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LETTERS

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Water in lunar anorthosites and evidence for a wet early Moon

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The Moon was thought to be anhydrous since the Apollo era[1], but this view has been challenged by detections of water on the lunar surface[2–4] and in volcanic rocks[5–9] and regolith[10]. Part of this water is thought to have been brought through solar-wind implantation[2–4,7,10] and meteorite impacts[2,3,7,11], long after the primary lunar crust formed from the cooling magma ocean[12,13]. Here we show that this primary crust of the Moon contains significant amounts of water. We analysed plagioclase grains in lunar anorthosites thought to sample the primary crust, obtained in the Apollo missions, using Fourier-transform infrared spectroscopy, and detected approximately 6 ppm water. We also detected up to 2.7 ppm water in plagioclase grains in troctolites also from the lunar highland upper crust. From these measurements, we estimate that the initial water content of the lunar magma ocean was approximately 320 ppm; water accumulating in the final residuum of the lunar magma ocean could have reached 1.4 wt%, an amount sufficient to explain water contents measured in lunar volcanic rocks. The presence of water in the primary crust implies a more prolonged crystallization of the lunar magma ocean than a dry moon scenario and suggests that water may have played a key role in the genesis of lunar basalts.

Dissolved water in silicates can alter their structure, and hence significantly change their physical and chemical properties[14–17], which can further influence geologic processes. One of the most important conclusions resulting from the Apollo and Luna missions was that no water was detected in returned samples or at the surface of the Moon[1]. The Moon was thought to have lost its volatiles as it formed from ejecta of the impact of a Mars-size planetesimal with the proto Earth, the favoured Moon formation scenario[18], and during degassing of an early planet-wide magma ocean[12,13]. This notion has been included in most geophysical and geochemical models of formation and evolution of the Moon[12,18]. The view of a dry lunar interior, however, has been challenged by recent discoveries of water in picritic glass beads[5], apatites[6–8] and olivine melt inclusions[9], which were facilitated by the improvements of the analytical detection limit of hydrogen. Indigenous water is suggested to be heterogeneously distributed in the lunar interior and some parts of lunar mantle may contain as much water as Earth's upper mantle[5,9]. Hydrogen isotopic compositions of apatites in mare basalts have been interpreted to indicate a hybrid source of the water, that is, a combination of lunar mantle, comets and solar-wind protons[7]. The chlorine isotope compositions in the lunar pyroclastic deposits, however, have been interpreted as suggesting an essentially anhydrous lunar interior[19]. It has been further suggested using magma ocean crystallization modelling that the water content of the bulk lunar magma ocean (LMO) was less (possibly far less) than 100 ppm and water was later added during mantle cumulate overturn or through impacts[11].

Here we have measured water in primary products of the LMO, thereby bypassing the processes of later addition of water to the Moon through impact events or during mantle overturn as suggested by previous studies[7,11]. These data are used to estimate the water content of the Moon's interior at the time of the magma ocean, as well as that of the mare magma source regions. So far, ferroan anorthosite (FAN) is the only available lithology that is believed to be a primary product of the LMO (refs 12,13). It is generally accepted that plagioclase, after crystallization, floated in the magma ocean and formed FAN as the original crust of the Moon[12]. Therefore, any indigenous water preserved in pristine FAN was partitioned from the magma ocean. Fourier-transform infrared spectroscopy (FTIR) was used to measure water contents in plagioclases of FANs 15415,238 and 60015,787 (see Supplementary Information S1). Both these samples have >98 vol% of plagioclase with anorthite contents >96%. To assess the water inventory in the lunar highland upper crust, nominally anhydrous minerals in troctolite 76535,164 (see Supplementary Information S1) were also analysed using FTIR. Troctolite, an olivine-rich end ember of the Mg suite that composes about half of the highland upper crust[20], is thought to be derived from the magma ocean crystallization products, but its detailed origin is under debate[20].

The mineral grains allocated for this study are from the interior portion of each individual rock. Therefore, potential hydrogen implanted by solar wind[10] was avoided because direct solar implantation is limited to 0.2 μm depth from the sample surfaces[21], and even though micrometeorite gardening and melting can transfer OH to some depth on the basis of a recent study of lunar agglutinitic glasses[10], such OH is in glasses from impact remelting, not pristine minerals. Layers of tens to a few hundred micrometres in thickness on both sides of each grain were also removed in the preparation of doubly polished parallel surfaces for FTIR analyses (see Methods), which further ensured the removal of any layer affected by solar implantation. Infrared spectra of plagioclase from FAN 15415,238 and 60015,787 are characterized by a small wide absorption band in the O–H region (Fig. 1 and Supplementary Fig. S2) that resembles those observed in terrestrial plagioclases[22,23]. This broad band (~3,700 to ~3,100 cm^{-1}) is interpreted as absorption by structural O–H bond vibrations in plagioclase for two reasons. First, one of our doubly polished grains of 15415 was heated to 1,000 °C for 24 h in a high-purity N_2 atmosphere at the University of Michigan and then cleaned with the same procedure (see Methods). The band is strongly diminished in the heated sample, demonstrating that

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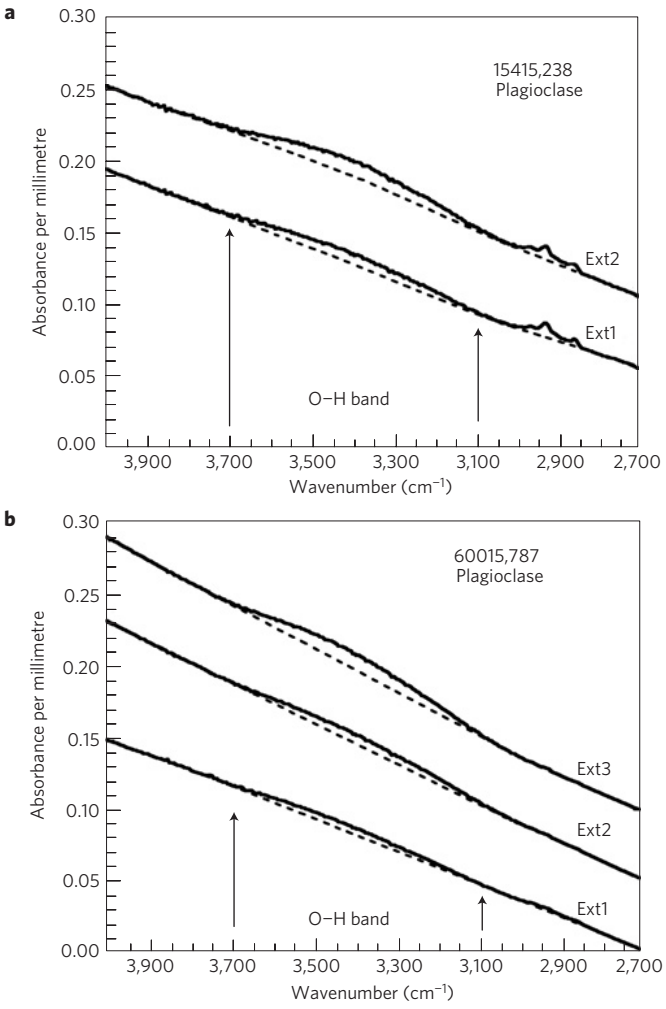

