	0	0.1655(2)	1.0000(8)	0.0186	0.028(2)	0.0119(7)	0.0153(8)	0	0.0012 (9)	
O3	0.6304(3)	0.1675(2)	0.3898(1)	1.0000(8)	0.0100	0.0097(7)	0.0084(4)	0.0125(4)	-0.0001(4)	0.0035(5)	0.0005(
F4	0.1306(5)	0	0.4003(2)	1.0000(8)	0.0129	0.0129(11)	0.0118(6)	0.0147(7)	0	0.0044(8)	
Sample	E1										
K	0	0	0	0.9131(7)	0.0314	0.0314(4)	0.0313(5)	0.0308(4)	0	0.0042(3)	
T[Si]	0.57457(6)	0.16688(4)	0.22366(4)	0.9819(9)	0.0089	0.0079(1)	0.0083(1)	0.0103(1)	0.0000(1)	0.0011(1)	0.0000(
M1[Mg]	0	0.5	0.5	0.7762(8)	0.0094	0.0077(2)	0.0070(2)	0.0136(3)	0	0.0024(2)	
M1[Fe]	0	0.5	0.5	0.2236(6)	0.0094	0.0077(2)	0.0070(2)	0.0136(3)	0	0.0024(2)	
M2[Mg]	0	0.83630(6)	0.5	0.8019(8)	0.0108	0.0066(2)	0.0138(2)	0.0118(2)	0	0.0007(1)	
M2[Fe]	0	0.83630(6)	0.5	0.1980(7)	0.0108	0.0066(2)	0.0138(2)	0.0118(2)	0	0.0007(1)	
O1	0.8170(2)	0.2377(1)	0.1651(1)	1.0000(8)	0.0177	0.0160(4)	0.0231(5)	0.0139(4)	-0.0015(3)	0.0027(3)	-0.0060(
O2	0.5303(3)	0	0.1657(2)	1.0000(8)	0.0179	0.0260(7)	0.0126(5)	0.0139(6)	0	0.0003(5)	
O3	0.6307(2)	0.1676(1)	0.38978(9)	1.0000(8)	0.0103	0.0097(3)	0.0102(3)	0.0107(3)	-0.0002(3)	0.0013(2)	0.0004(
F4	0.1298(2)	0	0.4008(1)	1.0000(8)	0.0128	0.0108(4)	0.0142(5)	0.0134(5)	0	0.0019(4)	

Table 3 Crystallographic coordinates and displacement parameters of the studied micas. In square brackets are quoted the cations used for occupancy refinement

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Atom	x/a	y/b	z/c	Occupancy	U_{iso}	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sample	E2										
K	0	0	0	0.8939(8)	0.0316	0.0317(5)	0.0311(5)	0.0319(5)	0	0.0052(4)	
T[Si]	0.57444(7)	0.16685(4)	0.22370(4)	0.9862(9)	0.0088	0.0086(1)	0.0076(1)	0.0104(2)	0.0000(1)	0.0020(1)	0.0001(
M1[Mg]	0	0.5	0.5	0.7759(8)	0.0092	0.0083(3)	0.0062(3)	0.0138(3)	0	0.0036(2)	
M1[Fe]	0	0.5	0.5	0.2237(6)	0.0092	0.0083(3)	0.0062(3)	0.0138(3)	0	0.0036(2)	
M2[Mg]	0	0.83595(7)	0.5	0.8068(8)	0.0104	0.0069(2)	0.0126(2)	0.0120(2)	0	0.0020(2)	
M2[Fe]	0	0.83595(7)	0.5	0.1932(7)	0.0104	0.0069(2)	0.0126(2)	0.0120(2)	0	0.0020(2)	
O1	0.8166(2)	0.2379(2)	0.1650(1)	1.0000(8)	0.0181	0.0167(5)	0.0227(6)	0.0151(5)	-0.0014(4)	0.0037(4)	-0.0059(
O2	0.5304(4)	0	0.1655(2)	1.0000(8)	0.0182	0.0261(8)	0.0132(6)	0.0145(7)	0	0.0014 (6)	
O3	0.6308(2)	0.1673(1)	0.3899(1)	1.0000(8)	0.0095	0.0088(3)	0.0085(3)	0.0114(4)	-0.0003(3)	0.0024(3)	0.0004(
F4	0.1307(3)	0	0.4004(2)	1.0009(8)	0.0130	0.0111(5)	0.0140(6)	0.0144(6)	0	0.0034(4)	
Sample	E3										
K	0	0	0	0.8970(7)	0.0311	0.0318(3)	0.0312(4)	0.0299(3)	0	0.0048(3)	
T[Si]	0.57472(5)	0.16681(3)	0.22370(3)	0.9808(9)	0.0090	0.0088(1)	0.0083(1)	0.0099(1)	0.0000(1)	0.0017(1)	0.0001(
M1[Mg]	0	0.5	0.5	0.7672(7)	0.0098	0.0087(2)	0.0078(2)	0.0135(2)	0	0.0032(1)	
M1[Fe]	0	0.5	0.5	0.2323(6)	0.0098	0.0087(2)	0.0078(2)	0.0135(2)	0	0.0032(1)	
M2[Mg]	0	0.83616(5)	0.5	0.7932(7)	0.0111	0.0077(1)	0.0140(2)	0.0115(1)	0	0.0014(1)	
M2[Mg]	0	0.83616(5)	0.5	0.2065(6)	0.0111	0.0077(1)	0.0140(2)	0.0115(1)	0	0.0014(1)	
O1	0.8170(2)	0.2379(1)	0.16507(8)	1.0000(8)	0.0187	0.0173(3)	0.0244(4)	0.0145(3)	-0.0016(3)	0.0032(2)	-0.0069(
O2	0.5306(3)	0	0.1658(1)	1.0000(8)	0.0183	0.0272(6)	0.0128(4)	0.0139(4)	0	0.0012(4)	
O3	0.6309(1)	0.16750(8)	0.38995(7)	1.0000(8)	0.0104	0.0105(2)	0.0104(2)	0.0102(4)	0.0001(2)	0.0016(2)	0.0005(
F4	0.1302(2)	0	0.4005(1)	1.0008(7)	0.0129	0.0119(3)	0.0142(4)	0.0127(3)	0	0.0023(3)	

Table 5 Selected distortion parameters of the studied crystals.

	E0	E1	E2	E3
t_{tet} [Å]	2.234	2.236	2.237	2.236
BLD $_T$	0.177	0.134	0.149	0.165
Volume $_T$ [Å^3]	2.30	2.30	2.30	2.30
TQE	1.0005	1.0005	1.0005	1.0005
TAV	1.944	1.948	2.077	1.887
τ [°]	110.66	110.66	110.71	110.64
α [°]	4.83	4.87	4.80	4.81
Δz [Å]	0.003	0.006	0.005	0.007
D.M. [Å]	0.477	0.480	0.478	0.482
Ψ_{M1} [°]	59.25	59.32	59.26	59.32
Ψ_{M2} [°]	59.03	59.04	59.04	59.07
BLD_{M1}	1.043	1.020	1.054	1.044
ELD_{M1}	5.412	5.495	5.423	5.502
BLD_{M2}	1.785	1.945	1.779	1.828
ELD_{M2}	5.153	5.167	5.175	5.202
$Shift_{M2}$ [Å]	0.0250	0.0274	0.0240	0.0261
Volume $_{M1}$ [Å]	11.71	11.75	11.68	11.72
OQE_{M1}	1.013	1.014	1.013	1.014
OAV_{M1}	42.616	44.107	43.018	44.219
Volume $_{M2}$ [Å^3]	11.50	11.49	11.49	11.49
OQE_{M2}	1.012	1.012	1.012	1.013
OAV_{M2}	39.242	39.598	39.476	39.938
e_uM1/ e_sM1	1.114	1.116	1.115	1.117
e_uM2/ e_sM2	1.109	1.109	1.109	1.110
t_{oct} [Å]	2.124	2.122	2.122	2.120
t_{int} [Å]	3.290	3.293	3.288	3.292
Δ_{K-O} [Å]	0.221	0.223	0.219	0.221
t_{K-O4} [Å]	3.920	3.929	3.922	3.923

Note: t_{tet}: tetrahedral sheet thickness calculated from z coordinates of basal and apical O atoms; TQE: tetrahedral quadratic elongation calculated as TQE = $\sum_i(l_i/l_0)^2/4$ where l_0 is the center to vertex distance for an undistorted tetrahedron (Robinson et al. 1971); TAV: tetrahedral angle variance defined as TAV = $\sum_i(\theta_i\text{-}109.47°)^2/5$ (Robinson et al. 1971); τ: tetrahedral flattening angle; α: tetrahedral rotation angle (Hazen and Burnham, 1973); Δz: departure from co planarity of the basal O atoms, calculated as $\Delta z = (z_{o2} - z_{o1})c\sin\beta$ ((Güven 1971)); D.M.: dimensional misfit between tetrahedral and octahedral sheets defined as D.M. = [$2\sqrt{3}$ <O – O > $_{bas}$ - $3\sqrt{2}$ <M – O>] (Toraya 1981); Ψ: octahedral flattening angles (Donnay et al. 1964); BLD: bond-length distortions calculated as BLD = 100/n * $\sum_i$ I(M – O)$_i$ -<M – O> I / <M – O> (Renner and Lehmann 1986); ELD: edge-length distortion defined as ELD = 100/n * $\sum_i$ I(O – O)$_i$ -<O – O> I / <O – O>) (Renner and Lehman 1986); $Shift_{M2}$: off-center shift of the M2 cation defined as the distance between the refined position of cation and the geometrical center of M2 site (coordinates: x/a = 0.0, y/b = 0.8333, z/c = 0.5); OQE: octahedral quadratic elongation calculated as OQE = $\sum_i(l_i/l_0)^2/6$ where l_0 is the center to vertex distance for an undistorted octahedron (Robinson et al. 1971); OAV: octahedral angle variance defined as OAV = $\sum_i(\theta_i\text{-}90°)^2/11$ (Robinson et al. 1971); e_u,e_s: mean lengths of unshared and shared edges (Toraya 1981), respectively; t_{oct}: octahedral sheet thickness (Toraya, 1981); t_{int} calculated from the z coordinates of basal O atoms; Δ_{K-O} = <K-O>$_{outer}$-<K-O>$_{inner}$; t_{K-O4}: projection of K-O4 distance along c*

Table 6 Octahedral cation distribution, mean atomic numbers (m.a.n.s, e^-) of cation sites, octahedral and tetrahedral mean distances, as determined by structure refinement (Xref) and chemical determinations (EMPA). See text for details.

	E0	E1	E2	E3
$e^-_{(M1+M2)X\text{-}ref}$	44.49(3)	44.67(3)	44.53(3)	45.02(2)
$e^-_{(M1+M2)\ EMPA}$	45.60	46.24	46.22	45.43
K $e^-_{X\text{-}ref}$	17.19(2)	17.35(2)	16.98(2)	17.04(1)
K e^-_{EMPA}	17.44	17.39	17.15	17.09
T $e^-_{X\text{-}ref}$	13.81(1)	13.75(1)	13.81(1)	13.73(1)
T e^-_{EMPA}	13.77	13.76	13.77	13.78
Σ^+	22.60	22.66	22.70	22.66
Σ^-	22.58	22.65	22.68	22.64
$\langle T\text{-}O\rangle_{X\text{-}ref}$	1.649(2)	1.650(1)	1.649(1)	1.650(1)
$\langle T\text{-}O\rangle_{EMPA}$	1.655	1.655	1.655	1.654
$\langle M\text{-}O\rangle_{X\text{-}ref}$	2.068(2)	2.068(1)	2.067(1)	2.068(1)
$\langle M\text{-}O\rangle_{EMPA}$	2.067	2.065	2.064	2.063

