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Figure DR1. Example calibration curves for SIMS analyses from different analytical sessions. For details see Hauri et al. (2002, 2006a,b). Calibration factors (e.g. line slopes) are listed adjacent to the run dates. Some glass standards > 2 wt% H$_2$O are listed for reference (see Hauri et al., 2006a and references therein for others), as are some cpx analyzed.
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**Figure DR2.** Correlation of water partitioning between pyroxene and melt, and the proportion of the tetrahedral site occupied by $\text{Al}^{IV}/(\text{IV})_T$. Data derived from laboratory experiments and analyses reported in Hauri et al. (2006a). Note both orthopyroxenes and clinopyroxenes are included in the regression. Gray line and shaded field are original regression and error envelope from Hauri et al. (2006a; their Fig. 2E). Solid line is the new regression (see discussion), dotted lines represent the calculated error envelope.
1. SIMS CALIBRATION OF H₂O IN CLINOPYROXENE

Data for this study were acquired by SIMS over several analytical sessions using the Cameca 6f ion microprobe at the Carnegie Institution of Washington’s Department of Terrestrial Magnetism, from 2006 to 2007. The $^{16}\text{O}^{1}\text{H}/^{30}\text{Si}$ signals in the clinopyroxenes were calibrated in reference to the following pyroxene "standards" and their independently-determined H₂O concentrations as given in parentheses (ppm) from Bell et al (1995) and Bell et al (2004): PMR-53 (268), KBH-1-opx (217), ROM271#10 (195), ROM271#16 (439) and ROM271#21 (490). In some sessions, calibration factors were determined directly from linear regression on pyroxene standards, yielding factors that varied from 0.48 – 0.43 ($\text{H}_2\text{O (wt\%)}/[^{16}\text{O}^{1}\text{H}/^{30}\text{Si}]$), as illustrated in Fig. 1b. Calibration involved simple multiplication of $^{16}\text{O}^{1}\text{H}/^{30}\text{Si}$ signals by these factors, without correction of Si explicitly (as in Hauri et al., 2006a and b). The scatter about the lines in Fig. 1b reflects uncertainty in the published H₂O contents of the pyroxene standards, and is not due to matrix effects within the pyroxene compositions (which should only on the order of a few percent, using the method of Hauri et al, 2006b). In other sessions, we used basaltic glass calibrations, which ranged from 0.31 to 0.40 (based on H₂O determinations reported in Hauri 2002; Hauri et al., 2006a and b), and then applied a correction of 1.3846 to account for the off-set in calibration factors between cpx/glass as determined in all sessions from 2006-2007. The off-set is presumably the manifestation of matrix effects between basaltic glass and cpx, possibly similar in cause to those that affect H isotopic fractionation (Hauri et al., 2006b). Fig. 1a shows an example of the glass
calibrations, which are in general better determined than the cpx calibration, but also based on H₂O concentrations well above the range measured in cpx.

2. PARTITIONING OF WATER BETWEEN CPX AND MAFIC MELT

A number of studies have recognized the importance of pyroxene composition, specifically Al, on the crystal/melt partitioning of H₂O (Aubaud et al., 2004; Hauri et al., 2006a; Mierdel et al., 2007; Grant et al., 2007). For example, Hauri et al (2006a) show a positive correlation between D₇H₂O px/liq and the fraction of the tetrahedral site occupied by Al (Al⁴/[IV]Total), and the expression developed therein is the basis for the calculations in this paper. We have modified the expression, however, to improve upon two related issues. One is that the original regression had the unfortunate result of predicting negative D at zero Al. Second, because of the lack of data for low Al pyroxenes, the errors on the regression became unacceptably large at low Al⁴/[IV]Total (and D), causing the calculated H₂O contents of liquid to have very high and uncertain values (because D is in the denominator of the calculation). In order to improve constraints near the origin of the regression of D on Al⁴/[IV]Total, we have modified it by estimating the D for Al-free pyroxenes using the solubility data of Bromily et al. (2004) for cpx and Rauch and Keppler (2002) for opx, coupled with a model for H₂O solubility in a basaltic melt (Dixon et al., 1995) calculated from 0.3-10 kb. This gives a D₇H₂O px/liq for Al-free pyroxene of 0.001 +/- 0.0005 (where the uncertainty derives from the range in pressure). The y-intercept of the regression is then forced near this point by strongly weighting it in the regression, leading to a slightly different partitioning relationship: D₇H₂O px/liq =
0.29(Al$^{IV}$/[IV]$^{Total}$) + 0.00099, with uncertainties on the slope and intercept as given in Figure 2. This has the effect of reducing errors in D's calculated when Al$^{IV}$/[IV]$^{Total} < 0.04$, and of predicting a finite D at zero Al, both of which mark improvements. Clearly further experimental data are welcome in constraining the $D_{H_2O}^{cpx/liq}$ for conditions relevant to erupting mafic magmas, but the general coherence in this study between H$_2$O measured in melt inclusions, and that calculated from cpx, supports a partitioning relationship similar to the one used here.