Figure 1 | Representative polarized FTIR spectra of plagioclase from FANs. a,**b**, Spectra for 15415,238 (**a**) and 60015,787 (**b**) at mutually perpendicular orientations (Ext1, Ext2 and Ext3: optical extinction directions 1, 2 and 3) are normalized to 1 mm and shifted vertically for comparison. The dashed line indicates the baseline position used for water content estimations. The narrow peaks (3,000–2,800 cm^{-1}) most probably come from organic contamination on the mineral surface during sample preparation[22,23]. The spectra with the same label (for example, Ext1) for different crystals were not taken at the same crystal orientation relative to the mineral crystallographic axis.

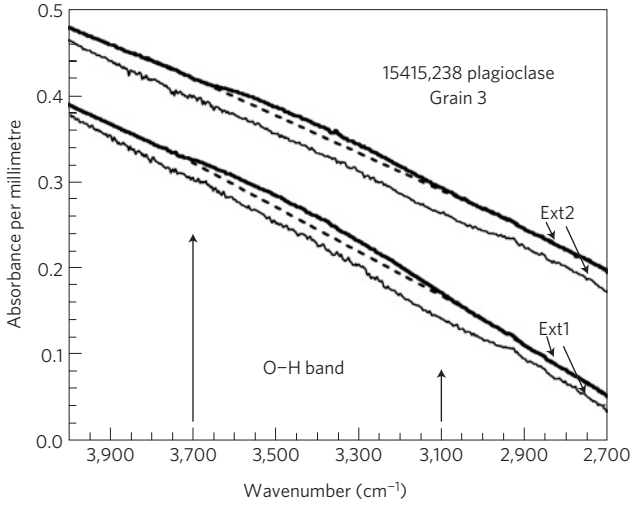

Figure 2 | FTIR spectra of plagioclase from 15415,238 before and after heating at 1,000 °C for 24 h. FTIR analyses were performed on the sample before (upper thick curve with the dashed baseline used for water content estimation) and after the heating experiment (lower thin curve) at the same orientations of the sample relative to the polarizer (Ext1 and Ext2, respectively). The diminished band (~3,700 to ~3,100 cm^{-1}) in the spectrum of the heated sample demonstrates that dehydration occurred. The band in the untreated sample is due to absorption of O–H bond vibration, and not an artefact in the baseline.

dehydration occurred (Fig. 2). Second, the anisotropy of the O–H absorption band height or absorbance area during rotation of the infrared polarizer relative to the plagioclase crystals, and the 90° interval between maximum and minimum (Supplementary Fig. S4) demonstrate that this band (Fig. 1 and Supplementary Fig. S2) cannot be caused by water in minute melt or fluid inclusions[23] or by contamination during sample preparation. Furthermore, O–H absorbance area does not seem related to the degree of plagioclase fracturing that was probably produced during impact. Total integrated absorption areas of the OH bands (A_{tot} in cm^{-2}) along three mutually perpendicular directions (see Supplementary Information S2) were converted to water contents (C_{H_2O} in parts per million by weight of H_2O) using the Beer–Lambert law in the form $C_{H_2O} = A_{tot}/I'$, where I' is the calibrated specific integral absorption coefficient (15.3 ± 0.7 ppm^{-1} cm^{-2} for feldspars[22]). Although infrared absorbances are typically reproducible between different laboratories[24], we nonetheless verified the accuracy of our measurement by analysing an external standard, the plagioclase used for the absorption coefficient calibration[22], GRR1968. The total absorbance area of spectra at three mutually perpendicular directions and with sample thickness normalized to 1 cm is 1,712 cm^{-2}, differing from that of 1,688 cm^{-2} in ref. 22 by 1.4% relative, thereby verifying inter-laboratory reproducibility. The water contents in plagioclases are ≥5.0 ppm H_2O by weight (grain Pl3) for 15415,238 and 6.4 ppm for 60015,787 (Supplementary Table S1). Note that there could be a systematic error in the calibration for plagioclase, which would act to underestimate water contents calculated using this calibration (see Supplementary Information S2).

The water partition coefficient between plagioclase and silicate melt is not well constrained (see Supplementary Information S3), which is a main source of uncertainty in the following discussion. Using a partition coefficient of 0.004 between plagioclase and silicate melt[25], the water content of a melt in equilibrium with 60015 plagioclase is calculated to be ~1,600 ppm H_2O. Co-crystallized pyroxene cumulates should contain ~11 ppm H_2O using a partition coefficient of 0.007 between pyroxene and silicate melt[26]. About 1,600 ppm represents the amount of water in the residual melt of the magma ocean, when floating plagioclase was forming the original lunar crust. At that point, approximately 80 vol% of the LMO is thought to have been solidified[12]. Using this degree of crystallization, the amount of water in the parental magma of FAN 60015, that is, in the initial magma ocean, is inferred to be ~320 ppm H_2O (Fig. 3). The first crystallized olivine cumulate in the LMO could have ~0.6 ppm of water using a partition coefficient of 0.002 between olivine and silicate melt[27], which is much higher than ~90 ppb of water in the lunar mantle inferred from Cl isotope studies[19]. As crystallization of the LMO continued, volatiles and other incompatible trace elements became enriched in the magma ocean residuum. On the basis of the LMO model[12], the final 2 vol% of the magma ocean residuum (urKREEP) that may be the source of the potassium, rare-earth and phosphorus (KREEP)-rich lithologies unique to the Moon potentially could have had as much as ~1.4 wt% of water. This is an order of magnitude higher than the thousands of parts per million maximum suggested previously[11], and also 1.5 orders of magnitude higher than 850–1,100 ppm in

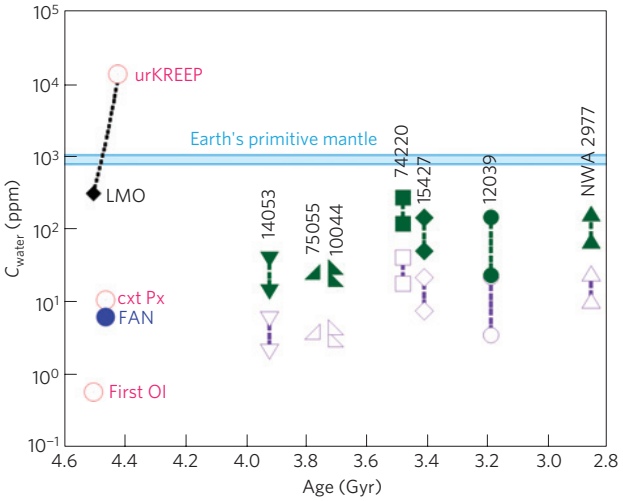

Figure 3 | Water contents in LMO products and mantle sources of basalts through time. Model ages are used for primary magma ocean products[12,13] and isochron ages for the basalts (Supplementary Table S2). Water contents of the initial magma ocean (LMO), the first crystallized olivine cumulate (first Ol), co-crystallized pyroxene cumulate (cxt Px) and urKREEP were estimated from the water content measured in FAN plagioclases. The black dashed line from LMO to urKREEP shows the water content evolution in magma ocean residua. The water contents of mantle sources with isochron ages <4.0 Gyr were calculated assuming 20% (green) or 3% partial melting (purple; Supplementary Table S2).