Scordari et al.: FLUOROPHLOGOPITE FROM PIANO DELLE CONCAZZE (Mt. ETNA, ITALY);
Deposit material (7 total items); AM-13-046; American Mineralogist May June 2013

```
data_global
_audit_creation_date        "11-02-12"
_audit_creation_method CRYSTALS_ver_14.06

_oxford_structure_analysis_title  'THIS IS THE DEFAULT SETTING FOR THE TITLE, P'
_chemical_name_systematic         ?
_chemical_melting_point           ?

#looking for refcif


# Check this file using the IUCr facility at:
# http://checkcif.iucr.org/

# The content below is held in the file 'script/refcif.dat'. This is a text
# file which you may edit to reflect local conditions.
# Items which need looking at are represented by a '?'.
# Items for which there are choices are prefixed with 'choose from'.

_publ_contact_letter
;
 Please consider this CIF submission for
 publication as a Short Format Paper in Acta
 Crystallographica E.
;
_publ_contact_author_name         'Anthony Other'
_publ_contact_author_address
;
 Chemical Crystallography Laboratory,
 Department of Chemistry,
 University of Nowhere,
 Nowhere, NO1 4T, UK.
;

_publ_contact_author_phone        '+44 1865 000000'
_publ_contact_author_fax          '+44 1865 000000'
_publ_contact_author_email        'a.n.other@chem.no.ac.uk'
_publ_requested_journal           'Section E'
_publ_requested_category          EO # choose from: FI FM FO CI CM CO AD
_publ_requested_coeditor_name     'Prof William Clegg'

_publ_section_title
# Title of paper - generally just the systematic or trivial name
; ?
;

# The loop structure below should contain the names and addresses of all
# authors, in the required order of publication. Repeat as necessary.
loop_
 _publ_author_name
 _publ_author_address
 _publ_author_footnote
```

```
 'Other, Anthony N.' # Author 1
;
 ? # Address for author 1
;
;
 ? # Footnote for author 1
;
 'Else, S. O.' # Author 2
;
 ? # Address 2
;
;
 ? # Footnote 2
;


_publ_section_abstract
# Text of the abstract
# (a) The Abstract must be self-contained and comprehensible
# without the rest of the paper. This means no references
# to atom names or to compound numbers; compounds must be
# identified as the title compound, or by name or by some other
# means such as derivatives of each other (e.g. "the
# corresponding ketone").
# (b) The chemical formula of "the title compound" must be given.
# (c) Any crystallographic molecular symmetry should be
# mentioned, and also the presence of more than one molecule
# in the asymmetric unit (i.e. anything other than Z'=1).
;
?
;

_publ_section_related_literature  # Not in version 2.3.1
##################################################################
# Essential references (e.g. to the origin of the material studied, related
# structural studies, and to information supporting the reported structure)
# should be cited in this section, with appropriate very brief explanatory
# text, for example `The synthesis of the complex is described by Jones
# (2001). The anhydrous complex has essentially the same coordination
# geometry (Smith, 2003).' In addition, all references included in the
# supplementary materials, but not elsewhere in the published article,
# should be given here. The simple text `For related literature, see:...'
# may be used, but further details should be provided in the Comment
# section, which forms part of the supplementary material.
##################################################################
;
?
;

_publ_section_comment
# Text of the paper
# Note that atoms are referenced as N2, not N(2) or N~2~
# If text containing () occur within (), the outer ones should be []
# Figures should be referenced as Fig.
;
```

```
?
;
_publ_section_acknowledgements    # Acknowledgments
;
?
;
_publ_section_figure_captions
# Captions to figures - Start each caption on a new line after a blank line
;
Fig. 1.
The title compound with displacement ellipsoids drawn at the 50%
probability level. H atoms are shown as spheres of
arbitary radius.

;


_publ_section_exptl_refinement
# Some potentially useful phrases are donated by Bill Clegg:
;
In the absence of significant anomalous scattering, Friedel pairs were
merged.

The absolute configuration was arbitrarily assigned.

The relatively large ratio of minimum to maximum corrections applied
in the multiscan process (1:nnn) reflect changes in the illuminated
volume of the crystal.

Changes in illuminated volume were kept to a minimum, and were
taken into account (G\"orbitz, 1999) by the multi-scan inter-frame
scaling (DENZO/SCALEPACK, Otwinowski & Minor, 1997).


G\"orbitz, C. H. (1999). Acta Cryst. B55, 1090-1098.




The H atoms were all located in a difference map, but those
attached to carbon atoms were repositioned geometrically.
The H atoms were initially refined with soft restraints on the
bond lengths and angles to regularise their geometry
(C---H in the range 0.93--0.98,
N---H in the range 0.86--0.89
N---H to 0.86
O---H = 0.82
\%A)
and
U~iso~(H) (in the range 1.2-1.5 times U~eq~ of the parent atom),
after which the positions were refined with riding constraints.

;

_publ_section_exptl_prep
```

```
# Brief details or a reference. Include solvent if known
;
?
;

# Hand-made tables can be put in the cif. The number of columns
# is set in the loop header.
# The contants of each column can either be a piece of text
# without any spaces (eg a number) or other text enclosed in " "
# Remove the leading # in the following example

#geom_extra_table_head_A
#;
#Table 2.
#Molecular parameters deviating most from MOGUL averages
#;
#
#loop_
#_geom_extra_tableA_col_1
#_geom_extra_tableA_col_2
#_geom_extra_tableA_col_3
#_geom_extra_tableA_col_4
#
#Parameter "Observed Value" "MOGUL Value" "MOGUL e.s.d"
#N5-C6-C4 129 124 "7 (\%)"
#C3-O10-C2 105 109 "2 (\%)"
#C6-O7 1.25 1.22 ".02 (\%A)"
#

data_1

_exptl_special_details
;
 The crystal was
placed in the cold stream of an Oxford
Cryosystems open-flow nitrogen cryostat (Cosier &
Glazer, 1986) with a nominal stability of 0.1K.

Cosier, J. & Glazer, A.M., 1986. J. Appl. Cryst. 105 107.
;
_refine_special_details
;
?
;
# End of 'script/refcif.dat'
#end of refcif
_cell_length_a                    5.3192(3)
_cell_length_b                    9.2181(5)
_cell_length_c                    10.1096(6)
_cell_angle_alpha                 90
_cell_angle_beta                  100.168(4)
_cell_angle_gamma                 90
_cell_volume                      487.92(5)

_symmetry_cell_setting            'Monoclinic'
```

```
_symmetry_space_group_name_H-M    'C 1 2/m 1 '
_symmetry_space_group_name_Hall   ?
loop_
 _symmetry_equiv_pos_as_xyz
 'x,y,z'
 '-x,-y,-z'
 'x+1/2,y+1/2,z'
 '-x+1/2,-y+1/2,-z'
 '-x,y,-z'
 'x,-y,z'
 '-x+1/2,y+1/2,-z'
 'x+1/2,-y+1/2,z'

loop_
_atom_type_symbol
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_Cromer_Mann_a1
_atom_type_scat_Cromer_Mann_b1
_atom_type_scat_Cromer_Mann_a2
_atom_type_scat_Cromer_Mann_b2
_atom_type_scat_Cromer_Mann_a3
_atom_type_scat_Cromer_Mann_b3
_atom_type_scat_Cromer_Mann_a4
_atom_type_scat_Cromer_Mann_b4
_atom_type_scat_Cromer_Mann_c
_atom_type_scat_source
 H       0.0000   0.0000   0.4930  10.5109   0.3229  26.1257   0.1402   3.1424
   0.0408  57.7997   0.0030 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Lip1    0.0000   0.0000   0.6968   4.6237   0.7888   1.9557   0.3414   0.6316
   0.1563  10.0953   0.0167 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fep2    0.3010   0.8450  11.0424   4.6538   7.3740   0.3053   4.1346  12.0546
   0.4399  31.2809   1.0097 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Cap2    0.2030   0.3060  15.6348  -0.0074   7.9518   0.6089   8.4372  10.3116
   0.8537  25.9905 -14.8750 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Nap1    0.0300   0.0250   3.2565   2.6671   3.9362   6.1153   1.3998   0.2001
   1.0032  14.0390   0.4040 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Kp1     0.1790   0.2500   7.9578  12.6331   7.4917   0.7674   6.3590  -0.0020
   1.1915  31.9128  -4.9978 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Mgp2    0.0420   0.0360   3.4988   2.1676   3.8378   4.7542   1.3284   0.1850
   0.8497  10.1411   0.4853 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fep3    0.3010   0.8450  11.1764   4.6147   7.3863   0.3005   3.3948  11.6729
   0.0724  38.5566   0.9707 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Alp3    0.0560   0.0520   4.1745   1.9382   3.3876   4.1455   1.2030   0.2288
   0.5281   8.2852   0.7068 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Crp3    0.2840   0.6240   9.6809   5.5946   7.8114   0.3344   2.8760  12.8288
   0.1136  32.8761   0.5183 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Zrp4   -2.9650   0.5600  18.1668   1.2148  10.0562  10.1483   1.0112  21.6054
  -2.6479  -0.1028   9.4145 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Tip4    0.2480   0.4460  19.5114   0.1789   8.2347   6.6702   2.0134  -0.2926
   1.5208  12.9464 -13.2800 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Si      0.0720   0.0710   6.2915   2.4386   3.0353  32.3337   1.9891   0.6785
   1.5410  81.6937   1.1407 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Sip4    0.0720   0.0710   4.4392   1.6417   3.2034   3.4376   1.1945   0.2149
   0.4165   6.6536   0.7463 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
```

```
 O       0.0080   0.0060   3.0485  13.2771   2.2868   5.7011   1.5463   0.3239
   0.8670  32.9089   0.2508 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Om2     0.0080   0.0060   3.7504  16.5151   2.8429   6.5920   1.5430   0.3192
   1.6209  43.3486   0.2421 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Om1     0.0080   0.0060   4.1916  12.8573   1.6397   4.1724   1.5267  47.0179
 -20.3070  -0.0140  21.9412 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 F       0.0140   0.0100   3.5392  10.2825   2.6412   4.2944   1.5170   0.2615
   1.0243  26.1476   0.2776 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fm1     0.0140   0.0100   3.6322   5.2776   3.5106  14.7353   1.2606   0.4423
   0.9407  47.3437   0.6534 'International Tables Vol C 4.2.6.8 and 6.1.1.4'


_cell_formula_units_Z                  8

# Given Formula = H1 Alp30.06 Cap20.24 Crp30.01 F0.50 Fep20.01 Fep30.10 Fm10.50
Kp10.50 Lip10.50
# Dc =      2.59 Fooo =    320.06 Mu =     50.60 M =     95.25
# Found Formula = Fep20.06 Fm10.16 Kp10.06 Mgp20.25 O1.09 Om21.25 Si0.58
Sip40.41
# Dc =      2.95 FOOO =    320.06 Mu =     49.69 M =    108.49

_chemical_formula_sum             'Fep20.06 Fm10.16 Kp10.06 Mgp20.25 O1.09
Om21.25 Si0.58 Sip40.41'
_chemical_formula_moiety          'Fep20.06 Fm10.16 Kp10.06 Mgp20.25 O1.09
Om21.25 Si0.58 Sip40.41'
_chemical_compound_source              ?
_chemical_formula_weight           108.49


_cell_measurement_reflns_used          0
_cell_measurement_theta_min            0
_cell_measurement_theta_max            0
_cell_measurement_temperature        293

_exptl_crystal_description        '?'
_exptl_crystal_colour             '?'
_exptl_crystal_size_min           ?
_exptl_crystal_size_mid           ?
_exptl_crystal_size_max           ?

_exptl_crystal_density_diffrn     2.954
_exptl_crystal_density_meas       ?
_exptl_crystal_density_method     'not measured'
# Non-dispersive F(000):
_exptl_crystal_F_000              320.058
_exptl_absorpt_coefficient_mu     4.969

# Sheldrick geometric approximatio 1.00 1.00
_exptl_absorpt_correction_type    none
_exptl_absorpt_correction_T_min   1.0000
_exptl_absorpt_correction_T_max   1.0000
_diffrn_measurement_device_type   'Unknown'
_diffrn_measurement_device        'Serial'
_diffrn_radiation_monochromator   'graphite'
_diffrn_radiation_type            'Mo K\a'
```

```
_diffrn_radiation_wavelength           0.71070
_diffrn_measurement_method        \w/2\q

# If a reference occurs more than once, delete the author
# and date from subsequent references.
_computing_data_collection        'USER DEFINED DATA COLLECTION'
_computing_cell_refinement        'USER DEFINED CELL REFINEMENT'
_computing_data_reduction         'USER DEFINED DATA REDUCTION'
_computing_structure_solution     'USER DEFINED STRUCTURE SOLUTION'
_computing_structure_refinement   'CRYSTALS (Betteridge et al., 2003)'
_computing_publication_material   'CRYSTALS (Betteridge et al., 2003)'
_computing_molecular_graphics     'CAMERON (Watkin et al., 1996)'

_diffrn_standards_interval_time   .
_diffrn_standards_interval_count  .
_diffrn_standards_number          0
_diffrn_standards_decay_%         ?

_diffrn_ambient_temperature        293
_diffrn_reflns_number             4265
_reflns_number_total              1647
_diffrn_reflns_av_R_equivalents   0.069
# Number of reflections without Friedels Law is 1647
# Number of reflections with Friedels Law is 0
# Theoretical number of reflections is about 3931


_diffrn_reflns_theta_min          2.047
_diffrn_reflns_theta_max          44.578
_diffrn_measured_fraction_theta_max 0.805

_diffrn_reflns_theta_full         28.084
_diffrn_measured_fraction_theta_full 0.997


_diffrn_reflns_limit_h_min        -10
_diffrn_reflns_limit_h_max        10
_diffrn_reflns_limit_k_min        0
_diffrn_reflns_limit_k_max        17
_diffrn_reflns_limit_l_min        0
_diffrn_reflns_limit_l_max        19
_reflns_limit_h_min               -10
_reflns_limit_h_max               10
_reflns_limit_k_min               0
_reflns_limit_k_max               17
_reflns_limit_l_min               0
_reflns_limit_l_max               19

_oxford_diffrn_Wilson_B_factor    0.00
_oxford_diffrn_Wilson_scale       0.00

_atom_sites_solution_primary      direct #heavy,direct,difmap,geom
# _atom_sites_solution_secondary  difmap
_atom_sites_solution_hydrogens    geom
```

```
_refine_diff_density_min         -1.81
_refine_diff_density_max         2.39


# The current dictionary definitions do not cover the
# situation where the reflections used for refinement were
# selected by a user-defined sigma threshold


# The values actually used during refinement
_oxford_reflns_threshold_expression_ref     I>3.0\s(I)
_refine_ls_number_reflns         1686
_refine_ls_number_restraints     24
_refine_ls_number_parameters     71
_oxford_refine_ls_R_factor_ref   0.0385
_refine_ls_wR_factor_ref         0.0415
_refine_ls_goodness_of_fit_ref   0.7699
_refine_ls_shift/su_max          0.0164582
_refine_ls_shift/su_mean         0.0014770



# choose from: rm (reference molecule of known chirality),
# ad (anomolous dispersion - Flack), rmad (rm and ad),
# syn (from synthesis), unk (unknown) or . (not applicable).
_chemical_absolute_configuration  '.'


_refine_ls_structure_factor_coef  F
_refine_ls_matrix_type            full
_refine_ls_hydrogen_treatment     none          # none, undef, noref, refall,
                                                # refxyz, refU, constr or mixed

# WARNING. The IUCr will not accept Unit Weights
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
;
 Method= Quasi-Unit weights
 W = 1.0 or 1./2F
;
# Insert your own references if required - in alphabetical order
_publ_section_references
;
User-defined structure solution reference

User-defined data collection reference

User defined data reduction

User defined cell refinement

Betteridge, P.W., Carruthers, J.R., Cooper, R.I.,
Prout, K. & Watkin, D.J. (2003). J. Appl. Cryst. 36, 1487.

Watkin, D.J., Prout, C.K. & Pearce, L.J. (1996). CAMERON, Chemical
```

```
Crystallography Laboratory, Oxford, UK.
;

# Uequiv = arithmetic mean of Ui i.e. Ueqiv = (U1+U2+U3)/3

# Replace last . with number of unfound hydrogen atomsattached to an atom.

# ..._refinement_flags_...
# . no refinement constraints          S special position constraint on site
# G rigid group refinement of site     R riding atom
# D distance or angle restraint on site T thermal displacement constraints
# U Uiso or Uij restraint (rigid bond)  P partial occupancy constraint


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_occupancy
_atom_site_adp_type
_atom_site_refinement_flags_posn
_atom_site_refinement_flags_adp
_atom_site_refinement_flags_occupancy
_atom_site_disorder_assembly
_atom_site_disorder_group
_oxford_atom_site_special_shape
_atom_site_attached_hydrogens
Kp11 Kp1 0.0000 0.5000 0.0000 0.0318 0.9049(9) Uani . . P . . . .
Mgp22 Mgp2 0.0000 0.0000 0.5000 0.0098 0.7726(8) Uani . . P . . . .
Fep22 Fep2 0.0000 0.0000 0.5000 0.0098 0.2277(7) Uani . . P . . . .
Mgp23 Mgp2 0.0000 0.33604(9) 0.5000 0.0105 0.8113(8) Uani . . P . . . .
Fep23 Fep2 0.0000 0.33604(9) 0.5000 0.0105 0.1889(7) Uani . . P . . . .
Sip44 Sip4 0.07452(13) 0.16681(5) 0.22369(5) 0.0092 0.4055(10) Uani . . P . . .
.
Si4 Si 0.07452(13) 0.16681(5) 0.22369(5) 0.0092 0.5806(10) Uani . . P . . . .
Om25 Om2 0.3176(4) 0.2381(2) 0.16523(16) 0.0183 0.5606(8) Uani . . P . . . .
O5 O 0.3176(4) 0.2381(2) 0.16523(16) 0.0183 0.4394(8) Uani . . P . . . .
Om26 Om2 0.0302(6) 0.0000 0.1655(2) 0.0186 0.1529(8) Uani . . P . . . .
O6 O 0.0302(6) 0.0000 0.1655(2) 0.0186 0.8471(8) Uani . . P . . . .
Om27 Om2 0.1304(3) 0.16751(15) 0.38981(13) 0.0100 0.5609(8) Uani . . P . . . .
O7 O 0.1304(3) 0.16751(15) 0.38981(13) 0.0100 0.4391(8) Uani . . P . . . .
Fm18 Fm1 0.1306(5) 0.5000 0.40027(19) 0.0129 0.6313(8) Uani . . P . . . .
Om28 Om2 0.1306(5) 0.5000 0.40027(19) 0.0129 0.3687(8) Uani . . P . . . .
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
Kp11 0.0331(10) 0.0300(6) 0.0325(6) 0.0000 0.0059(7) 0.0000
```

```
Mgp22 0.0088(6) 0.0060(3) 0.0154(4) 0.0000 0.0042(4) 0.0000
Fep22 0.0088(6) 0.0060(3) 0.0154(4) 0.0000 0.0042(4) 0.0000
Mgp23 0.0074(4) 0.0119(3) 0.0125(3) 0.0000 0.0025(3) 0.0000
Fep23 0.0074(4) 0.0119(3) 0.0125(3) 0.0000 0.0025(3) 0.0000
Sip44 0.0086(3) 0.00737(16) 0.01191(18) -0.00006(16) 0.00261(19) -0.00016(19)
Si4 0.0086(3) 0.00737(16) 0.01191(18) -0.00006(16) 0.00261(19) -0.00016(19)
Om25 0.0162(10) 0.0225(7) 0.0165(6) -0.0011(5) 0.0038(7) -0.0064(6)
O5 0.0162(10) 0.0225(7) 0.0165(6) -0.0011(5) 0.0038(7) -0.0064(6)
