

Isotopic fractionation of neon during magma degassing

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Supplementary Information

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Material and Method

Experimental samples:

Starting material. The initial material comprises basanitic scoria from the northeastern vent of Fasnía volcano, which erupted in January 1975 in Tenerife, Spain. This composition has previously been used for CO₂ solubility experiments (Jiménez-Mejías *et al.*, 2021) which facilitated our experiments. The chemical major element composition of the glass is shown in Table S-1. The natural sample was first crushed and powdered by using an agate mortar and acetone to prevent friction and damage to the mortar. The powdered sample was then melted at 1400 °C and 1 bar for 240 min in a platinum crucible. At the end of the experiment, the crucible was rapidly removed from the furnace and dropped into water ensuring a fast quenching. After grinding the resulting glass, this sequence was repeated once to ensure a homogeneous, anhydrous and bubble/crystal-free glass which was used as a starting material in all experiments described below.

Major elements. All glasses produced in this study were mounted in epoxy resin, polished, and analysed using a Cameca SX-Five electron microprobe at the Orléans Earth Sciences Institute (ISTO), France. Analytical conditions included an accelerating voltage of 15 kV, a beam current of 10 nA, counting times of 10 seconds per element, and a defocused beam diameter of 20 µm to minimise alkali migration. The calibration of major elements was performed using the following standards: albite (Si, Na), TiMnO₃ (Mn, Ti), Al₂O₃ (Al), FeO (Fe), MgO (Mg), orthoclase (K), andradite (Ca), and apatite (P). PET analyser crystals were used for elements with lower-energy emissions (K, Ti, P, and Ca), while LiF analyser crystals were employed for higher-energy emissions (Fe and Mn) (Di Carlo *et al.*, 2006). The results are presented in Table S-1.

Ne-bearing glass. A portion of the starting material was used to produce the neon-bearing glass used for the experiments. We followed the same technique as Jambon *et al.* (1986). The starting glass powder was

equilibrated in a temperature-regulated electric furnace at 1400 °C, under a constant flux of neon at 1 bar of pressure for 270 min and quenched following the same procedure as with the starting material.

To evaluate the homogeneity of the starting glass, the ^{22}Ne concentration and the isotopic composition of four chunks of glass of about 0.1–0.2 mg each were first measured using a QMS700 quadrupole-type mass spectrometer hosted at the ISTO. The sample holder where the chunks of glass were charged was baked at 100 °C and pumped for at least one day to desorb any noble gases of the glass after quenching and the inner surfaces of the laser chamber, to avoid air contamination. Once the analytical blank was low enough to start the analysis (^{22}Ne less than $2.5 \cdot 10^{-13} \text{ cm}^3$), a laser-type Ytterbium-doped fibre was used to melt the glass and release its dissolved volatiles. Each analysed piece was heated for two min to verify its complete degassing. The gas was purified and measured in the mass spectrometer. The ^{22}Ne concentration and the $^{21}\text{Ne}/^{22}\text{Ne}$ isotopic composition are detailed in Table S-2. The measured solubility was $3.16 \pm 0.38 \cdot 10^{-4} \text{ cm}^3\text{STP} \cdot \text{g}^{-1} \cdot \text{bar}^{-1}$.

Additionally, two pieces of the same neon-bearing glass were analysed using the newly acquired Noblesse 5F-5M noble gas mass spectrometer at ISTO Orleans, France. This instrument can resolve most isobaric interferences, including the critical $^{40}\text{Ar}^{++}$ interference with $^{20}\text{Ne}^+$, which is essential to this study. Importantly, the Noblesse 5F-5M was acquired after the analyses performed with the QMS700 and HELIX-SFT mass spectrometers discussed here. As such, its data serve as subsequent confirmation of the elemental and isotopic homogeneity of the neon in the starting material.

The neon-enriched glass contains neon at abundances too high for direct analysis with either the Noblesse 5F-5M or Helix-SFT mass spectrometers. To address this, the gas was expanded into a larger volume (1 l) prior to introduction into the purification line and a volume of 0.21 cm^3 was used to introduce only a small quantity of neon into the mass spectrometer. A schematic representation of the system is provided in Figure S-1. The gas extraction followed the same protocol as with the quadrupole. The same laser type as for the QMS700 was used to bring the glass into the liquid state for two minutes, releasing the gas until *manual valve a* (Figure S-1), which corresponds to a total volume of 1100 cm^3 , from which an aliquot of 0.21 cm^3 was extracted for subsequent mass spectrometer analysis. The purification protocol closely follows the procedure outlined for the Helix-SFT in the laser ablation section because the line used for the Noblesse is the same as for the Helix-SFT. Upon entering the purification line, the gas undergoes an initial stage of purification using two titanium sponge getters as it expands. In the first stage, a hot titanium getter (operating at 700 °C) absorbs gas species such as CO_2 , O_2 , and N_2 for five minutes. After cooling to ambient temperature, this getter continues to trap H_2 for an additional ten minutes (Stout and Gibbons, 1955). The second titanium getter further refines the gas before it is sent to cryogenic traps. At this stage, noble gases—except for neon—are captured for 15 minutes using two Cryoscan CryoSorb cold-head traps. The first trap, filled with stainless steel, adsorbs heavier noble gases (Ar, Kr, and Xe), while the second trap, made of activated charcoal, traps helium and neon and can be used to separate He from Ne. The results, summarised in Table S-3, include the $^{20}\text{Ne}/^{22}\text{Ne}$ and $^{21}\text{Ne}/^{22}\text{Ne}$ isotopic ratios derived from multiple gas aliquots (5 to 10): Table S-4 presents the run standards used throughout the analysis period. This procedure effectively minimised the blank contribution, which in this case was less than 21 %.

Capsule preparation and experimental set-up. Each capsule is set up from an $\text{Au}_{80}\text{Pd}_{20}$ tube of 2.9 mm outer diameter, 0.4 mm wall thickness and 1.5 cm length. A representative drawing of the experimental capsule is shown in Figure 4. The capsule was loaded with (1) Ne-free powdered basaltic glass, (2) silver oxalate as a source of CO_2 , and (3) Ne-bearing basaltic glass for those experiments carried out with Ne. The mass of each component is given in Table S-5. The experiments were run in Internally Heated Pressure Vessels (IHPV) at ISTO, France. The experimental conditions are shown in Table S-5. All experiments were terminated by drop-

quenching (Di Carlo *et al.*, 2006). A single experiment typically involved running two or more capsules simultaneously. This approach enabled us to test the same experimental conditions (P-T-duration) with varying amounts of CO₂ so as to observe its impact on vesicularity.