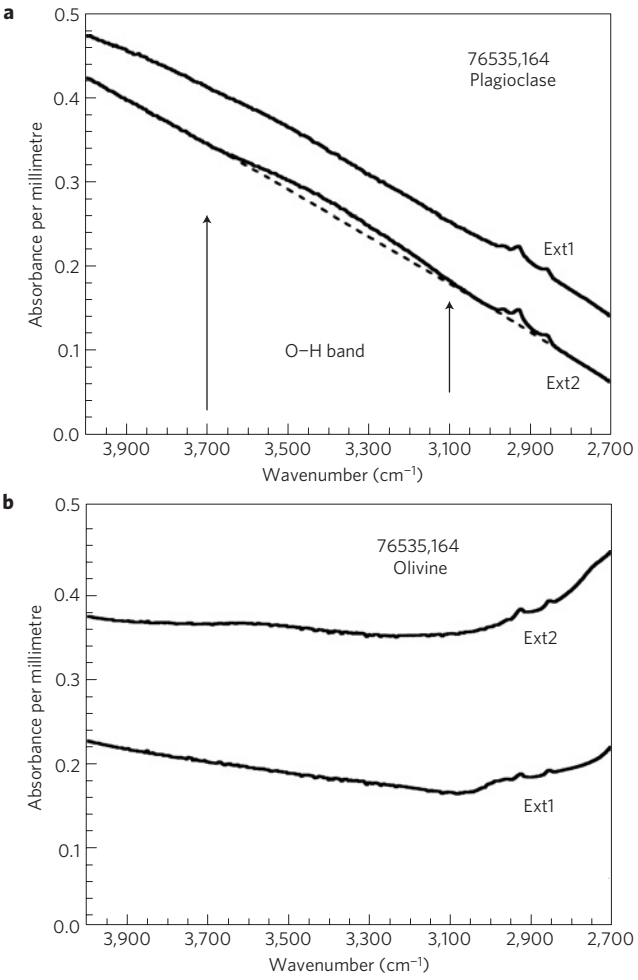

Figure 4 | Representative polarized FTIR spectra of troctolite 76535,164. **a**,**b**, Spectra of plagioclase (**a**) and olivine (**b**) at two mutually perpendicular orientations (Ext1 and Ext2) are normalized to 1 mm and shifted vertically for comparison. The dashed line indicates the baseline position used to calculate water concentrations (see Supplementary Information S2). No obvious OH bands are observed in the spectra taken with orientation Ext1 of plagioclase or in any of the olivine spectra. The narrow peaks between 3,000 and 2,800 cm^{-1} most probably come from organic contamination on the mineral surface during sample preparation[22,23].

Earth's primitive mantle[28] (Fig. 3). This implies that the LMO crystallization products could have spanned a wide range of water contents, from <1 ppm to ~1.4 wt% (Fig. 3). After the LMO solidification, these materials are thought to have undergone gravitational overturn driven by density difference[29]. Overturned lunar cumulate mantle provided the source regions for mare basalts[12,29]. Even assuming 20% of partial melting of the source regions of mare basalts in which water was detected[5–9], calculated water contents of their source regions are still well within the range of those we calculated for the primary magma ocean products inferred from water content in plagioclase from FANs (Fig. 3).

Even a small amount of water can change the liquid line of descent of melt and suppress crystallization of plagioclase relative to olivine and clinopyroxene, such as in mid-ocean ridge basalts[15]. Therefore, the amount of water we calculated could affect the LMO crystallization dynamics, especially for the last few tens of volume per cent of magma ocean residuum. The depression of the liquidus due to increased water contents would also prolong the crystallization of the LMO and potentially explain an extraordinarily young age (4,360 ± 3 Myr) for FAN 60025 (ref. 30) in the framework of a magma ocean model. Note that an alternative explanation of the young age has been suggested by challenging the existence of a global magma ocean[30]. Even if the latter argument is correct, the measured water concentration in plagioclase can still be used to infer water contents of parental magmas of cumulates that formed the earliest lunar crust. Moreover, the relatively high abundance of water (~1.4 wt%) in urKREEP can significantly change its physical properties, such as lowering density[16] and viscosity[17], which could affect the dynamics of magma ocean cumulate overturn[29]. In summary, the variable amounts of water in urKREEP and earlier cumulates (Fig. 3) could play a critical role on the genesis of lunar basalts, in which indigenous water has been recently discovered[5–9], in a similar way to the role of water in terrestrial oceanic mantle melting regimes[14].

Intrinsic water was also detected in plagioclase of troctolite 76535,164 (Fig. 4 and Supplementary Fig. S3). The minimum water contents vary from 0.8 to 2.7 ppm (Pl1 of 76535,164) (Supplementary Table S1). No O–H absorption band has been observed in a 1.055-mm-thick olivine from this troctolite (Fig. 4), implying a H_2O content of <1 ppm. The fact that plagioclase is more hydrous than olivine is consistent with the H_2O partition coefficient between olivine and melt being smaller than that between plagioclase and melt[25,27]. The minimum whole-rock water content of troctolite 76535 is ~2 ppm on the basis of its mineral modal abundance, which is lower than that calculated for the initial magma ocean from our FAN data.

The presence of indigenous water in FAN and troctolite suggests that the highland upper crust is not anhydrous. Considering the distributions of two major lithologies (FAN and Mg suite) in the highland upper crust[20] and assuming that the results for 60015 (~6 ppm) and 76535 (~2 ppm) are representative of FANs and the Mg suite, respectively, the upper crust may contain ~4 ppm of indigenous water. Incidentally, trace amounts of water/hydroxyl have been detected in the lunar highlands surface by various spacecrafts, although lack of hydroxyl reflectance calibration meant that it could not be quantified[3]. Hence, another implication of

the results presented here is that the water measured in lunar highland lithologies may contribute a significant portion of the water detected by spacecraft.