Om26 0.0278(16) 0.0119(7) 0.0153(8) 0.0000 0.0012(9) 0.0000
O6 0.0278(16) 0.0119(7) 0.0153(8) 0.0000 0.0012(9) 0.0000
Om27 0.0097(7) 0.0084(4) 0.0125(4) -0.0001(4) 0.0035(5) 0.0005(5)
O7 0.0097(7) 0.0084(4) 0.0125(4) -0.0001(4) 0.0035(5) 0.0005(5)
Fm18 0.0129(11) 0.0118(6) 0.0147(7) 0.0000 0.0044(8) 0.0000
Om28 0.0129(11) 0.0118(6) 0.0147(7) 0.0000 0.0044(8) 0.0000

_refine_ls_extinction_method
    'None'
_oxford_refine_ls_scale 0.592(3)

loop_
    _oxford_twin_element_scale_factors
         0.861(9)
         0.138(5)
         0.001(1)
loop_
 _geom_bond_atom_site_label_1
 _geom_bond_site_symmetry_1
 _geom_bond_atom_site_label_2
 _geom_bond_site_symmetry_2
 _geom_bond_distance
 _geom_bond_publ_flag
Mgp22 . Om28 3_445 2.044(3)    yes
Mgp22 . Om28 4_556 2.044(3)    yes
Mgp22 . Fm18 3_445 2.044(3)    yes
Mgp22 . Fm18 7_546 2.044(3)    yes
Mgp22 . O7 6_555 2.0926(14)    yes
Mgp22 . O7 5_556 2.0926(14)    yes
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Scordari et al.: FLUOROPHLOGOPITE FROM PIANO DELLE CONCAZZE (Mt. ETNA, ITALY);
Deposit material (7 total items); AM-13-046; American Mineralogist May June 2013

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_publ_section_related_literature  # Not in version 2.3.1
##################################################################
# Essential references (e.g. to the origin of the material studied, related
# structural studies, and to information supporting the reported structure)
# should be cited in this section, with appropriate very brief explanatory
# text, for example `The synthesis of the complex is described by Jones
# (2001). The anhydrous complex has essentially the same coordination
# geometry (Smith, 2003).' In addition, all references included in the
# supplementary materials, but not elsewhere in the published article,
# should be given here. The simple text `For related literature, see:...'
# may be used, but further details should be provided in the Comment
# section, which forms part of the supplementary material.
##################################################################
;
?
;

_publ_section_comment
# Text of the paper
# Note that atoms are referenced as N2, not N(2) or N~2~
# If text containing () occur within (), the outer ones should be []
# Figures should be referenced as Fig.
;
```

```
?
;
_publ_section_acknowledgements    # Acknowledgments
;
?
;
_publ_section_figure_captions
# Captions to figures - Start each caption on a new line after a blank line
;
Fig. 1.
The title compound with displacement ellipsoids drawn at the 50%
probability level. H atoms are shown as spheres of
arbitary radius.

;


_publ_section_exptl_refinement
# Some potentially useful phrases are donated by Bill Clegg:
;
In the absence of significant anomalous scattering, Friedel pairs were
merged.

The absolute configuration was arbitrarily assigned.

The relatively large ratio of minimum to maximum corrections applied
in the multiscan process (1:nnn) reflect changes in the illuminated
volume of the crystal.

Changes in illuminated volume were kept to a minimum, and were
taken into account (G\"orbitz, 1999) by the multi-scan inter-frame
scaling (DENZO/SCALEPACK, Otwinowski & Minor, 1997).


G\"orbitz, C. H. (1999). Acta Cryst. B55, 1090-1098.




The H atoms were all located in a difference map, but those
attached to carbon atoms were repositioned geometrically.
The H atoms were initially refined with soft restraints on the
bond lengths and angles to regularise their geometry
(C---H in the range 0.93--0.98,
N---H in the range 0.86--0.89
N---H to 0.86
O---H = 0.82
\%A)
and
U~iso~(H) (in the range 1.2-1.5 times U~eq~ of the parent atom),
after which the positions were refined with riding constraints.

;

_publ_section_exptl_prep
```

```
# Brief details or a reference. Include solvent if known
;
?
;

# Hand-made tables can be put in the cif. The number of columns
# is set in the loop header.
# The contants of each column can either be a piece of text
# without any spaces (eg a number) or other text enclosed in " "
# Remove the leading # in the following example

#geom_extra_table_head_A
#;
#Table 2.
#Molecular parameters deviating most from MOGUL averages
#;
#
#loop_
#_geom_extra_tableA_col_1
#_geom_extra_tableA_col_2
#_geom_extra_tableA_col_3
#_geom_extra_tableA_col_4
#
#Parameter "Observed Value" "MOGUL Value" "MOGUL e.s.d"
#N5-C6-C4 129 124 "7 (\%)"
#C3-O10-C2 105 109 "2 (\%)"
#C6-O7 1.25 1.22 ".02 (\%A)"
#

data_1

_exptl_special_details
;
 The crystal was
placed in the cold stream of an Oxford
Cryosystems open-flow nitrogen cryostat (Cosier &
Glazer, 1986) with a nominal stability of 0.1K.

Cosier, J. & Glazer, A.M., 1986. J. Appl. Cryst. 105 107.
;
_refine_special_details
;
?
;
# End of 'script/refcif.dat'
#end of refcif
_cell_length_a                   5.3239(3)
_cell_length_b                   9.2204(4)
_cell_length_c                   10.1192(5)
_cell_angle_alpha                90
_cell_angle_beta                 100.185(3)
_cell_angle_gamma                90
_cell_volume                     488.91(4)

_symmetry_cell_setting           'Monoclinic'
```

```
_symmetry_space_group_name_H-M    'C 1 2/m 1 '
_symmetry_space_group_name_Hall   ?
loop_
 _symmetry_equiv_pos_as_xyz
 'x,y,z'
 '-x,-y,-z'
 'x+1/2,y+1/2,z'
 '-x+1/2,-y+1/2,-z'
 '-x,y,-z'
 'x,-y,z'
 '-x+1/2,y+1/2,-z'
 'x+1/2,-y+1/2,z'

loop_
_atom_type_symbol
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_Cromer_Mann_a1
_atom_type_scat_Cromer_Mann_b1
_atom_type_scat_Cromer_Mann_a2
_atom_type_scat_Cromer_Mann_b2
_atom_type_scat_Cromer_Mann_a3
_atom_type_scat_Cromer_Mann_b3
_atom_type_scat_Cromer_Mann_a4
_atom_type_scat_Cromer_Mann_b4
_atom_type_scat_Cromer_Mann_c
_atom_type_scat_source
 H       0.0000   0.0000   0.4930  10.5109   0.3229  26.1257   0.1402   3.1424
   0.0408  57.7997   0.0030 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Lip1    0.0000   0.0000   0.6968   4.6237   0.7888   1.9557   0.3414   0.6316
   0.1563  10.0953   0.0167 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fep2    0.3010   0.8450  11.0424   4.6538   7.3740   0.3053   4.1346  12.0546
   0.4399  31.2809   1.0097 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Cap2    0.2030   0.3060  15.6348  -0.0074   7.9518   0.6089   8.4372  10.3116
   0.8537  25.9905 -14.8750 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Nap1    0.0300   0.0250   3.2565   2.6671   3.9362   6.1153   1.3998   0.2001
   1.0032  14.0390   0.4040 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Kp1     0.1790   0.2500   7.9578  12.6331   7.4917   0.7674   6.3590  -0.0020
   1.1915  31.9128  -4.9978 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Mgp2    0.0420   0.0360   3.4988   2.1676   3.8378   4.7542   1.3284   0.1850
   0.8497  10.1411   0.4853 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fep3    0.3010   0.8450  11.1764   4.6147   7.3863   0.3005   3.3948  11.6729
   0.0724  38.5566   0.9707 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Alp3    0.0560   0.0520   4.1745   1.9382   3.3876   4.1455   1.2030   0.2288
   0.5281   8.2852   0.7068 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Crp3    0.2840   0.6240   9.6809   5.5946   7.8114   0.3344   2.8760  12.8288
   0.1136  32.8761   0.5183 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Zrp4   -2.9650   0.5600  18.1668   1.2148  10.0562  10.1483   1.0112  21.6054
  -2.6479  -0.1028   9.4145 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Tip4    0.2480   0.4460  19.5114   0.1789   8.2347   6.6702   2.0134  -0.2926
   1.5208  12.9464 -13.2800 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Si      0.0720   0.0710   6.2915   2.4386   3.0353  32.3337   1.9891   0.6785
   1.5410  81.6937   1.1407 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Sip4    0.0720   0.0710   4.4392   1.6417   3.2034   3.4376   1.1945   0.2149
   0.4165   6.6536   0.7463 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
```

```
 O       0.0080   0.0060   3.0485  13.2771   2.2868   5.7011   1.5463   0.3239
   0.8670  32.9089   0.2508 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Om2     0.0080   0.0060   3.7504  16.5151   2.8429   6.5920   1.5430   0.3192
   1.6209  43.3486   0.2421 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Om1     0.0080   0.0060   4.1916  12.8573   1.6397   4.1724   1.5267  47.0179
 -20.3070  -0.0140  21.9412 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 F       0.0140   0.0100   3.5392  10.2825   2.6412   4.2944   1.5170   0.2615
   1.0243  26.1476   0.2776 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fm1     0.0140   0.0100   3.6322   5.2776   3.5106  14.7353   1.2606   0.4423
   0.9407  47.3437   0.6534 'International Tables Vol C 4.2.6.8 and 6.1.1.4'


_cell_formula_units_Z                    8

# Given Formula = H1 Alp30.06 Cap20.24 Crp30.01 F0.50 Fep20.01 Fep30.10 Fm10.50
Kp10.50 Lip10.50
# Dc =      2.59 Fooo =    325.13 Mu =     50.50 M =     95.25
# Found Formula = F0.17 Fep20.07 Kp10.06 Mgp20.24 O1.06 Om21.27 Si0.73 Sip40.26
# Dc =      2.96 FOOO =    325.13 Mu =     50.20 M =    109.12

_chemical_formula_sum             'F0.17 Fep20.07 Kp10.06 Mgp20.24 O1.06 Om21.27
Si0.73 Sip40.26'
_chemical_formula_moiety          'F0.17 Fep20.07 Kp10.06 Mgp20.24 O1.06 Om21.27
Si0.73 Sip40.26'
_chemical_compound_source               ?
_chemical_formula_weight            109.12


_cell_measurement_reflns_used            0
_cell_measurement_theta_min              0
_cell_measurement_theta_max              0
_cell_measurement_temperature          293

_exptl_crystal_description        '?'
_exptl_crystal_colour             '?'
_exptl_crystal_size_min           ?
_exptl_crystal_size_mid           ?
_exptl_crystal_size_max           ?

_exptl_crystal_density_diffrn     2.965
_exptl_crystal_density_meas       ?
_exptl_crystal_density_method     'not measured'
# Non-dispersive F(000):
_exptl_crystal_F_000              325.133
_exptl_absorpt_coefficient_mu     5.020

# Sheldrick geometric approximatio 1.00 1.00
_exptl_absorpt_correction_type    none
_exptl_absorpt_correction_T_min   0.5540
_exptl_absorpt_correction_T_max   1.0000
_diffrn_measurement_device_type   'Unknown'
_diffrn_measurement_device        'Serial'
_diffrn_radiation_monochromator   'graphite'
_diffrn_radiation_type            'Mo K\a'
_diffrn_radiation_wavelength           0.71070
```

```
_diffrn_measurement_method        \w/2\q

# If a reference occurs more than once, delete the author
# and date from subsequent references.
_computing_data_collection        'USER DEFINED DATA COLLECTION'
_computing_cell_refinement        'USER DEFINED CELL REFINEMENT'
_computing_data_reduction         'USER DEFINED DATA REDUCTION'
_computing_structure_solution     'USER DEFINED STRUCTURE SOLUTION'
_computing_structure_refinement   'CRYSTALS (Betteridge et al., 2003)'
_computing_publication_material   'CRYSTALS (Betteridge et al., 2003)'
_computing_molecular_graphics     'CAMERON (Watkin et al., 1996)'

_diffrn_standards_interval_time   .
_diffrn_standards_interval_count  .
_diffrn_standards_number          0
_diffrn_standards_decay_%         ?

_diffrn_ambient_temperature        293
_diffrn_reflns_number             4376
_reflns_number_total              0
_diffrn_reflns_av_R_equivalents   0.0525
# Number of reflections without Friedels Law is 0
# Number of reflections with Friedels Law is 0
# Theoretical number of reflections is about 2552


_diffrn_reflns_theta_min          2.045
_diffrn_reflns_theta_max          44.626
_diffrn_measured_fraction_theta_max 0.887

_diffrn_reflns_theta_full         37.486
_diffrn_measured_fraction_theta_full 0.996


_diffrn_reflns_limit_h_min        -10
_diffrn_reflns_limit_h_max        10
_diffrn_reflns_limit_k_min        0
_diffrn_reflns_limit_k_max        17
_diffrn_reflns_limit_l_min        0
_diffrn_reflns_limit_l_max        19
_reflns_limit_h_min               -10
_reflns_limit_h_max               10
_reflns_limit_k_min               0
_reflns_limit_k_max               17
_reflns_limit_l_min               0
_reflns_limit_l_max               19

_oxford_diffrn_Wilson_B_factor    0.00
_oxford_diffrn_Wilson_scale       0.00

_atom_sites_solution_primary      direct #heavy,direct,difmap,geom
# _atom_sites_solution_secondary  difmap
_atom_sites_solution_hydrogens    geom

_refine_diff_density_min          -1.89
```

```
_refine_diff_density_max         0.66


# The current dictionary definitions do not cover the
# situation where the reflections used for refinement were
# selected by a user-defined sigma threshold


# The values actually used during refinement
_oxford_reflns_threshold_expression_ref     I>3.0\s(I)
_refine_ls_number_reflns         2072
_refine_ls_number_restraints     24
_refine_ls_number_parameters     71
_oxford_refine_ls_R_factor_ref   0.0315
_refine_ls_wR_factor_ref         0.0324
_refine_ls_goodness_of_fit_ref   0.7173
_refine_ls_shift/su_max          0.0142040
_refine_ls_shift/su_mean         0.0015464



# choose from: rm (reference molecule of known chirality),
# ad (anomolous dispersion - Flack), rmad (rm and ad),
# syn (from synthesis), unk (unknown) or . (not applicable).
_chemical_absolute_configuration  '.'


_refine_ls_structure_factor_coef  F
_refine_ls_matrix_type           full
_refine_ls_hydrogen_treatment    none          # none, undef, noref, refall,
                                               # refxyz, refU, constr or mixed

# WARNING. The IUCr will not accept Unit Weights
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
;
 Method= Quasi-Unit weights
 W = 1.0 or 1./2F
;
# Insert your own references if required - in alphabetical order
_publ_section_references
;
User-defined structure solution reference

User-defined data collection reference

User defined data reduction

User defined cell refinement

Betteridge, P.W., Carruthers, J.R., Cooper, R.I.,
Prout, K. & Watkin, D.J. (2003). J. Appl. Cryst. 36, 1487.

Watkin, D.J., Prout, C.K. & Pearce, L.J. (1996). CAMERON, Chemical
Crystallography Laboratory, Oxford, UK.
```

```
;

# Uequiv = arithmetic mean of Ui i.e. Ueqiv = (U1+U2+U3)/3

# Replace last . with number of unfound hydrogen atomsattached to an atom.

# ..._refinement_flags_...
# . no refinement constraints           S special position constraint on site
# G rigid group refinement of site      R riding atom
# D distance or angle restraint on site T thermal displacement constraints
# U Uiso or Uij restraint (rigid bond)  P partial occupancy constraint


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_occupancy
_atom_site_adp_type
_atom_site_refinement_flags_posn
_atom_site_refinement_flags_adp
_atom_site_refinement_flags_occupancy
_atom_site_disorder_assembly
_atom_site_disorder_group
_oxford_atom_site_special_shape
_atom_site_attached_hydrogens
Kp11 Kp1 0.0000 0.5000 0.0000 0.0314 0.9131(7) Uani . . P . . . .
Mgp22 Mgp2 0.0000 0.0000 0.5000 0.0094 0.7762(7) Uani . . P . . . .
Fep22 Fep2 0.0000 0.0000 0.5000 0.0094 0.2236(6) Uani . . P . . . .
Mgp23 Mgp2 0.0000 0.33630(6) 0.5000 0.0108 0.8019(7) Uani . . P . . . .
Fep23 Fep2 0.0000 0.33630(6) 0.5000 0.0108 0.1980(7) Uani . . P . . . .
Sip44 Sip4 0.07457(6) 0.16688(4) 0.22366(4) 0.0089 0.2513(9) Uani . . P . . . .
Si4 Si 0.07457(6) 0.16688(4) 0.22366(4) 0.0089 0.7306(9) Uani . . P . . . .
Om25 Om2 0.3170(2) 0.23771(14) 0.16511(11) 0.0177 0.5044(8) Uani . . P . . . .
O5 O 0.3170(2) 0.23771(14) 0.16511(11) 0.0177 0.4956(8) Uani . . P . . . .
Om26 Om2 0.0303(3) 0.0000 0.16567(15) 0.0179 0.4771(8) Uani . . P . . . .
O6 O 0.0303(3) 0.0000 0.16567(15) 0.0179 0.5229(8) Uani . . P . . . .
Om27 Om2 0.13072(17) 0.16755(10) 0.38978(9) 0.0103 0.6124(8) Uani . . P . . . .
O7 O 0.13072(17) 0.16755(10) 0.38978(9) 0.0103 0.3876(8) Uani . . P . . . .
Om28 Om2 0.1298(2) 0.5000 0.40078(13) 0.0128 0.4022(8) Uani . . P . . . .
F8 F 0.1298(2) 0.5000 0.40078(13) 0.0128 0.5978(8) Uani . . P . . . .
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
Kp11 0.0314(4) 0.0313(5) 0.0308(4) 0.0000 0.0042(3) 0.0000
Mgp22 0.0077(2) 0.0070(2) 0.0136(3) 0.0000 0.00241(19) 0.0000
Fep22 0.0077(2) 0.0070(2) 0.0136(3) 0.0000 0.00241(19) 0.0000
```

```
Mgp23 0.00658(16) 0.0138(2) 0.01175(19) 0.0000 0.00073(13) 0.0000
Fep23 0.00658(16) 0.0138(2) 0.01175(19) 0.0000 0.00073(13) 0.0000
Sip44 0.00788(12) 0.00827(12) 0.01030(13) -0.00003(10) 0.00109(9) 0.00001(10)
Si4 0.00788(12) 0.00827(12) 0.01030(13) -0.00003(10) 0.00109(9) 0.00001(10)
Om25 0.0160(4) 0.0231(5) 0.0139(4) -0.0015(3) 0.0027(3) -0.0060(4)
O5 0.0160(4) 0.0231(5) 0.0139(4) -0.0015(3) 0.0027(3) -0.0060(4)
Om26 0.0260(7) 0.0126(5) 0.0139(6) 0.0000 0.0003(5) 0.0000
O6 0.0260(7) 0.0126(5) 0.0139(6) 0.0000 0.0003(5) 0.0000
Om27 0.0097(3) 0.0102(3) 0.0107(3) -0.0002(3) 0.0013(2) 0.0004(3)
O7 0.0097(3) 0.0102(3) 0.0107(3) -0.0002(3) 0.0013(2) 0.0004(3)
Om28 0.0108(4) 0.0142(5) 0.0134(5) 0.0000 0.0019(4) 0.0000
F8 0.0108(4) 0.0142(5) 0.0134(5) 0.0000 0.0019(4) 0.0000

_refine_ls_extinction_method
    'None'
_oxford_refine_ls_scale 0.600(3)

loop_
    _oxford_twin_element_scale_factors
         0.983(9)
         0.001(1)
         0.016(2)
loop_
 _geom_bond_atom_site_label_1
 _geom_bond_site_symmetry_1
 _geom_bond_atom_site_label_2
 _geom_bond_site_symmetry_2
 _geom_bond_distance
 _geom_bond_publ_flag
Mgp22 . Om28 4_556 2.0474(12)    yes
Mgp22 . Om28 3_445 2.0474(12)    yes
Mgp22 . F8 4_556 2.0474(12)    yes
Mgp22 . F8 3_445 2.0474(12)    yes
Mgp22 . O7 6_555 2.0951(9)    yes
Mgp22 . O7 5_556 2.0951(9)    yes
Mgp22 . Om27 6_555 2.0951(9)    yes
Mgp22 . O7 2_556 2.0951(9)    yes
Mgp22 . Om27 . 2.0951(9)    yes
Mgp22 . O7 . 2.0951(9)    yes
Fep22 . Om27 6_555 2.0951(9)    yes
Fep22 . O7 2_556 2.0951(9)    yes
Fep22 . O7 5_556 2.0951(9)    yes
Fep22 . O7 6_555 2.0951(9)    yes
Fep22 . Om27 2_556 2.0951(9)    yes
Fep22 . Om27 5_556 2.0951(9)    yes
Fep22 . F8 3_445 2.0474(12)    yes