X-ray microtomography acquisitions

X-ray computed tomography is a non-destructive technique that allows for the precise localisation of vesicles prior to their puncturing, enabling the analysis of the gas by mass spectrometry and the characterisation of the vesicle size distribution (VSD) of the quenched glasses. The data were collected at ISTO with a Nanotom Phoenix micro-tomography system, which is equipped with a 180 kV X-ray tube with variable filament currents (40 - 170 µA) and an operating voltage of up to 120 kV.

The biggest piece of glass of every experimental capsule (sometimes several pieces at the same time) was mounted on a carbon fibre rod with a thermoplastic adhesive and then placed into the instrument to be rotated 360° for the scan. Between 700 and 2000 projections were acquired with a resolution of 3 to 3.5 µm per pixel. The reconstruction of the acquired images in stacks was made via 3D with VGStudioMax. The first step is to correct for artefacts using a median filter and separating different density bands. Then we separated the portion of glass from the vesicles and the air surrounding the glass.

The microtomography images were processed using *ImageJ* to obtain the different VSDs in 2D. The “*analyse particles*” plugin was used after adjusting the threshold to obtain the surface of the glass and vesicles independently in each slice of the 2D image stack.

Vesicularity. The vesicularity of the samples was calculated using *VGStudioMax* by separating the volume of the glass from the volume occupied by the vesicles. Vesicularity is shown in Table S-9. Figure S-2 illustrates the evolution of vesicularity over time, with a representative example for each of the experiments.

Fourier-transform infrared spectroscopy (FTIR):

One or two chunks of glass for every quenched glass were doubly-polished for infrared spectroscopy analyses to thicknesses of 25-107 µm. The FTIR spectra were collected in transmission using a Nicolet 6700 FTIR spectrometer attached to an IR microscope (Nicolet Continuum) equipped with an MCT-detector at ISTO. CO₂ and H₂O contents were determined using a LEITZ DMR optical microscope equipped with an electronic X-Y stage. The absorbances of carbonate bands (≈1430 and ≈1525 cm⁻¹) and OH stretching vibration (≈3525 cm⁻¹) were determined by performing a first subtraction with the spectrum of an anhydrous decarbonated sample (the starting material in the mid-infrared (MIR) region, scaled to the same thickness, and then drawing a linear baseline below the H₂O bending and carbonate vibration bands). Typically, 128-256 scans were collected for each spectrum in the range of 1000 to 4000 cm⁻¹, with a window aperture of 40 x 40 and 20 x 20 µm and a spectral resolution of 4 cm⁻¹, collecting background on NaCl windows before each sample. The H₂O and CO₂ concentrations in the experimental glasses were determined using the Beer-Lambert law (*e.g.*, Stolper, 1982):

$$C_i = 100 \times (M_i \times A_j) / (d \times \rho \times \epsilon_j)$$

Where C_i is the concentration of the species i in wt.%, M_i is the molecular weight of the species, A_j is the absorbance (peak height), d is the thickness of the section at each analysed point, ρ is the density of the sample (in g · l⁻¹) and ϵ_j is the absorption coefficient of band j (in l · mol⁻¹ · cm⁻¹).

Despite that no water was added to the capsules, some FTIR spectra show a small amount of dissolved water generated through the reduction of Fe³⁺ of the melt during the synthesis. Thus, we decided to use the carbonate

peak of 1430 cm^{-1} , since the 1525 cm^{-1} band is slightly affected by the H_2O bending at 1630 cm^{-1} , to calculate the amount of CO_2 dissolved into the glass. Hence, the absorption coefficient considered for the 1430 cm^{-1} peak is $283 \pm 8\text{ l} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$. The results for CO_2 and water contents are shown in Table S-6.

Laser ablation analyses:

The experimental samples were loaded into the laser cell under vacuum and baked at $100\text{ }^\circ\text{C}$ until the blank in the line was low enough ($^{22}\text{Ne} \approx 3 \cdot 10^{-13}\text{ cm}^3\text{STP}$) to start with the analysis. The drilling was done with a diode-pumped air-cooled Q-switched laser of 263 nm wavelength and $35\text{ }\mu\text{m}$ diameter, which works by repetition of power plasma pulses. This tool allows us to drill by ablation our glass chunks which are mm thick (Figure S-3) and be very precise in reaching micrometric bubbles. The pressure in the laser cell was continuously measured (in millibars) with a manometer MKS Baratron® 1 Torr full scale. When the laser ablation starts, a smooth increase in pressure happens (corresponding to the ablation of the glass). When a bubble is reached, a sharp change in the pressure is observed (Fig. S-5), and the ablation is stopped. Gas purification then starts, as explained next.

Once the gas is released from a pierced bubble the first stage of purification is performed with two Ti-sponge getters while the volume of the gas expands into the line. As explained above, during five minutes, the first hot Ti-getter ($800\text{ }^\circ\text{C}$) adsorbs chemical species like CO_2 , O_2 , and N_2 and then, at ambient temperature after cooling, trap H_2 for ten minutes (Stout and Gibbons, 1955). A second Ti-getter repeats the process. The noble gases, except Ne, were adsorbed for 15 minutes on activated charcoal in two cold fingers at liquid N_2 temperature. One cold finger is positioned at the end of the purification line, trapping 99 % of Ar. An additional cold finger is placed at the entrance of the mass spectrometer to reduce Ar^{++} interference before and during the Ne analysis. Before introducing Ne into a Helix-Split Flight Tube (ThermoFisher) mass spectrometer, we measured two cycles of Ne isotopes to stabilize the magnet. ^{20}Ne was measured using a mass scan instead of setting the magnet on the required field to measure ^{20}Ne , using an acceleration voltage of 4.5 kV and a trap current of $250\text{ }\mu\text{A}$ for the secondary electron multiplier (SEM) configuration.