Methods

Plagioclase grains from FANs 15415,238 and 60015,787, and plagioclase and olivine grains from one troctolite 76535,164 were analysed by FTIR to determine water contents. Each mineral grain was embedded in crystal bond and a doubly polished section was prepared manually using sandpaper and alumina powder (down to 1 μm). The grain was not polished at any orientation relative to the crystallographic axis (that is, with polished surface perpendicular to the optic normal, acute bisectrix or obtuse bisectrix directions). Instead a mineral section of random orientation was made as thick as possible to get maximum infrared signal for O–H bond vibrations with a relatively good spectrum signal–noise ratio. One plagioclase grain of 60015 was large enough to be polished into a mineral cube with two sets of polished mutually perpendicular surfaces. The polished grains were cleaned successively with acetone, ethanol, deionized water and CH_2Cl_2 in an ultrasonic bath. A plagioclase (GRR1968) previously analysed by another laboratory[22] was also polished into a cube using the same procedure to check for inter-laboratory reproducibility. The thickness of each grain was measured using a Mitutoyo digital micrometer. Before FTIR analysis of each grain, the sample chamber was flushed with N_2 for at least an hour.

Polarized FTIR spectra with wavenumbers from 7,800 to 600 cm^{-1} were collected using a Hyperion 3000 microscope attached to a Bruker Vertex 70 FTIR spectrometer at the Astromaterial and Research Exploration Science (ARES) directorate of the NASA-Johnson Space Center. The standard mercury cadmium telluride detector and KBr beam splitter were used during FTIR analysis, as well as a Zn–Se wire-grid polarizer. Aperture size from 255 × 255 μm to 425 × 425 μm (mainly 340 × 340 μm) during each analysis was chosen on the basis of the grain size. During each analysis, 256 scans were performed for each infrared measurement under a nitrogen environment, to minimize interference from atmospheric water vapour. A new background was collected before each infrared measurement on a new grain or the same grain using a different polarizing angle. Polarized infrared spectra (wave numbers from 9,000 to 1,500 cm^{-1}) were also acquired using an AutoImage microscope on a Perkin-Elmer Spectrum GX FTIR spectrometer at the University of Michigan. A near-infrared source, CaF_2 beam splitter, KRS-5 infrared wire-grid polarizer, mercury cadmium telluride detector and N_2 gas purge and an aperture size of 340 × 340 μm were used during these FTIR analyses. The spectra from two different FTIR are consistent with each other.

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Author contributions

H.H. conceived this study and performed the analyses and experiments. Y.Z. provided the terrestrial plagioclase grains. A.H.P and Y.Z. assisted in experiments and FTIR analyses. H.H., A.H.P., Y.Z. and C.R.N. discussed the data and wrote the paper.

Additional information

Supplementary information is available in the online version of the paper. Reprints and permissions information is available online at www.nature.com/reprints. Correspondence and requests for materials should be addressed to H.H.

Competing financial interests

The authors declare no competing financial interests.

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SUPPLEMENTARY INFORMATION

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Water in lunar anorthosites and evidence for a wet early Moon

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Supplementary Information

1. Description of Ferroan Anorthosites 15415 and 61005, and Troctolite 76535

Ferroan anorthosite 15415 is one of the best known rocks of the Apollo collection, and is popularly called the "Genesis Rock" because the astronauts thought they had a piece of the Moon's primordial crust. It was collected on the rim of Apur Crater during the Apollo 15 mission[1]. Rock 15415 is a pristine coarse-grained, unbrecciated anorthosite composed of 99 vol.% calcic plagioclase ($An_{96.9}$)[2]. The plagioclase is homogenous with the variation of anorthite content being within 1 mol.%[2]. Besides plagioclase, accessory augite and traces of orthopyroxene and ilmenite have been observed[3]. No crystallization age has been determined for 15415[4,5] but ages for ferroan anorthosites range from 4.29 to 4.56 Ga[6]. This rock has experienced multiple episodes of shattering and impact[3]. However, the low abundance of siderophile elements supports the chemical pristinity of this ferroan anorthosite[7]. The aliquot of 15415,238 allocated to us is composed of plagioclase grains from the interior portion of this rock. These mineral grains are anhedral, full of fractures and tiny (<50 μm) mineral inclusions (Fig. S1). Melt inclusions are absent.

Rock 60015 is a highly shocked ferroan anorthosite, which was collected near the Lunar Module during the Apollo 16 mission. The rock is covered by a rind of thick black glass[8]. The interior consists almost entirely of anorthitic plagioclase (>98 vol.%), with accessory pyroxene (orthopyroxene and augite, ~1.3 vol.%) and trace amount of ilmenite (~0.1 vol.%)[8]. Plagioclase is homogenous ($An_{96.4\text{-}97.1}$)[8]. Metallic iron detected in the interior portion has low concentrations of Ni and Co[9] and the rock has extremely low concentrations of siderophile elements[10], which suggests there was no meteoritic contamination at least for the interior portion[9,11]. No crystallization age has been determined for 60015[12]. Our aliquot from 60015,787 obtained from the Lunar Samples Curation was a large (~3 mm) plagioclase grain from the interior portion. This mineral grain is anhedral, full of fractures and tiny mineral inclusions. A few bubble-like inclusions were also observed, which may indicate that shock-induced temperature was high enough to initiate partial melting[8].

Troctolite 76535 is a coarse-grained plutonic rock, collected at Station 6 during the Apollo 17 mission. It has an equilibrated texture, mainly composed of olivine (60 vol.%), anorthitic plagioclase (35 vol.%), and orthopyroxene (5 vol.%)[13]. Olivine is very homogeneous ($Fo_{87.3}$) as is plagioclase ($An_{96.2}$) and orthopyroxene ($En_{84.1}Wo_{0.9}$)[13]. Other

minor phases observed in this rock include augite, spinel, apatite, merrillite, metal and baddeleyite[13]. This rock has very low abundance of siderophile elements[14], which suggests it is chemically pristine. Isotopic U-Th-Pb studies yielded an age of 4.236±0.015 Ga[15], which is consistent with the Sm-Nd isotopic age of 4.26±0.06 Ga[16], but is younger than the Rb-Sr isotopic age of 4.61±0.07 Ga[17]. The aliquot 76535,164 allocated to us consists of plagioclase and olivine grains from the interior portion of 76535. Both olivine and plagioclase contain abundant acicular to rounded opaque inclusions. In general, the plagioclase grains contain more fractures than the olivine grains.