Fep22 . F8 4_556 2.0474(12)    yes
Fep22 . Om28 3_445 2.0474(12)    yes
Fep22 . Om27 . 2.0951(9)    yes
Fep22 . O7 . 2.0951(9)    yes
Mgp23 . Om28 2_566 2.0024(10)    yes
Mgp23 . F8 2_566 2.0024(10)    yes
Mgp23 . Om27 8_455 2.0820(9)    yes
Mgp23 . Om27 4_556 2.0820(9)    yes
Mgp23 . O7 8_455 2.0820(9)    yes
```

```
Mgp23 . O7 4_556 2.0820(9)    yes
Mgp23 . Om27 5_556 2.1033(10)    yes
Mgp23 . O7 5_556 2.1033(10)    yes
Mgp23 . Om27 . 2.1033(10)    yes
Mgp23 . O7 . 2.1033(10)    yes
Mgp23 . Om28 . 2.0024(10)    yes
Mgp23 . F8 . 2.0024(10)    yes
Fep23 . Om27 5_556 2.1033(10)    yes
Fep23 . O7 5_556 2.1033(10)    yes
Fep23 . Om27 4_556 2.0820(9)    yes
Fep23 . Om27 8_455 2.0820(9)    yes
Fep23 . O7 4_556 2.0820(9)    yes
Fep23 . O7 8_455 2.0820(9)    yes
Fep23 . F8 5_556 2.0024(10)    yes
Fep23 . Om28 5_556 2.0024(10)    yes
Fep23 . Om27 . 2.1033(10)    yes
Fep23 . O7 . 2.1033(10)    yes
Fep23 . Om28 . 2.0024(10)    yes
Fep23 . F8 . 2.0024(10)    yes
Sip44 . O5 8_455 1.6487(11)    yes
Sip44 . Om25 8_455 1.6487(11)    yes
Sip44 . Om25 . 1.6472(11)    yes
Sip44 . O5 . 1.6472(11)    yes
Sip44 . Om26 . 1.6488(7)    yes
Sip44 . O6 . 1.6488(7)    yes
Sip44 . Om27 . 1.6546(10)    yes
Sip44 . O7 . 1.6546(10)    yes
Si4 . O5 8_455 1.6487(11)    yes
Si4 . Om25 8_455 1.6487(11)    yes
Si4 . Om25 . 1.6472(11)    yes
Si4 . O5 . 1.6472(11)    yes
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Scordari et al.: FLUOROPHLOGOPITE FROM PIANO DELLE CONCAZZE (Mt. ETNA, ITALY);
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;
 Dipartimento Scienze della Terra,
 University of Bari,
 via Orabona, N.4 I-70125, IT.
;

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 Universit‡ di Bari,
 via Orabona, N.4 I-70125, IT.
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 'Ventruti G.'
;
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;

 'Schingaro E.'
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;

 'Mazziotti S.'
;
 Dipartimento Geomineralogico,
 Universit‡ di Roma,
 via Orabona, N.4 I-70125, IT.
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 Tip4    0.2480   0.4460  19.5114   0.1789   8.2347   6.6702   2.0134  -0.2926
   1.5208  12.9464 -13.2800 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Si      0.0720   0.0710   6.2915   2.4386   3.0353  32.3337   1.9891   0.6785
   1.5410  81.6937   1.1407 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Sip4    0.0720   0.0710   4.4392   1.6417   3.2034   3.4376   1.1945   0.2149
   0.4165   6.6536   0.7463 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 O       0.0080   0.0060   3.0485  13.2771   2.2868   5.7011   1.5463   0.3239
   0.8670  32.9089   0.2508 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Om2     0.0080   0.0060   3.7504  16.5151   2.8429   6.5920   1.5430   0.3192
   1.6209  43.3486   0.2421 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Om1     0.0080   0.0060   4.1916  12.8573   1.6397   4.1724   1.5267  47.0179
 -20.3070  -0.0140  21.9412 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 F       0.0140   0.0100   3.5392  10.2825   2.6412   4.2944   1.5170   0.2615
   1.0243  26.1476   0.2776 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fm1     0.0140   0.0100   3.6322   5.2776   3.5106  14.7353   1.2606   0.4423
   0.9407  47.3437   0.6534 'International Tables Vol C 4.2.6.8 and 6.1.1.4'


_cell_formula_units_Z                    8

# Given Formula = H1 Alp30.06 Cap20.24 Crp30.01 F0.50 Fep20.01 Fep30.10 Fm10.50
Kp10.50 Lip10.50
# Dc =      2.59 Fooo =    326.22 Mu =     50.61 M =     95.25
```

```
# Found Formula = Fep20.06 Fm10.18 Kp10.06 Mgp20.25 O1.14 Om21.18 Si0.80
Sip40.19
# Dc =      2.96 FOOO =    326.22 Mu =     49.83 M =    108.57

_chemical_formula_sum             'Fep20.06 Fm10.18 Kp10.06 Mgp20.25 O1.14
Om21.18 Si0.80 Sip40.19'
_chemical_formula_moiety          'Fep20.06 Fm10.18 Kp10.06 Mgp20.25 O1.14
Om21.18 Si0.80 Sip40.19'
_chemical_compound_source               ?
_chemical_formula_weight           108.57


_cell_measurement_reflns_used           0
_cell_measurement_theta_min             0
_cell_measurement_theta_max             0
_cell_measurement_temperature         293

_exptl_crystal_description        '?'
_exptl_crystal_colour             '?'
_exptl_crystal_size_min           ?
_exptl_crystal_size_mid           ?
_exptl_crystal_size_max           ?

_exptl_crystal_density_diffrn     2.956
_exptl_crystal_density_meas       ?
_exptl_crystal_density_method     'not measured'
# Non-dispersive F(000):
_exptl_crystal_F_000              326.221
_exptl_absorpt_coefficient_mu     4.983

# Sheldrick geometric approximatio 1.00 1.00
_exptl_absorpt_correction_type    none
_exptl_absorpt_correction_T_min   1.0000
_exptl_absorpt_correction_T_max   1.0000
_diffrn_measurement_device_type   'Unknown'
_diffrn_measurement_device        'Serial'
_diffrn_radiation_monochromator   'graphite'
_diffrn_radiation_type            'Mo K\a'
_diffrn_radiation_wavelength           0.71070
_diffrn_measurement_method        \w/2\q

# If a reference occurs more than once, delete the author
# and date from subsequent references.
_computing_data_collection        'USER DEFINED DATA COLLECTION'
_computing_cell_refinement        'USER DEFINED CELL REFINEMENT'
_computing_data_reduction         'USER DEFINED DATA REDUCTION'
_computing_structure_solution     'USER DEFINED STRUCTURE SOLUTION'
_computing_structure_refinement   'CRYSTALS (Betteridge et al., 2003)'
_computing_publication_material   'CRYSTALS (Betteridge et al., 2003)'
_computing_molecular_graphics     'CAMERON (Watkin et al., 1996)'

_diffrn_standards_interval_time   .
_diffrn_standards_interval_count  .
_diffrn_standards_number          0
_diffrn_standards_decay_%         ?
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```
_diffrn_ambient_temperature        293
_diffrn_reflns_number             2919
_reflns_number_total              1616
_diffrn_reflns_av_R_equivalents   0.013
# Number of reflections without Friedels Law is 0
# Number of reflections with Friedels Law is 1616
# Theoretical number of reflections is about 3287


_diffrn_reflns_theta_min          4.095
_diffrn_reflns_theta_max          41.409
_diffrn_measured_fraction_theta_max 0.941

_diffrn_reflns_theta_full         26.916
_diffrn_measured_fraction_theta_full 0.997


_diffrn_reflns_limit_h_min        -9
_diffrn_reflns_limit_h_max        9
_diffrn_reflns_limit_k_min        -17
_diffrn_reflns_limit_k_max        17
_diffrn_reflns_limit_l_min        -17
_diffrn_reflns_limit_l_max        18
_reflns_limit_h_min               -9
_reflns_limit_h_max               9
_reflns_limit_k_min               0
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_reflns_limit_l_min               0
_reflns_limit_l_max               18

_oxford_diffrn_Wilson_B_factor    0.00
_oxford_diffrn_Wilson_scale       0.00

_atom_sites_solution_primary      direct #heavy,direct,difmap,geom
# _atom_sites_solution_secondary  difmap
_atom_sites_solution_hydrogens    geom

_refine_diff_density_min          ?
_refine_diff_density_max          ?


# The current dictionary definitions do not cover the
# situation where the reflections used for refinement were
# selected by a user-defined sigma threshold


# The values actually used during refinement
_oxford_reflns_threshold_expression_ref     I>3.0\s(I)
_refine_ls_number_reflns          1250
_refine_ls_number_restraints      23
_refine_ls_number_parameters      68
_oxford_refine_ls_R_factor_ref    0.0296
_refine_ls_wR_factor_ref          0.0304
_refine_ls_goodness_of_fit_ref    0.5731
```

```
_refine_ls_shift/su_max           0.0139060
_refine_ls_shift/su_mean          0.0009775


# The values computed from all data
_oxford_reflns_number_all         1609
_refine_ls_R_factor_all           0.0424
_refine_ls_wR_factor_all          0.0507

# The values computed with a 2 sigma cutoff - a la SHELX
_reflns_threshold_expression      I>2.0\s(I)
_reflns_number_gt                 1304
_refine_ls_R_factor_gt            0.0312
_refine_ls_wR_factor_gt           0.0315

# choose from: rm (reference molecule of known chirality),
# ad (anomolous dispersion - Flack), rmad (rm and ad),
# syn (from synthesis), unk (unknown) or . (not applicable).
_chemical_absolute_configuration  '.'


_refine_ls_structure_factor_coef  F
_refine_ls_matrix_type            full
_refine_ls_hydrogen_treatment     none           # none, undef, noref, refall,
                                                 # refxyz, refU, constr or mixed

# WARNING. The IUCr will not accept Unit Weights
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
;
 Method= Quasi-Unit weights
 W = 1.0 or 1./2F
;
# Insert your own references if required - in alphabetical order
_publ_section_references
;
User-defined structure solution reference

User-defined data collection reference

User defined data reduction

User defined cell refinement

Betteridge, P.W., Carruthers, J.R., Cooper, R.I.,
Prout, K. & Watkin, D.J. (2003). J. Appl. Cryst. 36, 1487.

Watkin, D.J., Prout, C.K. & Pearce, L.J. (1996). CAMERON, Chemical
Crystallography Laboratory, Oxford, UK.
;

# Uequiv = arithmetic mean of Ui i.e. Ueqiv = (U1+U2+U3)/3

# Replace last . with number of unfound hydrogen atomsattached to an atom.
```

```
# ..._refinement_flags_...
# . no refinement constraints           S special position constraint on site
# G rigid group refinement of site      R riding atom
# D distance or angle restraint on site T thermal displacement constraints
# U Uiso or Uij restraint (rigid bond)  P partial occupancy constraint


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_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
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_atom_site_adp_type
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_atom_site_refinement_flags_adp
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_atom_site_disorder_assembly
_atom_site_disorder_group
_oxford_atom_site_special_shape
_atom_site_attached_hydrogens
Kp11 Kp1 0.0000 0.5000 0.0000 0.0316 0.8939(8) Uani . . P . . . .
Mgp22 Mgp2 0.0000 0.0000 0.5000 0.0092 0.7759(8) Uani . . P . . . .
Fep22 Fep2 0.0000 0.0000 0.5000 0.0092 0.2237(6) Uani . . P . . . .
Mgp23 Mgp2 0.0000 0.33595(7) 0.5000 0.0104 0.8068(8) Uani . . P . . . .
Fep23 Fep2 0.0000 0.33595(7) 0.5000 0.0104 0.1932(7) Uani . . P . . . .
Sip44 Sip4 0.07444(7) 0.16685(4) 0.22370(4) 0.0088 0.1888(9) Uani . . P . . . .
Si4 Si 0.07444(7) 0.16685(4) 0.22370(4) 0.0088 0.7974(9) Uani . . P . . . .
Om25 Om2 0.3166(2) 0.23791(15) 0.16500(12) 0.0181 0.2805(8) Uani . . P . . . .
O5 O 0.3166(2) 0.23791(15) 0.16500(12) 0.0181 0.7195(8) Uani . . P . . . .
Om26 Om2 0.0304(4) 0.0000 0.16551(18) 0.0182 0.2649(8) Uani . . P . . . .
O6 O 0.0304(4) 0.0000 0.16551(18) 0.0182 0.7354(8) Uani . . P . . . .
Om27 Om2 0.13084(18) 0.16731(11) 0.38986(10) 0.0095 0.7646(8) Uani . . P . . . .
O7 O 0.13084(18) 0.16731(11) 0.38986(10) 0.0095 0.2353(8) Uani . . P . . . .
Fm18 Fm1 0.1307(3) 0.5000 0.40044(15) 0.0130 0.7292(8) Uani . . P . . . .
Om28 Om2 0.1307(3) 0.5000 0.40044(15) 0.0130 0.2717(8) Uani . . P . . . .
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_atom_site_aniso_U_12
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Mgp22 0.0083(3) 0.0062(3) 0.0138(3) 0.0000 0.0036(2) 0.0000
Fep22 0.0083(3) 0.0062(3) 0.0138(3) 0.0000 0.0036(2) 0.0000
Mgp23 0.0069(2) 0.0126(2) 0.0120(2) 0.0000 0.00200(16) 0.0000
Fep23 0.0069(2) 0.0126(2) 0.0120(2) 0.0000 0.00200(16) 0.0000
Sip44 0.00859(14) 0.00756(14) 0.01042(15) 0.00004(13) 0.00200(11) -0.00011(13)
Si4 0.00859(14) 0.00756(14) 0.01042(15) 0.00004(13) 0.00200(11) -0.00011(13)
Om25 0.0167(5) 0.0227(6) 0.0151(5) -0.0014(4) 0.0037(4) -0.0059(4)
O5 0.0167(5) 0.0227(6) 0.0151(5) -0.0014(4) 0.0037(4) -0.0059(4)
```

```
Om26 0.0261(8) 0.0132(6) 0.0145(7) 0.0000 0.0014(6) 0.0000
O6 0.0261(8) 0.0132(6) 0.0145(7) 0.0000 0.0014(6) 0.0000
Om27 0.0088(3) 0.0085(3) 0.0114(4) -0.0003(3) 0.0024(3) 0.0004(3)
O7 0.0088(3) 0.0085(3) 0.0114(4) -0.0003(3) 0.0024(3) 0.0004(3)
Fm18 0.0111(5) 0.0140(6) 0.0144(6) 0.0000 0.0034(4) 0.0000
Om28 0.0111(5) 0.0140(6) 0.0144(6) 0.0000 0.0034(4) 0.0000

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 _geom_bond_site_symmetry_2
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 _geom_bond_publ_flag
Mgp22 . Om28 3_445 2.0424(14)    yes
Mgp22 . Om28 4_556 2.0424(14)    yes
Mgp22 . Fm18 3_445 2.0424(14)    yes
Mgp22 . Fm18 7_546 2.0424(14)    yes
Mgp22 . O7 6_555 2.0921(10)    yes
Mgp22 . O7 5_556 2.0921(10)    yes
Mgp22 . Om27 6_555 2.0921(10)    yes
Mgp22 . O7 2_556 2.0921(10)    yes
Mgp22 . Om27 . 2.0921(10)    yes
Mgp22 . O7 . 2.0921(10)    yes
Fep22 . Om27 6_555 2.0921(10)    yes
Fep22 . O7 2_556 2.0921(10)    yes
Fep22 . O7 5_556 2.0921(10)    yes
Fep22 . O7 6_555 2.0921(10)    yes
Fep22 . Om27 2_556 2.0921(10)    yes
Fep22 . Om27 5_556 2.0921(10)    yes
Fep22 . Fm18 7_546 2.0424(14)    yes
Fep22 . Fm18 3_445 2.0424(14)    yes
Fep22 . Om28 4_556 2.0424(14)    yes
Fep22 . Sip44 . 3.2743(4)    yes
Fep22 . Si4 . 3.2743(4)    yes
Fep22 . Om27 . 2.0921(10)    yes
Fep22 . O7 . 2.0921(10)    yes
Mgp23 . Om28 5_556 2.0076(11)    yes
Mgp23 . Fm18 5_556 2.0076(11)    yes
Mgp23 . Om27 8_455 2.0789(10)    yes
Mgp23 . O7 8_455 2.0789(10)    yes
Mgp23 . Om27 4_556 2.0789(10)    yes
Mgp23 . O7 4_556 2.0789(10)    yes
Mgp23 . Om27 5_556 2.1011(11)    yes
Mgp23 . O7 5_556 2.1011(11)    yes
Mgp23 . Om27 . 2.1011(11)    yes
Mgp23 . O7 . 2.1011(11)    yes
Mgp23 . Fm18 . 2.0076(11)    yes
Mgp23 . Om28 . 2.0076(11)    yes
Fep23 . Om27 5_556 2.1011(11)    yes
Fep23 . O7 5_556 2.1011(11)    yes
Fep23 . Om27 4_556 2.0789(10)    yes
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Fep23 . O7 4_556 2.0789(10)    yes
Fep23 . Om27 8_455 2.0789(10)    yes
Fep23 . O7 8_455 2.0789(10)    yes
Fep23 . Fm18 2_566 2.0076(11)    yes
Fep23 . Om28 2_566 2.0076(11)    yes
Fep23 . Sip44 . 3.2841(5)    yes
Fep23 . Si4 . 3.2841(5)    yes
Fep23 . Om27 . 2.1011(11)    yes
Fep23 . O7 . 2.1011(11)    yes
Fep23 . Fm18 . 2.0076(11)    yes
Fep23 . Om28 . 2.0076(11)    yes
Sip44 . O5 8_455 1.6475(13)    yes
Sip44 . Om25 8_455 1.6475(13)    yes
Sip44 . Om25 . 1.6471(12)    yes
Sip44 . O5 . 1.6471(12)    yes
Sip44 . Om26 . 1.6479(7)    yes
Sip44 . O6 . 1.6479(7)    yes
Sip44 . Om27 . 1.6537(11)    yes
Sip44 . O7 . 1.6537(11)    yes
Si4 . O5 8_455 1.6475(13)    yes
Si4 . Om25 8_455 1.6475(13)    yes
Si4 . Si4 8_455 3.0697(4)    yes
Si4 . Si4 8_555 3.0697(4)    yes
Si4 . Om25 . 1.6471(12)    yes
Si4 . O5 . 1.6471(12)    yes
Si4 . Om26 . 1.6479(7)    yes
Si4 . O6 . 1.6479(7)    yes
Si4 . Om27 . 1.6537(11)    yes
Si4 . O7 . 1.6537(11)    yes
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Om28 3_445 Mgp22 . Om28 4_556 179.994    yes
Om28 4_556 Mgp22 . Fm18 3_445 179.994    yes
Om28 3_445 Mgp22 . Fm18 7_546 179.994    yes
Fm18 3_445 Mgp22 . Fm18 7_546 179.994    yes
Om28 3_445 Mgp22 . O7 6_555 96.86(4)    yes
Om28 4_556 Mgp22 . O7 6_555 83.14(4)    yes
Fm18 3_445 Mgp22 . O7 6_555 96.86(4)    yes
Fm18 7_546 Mgp22 . O7 6_555 83.14(4)    yes
Om28 3_445 Mgp22 . O7 5_556 83.14(4)    yes
Om28 4_556 Mgp22 . O7 5_556 96.86(4)    yes
Fm18 3_445 Mgp22 . O7 5_556 83.14(4)    yes
Fm18 7_546 Mgp22 . O7 5_556 96.86(4)    yes
O7 6_555 Mgp22 . O7 5_556 179.995    yes
Om28 3_445 Mgp22 . Om27 6_555 96.86(4)    yes
Om28 4_556 Mgp22 . Om27 6_555 83.14(4)    yes
Fm18 3_445 Mgp22 . Om27 6_555 96.86(4)    yes
Fm18 7_546 Mgp22 . Om27 6_555 83.14(4)    yes
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Om28 3_445 Mgp22 . O7 2_556 83.14(4)    yes
Om28 4_556 Mgp22 . O7 2_556 96.86(4)    yes
Fm18 3_445 Mgp22 . O7 2_556 83.14(4)    yes
Fm18 7_546 Mgp22 . O7 2_556 96.86(4)    yes
O7 6_555 Mgp22 . O7 2_556 85.07(6)    yes
Om28 3_445 Mgp22 . Om27 . 96.86(4)    yes
Om28 4_556 Mgp22 . Om27 . 83.14(4)    yes
Fm18 3_445 Mgp22 . Om27 . 96.86(4)    yes
Fm18 7_546 Mgp22 . Om27 . 83.14(4)    yes
O7 6_555 Mgp22 . Om27 . 94.93(6)    yes
Om28 3_445 Mgp22 . O7 . 96.86(4)    yes
Om28 4_556 Mgp22 . O7 . 83.14(4)    yes
Fm18 3_445 Mgp22 . O7 . 96.86(4)    yes
Fm18 7_546 Mgp22 . O7 . 83.14(4)    yes
O7 6_555 Mgp22 . O7 . 94.93(6)    yes
O7 5_556 Mgp22 . Om27 6_555 179.995    yes
O7 5_556 Mgp22 . O7 2_556 94.93(6)    yes
Om27 6_555 Mgp22 . O7 2_556 85.07(6)    yes
O7 5_556 Mgp22 . Om27 . 85.07(6)    yes
Om27 6_555 Mgp22 . Om27 . 94.93(6)    yes
O7 2_556 Mgp22 . Om27 . 179.995    yes
O7 5_556 Mgp22 . O7 . 85.07(6)    yes
Om27 6_555 Mgp22 . O7 . 94.93(6)    yes
O7 2_556 Mgp22 . O7 . 179.995    yes
Om27 6_555 Fep22 . O7 2_556 85.07(6)    yes
Om27 6_555 Fep22 . O7 5_556 179.995    yes
O7 2_556 Fep22 . O7 5_556 94.93(6)    yes
O7 2_556 Fep22 . O7 6_555 85.07(6)    yes
O7 5_556 Fep22 . O7 6_555 179.995    yes
Om27 6_555 Fep22 . Om27 2_556 85.07(6)    yes
O7 5_556 Fep22 . Om27 2_556 94.93(6)    yes
O7 6_555 Fep22 . Om27 2_556 85.07(6)    yes
Om27 6_555 Fep22 . Om27 5_556 179.995    yes
O7 2_556 Fep22 . Om27 5_556 94.93(6)    yes
O7 6_555 Fep22 . Om27 5_556 179.995    yes
Om27 2_556 Fep22 . Om27 5_556 94.93(6)    yes
Om27 6_555 Fep22 . Fm18 7_546 83.14(4)    yes
O7 2_556 Fep22 . Fm18 7_546 96.86(4)    yes
O7 5_556 Fep22 . Fm18 7_546 96.86(4)    yes
O7 6_555 Fep22 . Fm18 7_546 83.14(4)    yes
Om27 2_556 Fep22 . Fm18 7_546 96.86(4)    yes
Om27 6_555 Fep22 . Fm18 3_445 96.86(4)    yes
O7 2_556 Fep22 . Fm18 3_445 83.14(4)    yes
O7 5_556 Fep22 . Fm18 3_445 83.14(4)    yes
O7 6_555 Fep22 . Fm18 3_445 96.86(4)    yes
Om27 2_556 Fep22 . Fm18 3_445 83.14(4)    yes
Om27 6_555 Fep22 . Om28 4_556 83.14(4)    yes
O7 2_556 Fep22 . Om28 4_556 96.86(4)    yes
O7 5_556 Fep22 . Om28 4_556 96.86(4)    yes
O7 6_555 Fep22 . Om28 4_556 83.14(4)    yes
Om27 2_556 Fep22 . Om28 4_556 96.86(4)    yes
Om27 6_555 Fep22 . Sip44 . 77.85(3)    yes
O7 2_556 Fep22 . Sip44 . 154.48(3)    yes
O7 5_556 Fep22 . Sip44 . 102.15(3)    yes
O7 6_555 Fep22 . Sip44 . 77.85(3)    yes
```