Then Ne is introduced into the mass spectrometer. In addition to masses 20, 21, and 22, we also measured masses 40 and 44 for correcting isobaric interferences between Ar^{++} and $^{20}\text{Ne}^+$ and CO_2^{++} and ^{22}Ne . Fifteen cycles of measurement were done for every single vesicle, blank or standard. The acquisition software and the data processing were homemade using C# and MATLAB© respectively. The static background in the mass spectrometer was checked and estimated regularly ($^{20}\text{Ne} = 14.26 \cdot 10^{-12}$ and $^{22}\text{Ne} = 1.19 \cdot 10^{-13}\text{ cm}^3\text{STP}$). We determined the ionisation ratios $\text{Ar}^{++}/\text{Ar}^+ = 0.03$ and $\text{CO}_2^{++}/\text{CO}_2^+ = 0.0015$ for the Helix-SFT and corrected the partial contribution by introducing the parameters in the data processing code. The mass discrimination of the mass spectrometer is determined by averaging the standards measured over the course of the analysis of the vesicles. The standard used comes from the dilution of a volume of “one pipette” (0.1 cm^3) of atmospheric air in a reservoir of 2 l. The uncertainty in the mass discrimination is represented by the standard deviation of these standards (Table S-7). The Ne blank contribution was between 3 and 20 % except for V_7 in sample *ESFa-3B-240min* for which it was 30 %. After ablating the matrix for 10 min the matrix blank contributed to 70 %. Therefore, the correction was done directly using the line blank, since the matrix contribution can be considered negligible. Uncertainties on the isotopic ratios in the samples are derived from the propagation of the measured uncertainty, the blank correction, and the mass discrimination correction. The results of the vesicle analyses are shown in Table S-8.

Calculations:

Maximum mass fractionation factor (MFF_{Ne}). The maximum fractionation factor, was calculated using Graham’s law, resulting in values of $^{20}\text{Ne}/^{22}\text{Ne} = 10.28$ and $^{21}\text{Ne}/^{22}\text{Ne} = 0.0297$. These were determined as $^{20}\text{Ne}/^{22}\text{Ne}_{\text{Air}} \cdot (m_{22}/m_{20})^{1/2}$ and $^{21}\text{Ne}/^{22}\text{Ne}_{\text{Air}} \cdot (m_{22}/m_{21})^{1/2}$ respectively.

To study the relationship between CO₂ diffusion in the melt, the displacement of bubbles along the capsule and the average growth of vesicles in the magma before quenching (Fig. 1), we calculate the following parameters.

Bubble radius growth (G_r). The growth rate of the bubbles, G_0 , can provide useful information about the experimental samples. This parameter is independent of bubble size and gives information about the residence time of the bubbles in the magma, τ . Watson *et al.* (1982) calculated this value for basaltic melts using the diffusivity laws for dissolved CO₂, but Sarda and Graham (1990) observed that this value is significantly influenced by the initial CO₂ content of the magma, which is currently unknown. Consequently, the value of G_0 can vary by several orders of magnitude. In our experiments, the initial CO₂ content is known. The formula of τ , was deconvolved in order to calculate G_r in our experimental samples, and τ was fixed at 1800 minutes which is the time at which the run product glasses showed no more vesicles (sample EN-E3).

$$G_r = [1 / (\tau \times (-a))] \times time_{exp}$$

Where a is the slope of the vesicle size distribution (VSD) trend of each sample in cm and $time_{exp}$ is the time that the experiments run. These parameters, obtained by the 2D-image scan analyses are reported in Table S-9).

CO₂ diffusion in the melt (D_{CO_2}). The predominant C-bearing species in basaltic melts is CO₃²⁻ (Zhang *et al.*, 2007). Because of the similarity in atomic size and mass to Ar, CO₂ diffusion is approximately the same as the chemical diffusivity of Ar in silicate melts (Zhang and Xu, 1995; Behrens and Zhang, 2001; Nowak *et al.*, 2004; Zhang *et al.*, 2007), and is described as:

$$\ln D_{melt} = -13.99 - (17367 + 1.9448 \times P) / T + [(855.2 + 0.2712 \times P) \times Cw] / T$$

$$D_{CO_2} = D_{melt} \times time_{exp}$$

Where D_{melt} is CO₂ diffusivity in m² s⁻¹, P is the pressure in MPa, T is the temperature in Kelvins, and Cw is the weight per cent of H₂O.

Bubble displacement (B_d). The bubble ascent rate, v_b was calculated using Stokes' Law, which describes the motion of a spherical particle through a viscous fluid.

$$v_b = -2/9 \times (r^2 \times g \times (\rho_b - \rho_L)) / \eta$$

Where r is the mean radius of the vesicles in meters, g is the gravity in m · s⁻¹, ρ_b and ρ_L are the densities of the bubble (from Bottinga and Richet (1981) for the conditions of the experiment and the liquid respectively in kg · m⁻³, and η is the viscosity of the magma in Pa · s⁻¹, calculated for each experiment using the amount of dissolved water and the experimental pressure, following the method of Giordano *et al.* (2008). The average distance of bubbles displacement during each experiment is given by:

$$B_d = v_b \times time_{exp}$$

The results are shown in Table S-10 and plotted in Figure 1. The parameters needed for the calculations can be found in Tables S-6 and S-9. In addition, the evolution of vesicularity in 3D over time is shown in Figure S-2.

Supplementary Tables

Table S-1 To ensure consistency with the data of the natural sample published by Jiménez-Mejías *et al.* (2021) (Basanite FAS*) the composition of the starting glass and the Ne-bearing glass were normalized to 100 % anhydrous (**). The sum of oxides (original totals ***) was as high as 99 %. All Fe is reported as FeO, and the standard deviation. Oxide concentrations (wt. %) measured by electron microprobe analysis at Orléans Earth Sciences Institute, France (ISTO).

| | Basanite FAS* | Starting mat. ** | Ne-bearing glass** | Starting mat. *** | Ne-bearing glass*** |
|--------------------------------------|---------------|---------------------|-----------------------|----------------------|------------------------|
| SiO ₂ | 45.08 ± 0.14 | 44.86 ± 0.23 | 45.47 ± 0.15 | 44.27 ± 0.33 | 44.95 ± 0.28 |
| TiO ₂ | 3.82 ± 0.13 | 3.70 ± 0.18 | 3.78 ± 0.14 | 3.65 ± 0.16 | 3.74 ± 0.14 |
| Al ₂ O ₃ | 14.45 ± 0.17 | 14.87 ± 0.17 | 14.94 ± 0.17 | 14.67 ± 0.17 | 14.77 ± 0.12 |
| FeO* | 12.51 ± 0.27 | 12.39 ± 0.24 | 11.49 ± 0.43 | 12.22 ± 0.26 | 11.36 ± 0.48 |
| MnO | 0.28 ± 0.05 | 0.17 ± 0.07 | 0.11 ± 0.06 | 0.17 ± 0.07 | 0.11 ± 0.06 |
| MgO | 7.15 ± 0.22 | 7.80 ± 0.05 | 7.8 ± 0.11 | 7.69 ± 0.04 | 7.71 ± 0.10 |
| CaO | 10.82 ± 0.17 | 10.38 ± 0.10 | 10.43 ± 0.16 | 10.24 ± 0.12 | 10.31 ± 0.11 |
| Na ₂ O | 3.6 ± 0.10 | 3.63 ± 0.07 | 3.77 ± 0.08 | 3.59 ± 0.08 | 3.73 ± 0.06 |
| K ₂ O | 1.57 ± 0.07 | 1.47 ± 0.15 | 1.49 ± 0.04 | 1.45 ± 0.15 | 1.47 ± 0.05 |
| P ₂ O ₅ | 0.71 ± 0.07 | 0.73 ± 0.09 | 0.71 ± 0.05 | 0.72 ± 0.08 | 0.71 ± 0.05 |
| Na ₂ O + K ₂ O | 5.17 | 5.10 | 5.26 | 5.03 | 5.20 |
| Total | 100 | 100 | 100 | 98.67 | 98.85 |