2. Quantitative determination of water contents

Quantitative water estimations in nominally anhydrous biaxial minerals by FTIR are based on the total integrated absorption intensities in the OH region, which is typically taken as the sum of the integrated absorption intensities of polarized spectra in three principal optical orientations (i.e., perpendicular to the optic normal, acute bisectrix or obtuse bisectrix directions) of the mineral[18]. However, it was not possible to prepare our lunar crystals with polished faces oriented in the three principal optic orientations. On the other hand, the sum of the integrated absorption intensities of any three mutually perpendicular directions can also be used[19,20]. Even then it is still difficult to get two sets of mutually perpendicular surfaces in these randomly oriented highland mineral grains because they tend to break along fractures. Four large plagioclase grains of 15415, and four large plagioclase grains and two olivine grains in 76535 were doubly polished to thick sections for FTIR analysis (Table S1). Only one plagioclase grain of 60015 could be polished into a cube with two sets of mutually perpendicular surfaces. The fractures observed from the petrographic microscope, especially those extended to polished surfaces and the large ones within crystal were avoided during FTIR analyses when possible.

Infrared spectra of plagioclase from FAN 15415,238 and 60015,787, and troctolite 76535,164 are characterized by a small wide absorption band in the O-H region (Figs. 1, 3, S2 and S3) and resemble those observed in terrestrial plagioclases from the literature[19,20]. Several tests were made to prove that the small band is due to intrinsic OH vibration absorption and not an artifact of the baseline. Plagioclase Grain 3 of FAN 15415,238 was heated at 1000 °C for 24 hours in a Deltech furnace in a purified N_2 environment and then cleaned with the same procedure used for untreated samples. This sample was analyzed with the same FTIR setting before and after heating. The spectra of heated sample is essentially free of the small OH band compared with that of untreated sample (Fig. 2), indicating that the O-H bands observed in IR spectra of lunar plagioclase (Figs. 1, 4, S2 and S3) are caused by O-H bond vibrations, and that the water was lost during the heating experiment. To test that the observed band is structural OH, IR spectra were taken on the same spot by varying the polarizer direction (E-vector direction). The anisotropy of the OH absorption band area during rotation of the infrared polarizer in plagioclase (Fig. S4) demonstrates that the bands (Figs. 1, 4, S2 and S3) are caused by structural hydroxyl species in the plagioclase grains and not by water in melt inclusions[20]. These tests also demonstrate that the OH absorption band is unlikely caused by contamination during sample preparation. No absorption bands were observed at 5240 cm^{-1} (Fig. S5), which would have indicated the presence of molecular water if

sufficient amounts of water were incorporated in plagioclase. Consequently, the wide absorption bands (~3700 - ~3100 cm^{-1}; Figs.1, 4, S2 and S3) are caused by structural hydroxyl species in plagioclase[20,21]. No OH band was detected in olivine spectra of 76535 (Fig. 4). Using the Omnic© software, all the spectra were normalized to 1 cm thickness and the baseline for each spectrum was manually drawn following the procedure described by Johnson and Rossman[19,20]. Our results indicate that OH is roughly homogenously distributed within each plagioclase grain for FAN 15415 and 60015 (Table S1). Only one aggregate plagioclase grain (Pl1) from troctolite 76535 has detectable OH bands in its spectra. It may be because this grain is an aggregate that the spectra from different locations have variable OH band intensities (Figs. 4 and S3).

The absorption intensity in the OH region was determined by integrating the area above the manually drawn baseline (between 3700 and 3100 cm^{-1} for plagioclase). The overall absorption band area represents the sum of that for different mutually perpendicular orientations (Table S1). A piece of plagioclase crystal GRR1968, which was used to calibrate the specific integral absorption coefficient of water in plagioclase by Johnson and Rossman[19], was also analyzed along three mutually perpendicular directions in this study for comparison. The total IR band area obtained from our own analysis and for a sample thickness normalized to 1 cm is 1712 cm^{-2}, which agrees with the result (1688 cm^{-2}) in Johnson and Rossman[19] with 1.4% relative difference. Total absorption areas of the OH bands were converted to water contents (C_{H2O} in ppm by weight of H_2O) using the Beer-Lambert law in the form $C_{H2O} = A_{tot}/I'$, where I' is the specific integral absorption coefficient of 15.3±0.7 $ppm^{-1}cm^{-2}$ calibrated for feldspars[19] (Table S1). Note that this calibration for plagioclase used here includes 7 data points of plagioclase, sanidine and microcline and there is scatter in the fitting[19]. If only the single point for plagioclase (GRR1968, with anorthite content of 94%, similar to lunar plagioclase with that of ~97%)[19] is used to calibrate the specific integral absorption coefficient, the coefficient is 8.0 $ppm^{-1}cm^{-2}$ and the water content of plagioclase from 60015 (98.5 cm^{-2} of total absorbance area; Table S1) is 12.3 ppm. If only the 4 points for various plagioclase crystals are used for calibration, then the specific integral absorption coefficient is 10.7 $ppm^{-1}cm^{-2}$ and the water content of plagioclase from 60015 is 9.2 ppm. For the paper, we use the published values (using the 7 data points of plagioclase, sanidine and microcline), so our results may represent minimum values.

The FTIR detection limit for water in plagioclase used this study was determined using the signal variability (3σ) in the FTIR spectra (3700 – 3100 cm^{-1}) in which no OH band could be detected. The detection limit thus determined is 0.46±0.22 (1σ) ppm in terms of water, which is consistent with that of < 1 ppm typically given in the literature[22]. The propagated uncertainty on the measured water contents is ~50% (1σ), due to uncertainties in the OH band area determination based on manually drawn baselines, the uncertainties on the absorption coefficient (see discussion above) and thickness estimation (± 3 microns), and the fact that except for the 60015 plagioclase grain, three mutually perpendicular measurements could not be made, resulting in minimum water content estimates for each grain respectively. Even with this error, it is evident that there is detectable OH in plagioclase from the samples analyzed here.

3. Partition Coefficient between Plagioclase and Silicate Melt

In order to estimate the composition of a silicate melt in equilibrium with

plagioclase, a partition coefficient for the element of interest must be used. Partition coefficients are a function of temperature, pressure, melt composition, and plagioclase composition[23-25]. However, such dependence is not known, and only two empirically determined partition coefficients of hydrogen between plagioclase and silicate melt have been published in the literature[26]. Both partition coefficients were established by comparing the water contents in phenocrysts and those in melt inclusions, but there is a difference of two orders of magnitude between these two values, 0.004 (ref. 27) versus 0.1 (ref. 28). In the first case, a linear function was established between water in plagioclase phenocrysts and that in plagioclase melt inclusions and 0.004 was calculated from this linear function[27]. For the latter, however, the averages of water contents in plagioclase phenocrysts and plagioclase melt inclusions were used to calculate the partition coefficient (= 0.1) (ref. 28). As suggested by the authors[28], this partition coefficient may be meaningless because an average of water contents in melt inclusions, which had an order of magnitude of variation (410 – 3630 ppm H_2O), was used. Furthermore, a partition coefficient of 0.1 appears unrealistically high, being much higher than for typical nominally anhydrous minerals (~0.002 for olivine and ~0.02 for pyroxene)[29] but similar to that of apatite (0.1 – 0.25 (ref. 30), 0.13 – 0.85 (ref. 31)), a hydrous mineral. Therefore, we chose 0.004 as partition coefficient of H_2O between plagioclase and melt in this study. Note that the melt compositions for the estimated plagioclase-melt partition coefficient (0.004) are dacite to rhyolite[32], which may not be ideal for lunar magma ocean composition, although this partition coefficient (0.004) has also been applied to terrestrial basalt[33].