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Om27 2_556 Fep22 . Sip44 . 154.48(3)    yes
Om27 6_555 Fep22 . Si4 . 77.85(3)    yes
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O7 5_556 Fep22 . Si4 . 102.15(3)    yes
O7 6_555 Fep22 . Si4 . 77.85(3)    yes
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O7 2_556 Fep22 . Om27 . 179.995    yes
O7 5_556 Fep22 . Om27 . 85.07(6)    yes
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Om27 2_556 Fep22 . Om27 . 179.995    yes
Om27 6_555 Fep22 . O7 . 94.93(6)    yes
O7 2_556 Fep22 . O7 . 179.995    yes
O7 5_556 Fep22 . O7 . 85.07(6)    yes
O7 6_555 Fep22 . O7 . 94.93(6)    yes
Om27 2_556 Fep22 . O7 . 179.995    yes
Om27 5_556 Fep22 . Fm18 7_546 96.86(4)    yes
Om27 5_556 Fep22 . Fm18 3_445 83.14(4)    yes
Fm18 7_546 Fep22 . Fm18 3_445 179.994    yes
Om27 5_556 Fep22 . Om28 4_556 96.86(4)    yes
Fm18 3_445 Fep22 . Om28 4_556 179.994    yes
Om27 5_556 Fep22 . Sip44 . 102.15(3)    yes
Fm18 7_546 Fep22 . Sip44 . 99.74(4)    yes
Fm18 3_445 Fep22 . Sip44 . 80.26(4)    yes
Om28 4_556 Fep22 . Sip44 . 99.74(4)    yes
Om27 5_556 Fep22 . Si4 . 102.15(3)    yes
Fm18 7_546 Fep22 . Si4 . 99.74(4)    yes
Fm18 3_445 Fep22 . Si4 . 80.26(4)    yes
Om28 4_556 Fep22 . Si4 . 99.74(4)    yes
Om27 5_556 Fep22 . Om27 . 85.07(6)    yes
Fm18 7_546 Fep22 . Om27 . 83.14(4)    yes
Fm18 3_445 Fep22 . Om27 . 96.86(4)    yes
Om28 4_556 Fep22 . Om27 . 83.14(4)    yes
Sip44 . Fep22 . Om27 . 25.52(3)    yes
Om27 5_556 Fep22 . O7 . 85.07(6)    yes
Fm18 7_546 Fep22 . O7 . 83.14(4)    yes
Fm18 3_445 Fep22 . O7 . 96.86(4)    yes
Om28 4_556 Fep22 . O7 . 83.14(4)    yes
Sip44 . Fep22 . O7 . 25.52(3)    yes
Si4 . Fep22 . Om27 . 25.52(3)    yes
Si4 . Fep22 . O7 . 25.52(3)    yes
Om28 5_556 Mgp23 . Om27 8_455 84.33(5)    yes
Fm18 5_556 Mgp23 . Om27 8_455 84.33(5)    yes
Om28 5_556 Mgp23 . O7 8_455 84.33(5)    yes
Fm18 5_556 Mgp23 . O7 8_455 84.33(5)    yes
Om28 5_556 Mgp23 . Om27 4_556 96.92(5)    yes
Fm18 5_556 Mgp23 . Om27 4_556 96.92(5)    yes
Om27 8_455 Mgp23 . Om27 4_556 178.34(7)    yes
O7 8_455 Mgp23 . Om27 4_556 178.34(7)    yes
Om28 5_556 Mgp23 . O7 4_556 96.92(5)    yes
Fm18 5_556 Mgp23 . O7 4_556 96.92(5)    yes
Om27 8_455 Mgp23 . O7 4_556 178.34(7)    yes
O7 8_455 Mgp23 . O7 4_556 178.34(7)    yes
Om28 5_556 Mgp23 . Om27 5_556 96.55(4)    yes
Fm18 5_556 Mgp23 . Om27 5_556 96.55(4)    yes
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Om27 8_455 Mgp23 . Om27 5_556 84.43(4)    yes
O7 8_455 Mgp23 . Om27 5_556 84.43(4)    yes
Om27 4_556 Mgp23 . Om27 5_556 94.34(4)    yes
Om28 5_556 Mgp23 . O7 5_556 96.55(4)    yes
Fm18 5_556 Mgp23 . O7 5_556 96.55(4)    yes
Om27 8_455 Mgp23 . O7 5_556 84.43(4)    yes
O7 8_455 Mgp23 . O7 5_556 84.43(4)    yes
Om27 4_556 Mgp23 . O7 5_556 94.34(4)    yes
Om28 5_556 Mgp23 . Fep22 3_555 90.38(4)    yes
Fm18 5_556 Mgp23 . Fep22 3_555 90.38(4)    yes
Om27 8_455 Mgp23 . Fep22 3_555 138.23(3)    yes
O7 8_455 Mgp23 . Fep22 3_555 138.23(3)    yes
Om27 4_556 Mgp23 . Fep22 3_555 42.99(3)    yes
Om28 5_556 Mgp23 . Om27 . 178.13(5)    yes
Fm18 5_556 Mgp23 . Om27 . 178.13(5)    yes
Om27 8_455 Mgp23 . Om27 . 94.34(4)    yes
O7 8_455 Mgp23 . Om27 . 94.34(4)    yes
Om27 4_556 Mgp23 . Om27 . 84.43(4)    yes
Om28 5_556 Mgp23 . O7 . 178.13(5)    yes
Fm18 5_556 Mgp23 . O7 . 178.13(5)    yes
Om27 8_455 Mgp23 . O7 . 94.34(4)    yes
O7 8_455 Mgp23 . O7 . 94.34(4)    yes
Om27 4_556 Mgp23 . O7 . 84.43(4)    yes
Om28 5_556 Mgp23 . Fm18 . 82.32(7)    yes
Fm18 5_556 Mgp23 . Fm18 . 82.32(7)    yes
Om27 8_455 Mgp23 . Fm18 . 96.92(5)    yes
O7 8_455 Mgp23 . Fm18 . 96.92(5)    yes
Om27 4_556 Mgp23 . Fm18 . 84.33(5)    yes
Om28 5_556 Mgp23 . Om28 . 82.32(7)    yes
Fm18 5_556 Mgp23 . Om28 . 82.32(7)    yes
Om27 8_455 Mgp23 . Om28 . 96.92(5)    yes
O7 8_455 Mgp23 . Om28 . 96.92(5)    yes
Om27 4_556 Mgp23 . Om28 . 84.33(5)    yes
O7 4_556 Mgp23 . Om27 5_556 94.34(4)    yes
O7 4_556 Mgp23 . O7 5_556 94.34(4)    yes
O7 4_556 Mgp23 . Fep22 3_555 42.99(3)    yes
Om27 5_556 Mgp23 . Fep22 3_555 137.33(3)    yes
O7 5_556 Mgp23 . Fep22 3_555 137.33(3)    yes
O7 4_556 Mgp23 . Om27 . 84.43(4)    yes
Om27 5_556 Mgp23 . Om27 . 84.62(6)    yes
O7 5_556 Mgp23 . Om27 . 84.62(6)    yes
Fep22 3_555 Mgp23 . Om27 . 89.74(3)    yes
O7 4_556 Mgp23 . O7 . 84.43(4)    yes
Om27 5_556 Mgp23 . O7 . 84.62(6)    yes
O7 5_556 Mgp23 . O7 . 84.62(6)    yes
Fep22 3_555 Mgp23 . O7 . 89.74(3)    yes
O7 4_556 Mgp23 . Fm18 . 84.33(5)    yes
Om27 5_556 Mgp23 . Fm18 . 178.13(5)    yes
O7 5_556 Mgp23 . Fm18 . 178.13(5)    yes
Fep22 3_555 Mgp23 . Fm18 . 41.37(4)    yes
Om27 . Mgp23 . Fm18 . 96.55(4)    yes
O7 4_556 Mgp23 . Om28 . 84.33(5)    yes
Om27 5_556 Mgp23 . Om28 . 178.13(5)    yes
O7 5_556 Mgp23 . Om28 . 178.13(5)    yes
Fep22 3_555 Mgp23 . Om28 . 41.37(4)    yes
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Om27 . Mgp23 . Om28 . 96.55(4)    yes
O7 . Mgp23 . Fm18 . 96.55(4)    yes
O7 . Mgp23 . Om28 . 96.55(4)    yes
Om27 5_556 Fep23 . Om27 4_556 94.34(4)    yes
O7 5_556 Fep23 . Om27 4_556 94.34(4)    yes
Om27 5_556 Fep23 . O7 4_556 94.34(4)    yes
O7 5_556 Fep23 . O7 4_556 94.34(4)    yes
Om27 5_556 Fep23 . Om27 8_455 84.43(4)    yes
O7 5_556 Fep23 . Om27 8_455 84.43(4)    yes
Om27 4_556 Fep23 . Om27 8_455 178.34(7)    yes
O7 4_556 Fep23 . Om27 8_455 178.34(7)    yes
Om27 5_556 Fep23 . O7 8_455 84.43(4)    yes
O7 5_556 Fep23 . O7 8_455 84.43(4)    yes
Om27 4_556 Fep23 . O7 8_455 178.34(7)    yes
O7 4_556 Fep23 . O7 8_455 178.34(7)    yes
Om27 5_556 Fep23 . Fm18 2_566 96.55(4)    yes
O7 5_556 Fep23 . Fm18 2_566 96.55(4)    yes
Om27 4_556 Fep23 . Fm18 2_566 96.92(5)    yes
O7 4_556 Fep23 . Fm18 2_566 96.92(5)    yes
Om27 8_455 Fep23 . Fm18 2_566 84.33(5)    yes
Om27 5_556 Fep23 . Om28 2_566 96.55(4)    yes
O7 5_556 Fep23 . Om28 2_566 96.55(4)    yes
Om27 4_556 Fep23 . Om28 2_566 96.92(5)    yes
O7 4_556 Fep23 . Om28 2_566 96.92(5)    yes
Om27 8_455 Fep23 . Om28 2_566 84.33(5)    yes
Om27 5_556 Fep23 . Fep22 7_556 137.33(3)    yes
O7 5_556 Fep23 . Fep22 7_556 137.33(3)    yes
Om27 4_556 Fep23 . Fep22 7_556 42.99(3)    yes
O7 4_556 Fep23 . Fep22 7_556 42.99(3)    yes
Om27 8_455 Fep23 . Fep22 7_556 138.23(3)    yes
Om27 5_556 Fep23 . Sip44 . 101.63(4)    yes
O7 5_556 Fep23 . Sip44 . 101.63(4)    yes
Om27 4_556 Fep23 . Sip44 . 101.76(3)    yes
O7 4_556 Fep23 . Sip44 . 101.76(3)    yes
Om27 8_455 Fep23 . Sip44 . 77.44(3)    yes
Om27 5_556 Fep23 . Si4 . 101.63(4)    yes
O7 5_556 Fep23 . Si4 . 101.63(4)    yes
Om27 4_556 Fep23 . Si4 . 101.76(3)    yes
O7 4_556 Fep23 . Si4 . 101.76(3)    yes
Om27 8_455 Fep23 . Si4 . 77.44(3)    yes
Om27 5_556 Fep23 . Om27 . 84.62(6)    yes
O7 5_556 Fep23 . Om27 . 84.62(6)    yes
Om27 4_556 Fep23 . Om27 . 84.43(4)    yes
O7 4_556 Fep23 . Om27 . 84.43(4)    yes
Om27 8_455 Fep23 . Om27 . 94.34(4)    yes
Om27 5_556 Fep23 . O7 . 84.62(6)    yes
O7 5_556 Fep23 . O7 . 84.62(6)    yes
Om27 4_556 Fep23 . O7 . 84.43(4)    yes
O7 4_556 Fep23 . O7 . 84.43(4)    yes
Om27 8_455 Fep23 . O7 . 94.34(4)    yes
Om27 5_556 Fep23 . Fm18 . 178.13(5)    yes
O7 5_556 Fep23 . Fm18 . 178.13(5)    yes
Om27 4_556 Fep23 . Fm18 . 84.33(5)    yes
O7 4_556 Fep23 . Fm18 . 84.33(5)    yes
Om27 8_455 Fep23 . Fm18 . 96.92(5)    yes
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Om27 5_556 Fep23 . Om28 . 178.13(5)    yes
O7 5_556 Fep23 . Om28 . 178.13(5)    yes
Om27 4_556 Fep23 . Om28 . 84.33(5)    yes
O7 4_556 Fep23 . Om28 . 84.33(5)    yes
Om27 8_455 Fep23 . Om28 . 96.92(5)    yes
O7 8_455 Fep23 . Fm18 2_566 84.33(5)    yes
O7 8_455 Fep23 . Om28 2_566 84.33(5)    yes
O7 8_455 Fep23 . Fep22 7_556 138.23(3)    yes
Fm18 2_566 Fep23 . Fep22 7_556 90.38(4)    yes
Om28 2_566 Fep23 . Fep22 7_556 90.38(4)    yes
O7 8_455 Fep23 . Sip44 . 77.44(3)    yes
Fm18 2_566 Fep23 . Sip44 . 152.72(4)    yes
Om28 2_566 Fep23 . Sip44 . 152.72(4)    yes
Fep22 7_556 Fep23 . Sip44 . 89.896(7)    yes
O7 8_455 Fep23 . Si4 . 77.44(3)    yes
Fm18 2_566 Fep23 . Si4 . 152.72(4)    yes
Om28 2_566 Fep23 . Si4 . 152.72(4)    yes
Fep22 7_556 Fep23 . Si4 . 89.896(7)    yes
O7 8_455 Fep23 . Om27 . 94.34(4)    yes
Fm18 2_566 Fep23 . Om27 . 178.13(5)    yes
Om28 2_566 Fep23 . Om27 . 178.13(5)    yes
Fep22 7_556 Fep23 . Om27 . 89.74(3)    yes
Sip44 . Fep23 . Om27 . 25.41(3)    yes
O7 8_455 Fep23 . O7 . 94.34(4)    yes
Fm18 2_566 Fep23 . O7 . 178.13(5)    yes
Om28 2_566 Fep23 . O7 . 178.13(5)    yes
Fep22 7_556 Fep23 . O7 . 89.74(3)    yes
Sip44 . Fep23 . O7 . 25.41(3)    yes
O7 8_455 Fep23 . Fm18 . 96.92(5)    yes
Fm18 2_566 Fep23 . Fm18 . 82.32(7)    yes
Om28 2_566 Fep23 . Fm18 . 82.32(7)    yes
Fep22 7_556 Fep23 . Fm18 . 41.37(4)    yes
Sip44 . Fep23 . Fm18 . 79.96(3)    yes
O7 8_455 Fep23 . Om28 . 96.92(5)    yes
Fm18 2_566 Fep23 . Om28 . 82.32(7)    yes
Om28 2_566 Fep23 . Om28 . 82.32(7)    yes
Fep22 7_556 Fep23 . Om28 . 41.37(4)    yes
Sip44 . Fep23 . Om28 . 79.96(3)    yes
Si4 . Fep23 . Om27 . 25.41(3)    yes
Si4 . Fep23 . O7 . 25.41(3)    yes
Si4 . Fep23 . Fm18 . 79.96(3)    yes
Om27 . Fep23 . Fm18 . 96.55(4)    yes
O7 . Fep23 . Fm18 . 96.55(4)    yes
Si4 . Fep23 . Om28 . 79.96(3)    yes
Om27 . Fep23 . Om28 . 96.55(4)    yes
O7 . Fep23 . Om28 . 96.55(4)    yes
O5 8_455 Sip44 . Sip44 8_455 21.29(4)    yes
Om25 8_455 Sip44 . Sip44 8_455 21.29(4)    yes
O5 8_455 Sip44 . Si4 8_455 21.29(4)    yes
Om25 8_455 Sip44 . Si4 8_455 21.29(4)    yes
O5 8_455 Sip44 . Si4 8_555 113.58(5)    yes
Om25 8_455 Sip44 . Si4 8_555 113.58(5)    yes
Sip44 8_455 Sip44 . Si4 8_555 120.11(3)    yes
Si4 8_455 Sip44 . Si4 8_555 120.11(3)    yes
O5 8_455 Sip44 . Sip44 8_555 113.58(5)    yes
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Om25 8_455 Sip44 . Sip44 8_555 113.58(5)    yes
Sip44 8_455 Sip44 . Sip44 8_555 120.11(3)    yes
Si4 8_455 Sip44 . Sip44 8_555 120.11(3)    yes
O5 8_455 Sip44 . Fep23 8_555 135.89(5)    yes
Om25 8_455 Sip44 . Fep23 8_555 135.89(5)    yes
Sip44 8_455 Sip44 . Fep23 8_555 118.198(11)    yes
Si4 8_455 Sip44 . Fep23 8_555 118.198(11)    yes
Si4 8_555 Sip44 . Fep23 8_555 62.314(9)    yes
O5 8_455 Sip44 . Fep22 . 109.78(5)    yes
Om25 8_455 Sip44 . Fep22 . 109.78(5)    yes
Sip44 8_455 Sip44 . Fep22 . 89.899(7)    yes
Si4 8_455 Sip44 . Fep22 . 89.899(7)    yes
Si4 8_555 Sip44 . Fep22 . 118.064(7)    yes
O5 8_455 Sip44 . Fep23 . 80.53(5)    yes
Om25 8_455 Sip44 . Fep23 . 80.53(5)    yes
Sip44 8_455 Sip44 . Fep23 . 61.822(10)    yes
Si4 8_455 Sip44 . Fep23 . 61.822(10)    yes
Si4 8_555 Sip44 . Fep23 . 89.920(14)    yes
O5 8_455 Sip44 . Om25 . 108.23(5)    yes
Om25 8_455 Sip44 . Om25 . 108.23(5)    yes
Sip44 8_455 Sip44 . Om25 . 122.34(5)    yes
Si4 8_455 Sip44 . Om25 . 122.34(5)    yes
Si4 8_555 Sip44 . Om25 . 21.29(4)    yes
O5 8_455 Sip44 . O5 . 108.23(5)    yes
Om25 8_455 Sip44 . O5 . 108.23(5)    yes
Sip44 8_455 Sip44 . O5 . 122.34(5)    yes
Si4 8_455 Sip44 . O5 . 122.34(5)    yes
Si4 8_555 Sip44 . O5 . 21.29(4)    yes
O5 8_455 Sip44 . Om26 . 108.17(8)    yes
Om25 8_455 Sip44 . Om26 . 108.17(8)    yes
Sip44 8_455 Sip44 . Om26 . 113.42(7)    yes
Si4 8_455 Sip44 . Om26 . 113.42(7)    yes
Si4 8_555 Sip44 . Om26 . 122.26(7)    yes
O5 8_455 Sip44 . O6 . 108.17(8)    yes
Om25 8_455 Sip44 . O6 . 108.17(8)    yes
Sip44 8_455 Sip44 . O6 . 113.42(7)    yes
Si4 8_455 Sip44 . O6 . 113.42(7)    yes
Si4 8_555 Sip44 . O6 . 122.26(7)    yes
O5 8_455 Sip44 . Om27 . 110.73(6)    yes
Om25 8_455 Sip44 . Om27 . 110.73(6)    yes
Sip44 8_455 Sip44 . Om27 . 89.98(4)    yes
Si4 8_455 Sip44 . Om27 . 89.98(4)    yes
Si4 8_555 Sip44 . Om27 . 89.88(4)    yes
O5 8_455 Sip44 . O7 . 110.73(6)    yes
Om25 8_455 Sip44 . O7 . 110.73(6)    yes
Sip44 8_455 Sip44 . O7 . 89.98(4)    yes
Si4 8_455 Sip44 . O7 . 89.98(4)    yes
Si4 8_555 Sip44 . O7 . 89.88(4)    yes
Sip44 8_555 Sip44 . Fep23 8_555 62.314(9)    yes
Sip44 8_555 Sip44 . Fep22 . 118.064(7)    yes
Fep23 8_555 Sip44 . Fep22 . 55.752(9)    yes
Sip44 8_555 Sip44 . Fep23 . 89.920(14)    yes
Fep23 8_555 Sip44 . Fep23 . 56.382(12)    yes
Fep22 . Sip44 . Fep23 . 56.325(12)    yes
Sip44 8_555 Sip44 . Om25 . 21.29(4)    yes
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Fep23 8_555 Sip44 . Om25 . 81.02(4)    yes
Fep22 . Sip44 . Om25 . 135.65(5)    yes
Fep23 . Sip44 . Om25 . 109.77(5)    yes
Sip44 8_555 Sip44 . O5 . 21.29(4)    yes
Fep23 8_555 Sip44 . O5 . 81.02(4)    yes
Fep22 . Sip44 . O5 . 135.65(5)    yes
Fep23 . Sip44 . O5 . 109.77(5)    yes
Sip44 8_555 Sip44 . Om26 . 122.26(7)    yes
Fep23 8_555 Sip44 . Om26 . 109.31(6)    yes
Fep22 . Sip44 . Om26 . 80.54(6)    yes
Fep23 . Sip44 . Om26 . 135.68(6)    yes
Om25 . Sip44 . Om26 . 108.22(8)    yes
Sip44 8_555 Sip44 . O6 . 122.26(7)    yes
Fep23 8_555 Sip44 . O6 . 109.31(6)    yes
Fep22 . Sip44 . O6 . 80.54(6)    yes
Fep23 . Sip44 . O6 . 135.68(6)    yes
Om25 . Sip44 . O6 . 108.22(8)    yes
Sip44 8_555 Sip44 . Om27 . 89.88(4)    yes
Fep23 8_555 Sip44 . Om27 . 32.68(3)    yes
Fep22 . Sip44 . Om27 . 33.03(4)    yes
Fep23 . Sip44 . Om27 . 33.04(4)    yes
Om25 . Sip44 . Om27 . 110.66(6)    yes
Sip44 8_555 Sip44 . O7 . 89.88(4)    yes
Fep23 8_555 Sip44 . O7 . 32.68(3)    yes
Fep22 . Sip44 . O7 . 33.03(4)    yes
Fep23 . Sip44 . O7 . 33.04(4)    yes
Om25 . Sip44 . O7 . 110.66(6)    yes
O5 . Sip44 . Om26 . 108.22(8)    yes
O5 . Sip44 . O6 . 108.22(8)    yes
O5 . Sip44 . Om27 . 110.66(6)    yes
Om26 . Sip44 . Om27 . 110.73(7)    yes
O6 . Sip44 . Om27 . 110.73(7)    yes
O5 . Sip44 . O7 . 110.66(6)    yes
Om26 . Sip44 . O7 . 110.73(7)    yes
O6 . Sip44 . O7 . 110.73(7)    yes
O5 8_455 Si4 . Sip44 8_455 21.29(4)    yes
Om25 8_455 Si4 . Sip44 8_455 21.29(4)    yes
O5 8_455 Si4 . Si4 8_455 21.29(4)    yes
Om25 8_455 Si4 . Si4 8_455 21.29(4)    yes
O5 8_455 Si4 . Si4 8_555 113.58(5)    yes
Om25 8_455 Si4 . Si4 8_555 113.58(5)    yes
Sip44 8_455 Si4 . Si4 8_555 120.11(3)    yes
Si4 8_455 Si4 . Si4 8_555 120.11(3)    yes
O5 8_455 Si4 . Sip44 8_555 113.58(5)    yes
Om25 8_455 Si4 . Sip44 8_555 113.58(5)    yes
Sip44 8_455 Si4 . Sip44 8_555 120.11(3)    yes
Si4 8_455 Si4 . Sip44 8_555 120.11(3)    yes
O5 8_455 Si4 . Fep23 8_555 135.89(5)    yes
Om25 8_455 Si4 . Fep23 8_555 135.89(5)    yes
Sip44 8_455 Si4 . Fep23 8_555 118.198(11)    yes
Si4 8_455 Si4 . Fep23 8_555 118.198(11)    yes
Si4 8_555 Si4 . Fep23 8_555 62.314(9)    yes
O5 8_455 Si4 . Fep22 . 109.78(5)    yes
Om25 8_455 Si4 . Fep22 . 109.78(5)    yes
Sip44 8_455 Si4 . Fep22 . 89.899(7)    yes
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Si4 8_455 Si4 . Fep22 . 89.899(7)    yes
Si4 8_555 Si4 . Fep22 . 118.064(7)    yes
O5 8_455 Si4 . Fep23 . 80.53(5)    yes
Om25 8_455 Si4 . Fep23 . 80.53(5)    yes
Sip44 8_455 Si4 . Fep23 . 61.822(10)    yes
Si4 8_455 Si4 . Fep23 . 61.822(10)    yes
Si4 8_555 Si4 . Fep23 . 89.920(14)    yes
O5 8_455 Si4 . Om25 . 108.23(5)    yes
Om25 8_455 Si4 . Om25 . 108.23(5)    yes
Sip44 8_455 Si4 . Om25 . 122.34(5)    yes
Si4 8_455 Si4 . Om25 . 122.34(5)    yes
Si4 8_555 Si4 . Om25 . 21.29(4)    yes
O5 8_455 Si4 . O5 . 108.23(5)    yes
Om25 8_455 Si4 . O5 . 108.23(5)    yes
Sip44 8_455 Si4 . O5 . 122.34(5)    yes
Si4 8_455 Si4 . O5 . 122.34(5)    yes
Si4 8_555 Si4 . O5 . 21.29(4)    yes
O5 8_455 Si4 . Om26 . 108.17(8)    yes
Om25 8_455 Si4 . Om26 . 108.17(8)    yes
Sip44 8_455 Si4 . Om26 . 113.42(7)    yes
Si4 8_455 Si4 . Om26 . 113.42(7)    yes
Si4 8_555 Si4 . Om26 . 122.26(7)    yes
O5 8_455 Si4 . O6 . 108.17(8)    yes
Om25 8_455 Si4 . O6 . 108.17(8)    yes
Sip44 8_455 Si4 . O6 . 113.42(7)    yes
Si4 8_455 Si4 . O6 . 113.42(7)    yes
Si4 8_555 Si4 . O6 . 122.26(7)    yes
O5 8_455 Si4 . Om27 . 110.73(6)    yes
Om25 8_455 Si4 . Om27 . 110.73(6)    yes
Sip44 8_455 Si4 . Om27 . 89.98(4)    yes
Si4 8_455 Si4 . Om27 . 89.98(4)    yes
Si4 8_555 Si4 . Om27 . 89.88(4)    yes
O5 8_455 Si4 . O7 . 110.73(6)    yes
Om25 8_455 Si4 . O7 . 110.73(6)    yes
Sip44 8_455 Si4 . O7 . 89.98(4)    yes
Si4 8_455 Si4 . O7 . 89.98(4)    yes
Si4 8_555 Si4 . O7 . 89.88(4)    yes
Sip44 8_555 Si4 . Fep23 8_555 62.314(9)    yes
Sip44 8_555 Si4 . Fep22 . 118.064(7)    yes
Fep23 8_555 Si4 . Fep22 . 55.752(9)    yes
Sip44 8_555 Si4 . Fep23 . 89.920(14)    yes
Fep23 8_555 Si4 . Fep23 . 56.382(12)    yes
Fep22 . Si4 . Fep23 . 56.325(12)    yes
Sip44 8_555 Si4 . Om25 . 21.29(4)    yes
Fep23 8_555 Si4 . Om25 . 81.02(4)    yes
Fep22 . Si4 . Om25 . 135.65(5)    yes
Fep23 . Si4 . Om25 . 109.77(5)    yes
Sip44 8_555 Si4 . O5 . 21.29(4)    yes
Fep23 8_555 Si4 . O5 . 81.02(4)    yes
Fep22 . Si4 . O5 . 135.65(5)    yes
Fep23 . Si4 . O5 . 109.77(5)    yes
Sip44 8_555 Si4 . Om26 . 122.26(7)    yes
Fep23 8_555 Si4 . Om26 . 109.31(6)    yes
Fep22 . Si4 . Om26 . 80.54(6)    yes
Fep23 . Si4 . Om26 . 135.68(6)    yes
```