The use of Al$^{IV}$/[IV]$^{Total}$ as a pyroxene component follows directly from that published in Hauri et al (2006a), in their Figure 2. In calculating Al$^{IV}$/[IV]$^{Total}$, pyroxene analyses were first converted from weight percent to single cation fractions, assuming all Fe as Fe$^{2+}$. The sum of cations on the tetrahedral site include all of the Si, and the sum of the Al$^{IV}$ in the Ca-Tschermak component (CaTs: CaAl$_2$SiO$_6$), the Cr-Tschermak component (CrTs: CaCrAlSiO$_6$) and Ti-Tschermak component (TiTs: CaTiAl$_2$O$_6$). The Al$^{IV}$ in each of these components was calculated as follows:

$$CrTs^{IV} = Cr \text{ fraction}$$
$$TiTs^{IV} = \text{twice the Ti fraction}$$
$$CaTs^{IV} = 0.5\times(Al-[Na+Cr+2Ti]), \text{ or the excess Al after assignment to jadeite (NaAlSi$_2$O$_6$), CrTs and TiTs.}$$

Al$^{IV}$/[IV]$^{Total}$ is then equal to:

$$\frac{(CaTs^{IV} + CrTs^{IV} + TiTs^{IV})}{(Si^{IV} + CaTs^{IV} + CrTs^{IV} + TiTs^{IV})}$$
3. ERRORS IN MAGMATIC WATER ESTIMATES CALCULATED FROM CLINOOPYROXENE MEASUREMENTS

There are many sources of error in calculating magmatic water concentrations ($C_{H_2O}^{\text{liq}}$) from water measured in clinopyroxene ($C_{H_2O}^{\text{cpx}}$), which include the combined uncertainties in (a) the SIMS measurements of $H_2O$ within an analytical session, (b) the calibrations between sessions, (c) the $Al^{IV}$ measurements, and (d) the partitioning relationship outlined above. Here we assess the likely magnitude of these uncertainties, and how they may bear on $C_{H_2O}^{\text{liq}}$.

Analytical errors (a) and (c) are the smallest, and can be assessed from multiple analyses collected within nominally homogeneous grains, such as the one shown in Figure 1 (main paper). For example the uncertainty on the measurements at spots h and i within the core of A03-02-cpx16 is < 1% relative for both $C_{H_2O}^{\text{cpx}}$ and $Al^{IV}$ (Table 1), and so within-run analytical precision can be excellent. Based on replicate analyses over several years of analysis, we take 10% as a conservative estimate of the internal precision. External precision of the $H_2O$ measurements (b), however, approaches 15% relative (2-sigma), based on nearby spots analyzed in different SIMS sessions, reflecting the uncertainties in the different session calibrations (as in Fig 1b of the DR). The calibration slopes in Fig. 1b are indeed reproducible at the 10-15% level, as indicated in the small range in slopes determined over many sessions (0.43 - 0.48), even though the 2-sigma uncertainty on the slope is large (~ 33%) for any given analytical session. This large uncertainty in the absolute value of the slope reflects the scatter of points about the calibration line (Fig 1b), which as discussed above, likely reflects uncertainty in the
published data on the standards derived from different labs by different techniques. We thus consider the precision of the measurements, all made at CIW, to be 15%, but accuracy of the measurements with respect to the range reported by different labs to be 33%.