Table S-2 ²²Ne abundances (in cm³ STP/g) and ²¹Ne/²²Ne ratio obtained with the quadrupole mass spectrometer for the Ne-bearing glass chunks analysed for checking homogeneity. The glass was free of vesicles and melted for 2 min under vacuum. The error associated with the abundance and the isotopic values is calculated as 1σ. The error associated with the average is obtained by the standard deviation of the four values.

| Sample | mass · 10 ⁻⁴ g | ²² Ne · 10 ⁻⁹ cm ³ STP / g | ²¹ Ne/ ²² Ne | Solubility · 10 ⁻⁴ cm ³ STP / g · bar |
|----------------|---------------------------|---|------------------------------------|---|
| Test6-s1 | 2.32 | 9.67 ± 0.04 | 0.0296 ± 0.0015 | 3.26 |
| Test6-s2 | 1.24 | 10.79 ± 0.04 | 0.0285 ± 0.0014 | 3.63 |
| Test6-s3 | 2.04 | 8.86 ± 0.02 | 0.0299 ± 0.0015 | 2.98 |
| Test6-s4 | 1.40 | 8.19 ± 0.02 | 0.0295 ± 0.0015 | 2.76 |
| Average | | 9.38 ± 1.12 | 0.0294 ± 0.0006 | 3.16 ± 0.38 |

Table S-3 ^{22}Ne abundances (in cm^3 STP/g) and $^{20}\text{Ne}/^{22}\text{Ne}$ and $^{21}\text{Ne}/^{22}\text{Ne}$ ratios measured with the Noblesse 5F-5M mass spectrometer for two neon-bearing glass samples (Test7-s1 and Test7-s2), analysed to check their isotopic composition and homogeneity. The # symbol denotes repeated measurements to test reproducibility, with each measurement corresponding to a new set of aliquots (see Vol 2 in Figure S-1). The glass, free of vesicles, was brought back to liquid state under vacuum for 2 minutes. The error associated with the abundance and the isotopic values is calculated as 1σ . The error associated with the average is obtained by the standard deviation of the test values.

| Sample | mass · 10^{-4} g | Number of Aliquots | Blank | measured $^{22}\text{Ne} \cdot 10^{-8}$ cm^3 STP / g | $^{20}\text{Ne}/^{22}\text{Ne}$ | $^{21}\text{Ne}/^{22}\text{Ne}$ |
|-------------|--------------------|--------------------|---------|---|-----------------------------------|---------------------------------------|
| Test7-s1 #1 | 1.18 | 5 | 21.07 % | 1.22 | 9.87 ± 0.07 | 0.0287 ± 0.0010 |
| Test7-s1 #2 | 1.18 | 10 | 14.46 % | 1.84 | 9.78 ± 0.05 | 0.0290 ± 0.0008 |
| Test7-s1 #3 | 1.18 | 10 | 14.71 % | 1.81 | 9.87 ± 0.05 | 0.0291 ± 0.0009 |
| | | | | Average | 9.84 ± 0.05 | 0.0289 ± 0.0002 |
| Test7-s2 #1 | 2.30 | 10 | 5.81 % | 2.59 | 9.82 ± 0.03 | 0.0288 ± 0.0005 |
| Test7-s2 #2 | 2.30 | 10 | 5.84 % | 2.58 | 9.80 ± 0.03 | 0.0287 ± 0.0005 |
| | | | | Average | 9.81 ± 0.01 | 0.0287 ± 0.0001 |

Table S-4 Measured neon isotopic ratios (corrected for blanks) of the Ne-standard (for the Noblesse 5F-5M mass spectrometer). This set was analysed over ten days, the averages for the two isotopic ratios are represented with the associated 1sigma uncertainties. To correct for mass discrimination, averages of isotopic ratios measured on standards during the same period as for ne-bearing glass were taken as well as an average of $7.02 \cdot 10^{-15} \text{ cm}^3 \text{ STP} \cdot \text{cps}^{-1}$ for the sensitivity of ^{22}Ne . The uncertainty of the average represents the standard deviation of the reported set standards.