```
Om25 . Si4 . Om26 . 108.22(8)    yes
Sip44 8_555 Si4 . O6 . 122.26(7)    yes
Fep23 8_555 Si4 . O6 . 109.31(6)    yes
Fep22 . Si4 . O6 . 80.54(6)    yes
Fep23 . Si4 . O6 . 135.68(6)    yes
Om25 . Si4 . O6 . 108.22(8)    yes
Sip44 8_555 Si4 . Om27 . 89.88(4)    yes
Fep23 8_555 Si4 . Om27 . 32.68(3)    yes
Fep22 . Si4 . Om27 . 33.03(4)    yes
Fep23 . Si4 . Om27 . 33.04(4)    yes
Om25 . Si4 . Om27 . 110.66(6)    yes
Sip44 8_555 Si4 . O7 . 89.88(4)    yes
Fep23 8_555 Si4 . O7 . 32.68(3)    yes
Fep22 . Si4 . O7 . 33.03(4)    yes
Fep23 . Si4 . O7 . 33.04(4)    yes
Om25 . Si4 . O7 . 110.66(6)    yes
O5 . Si4 . Om26 . 108.22(8)    yes
O5 . Si4 . O6 . 108.22(8)    yes
O5 . Si4 . Om27 . 110.66(6)    yes
Om26 . Si4 . Om27 . 110.73(7)    yes
O6 . Si4 . Om27 . 110.73(7)    yes
O5 . Si4 . O7 . 110.66(6)    yes
Om26 . Si4 . O7 . 110.73(7)    yes
O6 . Si4 . O7 . 110.73(7)    yes
Si4 8_555 Om25 . Si4 . 137.42(8)    yes
Sip44 8_555 Om25 . Si4 . 137.42(8)    yes
Si4 8_555 Om25 . Sip44 . 137.42(8)    yes
Sip44 8_555 Om25 . Sip44 . 137.42(8)    yes
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Sip44 8_555 O5 . Si4 . 137.42(8)    yes
Si4 8_555 O5 . Sip44 . 137.42(8)    yes
Sip44 8_555 O5 . Sip44 . 137.42(8)    yes
Si4 6_555 Om26 . Si4 . 137.78(12)    yes
Sip44 6_555 Om26 . Si4 . 137.78(12)    yes
Si4 6_555 Om26 . Sip44 . 137.78(12)    yes
Sip44 6_555 Om26 . Sip44 . 137.78(12)    yes
Si4 6_555 O6 . Si4 . 137.78(12)    yes
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Si4 6_555 O6 . Sip44 . 137.78(12)    yes
Sip44 6_555 O6 . Sip44 . 137.78(12)    yes
Fep23 . Om27 . Fep22 . 95.15(4)    yes
Fep23 . Om27 . Mgp23 4_556 95.57(4)    yes
Fep22 . Om27 . Mgp23 4_556 94.36(4)    yes
Fep23 . Om27 . Fep23 4_556 95.57(4)    yes
Fep22 . Om27 . Fep23 4_556 94.36(4)    yes
Fep23 . Om27 . Mgp22 . 95.15(4)    yes
Mgp23 4_556 Om27 . Mgp22 . 94.36(4)    yes
Fep23 4_556 Om27 . Mgp22 . 94.36(4)    yes
Fep22 . Om27 . Mgp23 . 95.15(4)    yes
Mgp23 4_556 Om27 . Mgp23 . 95.57(4)    yes
Fep23 4_556 Om27 . Mgp23 . 95.57(4)    yes
Mgp22 . Om27 . Mgp23 . 95.15(4)    yes
Fep23 . Om27 . Sip44 . 121.54(6)    yes
Fep22 . Om27 . Sip44 . 121.44(6)    yes
Mgp23 4_556 Om27 . Sip44 . 121.87(5)    yes
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Mgp22 . Om27 . Sip44 . 121.44(6)    yes
Fep23 . Om27 . Si4 . 121.54(6)    yes
Fep22 . Om27 . Si4 . 121.44(6)    yes
Mgp23 4_556 Om27 . Si4 . 121.87(5)    yes
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Mgp22 . Om27 . Si4 . 121.44(6)    yes
Mgp23 . Om27 . Sip44 . 121.54(6)    yes
Mgp23 . Om27 . Si4 . 121.54(6)    yes
Fep23 . O7 . Fep22 . 95.15(4)    yes
Fep23 . O7 . Mgp23 4_556 95.57(4)    yes
Fep22 . O7 . Mgp23 4_556 94.36(4)    yes
Fep23 . O7 . Fep23 4_556 95.57(4)    yes
Fep22 . O7 . Fep23 4_556 94.36(4)    yes
Fep23 . O7 . Mgp22 . 95.15(4)    yes
Mgp23 4_556 O7 . Mgp22 . 94.36(4)    yes
Fep23 4_556 O7 . Mgp22 . 94.36(4)    yes
Fep22 . O7 . Mgp23 . 95.15(4)    yes
Mgp23 4_556 O7 . Mgp23 . 95.57(4)    yes
Fep23 4_556 O7 . Mgp23 . 95.57(4)    yes
Mgp22 . O7 . Mgp23 . 95.15(4)    yes
Fep23 . O7 . Sip44 . 121.54(6)    yes
Fep22 . O7 . Sip44 . 121.44(6)    yes
Mgp23 4_556 O7 . Sip44 . 121.87(5)    yes
Fep23 4_556 O7 . Sip44 . 121.87(5)    yes
Mgp22 . O7 . Sip44 . 121.44(6)    yes
Fep23 . O7 . Si4 . 121.54(6)    yes
Fep22 . O7 . Si4 . 121.44(6)    yes
Mgp23 4_556 O7 . Si4 . 121.87(5)    yes
Fep23 4_556 O7 . Si4 . 121.87(5)    yes
Mgp22 . O7 . Si4 . 121.44(6)    yes
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Mgp23 . O7 . Si4 . 121.54(6)    yes
Mgp23 2_566 Fm18 . Mgp23 . 97.68(7)    yes
Mgp23 2_566 Fm18 . Fep22 7_556 98.12(5)    yes
Mgp23 . Fm18 . Fep22 7_556 98.12(5)    yes
Mgp23 2_566 Fm18 . Mgp22 3_555 98.12(5)    yes
Mgp23 . Fm18 . Mgp22 3_555 98.12(5)    yes
Mgp23 2_566 Fm18 . Fep23 . 97.68(7)    yes
Fep22 7_556 Fm18 . Fep23 . 98.12(5)    yes
Mgp22 3_555 Fm18 . Fep23 . 98.12(5)    yes
Mgp23 2_566 Om28 . Mgp23 . 97.68(7)    yes
Mgp23 2_566 Om28 . Mgp22 3_555 98.12(5)    yes
Mgp23 . Om28 . Mgp22 3_555 98.12(5)    yes
Mgp23 2_566 Om28 . Fep22 3_555 98.12(5)    yes
Mgp23 . Om28 . Fep22 3_555 98.12(5)    yes
Mgp23 2_566 Om28 . Fep23 . 97.68(7)    yes
Mgp22 3_555 Om28 . Fep23 . 98.12(5)    yes
Fep22 3_555 Om28 . Fep23 . 98.12(5)    yes
```