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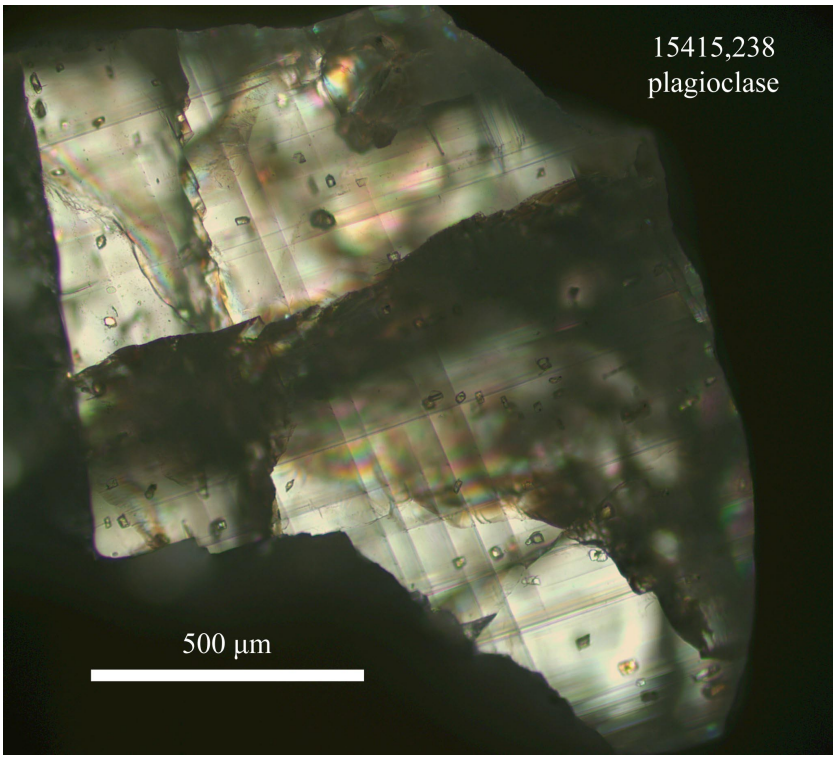

Fig. S1. Cross-polarized light image of plagioclase Grain 4 of FAN 15415,238. The thickness of this mineral section is 0.549 mm.

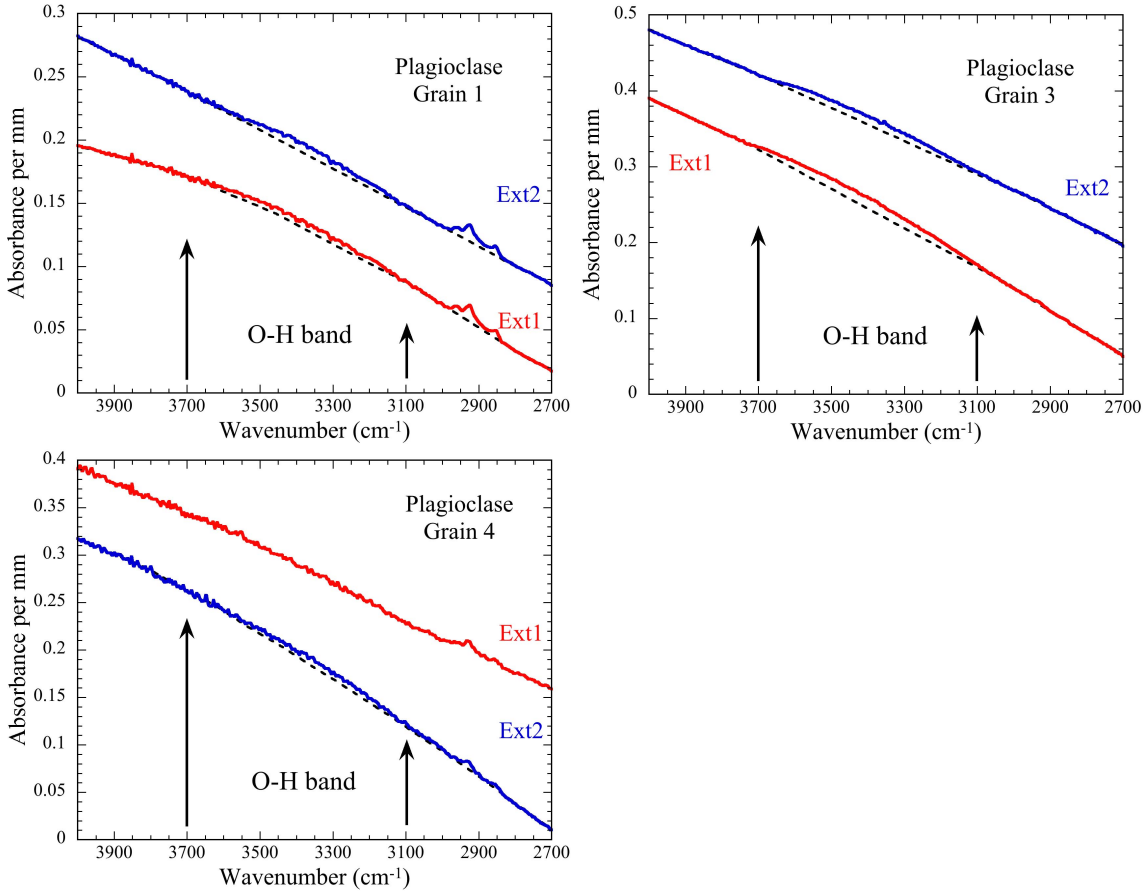

Fig. S2. Representative polarized FTIR spectra of plagioclase in FAN 15415,238 at mutually perpendicular orientations (Ext1 and Ext2). Spectra Ext1 and Ext2 in each crystal (Grain 1, 3 and 4) are from the same location for each grain. The broad band (~3700 - ~3100 cm^{-1}) in each spectrum is caused by O-H bond vibrations. The dashed line indicates the baseline position used for water content estimations. The small peaks between 3000 and 2800 cm^{-1} come from organic contamination on the mineral surface during sample preparation. Each spectrum is normalized to 1 mm.