| | | Sensitivity $^{22}\text{Ne} \cdot 10^{-15}$ ($\text{cm}^3\text{STP} / \text{cps}$) | $^{20}\text{Ne}/^{22}\text{Ne}$ | $^{21}\text{Ne}/^{22}\text{Ne}$ |
|-------|----------------|--|------------------------------------|---------------------------------------|
| Day 1 | STD_1 | 6.99 | 10.05 ± 0.02 | 0.0288 ± 0.0004 |
| | STD_2 | 7.01 | 10.04 ± 0.02 | 0.0300 ± 0.0004 |
| | STD_3 | 7.03 | 10.05 ± 0.02 | 0.0293 ± 0.0003 |
| | STD_4 | 7.03 | 10.04 ± 0.02 | 0.0298 ± 0.0003 |
| | STD_5 | 7.01 | 10.03 ± 0.02 | 0.0300 ± 0.0004 |
| | Average | | 10.04 ± 0.01 | 0.0296 ± 0.0005 |
| Day 2 | STD_6 | 6.96 | 10.00 ± 0.02 | 0.0295 ± 0.0003 |
| | STD_7 | 6.96 | 10.02 ± 0.02 | 0.0292 ± 0.0004 |
| | STD_8 | 6.92 | 10.00 ± 0.02 | 0.0298 ± 0.0003 |
| | STD_9 | 6.88 | 9.97 ± 0.02 | 0.0298 ± 0.0003 |
| | STD_10 | 6.92 | 10.05 ± 0.02 | 0.0289 ± 0.0003 |
| | STD_11 | 6.92 | 10.05 ± 0.02 | 0.0295 ± 0.0003 |
| | Average | | 10.01 ± 0.03 | 0.0294 ± 0.0003 |
| Day 3 | STD_12 | 7.20 | 10.04 ± 0.02 | 0.0292 ± 0.0004 |
| | STD_13 | 7.12 | 10.00 ± 0.02 | 0.0297 ± 0.0004 |
| | STD_14 | 7.13 | 10.05 ± 0.02 | 0.0294 ± 0.0003 |
| | STD_15 | 7.11 | 10.05 ± 0.02 | 0.0298 ± 0.0004 |
| | STD_16 | 7.10 | 10.09 ± 0.02 | 0.0300 ± 0.0004 |
| | STD_17 | 7.08 | 10.04 ± 0.02 | 0.0294 ± 0.0003 |
| | STD_18 | 7.00 | 10.04 ± 0.02 | 0.0296 ± 0.0004 |
| | | mean | | 10.04 ± 0.02 |
| Day 4 | STD_19 | 7.05 | 10.0 ± 0.02 | 0.0292 ± 0.0004 |
| | STD_20 | 7.04 | 10.03 ± 0.02 | 0.0298 ± 0.0004 |
| | STD_21 | 7.03 | 10.03 ± 0.02 | 0.0296 ± 0.0003 |
| | STD_22 | 7.05 | 10.06 ± 0.02 | 0.0294 ± 0.0003 |
| | mean | | 10.04 ± 0.01 | 0.0295 ± 0.0003 |
| Day 5 | STD_23 | 6.90 | 10.04 ± 0.02 | 0.0294 ± 0.0003 |
| | STD_24 | 6.96 | 10.15 ± 0.02 | 0.0295 ± 0.0003 |
| | STD_25 | 6.98 | 10.14 ± 0.02 | 0.0297 ± 0.0003 |
| | STD_26 | 7.02 | 10.14 ± 0.02 | 0.0296 ± 0.0004 |
| | STD_27 | 7.06 | 10.14 ± 0.02 | 0.0289 ± 0.0004 |
| | STD_28 | 7.08 | 10.13 ± 0.02 | 0.0298 ± 0.0003 |
| | mean | | 10.12 ± 0.04 | 0.0295 ± 0.0003 |

Table S-5 Experimental conditions. # indicates the experimental run; CO₂, Ne-bearing glass and powder of starting material loaded into the capsule in mg. *P* and *T* are the conditions in the IHPV. *time_{exp}* is the time at which the experiment was quenched.

| Sample | Run | Starting mat. · 10 ⁻³ g | CO ₂ added · 10 ⁻³ g | Ne-bearing glass · 10 ⁻³ g | T °C | P _{exp} bar | time _{exp} min |
|--|----------|------------------------------------|--|---------------------------------------|------|----------------------|-------------------------|
| <i>CO₂-bearing glass samples</i> | | | | | | | |
| ESFa-4 | #2 | 63.20 | 0.75 | - | 1200 | 1598 | 30 |
| ESFa-8 | #3 | 60.10 | 0.84 | - | 1200 | 1609 | 240 |
| ESFa-10 | #3 | 58.10 | 4.49 | - | 1200 | 1609 | 240 |
| ESFa-10E | #7 | 62.00 | 0.87 | - | 1200 | 1726 | 1140 |
| ESFa-2D | #7 | 62.30 | 3.59 | - | 1200 | 1726 | 1140 |
| ESFa-3D | #8 + 9 | 55.20 | 0.38 | - | 1200 | 1725 | 130 |
| ESFa-8D | #10 + 11 | 63.70 | 2.96 | - | 1200 | 1674 | 360 |
| ESFa-1C | #12 | 58.90 | 1.56 | - | 1200 | 1671 | 10 |
| ESFa-2C | #12 | 56.50 | 3.10 | - | 1200 | 1671 | 10 |
| ESFa-4C | #13 | 61.80 | 0.93 | - | 1200 | 1683 | 60 |
| ESFa-5C | #13 | 61.80 | 3.56 | - | 1200 | 1683 | 60 |
| ESFa-6C | #15 | 58.60 | 0.41 | - | 1200 | 1605 | 240 |
| ESFa-7C | #15 | 61.30 | 4.00 | - | 1200 | 1605 | 240 |
| ESFa-9C | #14 | 78.20 | 3.42 | - | 1200 | 1660 | 10 |
| ESFa-10C | #15 | 61.80 | 0.81 | - | 1200 | 1605 | 240 |
| ESFa-1B | #14 | 64.60 | 0.87 | - | 1200 | 1660 | 10 |
| <i>CO₂+Ne-bearing glass samples</i> | | | | | | | |
| ESFa-1B | #1 | 60.70 | 1.83 | 6.60 | 1200 | 1535 | 240 |
| ESFa-2B | #1 | 63.80 | 0.84 | 6.50 | 1200 | 1535 | 240 |
| ESFa-3B | #1 | 51.60 | 0.84 | 5.40 | 1200 | 1535 | 240 |
| ESFa-5B | #2 | 60.40 | 1.80 | 6.00 | 1200 | 1573 | 10 |
| ESFa-6B | #2 | 63.50 | 3.74 | 6.30 | 1200 | 1573 | 10 |
| ESFa-7B | #4 | 63.50 | 0.90 | 6.50 | 1200 | 1605 | 240 |
| ESFa-10B | #4 | 64.10 | 3.83 | 6.30 | 1200 | 1605 | 240 |
| ESFa-2A | #4 | 59.90 | 0.96 | 6.30 | 1200 | 1605 | 240 |
| ESFa-5A | #5 | 62.50 | 3.65 | 6.60 | 1200 | 1669 | 10 |
| EN-E3 | #7 | 57.90 | 0.17 | 5.90 | 1200 | 2015 | 1800 |

Table S-6 FTIR data. The average of H₂O and CO₂ concentration was calculated from the number of analytical spots in each sample, the results shown below were calculated using the error propagator developed by the « Digital modelling platform» of ISTO. The thickness belongs to the spot where the analysis was done. Thickness was measured five times at every analysed spot, and the thickness uncertainty corresponds to the standard deviation of these measurements. The density of the silicate liquid, δ_L , was determined using the model of Lange (1994) and Warren (1995). A_{1430} and A_{3525} are the absorbance of the fundamental OH-stretching vibration (~ 3525 cm⁻¹) and the carbonate doublet (~ 1430 cm⁻¹) in the mid-infrared (MIR) region.