```
Scordari et al.: FLUOROPHLOGOPITE FROM PIANO DELLE CONCAZZE (Mt. ETNA, ITALY);
Deposit material (7 total items); AM-13-046; American Mineralogist May June 2013

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_audit_creation_date        "10-02-11"
_audit_creation_method CRYSTALS_ver_14.06

_oxford_structure_analysis_title  'THIS IS THE DEFAULT SETTING FOR THE TITLE, P'
_chemical_name_systematic         ?
_chemical_melting_point           ?

#looking for refcif


# Check this file using the IUCr facility at:
# http://checkcif.iucr.org/

# The content below is held in the file 'script/refcif.dat'. This is a text
# file which you may edit to reflect local conditions.
# Items which need looking at are represented by a '?'.
# Items for which there are choices are prefixed with 'choose from'.

_publ_contact_letter
;
 Please consider this CIF submission for
 publication as a Short Format Paper in Acta
 Crystallographica E.
;
_publ_contact_author_name         'Anthony Other'
_publ_contact_author_address
;
 Chemical Crystallography Laboratory,
 Department of Chemistry,
 University of Nowhere,
 Nowhere, NO1 4T, UK.
;

_publ_contact_author_phone        '+44 1865 000000'
_publ_contact_author_fax          '+44 1865 000000'
_publ_contact_author_email        'a.n.other@chem.no.ac.uk'
_publ_requested_journal           'Section E'
_publ_requested_category          EO # choose from: FI FM FO CI CM CO AD
_publ_requested_coeditor_name     'Prof William Clegg'

_publ_section_title
# Title of paper - generally just the systematic or trivial name
; ?
;

# The loop structure below should contain the names and addresses of all
# authors, in the required order of publication. Repeat as necessary.
loop_
 _publ_author_name
 _publ_author_address
 _publ_author_footnote
```

```
 'Other, Anthony N.' # Author 1
;
 ? # Address for author 1
;
;
 ? # Footnote for author 1
;
 'Else, S. O.' # Author 2
;
 ? # Address 2
;
;
 ? # Footnote 2
;


_publ_section_abstract
# Text of the abstract
# (a) The Abstract must be self-contained and comprehensible
# without the rest of the paper. This means no references
# to atom names or to compound numbers; compounds must be
# identified as the title compound, or by name or by some other
# means such as derivatives of each other (e.g. "the
# corresponding ketone").
# (b) The chemical formula of "the title compound" must be given.
# (c) Any crystallographic molecular symmetry should be
# mentioned, and also the presence of more than one molecule
# in the asymmetric unit (i.e. anything other than Z'=1).
;
?
;

_publ_section_related_literature  # Not in version 2.3.1
##################################################################
# Essential references (e.g. to the origin of the material studied, related
# structural studies, and to information supporting the reported structure)
# should be cited in this section, with appropriate very brief explanatory
# text, for example `The synthesis of the complex is described by Jones
# (2001). The anhydrous complex has essentially the same coordination
# geometry (Smith, 2003).' In addition, all references included in the
# supplementary materials, but not elsewhere in the published article,
# should be given here. The simple text `For related literature, see:...'
# may be used, but further details should be provided in the Comment
# section, which forms part of the supplementary material.
##################################################################
;
?
;

_publ_section_comment
# Text of the paper
# Note that atoms are referenced as N2, not N(2) or N~2~
# If text containing () occur within (), the outer ones should be []
# Figures should be referenced as Fig.
;
```

```
?
;
_publ_section_acknowledgements    # Acknowledgments
;
?
;
_publ_section_figure_captions
# Captions to figures - Start each caption on a new line after a blank line
;
Fig. 1.
The title compound with displacement ellipsoids drawn at the 50%
probability level. H atoms are shown as spheres of
arbitary radius.

;


_publ_section_exptl_refinement
# Some potentially useful phrases are donated by Bill Clegg:
;
In the absence of significant anomalous scattering, Friedel pairs were
merged.

The absolute configuration was arbitrarily assigned.

The relatively large ratio of minimum to maximum corrections applied
in the multiscan process (1:nnn) reflect changes in the illuminated
volume of the crystal.

Changes in illuminated volume were kept to a minimum, and were
taken into account (G\"orbitz, 1999) by the multi-scan inter-frame
scaling (DENZO/SCALEPACK, Otwinowski & Minor, 1997).


G\"orbitz, C. H. (1999). Acta Cryst. B55, 1090-1098.




The H atoms were all located in a difference map, but those
attached to carbon atoms were repositioned geometrically.
The H atoms were initially refined with soft restraints on the
bond lengths and angles to regularise their geometry
(C---H in the range 0.93--0.98,
N---H in the range 0.86--0.89
N---H to 0.86
O---H = 0.82
\%A)
and
U~iso~(H) (in the range 1.2-1.5 times U~eq~ of the parent atom),
after which the positions were refined with riding constraints.

;

_publ_section_exptl_prep
```

```
# Brief details or a reference. Include solvent if known
;
?
;

# Hand-made tables can be put in the cif. The number of columns
# is set in the loop header.
# The contants of each column can either be a piece of text
# without any spaces (eg a number) or other text enclosed in " "
# Remove the leading # in the following example

#geom_extra_table_head_A
#;
#Table 2.
#Molecular parameters deviating most from MOGUL averages
#;
#
#loop_
#_geom_extra_tableA_col_1
#_geom_extra_tableA_col_2
#_geom_extra_tableA_col_3
#_geom_extra_tableA_col_4
#
#Parameter "Observed Value" "MOGUL Value" "MOGUL e.s.d"
#N5-C6-C4 129 124 "7 (\%)"
#C3-O10-C2 105 109 "2 (\%)"
#C6-O7 1.25 1.22 ".02 (\%A)"
#

data_1

_exptl_special_details
;
 The crystal was
placed in the cold stream of an Oxford
Cryosystems open-flow nitrogen cryostat (Cosier &
Glazer, 1986) with a nominal stability of 0.1K.