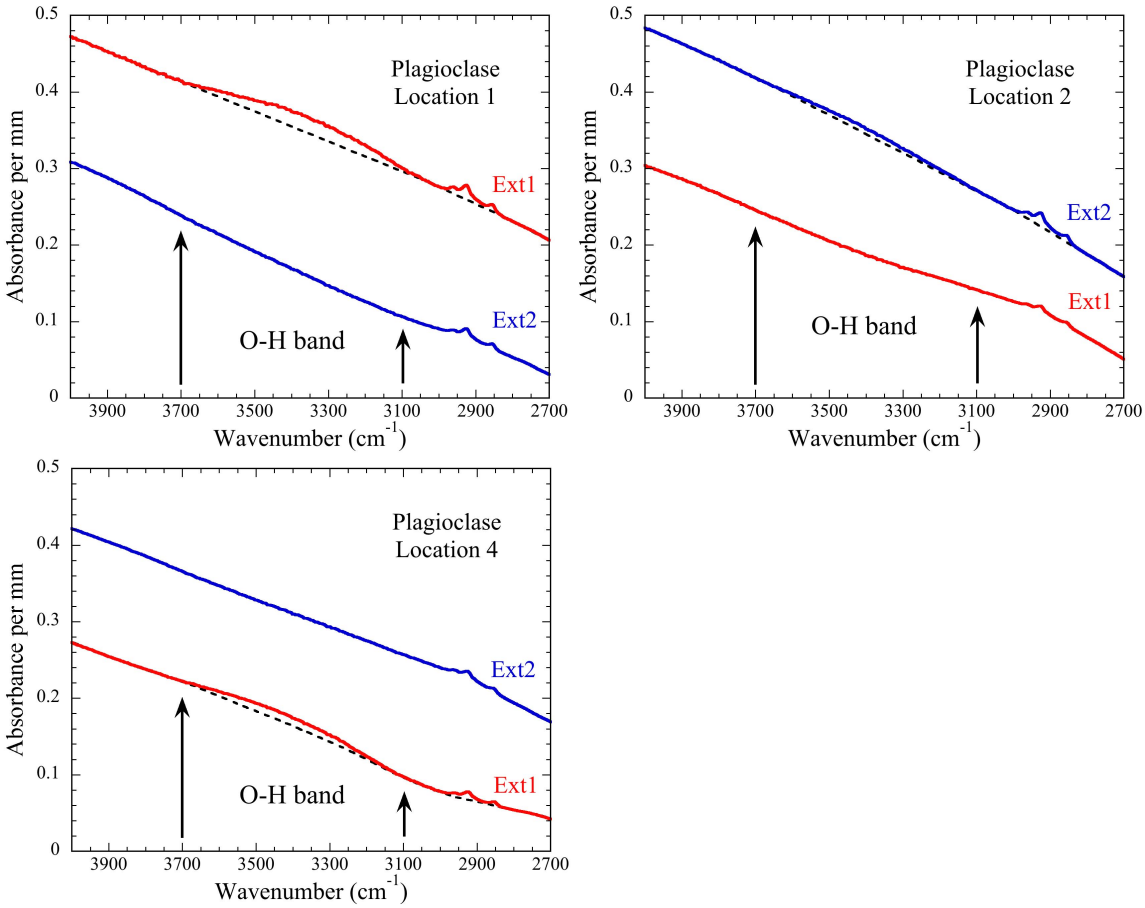

Fig. S3. Representative polarized FTIR spectra of plagioclase grain 1 in troctolite 76535,164 at mutually perpendicular orientations (Ext1 and Ext2). Spectra Ext1 and Ext2 in each plot (location 1, 2 and 4) were measured on the same spot on the plagioclase grains but with the infrared polarizer at two perpendicular orientations. Spectra Ext1 (or Ext2) at different locations may not be obtained with the same polarized infrared. The dashed line indicates the baseline position used for water content estimations. The small peaks between 3000 and 2800 cm^{-1} come from organic contamination on the mineral surface during sample preparation. Each spectrum is normalized to 1 mm.

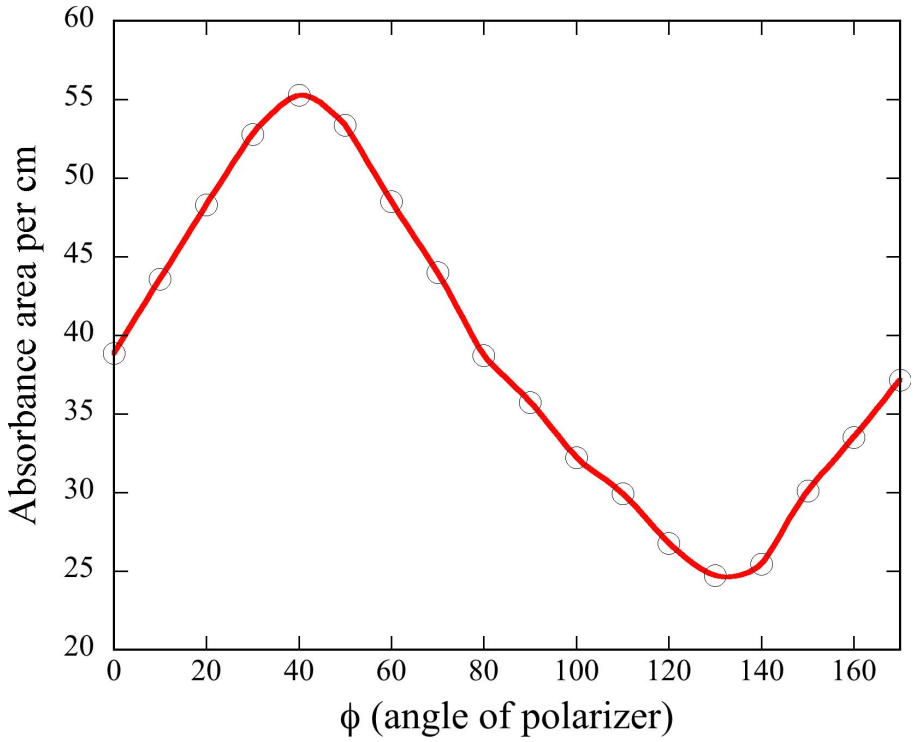

Fig. S4. Variation in OH absorbance area normalized to 1 cm with the angle of the infrared polarizer. FTIR measurements were performed at a single location on Grain 3 from FAN 15415,238. This plagioclase grain was not oriented relative to any major axis and its orientation was fixed during this series of measurements. Circles show the absorbance calculated as the area beneath the OH bands (~3700 to ~3100 cm^{-1}).