| Sample | Thickness · 10 ⁻³ cm | δ_L g · l ⁻¹ | A_{1430} | <i>sd</i> | A_{3525} | <i>sd</i> | [CO ₂] ppm | H ₂ O wt. % |
|-------------------------------------|---------------------------------|--------------------------------|------------|-----------|------------|-----------|------------------------|------------------------|
| <i>CO₂-bearing glass</i> | | | | | | | | |
| ESFa-4.3 | 3.32 ± 0.36 | 2722 ± 15 | 0.060 | 0.002 | - | - | 722 ± 429 | |
| ESFa-4.4 | 5.10 ± 0.40 | 2722 ± 15 | 0.037 | 0.003 | - | - | | |
| ESFa-8.1 | 5.58 ± 0.13 | 2717 ± 7 | 0.087 | 0.005 | 0.063 | 0.002 | 893 | 0.13 |
| ESFa-10.1 | 4.98 ± 0.18 | 2734 ± 41 | 0.114 | 0.004 | 0.064 | 0.003 | 1022 ± 252 | 0.10 ± 0.03 |
| ESFa-10.2 | 6.20 ± 0.21 | 2734 ± 41 | 0.088 | 0.003 | 0.044 | 0.002 | | |
| ESFa-10.3 | 5.58 ± 0.13 | 2734 ± 41 | 0.095 | 0.005 | 0.044 | 0.003 | | |
| ESFa-10E.1 | 6.06 ± 0.24 | 2739 ± 4 | 0.121 | 0.007 | 0.239 | 0.003 | 1315 ± 169 | 0.47 ± 0.04 |
| ESFa-10E.4 | 4.94 ± 0.36 | 2739 ± 4 | 0.117 | 0.004 | 0.231 | 0.001 | | |
| ESFa-10E.5 | 4.90 ± 0.25 | 2739 ± 4 | 0.136 | 0.002 | 0.217 | 0.002 | | |
| ESFa-10E.6 | 5.52 ± 0.20 | 2739 ± 4 | 0.118 | 0.006 | 0.217 | 0.001 | | |
| ESFa-2D.1 | 8.46 ± 0.19 | 2751 ± 7 | 0.162 | 0.006 | 0.303 | 0.002 | 1059 ± 111 | 0.39 ± 0.04 |
| ESFa-2D.2 | 8.68 ± 3.40 | 2751 ± 7 | 0.144 | 0.007 | 0.274 | 0.002 | | |
| ESFa-2D.3 | 8.58 ± 0.31 | 2751 ± 7 | 0.175 | 0.004 | 0.336 | 0.002 | | |
| ESFa-3D.1 | 3.04 ± 0.67 | 2772 ± 44 | 0.063 | 0.002 | - | - | 925 ± 200 | |
| ESFa-3D.2 | 5.92 ± 0.47 | 2772 ± 44 | 0.085 | 0.002 | - | - | | |
| ESFa-3D.3 | 5.92 ± 0.47 | 2772 ± 44 | 0.086 | 0.002 | - | - | | |
| ESFa-8D.1 | 9.68 ± 0.11 | 2735 ± 13 | 0.111 | 0.005 | - | - | 702 ± 114 | |
| ESFa-8D.3 | 9.16 ± 0.17 | 2735 ± 13 | 0.113 | 0.004 | - | - | | |
| ESFa-8D.4 | 9.40 ± 0.16 | 2735 ± 13 | 0.126 | 0.009 | - | - | | |
| ESFa-8D.4.2 | 9.66 ± 0.11 | 2735 ± 13 | 0.117 | 0.004 | - | - | | |

| Sample | Thickness · 10 ⁻³ cm | δ _L g · l ⁻¹ | A ₁₄₃₀ | sd | A ₃₅₂₅ | sd | [CO ₂] ppm | H ₂ O wt. % |
|-------------|---------------------------------|------------------------------------|-------------------|-------|-------------------|-------|------------------------|------------------------|
| ESFa-8D.5 | 10.66 ± 0.38 | 2735 ± 13 | 0.164 | 0.005 | - | - | | |
| ESFa-8D.6 | 9.64 ± 0.18 | 2735 ± 13 | 0.090 | 0.002 | - | - | | |
| ESFa-1C.1 | 5.50 ± 0.24 | 2762 ± 56 | 0.086 | 0.002 | - | - | 971 ± 159 | |
| ESFa-1C.2 | 5.36 ± 0.13 | 2762 ± 56 | 0.084 | 0.003 | - | - | | |
| ESFa-1C.3 | 6.68 ± 0.41 | 2762 ± 56 | 0.137 | 0.004 | - | - | | |
| ESFa-2C.1 | 3.68 ± 0.29 | 2761 ± 31 | 0.024 | 0.003 | - | - | 668 ± 304 | |
| ESFa-2C.2 | 4.24 ± 0.27 | 2761 ± 31 | 0.049 | 0.003 | - | - | | |
| ESFa-2C.3 | 4.26 ± 0.81 | 2761 ± 31 | 0.074 | 0.006 | - | - | | |
| ESFa-4C.2 | 2.64 ± 0.19 | 2742 ± 18 | 0.080 | 0.005 | - | - | 1710 ± 3 | |
| ESFa-5C.1 | 4.96 ± 0.19 | 2754 ± 32 | 0.063 | 0.003 | - | - | 722 | |
| ESFa-6C.1.1 | 6.68 ± 0.33 | 2730 ± 21 | 0.089 | 0.005 | - | - | 700 ± 84 | |
| ESFa-6C.1.2 | 6.72 ± 0.19 | 2730 ± 21 | 0.086 | 0.004 | - | - | | |
| ESFa-6C.2.1 | 7.00 ± 0.35 | 2730 ± 21 | 0.081 | 0.004 | - | - | | |
| ESFa-6C.2.2 | 7.00 ± 0.35 | 2730 ± 21 | 0.071 | 0.004 | - | - | | |
| ESFa-6C.2.3 | 7.02 ± 0.22 | 2730 ± 21 | 0.096 | 0.006 | - | - | | |
| ESFa-7C.1 | 7.18 ± 0.31 | 2773 ± 57 | 0.106 | 0.006 | - | - | 915 ± 137 | |
| ESFa-7C.2 | 5.78 ± 0.36 | 2773 ± 57 | 0.088 | 0.010 | - | - | | |
| ESFa-7C.3 | 5.72 ± 0.19 | 2773 ± 57 | 0.109 | 0.006 | - | - | | |
| ESFa-7C.4 | 5.78 ± 0.36 | 2773 ± 57 | 0.108 | 0.007 | - | - | | |
| ESFa-7C.5 | 7.82 ± 0.22 | 2773 ± 57 | 0.107 | 0.013 | - | - | | |
| ESFa-9C.1 | 5.00 ± 0.21 | 2762 ± 12 | 0.064 | 0.003 | 0.082 | 0.004 | 585 ± 178 | 0.21 ± 0.04 |
| ESFa-9C.2 | 4.56 ± 0.55 | 2762 ± 12 | 0.026 | 0.002 | 0.082 | 0.002 | | |
| ESFa-9C.3 | 4.56 ± 0.55 | 2762 ± 12 | 0.052 | 0.002 | 0.081 | 0.002 | | |
| ESFa-9C.4 | 3.72 ± 0.16 | 2762 ± 12 | 0.043 | 0.003 | 0.093 | 0.002 | | |
| ESFa-10C.1 | 5.22 ± 0.26 | 2731 ± 13 | 0.078 | 0.006 | - | - | 931 ± 97 | |
| ESFa-10C.2 | 5.22 ± 0.26 | 2731 ± 13 | 0.083 | 0.007 | - | - | | |
| ESFa-10C.3 | 5.40 ± 0.21 | 2731 ± 13 | 0.098 | 0.007 | - | - | | |