The total error on $C_{H2O}^{liq}$ also includes (d) the uncertainty on $D_{H2O}^{cpx/liq}$, and as discussed in the prior section, this is difficult to assess. While we have tried to improve the errors that result from the $D_{H2O}^{cpx/liq}$-$Al^{iv}$ regression (Fig. 2), we do not recommend using the partitioning relationship in Fig 2 (herein) for pyroxenes with $Al^{IV}/[IV]_{Total} < 0.02$, for which there are no experimental data from pyroxene-melt pairs. Such low-Al pyroxenes are not uncommon among evolved, low-pressure arc phenocryst populations. For example, IZ03-17a-cpx2c predicts anomalously high $C_{H2O}^{liq} (> 4 \text{ wt\%})$ due to a likely spurious low $D_{H2O}^{cpx/liq}$ (0.0046). It is certainly possible that at such low Al contents other substitution mechanisms (e.g., Fe$^{3+}$) may affect $D_{H2O}^{cpx/liq}$, but these have yet to be determined in such pyroxene compositions. Thus, we recommend using the $D_{H2O}^{cpx/liq}$ expression here, with its error envelope, only for pyroxenes with $Al^{IV}/[IV]_{Total} > 0.02$. The error envelope is derived from a standard York regression, as in Hauri et al (2006a), but the uncertainty is somewhat better than the original due to the Y-intercept constraints discussed above. As shown in Fig. 2, the error on the regression leads to an uncertainty of approximately 10% on $D_{H2O}^{cpx/liq}$.

Propagating all uncertainties into $C_{H2O}^{liq}$ leads to a total error of 35% (2-sigma), which is dominated by the uncertainty in the accuracy of the concentration measurements. For the purposes of comparing relative variations in the data (Figs. 1, 2 and 4 in the main
text), we plot error bars of 17%, which reflect the 2-sigma uncertainty in the external precision and in D, recognizing that the additional uncertainty in the accuracy affects all the data equally (including the melt inclusion data, since these were measured in the same lab as the D's and the cpx, with internally consistent calibration strategies). There is no doubt that future improvements in the SIMS calibrations, in the determination of D, and in the interlaboratory biases, may lead to total uncertainties that approach the precision of the measurements. Nonetheless, even at this time, the method shows promise in the ~15% agreement between the cpx population means and those derived independently from melt inclusion data (Fig 3, main text). This provides evidence that the large errors currently associated with the cpx calibrations are not systematic, and lead to accurate population means relative to melt inclusion data collected in the same laboratory.
4. REFERENCES


### Data Repository Table DR1

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<th>D_{H₂O}</th>
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**Data Repository Table DR1**

AR03-02 is scoria lapilli from Arenal volcano, Costa Rica (Wade et al., 2006)
IZ03-17a is scoria lapilli from Irazu volcano, Costa Rica (Benjamin et al., 2007)
GL-G2 is basaltic bomb from Galunggung volcano, Java (Kelley et al., in prep)
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Rims were confirmed in most grains from adhered glass at the grain edge, and in these cases the transect ran from the rim towards the core.
Some cores were confirmed by whole-grain compositional mapping by EMP.

If a grain was broken, the minimum crystal length and distance to the closest rim are reported.

H₂O (ppm) in cpx measured by SIMS ion microprobe at the Carnegie Institution of Washington (methods in text that accompany this table)
Al(IV)/(IV)total is the molar fraction of the tetrahedra site occupied by Al. Calculated as given in the text that accompanies this table
D(H₂O) is cpx/liq partition coefficient, calculated from:  \( D = (\text{Al in IV}) \times 0.29 + 0.00099 \), after Hauri et al (2006a)

\[ \text{H}_2\text{O}_{\text{liq}} \text{ calculated from: } \frac{\text{H}_2\text{O}(\text{cpx})}{D} \]

Error on H₂O concentration in cpx is 15%, based on external precision of the SIMS measurements.

Uncertainty on the accuracy approaches 33%, given the scatter in the cpx calibration shown in Fig 1h, due to interlaboratory differences.

H₂O-liq calculated from:  \( \text{H}_2\text{O}(\text{cpx})/D \)

Major element analyses by electron microprobe at MIT; in spots adjacent to SIMS analyses
Mg# is molar ratio of Mg to (Mg+Fe) in cpx

Error on C(H₂O)liq calculated by propagating errors in the numerator (+/-15% on the H₂O measurements) and in the denominator
(from the – 10% error on D error given by the error on the regression in Fig. 2).
## Data Repository Table DR1

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H2O-liq calculated from: H2O(cpx)/D

Major element analyses by electron microprobe at MIT; in spots adjacent to SIMS analyses

Mg# is molar ratio of Mg to (Mg+Fe) in cpx

Error on (CH2O)liq calculated by propagating errors in the numerator (+/-15% on the H2O measurements) and in the denominator (from the ~10% error on D error given by the error on the regression in Fig. 2).
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<th>μm from rim</th>
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