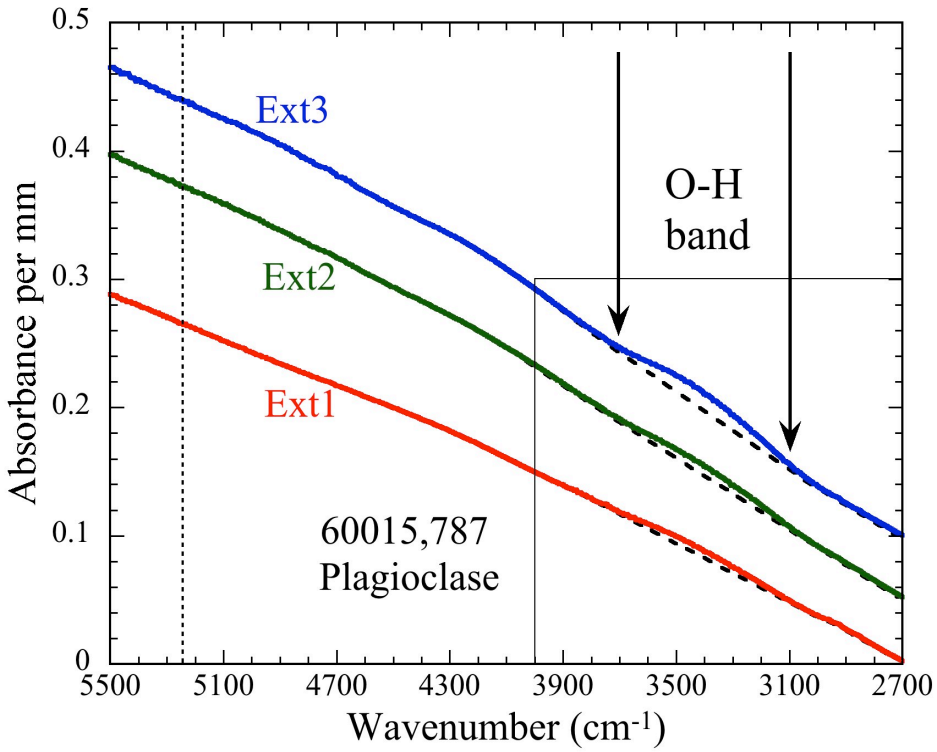

Fig. S5. Representative polarized FTIR spectra in the OH band region of FAN 60015 plagioclase. No bands were observed at ~5240 cm^{-1} in all the plagioclase spectra in this study, which is consistent with that the 3700-3100 cm^{-1} bands are for hydroxyl species, not molecule water nor water in melt inclusions. The portions of spectra in the box are the same as those in Fig. 1b.

Table S1. Integrated OH absorbance values and water contents obtained from FTIR analyses of lunar plagioclases and olivines.

Sample	Grain	Location	Thickness (mm)	Ext1 band[a] area (cm^{-2})	Ext2 band[a] area (cm^{-2})	Ext3 band[a] area (cm^{-2})	Total band[a] area (cm^{-2})	Water[b] (H_2O ppm)
15415	Pl1[c]	1	0.679	19.9	26.3		46.2	3.0
15415	Pl2[c]	1	0.907	21.5	24.9			
		2	0.907	21.1	28.4			
		3	0.907	18.2	28.6			
		Average		20.3	27.3		47.6	3.1
15415	Pl3[c]	1	1.021	41.6	30.7			
		2	1.021	42.2	34.1			
		3	1.021	44.8	29.2			
		4	1.021	48.1	28.5			
		5	1.021	49.2	32.8			
		Average		45.2	31.1		76.2	5.0
15415	Pl4[c,d]	1	0.549	n.d.[f]	6.4			
		2	0.549	n.d.[f]	8.6			
		3	0.549	n.d.[f]	8.3			
		Average			7.8		7.8	0.5
60015	Pl	1	1.061		26.5	58.6		
		2	1.061		29.5	50.1		
		3	1.061		22.1	55.9		
		4	1.061		26.2	58.7		
		5	1.641	14.8	22.1			
		6	1.641	18.7	23.7			

		7	1.641	17.5	29.3			
		8	1.641	16.2	27.9			
		Average		16.8	25.9	55.8	98.5	6.4
76535	Pl1[c,e]	1	1.086	41.7	n.d.[f]		41.7	2.7
		2	1.086	n.d.[f]	13.0		13.0	0.8
		3	1.086	n.d.[f]	41.1		41.1	2.7
		4	1.086	37.4	n.d.[f]		37.4	2.4
76535	Pl2[c]		1.194	n.d.[f]	n.d.[f]			
76535	Pl3[c]		1.346	n.d.[f]	n.d.[f]			
76535	Pl4[c]		0.796	n.d.[f]	n.d.[f]			
76535	Ol1[c]		1.055	n.d.[f]	n.d.[f]			
76535	Ol2[c]		0.604	n.d.[f]	n.d.[f]			

a) Area of integrated absorbances presented here are for spectra normalized to 1 cm and at mutually perpendicular orientations (Ext1, Ext2, Ext3) of the infrared polarizer relative to the grain.
b) The 1σ uncertainty on the measured water content is ~50% (see supplementary information).
c) We could only obtain spectra at two mutually perpendicular orientations. Therefore, the water content shown here (or location for Pl1 of 76535,164) represents a minimum value.
d) OH was detected at only one of the two orientations probably because the grain section was too thin (0.549 mm).
e) The highly variable total band area of this grain (Pl1) may be in part due to the fact that it is a plagioclase crystal aggregate. Location 1, 3 and 4 were analyzed at the same sample orientation, but different from one for location 2 during FTIR measurements.
f) n.d. = no OH bands observed in the infrared spectra.

Table S2. Water concentrations in the source regions of lunar basalts for which indigenous water has been measured and their isochron ages.

Sample	Age[a] Ga	Parental melt[b] Water ppm	3% partial melting[c] Water in source ppm	20% partial melting[c] Water in source ppm
74220	3.48 (Pb-Pb)	615 – 1410	18.45 – 42.3	123 – 282
15427	3.41 (Pb-Pb)	260 – 745	7.8 – 22.35	52 – 149
NWA 2977	2.86 (Sm-Nd)	360 – 850	10.8 – 25.5	72 – 170
14053	3.96 (Rb-Sr)	71 – 200	2.13 – 6	14.2 – 40
12039	3.19 (Rb-Sr)	120 – 756	3.6 – 22.68	24 – 151.2
10044	3.71 (Rb-Sr)	106 – 156	3.18 – 4.68	21.2 – 31.2
75055	3.77 (Rb-Sr)	~ 134	~ 4.02	~ 26.8

a) The ages for the basalts are isochron ages and come from literature data: 74220[34], 15427[35], NWA 2977[36], 14053[37], 12039[38], 10044[39] and 75055[40]. Isotopic dating systems used are shown in parentheses.
b) The water concentrations in parental melts of these basalts are based on literature data: 74220[41], 15427[42], NWA 2977[30], 14053[42,43], 12039[43], 10044[43] and 75055[43].
c) The water concentrations in the source regions assuming 3% or 20% partial melting to form the parental melts of these basalts.