| Sample | Thickness · 10 ⁻³ cm | δ _L g · l ⁻¹ | A ₁₄₃₀ | sd | A ₃₅₂₅ | sd | [CO ₂] ppm | H ₂ O wt. % |
|--|---------------------------------|------------------------------------|-------------------|-------|-------------------|-------|------------------------|------------------------|
| ESFa-10C.3 | 5.40 ± 0.21 | 2731 ± 13 | 0.098 | 0.007 | - | - | | |
| ESFa-1B.1 | 4.40 ± 0.17 | 2731 ± 13 | 0.034 | 0.003 | 0.099 | 0.009 | 707 ± 267 | 0.24 ± 0.04 |
| ESFa-1B.2 | 4.92 ± 0.16 | 2731 ± 13 | 0.060 | 0.003 | 0.124 | 0.006 | | |
| ESFa-1B.3 | 4.92 ± 0.16 | 2731 ± 13 | 0.085 | 0.004 | 0.086 | 0.006 | | |
| <i>CO₂+Ne-bearing glass</i> | | | | | | | | |
| ESFa-1B.1 | 5.00 ± 0.25 | 2678 ± 46 | 0.105 | 0.010 | | | 1328 ± 158 | |
| ESFa-1B.2 | 4.92 ± 0.27 | 2678 ± 46 | 0.122 | 0.006 | | | | |
| ESFa-2B.1 | 3.52 ± 0.19 | 2739 ± 75 | 0.079 | 0.004 | | | 1023 ± 259 | |
| ESFa-2B.2 | 3.52 ± 0.19 | 2739 ± 75 | 0.064 | 0.006 | | | | |
| ESFa-2B.3 | 4.08 ± 0.30 | 2739 ± 75 | 0.055 | 0.004 | | | | |
| ESFa-3B.1 | 8.22 ± 0.11 | 2691 ± 7 | 0.099 | 0.003 | | | 721 ± 188 | |
| ESFa-3B.2 | 8.22 ± 0.11 | 2691 ± 7 | 0.108 | 0.001 | | | | |
| ESFa-3B.3 | 8.22 ± 0.11 | 2691 ± 7 | 0.128 | 0.002 | | | | |
| ESFa-3B.4 | 7.32 ± 0.19 | 2691 ± 7 | 0.053 | 0.009 | | | | |
| ESFa-3B.5 | 5.68 ± 0.28 | 2691 ± 7 | 0.082 | 0.003 | | | | |
| ESFa-5B.1 | 2.58 ± 0.25 | 2755 ± 4 | 0.081 | 0.004 | | | 1705 ± 101 | |
| ESFa-5B.2 | 2.66 ± 0.22 | 2755 ± 4 | 0.077 | 0.004 | | | | |
| ESFa-6B.1 | 4.16 ± 0.38 | 2758 ± 3 | 0.052 | 0.002 | 0.120 | 0.000 | 880 ± 240 | 0.30 ± 0.02 |
| ESFa-6B.2 | 4.16 ± 0.38 | 2758 ± 3 | 0.077 | 0.001 | 0.107 | 0.001 | | |
| ESFa-7B.1 | 3.42 ± 0.22 | 2728 ± 8 | 0.090 | 0.002 | | | 1546 ± 66 | |
| ESFa-7B.2 | 3.40 ± 0.43 | 2728 ± 8 | 0.095 | 0.003 | | | | |
| ESFa-10B.1 | 3.90 ± 0.22 | 2707 ± 5 | 0.087 | 0.004 | | | 1197 ± 119 | |
| ESFa-10B.2 | 4.18 ± 0.18 | 2707 ± 5 | 0.081 | 0.003 | | | | |
| ESFa-2A.1 | 6.42 ± 0.20 | 2715 ± 20 | 0.120 | 0.004 | | | 1023 ± 63 | |
| ESFa-2A.6 | 3.28 ± 0.13 | 2715 ± 20 | 0.056 | 0.002 | | | | |
| ESFa-5A.1 | 3.38 ± 0.84 | 2770 ± 36 | 0.064 | 0.003 | | | 1043 ± 47 | |

| Sample | Thickness · 10 ⁻³ cm | δ _L g · l ⁻¹ | A ₁₄₃₀ | <i>sd</i> | A ₃₅₂₅ | <i>sd</i> | [CO ₂] ppm | H ₂ O wt. % |
|-----------|---------------------------------|------------------------------------|-------------------|-----------|-------------------|-----------|------------------------|------------------------|
| ESFa-5A.2 | 3.40 ± 0.71 | 2770 ± 36 | 0.060 | 0.005 | | | | |
| EN-E3.1 | 4.76 ± 0.17 | 2770 ± 6 | 0.213 | 0.001 | 0.4330 | 0.0019 | 2385 ± 162 | 0.91 ± 0.06 |
| EN-E3.2 | 4.76 ± 0.17 | 2770 ± 6 | 0.187 | 0.003 | 0.3600 | 0.0007 | | |
| EN-E3.3 | 4.76 ± 0.17 | 2770 ± 6 | 0.189 | 0.003 | 0.3932 | 0.0016 | | |
| EN-E3.4 | 4.76 ± 0.17 | 2770 ± 6 | 0.205 | 0.004 | 0.3864 | 0.0009 | | |
| EN-E3.5 | 4.76 ± 0.17 | 2770 ± 6 | 0.217 | 0.004 | 0.4038 | 0.0013 | | |

Table S-7 Measured neon isotopic ratios (corrected for blanks) of the Ne-standard (for the Helix-SDT mass spectrometer). This set was analysed over sixteen months, the averages for the two isotopic ratios are represented with the associated 1sigma uncertainties. To correct for mass discrimination, averages of isotopic ratios and sensitivity measured on standards during the same period as for vesicles (Table S-8) were taken. The uncertainty of the average represents the standard deviation of the reported set standards.