Cosier, J. & Glazer, A.M., 1986. J. Appl. Cryst. 105 107.
;
_refine_special_details
;
?
;
# End of 'script/refcif.dat'
#end of refcif
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_cell_length_c                   10.1159(3)
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_cell_angle_beta                 100.249(2)
_cell_angle_gamma                90
_cell_volume                     488.72(3)

_symmetry_cell_setting           'Monoclinic'
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_symmetry_space_group_name_Hall   ?
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 'x+1/2,y+1/2,z'
 '-x+1/2,-y+1/2,-z'
 '-x,y,-z'
 'x,-y,z'
 '-x+1/2,y+1/2,-z'
 'x+1/2,-y+1/2,z'

loop_
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_atom_type_scat_Cromer_Mann_b1
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_atom_type_scat_Cromer_Mann_b2
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_atom_type_scat_Cromer_Mann_c
_atom_type_scat_source
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   0.0408  57.7997   0.0030 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Lip1    0.0000   0.0000   0.6968   4.6237   0.7888   1.9557   0.3414   0.6316
   0.1563  10.0953   0.0167 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fep2    0.3010   0.8450  11.0424   4.6538   7.3740   0.3053   4.1346  12.0546
   0.4399  31.2809   1.0097 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Cap2    0.2030   0.3060  15.6348  -0.0074   7.9518   0.6089   8.4372  10.3116
   0.8537  25.9905 -14.8750 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Nap1    0.0300   0.0250   3.2565   2.6671   3.9362   6.1153   1.3998   0.2001
   1.0032  14.0390   0.4040 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Kp1     0.1790   0.2500   7.9578  12.6331   7.4917   0.7674   6.3590  -0.0020
   1.1915  31.9128  -4.9978 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Mgp2    0.0420   0.0360   3.4988   2.1676   3.8378   4.7542   1.3284   0.1850
   0.8497  10.1411   0.4853 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fep3    0.3010   0.8450  11.1764   4.6147   7.3863   0.3005   3.3948  11.6729
   0.0724  38.5566   0.9707 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Alp3    0.0560   0.0520   4.1745   1.9382   3.3876   4.1455   1.2030   0.2288
   0.5281   8.2852   0.7068 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Crp3    0.2840   0.6240   9.6809   5.5946   7.8114   0.3344   2.8760  12.8288
   0.1136  32.8761   0.5183 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Zrp4   -2.9650   0.5600  18.1668   1.2148  10.0562  10.1483   1.0112  21.6054
  -2.6479  -0.1028   9.4145 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Tip4    0.2480   0.4460  19.5114   0.1789   8.2347   6.6702   2.0134  -0.2926
   1.5208  12.9464 -13.2800 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Si      0.0720   0.0710   6.2915   2.4386   3.0353  32.3337   1.9891   0.6785
   1.5410  81.6937   1.1407 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Sip4    0.0720   0.0710   4.4392   1.6417   3.2034   3.4376   1.1945   0.2149
   0.4165   6.6536   0.7463 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
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 O       0.0080   0.0060   3.0485  13.2771   2.2868   5.7011   1.5463   0.3239
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 Om2     0.0080   0.0060   3.7504  16.5151   2.8429   6.5920   1.5430   0.3192
   1.6209  43.3486   0.2421 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Om1     0.0080   0.0060   4.1916  12.8573   1.6397   4.1724   1.5267  47.0179
 -20.3070  -0.0140  21.9412 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 F       0.0140   0.0100   3.5392  10.2825   2.6412   4.2944   1.5170   0.2615
   1.0243  26.1476   0.2776 'International Tables Vol C 4.2.6.8 and 6.1.1.4'
 Fm1     0.0140   0.0100   3.6322   5.2776   3.5106  14.7353   1.2606   0.4423
   0.9407  47.3437   0.6534 'International Tables Vol C 4.2.6.8 and 6.1.1.4'


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Kp10.50 Lip10.50
# Dc =      2.59 Fooo =    320.05 Mu =     50.52 M =     95.25
# Found Formula = F0.16 Fep20.07 Kp10.06 Mgp20.25 O1.06 Om21.28 Si0.60 Sip40.39
# Dc =      2.95 FOOO =    320.05 Mu =     49.81 M =    108.55

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Si0.60 Sip40.39'
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Si0.60 Sip40.39'
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_cell_measurement_temperature          293

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_exptl_crystal_density_diffrn     2.950
_exptl_crystal_density_meas       ?
_exptl_crystal_density_method     'not measured'
# Non-dispersive F(000):
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_exptl_absorpt_coefficient_mu     4.981

# Sheldrick geometric approximatio 1.00 1.00
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_diffrn_radiation_type            'Mo K\a'
_diffrn_radiation_wavelength           0.71070
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# If a reference occurs more than once, delete the author
# and date from subsequent references.
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_computing_cell_refinement        'USER DEFINED CELL REFINEMENT'
_computing_data_reduction         'USER DEFINED DATA REDUCTION'
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_computing_structure_refinement   'CRYSTALS (Betteridge et al., 2003)'
_computing_publication_material   'CRYSTALS (Betteridge et al., 2003)'
_computing_molecular_graphics     'CAMERON (Watkin et al., 1996)'

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# Number of reflections without Friedels Law is 0
# Number of reflections with Friedels Law is 1912
# Theoretical number of reflections is about 1953


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_diffrn_measured_fraction_theta_full 0.995


_diffrn_reflns_limit_h_min        -10
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_diffrn_reflns_limit_l_min        0
_diffrn_reflns_limit_l_max        19
_reflns_limit_h_min               -10
_reflns_limit_h_max               10
_reflns_limit_k_min               0
_reflns_limit_k_max               17
_reflns_limit_l_min               0
_reflns_limit_l_max               19

_oxford_diffrn_Wilson_B_factor    0.00
_oxford_diffrn_Wilson_scale       0.00

_atom_sites_solution_primary      direct #heavy,direct,difmap,geom
# _atom_sites_solution_secondary  difmap
_atom_sites_solution_hydrogens    geom

_refine_diff_density_min          -0.55
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```
_refine_diff_density_max         0.58


# The current dictionary definitions do not cover the
# situation where the reflections used for refinement were
# selected by a user-defined sigma threshold


# The values actually used during refinement
_oxford_reflns_threshold_expression_ref     I>3.0\s(I)
_refine_ls_number_reflns         2159
_refine_ls_number_restraints     24
_refine_ls_number_parameters     71
_oxford_refine_ls_R_factor_ref   0.0267
_refine_ls_wR_factor_ref         0.0258
_refine_ls_goodness_of_fit_ref   0.6985
_refine_ls_shift/su_max          0.0145265
_refine_ls_shift/su_mean         0.0015554



# choose from: rm (reference molecule of known chirality),
# ad (anomolous dispersion - Flack), rmad (rm and ad),
# syn (from synthesis), unk (unknown) or . (not applicable).
_chemical_absolute_configuration  '.'


_refine_ls_structure_factor_coef  F
_refine_ls_matrix_type           full
_refine_ls_hydrogen_treatment    none          # none, undef, noref, refall,
                                               # refxyz, refU, constr or mixed

# WARNING. The IUCr will not accept Unit Weights
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
;
 Method= Quasi-Unit weights
 W = 1.0 or 1./2F
;
# Insert your own references if required - in alphabetical order
_publ_section_references
;
User-defined structure solution reference

User-defined data collection reference

User defined data reduction

User defined cell refinement

Betteridge, P.W., Carruthers, J.R., Cooper, R.I.,
Prout, K. & Watkin, D.J. (2003). J. Appl. Cryst. 36, 1487.

Watkin, D.J., Prout, C.K. & Pearce, L.J. (1996). CAMERON, Chemical
Crystallography Laboratory, Oxford, UK.
```

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;

# Uequiv = arithmetic mean of Ui i.e. Ueqiv = (U1+U2+U3)/3

# Replace last . with number of unfound hydrogen atomsattached to an atom.

# ..._refinement_flags_...
# . no refinement constraints          S special position constraint on site
# G rigid group refinement of site     R riding atom
# D distance or angle restraint on site T thermal displacement constraints
# U Uiso or Uij restraint (rigid bond)  P partial occupancy constraint


loop_
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_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_occupancy
_atom_site_adp_type
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_atom_site_disorder_assembly
_atom_site_disorder_group
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Fep22 Fep2 0.0000 0.0000 0.5000 0.0098 0.2323(6) Uani . . P . . . .
Mgp23 Mgp2 0.0000 0.33616(5) 0.5000 0.0111 0.7932(7) Uani . . P . . . .
Fep23 Fep2 0.0000 0.33616(5) 0.5000 0.0111 0.2065(6) Uani . . P . . . .
Sip44 Sip4 0.07472(5) 0.16681(3) 0.22370(3) 0.0090 0.3878(9) Uani . . P . . . .
Si4 Si 0.07472(5) 0.16681(3) 0.22370(3) 0.0090 0.5930(9) Uani . . P . . . .
Om25 Om2 0.3170(2) 0.2379(1) 0.16507(8) 0.0187 0.4592(8) Uani . . P . . . .
O5 O 0.3170(2) 0.2379(1) 0.16507(8) 0.0187 0.5408(8) Uani . . P . . . .
Om26 Om2 0.0306(3) 0.0000 0.16507(8) 0.0183 0.4776(8) Uani . . P . . . .
O6 O 0.0306(3) 0.0000 0.16507(8) 0.0183 0.5224(8) Uani . . P . . . .
Om27 Om2 0.1309(1) 0.16750(8) 0.38995(7) 0.0104 0.6138(8) Uani . . P . . . .
O7 O 0.1309(1) 0.16750(8) 0.38995(7) 0.0104 0.3862(8) Uani . . P . . . .
Om28 Om2 0.1302(2) 0.5000 0.4005(1) 0.0129 0.3591(7) Uani . . P . . . .
F8 F 0.1302(2) 0.5000 0.4005(1) 0.0129 0.6417(7) Uani . . P . . . .
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_atom_site_aniso_U_22
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_atom_site_aniso_U_13
_atom_site_aniso_U_12
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Mgp22 0.00868(19) 0.00776(19) 0.0135(2) 0.0000 0.00323(14) 0.0000
Fep22 0.00868(19) 0.00776(19) 0.0135(2) 0.0000 0.00323(14) 0.0000
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Fep23 0.00765(13) 0.01402(16) 0.01150(14) 0.0000 0.00138(10) 0.0000
Sip44 0.00876(9) 0.00829(10) 0.00993(10) 0.00002(8) 0.00169(7) 0.00007(8)
Si4 0.00876(9) 0.00829(10) 0.00993(10) 0.00002(8) 0.00169(7) 0.00007(8)
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O5 0.0173(3) 0.0244(4) 0.0145(3) -0.0016(3) 0.0032(2) -0.0069(3)
Om26 0.0272(6) 0.0128(4) 0.0139(4) 0.0000 0.0012(4) 0.0000
O6 0.0272(6) 0.0128(4) 0.0139(4) 0.0000 0.0012(4) 0.0000
Om27 0.0105(2) 0.0104(2) 0.0102(2) -0.00008(19) 0.00164(18) 0.00053(19)
O7 0.0105(2) 0.0104(2) 0.0102(2) -0.00008(19) 0.00164(18) 0.00053(19)
Om28 0.0119(3) 0.0142(4) 0.0127(3) 0.0000 0.0023(3) 0.0000
F8 0.0119(3) 0.0142(4) 0.0127(3) 0.0000 0.0023(3) 0.0000

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Mgp22 . O7 . 2.0944(7)    yes
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Fep22 . O7 2_556 2.0944(7)    yes
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Fep22 . F8 3_445 2.0455(10)    yes
Fep22 . Om28 7_546 2.0455(10)    yes
Fep22 . Om28 3_445 2.0455(10)    yes
Fep22 . Om27 . 2.0944(7)    yes
Fep22 . O7 . 2.0944(7)    yes
Mgp23 . Om28 2_566 2.0064(7)    yes
Mgp23 . F8 2_566 2.0064(7)    yes
Mgp23 . Om27 8_455 2.0795(7)    yes
Mgp23 . Om27 4_556 2.0795(7)    yes
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Mgp23 . O7 . 2.1023(8)    yes
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Fep23 . O7 5_556 2.1023(8)    yes
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Fep23 . O7 . 2.1023(8)    yes
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Sip44 . O6 . 1.6480(5)    yes
Sip44 . Om27 . 1.6550(7)    yes
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Si4 . Om26 . 1.6480(5)    yes
Si4 . O6 . 1.6480(5)    yes
Si4 . Om27 . 1.6550(7)    yes
Si4 . O7 . 1.6550(7)    yes
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F8 4_556 Mgp22 . O7 . 83.09(3) yes
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O7 2_556 Fep22 . F8 4_556 96.91(3) yes
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O7 2_556 Fep22 . Om28 3_445 83.09(3)    yes
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O7 2_556 Fep22 . Om27 . 179.995    yes
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Om27 5_556 Fep22 . F8 3_445 83.09(3)    yes
F8 4_556 Fep22 . F8 3_445 179.994    yes
Om27 5_556 Fep22 . Om28 7_546 96.91(3)    yes
F8 3_445 Fep22 . Om28 7_546 179.994    yes
Om27 5_556 Fep22 . Om28 3_445 83.09(3)    yes
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Om27 5_556 Fep23 . O7 4_556 94.31(3)    yes
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Om27 8_455 Fep23 . O7 4_556 178.14(5)    yes
Om27 5_556 Fep23 . O7 8_455 84.31(3)    yes
O7 5_556 Fep23 . O7 8_455 84.31(3)    yes
Om27 4_556 Fep23 . O7 8_455 178.14(5)    yes
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Om27 5_556 Fep23 . F8 5_556 96.59(3)    yes
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Om27 5_556 Fep23 . Om28 5_556 96.59(3)    yes
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Om27 4_556 Fep23 . Om28 5_556 96.98(3)    yes
Om27 8_455 Fep23 . Om28 5_556 84.43(3)    yes
O7 4_556 Fep23 . Om28 5_556 96.98(3)    yes
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Om27 4_556 Fep23 . Om27 . 84.31(3)    yes
Om27 8_455 Fep23 . Om27 . 94.31(3)    yes
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Om27 5_556 Fep23 . O7 . 84.56(4)    yes
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Om27 8_455 Fep23 . O7 . 94.31(3)    yes
O7 4_556 Fep23 . O7 . 84.31(3)    yes
O7 8_455 Fep23 . F8 5_556 84.43(3)    yes
O7 8_455 Fep23 . Om28 5_556 84.43(3)    yes
O7 8_455 Fep23 . Om27 . 94.31(3)    yes
F8 5_556 Fep23 . Om27 . 178.19(3)    yes
Om28 5_556 Fep23 . Om27 . 178.19(3)    yes
O7 8_455 Fep23 . O7 . 94.31(3)    yes
F8 5_556 Fep23 . O7 . 178.19(3)    yes
Om28 5_556 Fep23 . O7 . 178.19(3)    yes
O5 8_455 Sip44 . Om25 . 108.24(3)    yes
Om25 8_455 Sip44 . Om25 . 108.24(3)    yes
O5 8_455 Sip44 . O5 . 108.24(3)    yes
Om25 8_455 Sip44 . O5 . 108.24(3)    yes
O5 8_455 Sip44 . Om26 . 108.24(6)    yes
Om25 8_455 Sip44 . Om26 . 108.24(6)    yes
O5 8_455 Sip44 . O6 . 108.24(6)    yes
Om25 8_455 Sip44 . O6 . 108.24(6)    yes
O5 8_455 Sip44 . Om27 . 110.60(4)    yes
Om25 8_455 Sip44 . Om27 . 110.60(4)    yes
O5 8_455 Sip44 . O7 . 110.60(4)    yes
Om25 8_455 Sip44 . O7 . 110.60(4)    yes
Om25 . Sip44 . Om26 . 108.32(6)    yes
Om25 . Sip44 . O6 . 108.32(6)    yes
Om25 . Sip44 . Om27 . 110.66(4)    yes
Om25 . Sip44 . O7 . 110.66(4)    yes
O5 . Sip44 . Om26 . 108.32(6)    yes
O5 . Sip44 . O6 . 108.32(6)    yes
O5 . Sip44 . Om27 . 110.66(4)    yes
Om26 . Sip44 . Om27 . 110.69(5)    yes
O6 . Sip44 . Om27 . 110.69(5)    yes
O5 . Sip44 . O7 . 110.66(4)    yes
Om26 . Sip44 . O7 . 110.69(5)    yes
O6 . Sip44 . O7 . 110.69(5)    yes
O5 8_455 Si4 . Om25 . 108.24(3)    yes
Om25 8_455 Si4 . Om25 . 108.24(3)    yes
O5 8_455 Si4 . O5 . 108.24(3)    yes
Om25 8_455 Si4 . O5 . 108.24(3)    yes
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O5 8_455 Si4 . Om26 . 108.24(6)    yes
Om25 8_455 Si4 . Om26 . 108.24(6)    yes
O5 8_455 Si4 . O6 . 108.24(6)    yes
Om25 8_455 Si4 . O6 . 108.24(6)    yes
O5 8_455 Si4 . Om27 . 110.60(4)    yes
Om25 8_455 Si4 . Om27 . 110.60(4)    yes
O5 8_455 Si4 . O7 . 110.60(4)    yes
Om25 8_455 Si4 . O7 . 110.60(4)    yes
Om25 . Si4 . Om26 . 108.32(6)    yes
Om25 . Si4 . O6 . 108.32(6)    yes
Om25 . Si4 . Om27 . 110.66(4)    yes
Om25 . Si4 . O7 . 110.66(4)    yes
O5 . Si4 . Om26 . 108.32(6)    yes
O5 . Si4 . O6 . 108.32(6)    yes
O5 . Si4 . Om27 . 110.66(4)    yes
Om26 . Si4 . Om27 . 110.69(5)    yes
O6 . Si4 . Om27 . 110.69(5)    yes
O5 . Si4 . O7 . 110.66(4)    yes
Om26 . Si4 . O7 . 110.69(5)    yes
O6 . Si4 . O7 . 110.69(5)    yes
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