| | Sensitivity $^{22}\text{Ne} \cdot 10^{-15}$ (cm ³ STP / cps) | $^{20}\text{Ne}/^{22}\text{Ne}$ | $^{21}\text{Ne}/^{22}\text{Ne}$ |
|-------------|---|---------------------------------|---------------------------------|
| STD_1 | 3.65 | 10.04 ± 0.02 | 0.0291 ± 0.0003 |
| STD_2 | 3.69 | 10.30 ± 0.04 | 0.0289 ± 0.0002 |
| STD_3 | 3.70 | 10.23 ± 0.03 | 0.0292 ± 0.0003 |
| STD_4 | 3.67 | 10.20 ± 0.02 | 0.0290 ± 0.0001 |
| STD_5 | 2.43 | 10.22 ± 0.03 | 0.0291 ± 0.0003 |
| STD_6 | 3.78 | 10.34 ± 0.03 | 0.0287 ± 0.0002 |
| STD_7 | 3.69 | 10.31 ± 0.03 | 0.0292 ± 0.0002 |
| STD_8 | 3.70 | 10.23 ± 0.02 | 0.0288 ± 0.0002 |
| STD_9 | 3.72 | 10.32 ± 0.04 | 0.0285 ± 0.0002 |
| STD_10 | 3.68 | 10.27 ± 0.03 | 0.0294 ± 0.0002 |
| STD_11 | 3.67 | 10.26 ± 0.03 | 0.0288 ± 0.0002 |
| STD_12 | 3.61 | 10.20 ± 0.03 | 0.0290 ± 0.0003 |
| STD_13 | 3.64 | 10.24 ± 0.02 | 0.0290 ± 0.0003 |
| STD_14 | 3.73 | 10.27 ± 0.03 | 0.0283 ± 0.0003 |
| STD_15 | 3.76 | 10.22 ± 0.03 | 0.0288 ± 0.0003 |
| STD_16 | 3.73 | 10.18 ± 0.02 | 0.0286 ± 0.0002 |
| STD_17 | 3.65 | 10.28 ± 0.03 | 0.0288 ± 0.0003 |
| mean | | 10.24 ± 0.07 | 0.0289 ± 0.0003 |
| STD_18 | 3.66 | 10.13 ± 0.03 | 0.0290 ± 0.0003 |
| STD_19 | 3.64 | 10.02 ± 0.04 | 0.0287 ± 0.0002 |
| STD_20 | 3.69 | 10.27 ± 0.03 | 0.0288 ± 0.0003 |
| STD_21 | 3.74 | 10.38 ± 0.03 | 0.0288 ± 0.0003 |
| STD_22 | 3.69 | 10.20 ± 0.03 | 0.0288 ± 0.0003 |
| STD_23 | 3.68 | 10.29 ± 0.03 | 0.0288 ± 0.0003 |
| STD_24 | 3.67 | 10.32 ± 0.04 | 0.0293 ± 0.0004 |
| STD_25 | 3.68 | 10.32 ± 0.04 | 0.0284 ± 0.0003 |
| STD_26 | 3.64 | 9.92 ± 0.03 | 0.0286 ± 0.0003 |
| STD_27 | 3.65 | 10.03 ± 0.04 | 0.0286 ± 0.0002 |
| STD_28 | 3.66 | 10.07 ± 0.04 | 0.0289 ± 0.0004 |
| STD_29 | 3.66 | 10.15 ± 0.03 | 0.0286 ± 0.0002 |
| STD_30 | 3.66 | 10.21 ± 0.02 | 0.0289 ± 0.0002 |
| STD_31 | 3.71 | 10.09 ± 0.04 | 0.0293 ± 0.0003 |
| STD_32 | 3.70 | 10.24 ± 0.04 | 0.0291 ± 0.0003 |
| STD_33 | 3.72 | 10.19 ± 0.02 | 0.0292 ± 0.0004 |
| mean | | 10.18 ± 0.13 | 0.0289 ± 0.0003 |

Table S-8 Measured ^{22}Ne abundances and Ne isotopic compositions obtained with the HELIX-SFT mass spectrometer of the vesicles for the experimental samples *ESFa-3B-204min*, *ESFa-5B-10min* and *ESFa-6B-10min*. Errors are 1 sigma uncertainties. $P_{b\text{Tot}}$ is the partial pressure resulting from vesicle gas expansion to the purification line when piercing. *Air* composition from (Ozima and Podosek, 2002). **The gas extracted in these vesicles corresponds to three vesicles pierced at the same time.

| Sample | $P_{b\text{Tot}} \cdot 10^{-7}$ bar | $^{22}\text{Ne} \cdot 10^{-12} \text{ cm}^3$ STP | $^{20}\text{Ne}/^{22}\text{Ne}$ | $^{21}\text{Ne}/^{22}\text{Ne}$ |
|-----------------------------------|--|---|---------------------------------|---------------------------------|
| ESFa-3B vesicles (240 min) | | | | |
| V1 | 40 | 5.00 ± 0.10 | 9.97 ± 0.06 | 0.0288 ± 0.0006 |
| V2** | 79 | 6.36 ± 0.12 | 10.28 ± 0.05 | 0.0303 ± 0.0006 |
| V3 | 26 | 3.33 ± 0.06 | 9.97 ± 0.09 | 0.0303 ± 0.0007 |
| V4 | 22 | 4.00 ± 0.08 | 9.8 ± 0.06 | 0.0285 ± 0.0007 |
| V5 | 165 | 18.25 ± 0.35 | 10.05 ± 0.05 | 0.0296 ± 0.0005 |
| V6 | 14 | 2.51 ± 0.05 | 9.75 ± 0.08 | 0.0286 ± 0.0007 |
| V7 | 11 | 1.93 ± 0.04 | 9.85 ± 0.12 | 0.0282 ± 0.0007 |
| V1 ALA | 19 | 2.08 ± 0.04 | 9.82 ± 0.08 | 0.0307 ± 0.0007 |
| V2 ALA | 15 | 2.51 ± 0.05 | 10.50 ± 0.13 | 0.0303 ± 0.0005 |
| V3 ALA | 350 | 3.84 ± 0.07 | 10.26 ± 0.10 | 0.0298 ± 0.0004 |
| V4 ALA | 9 | 1.73 ± 0.03 | 9.84 ± 0.08 | 0.0294 ± 0.0007 |
| V5 ALA | 11 | 1.67 ± 0.02 | 9.88 ± 0.10 | 0.0304 ± 0.0009 |
| V6 ALA | 9 | 1.20 ± 0.02 | 10.08 ± 0.11 | 0.0282 ± 0.0010 |
| V7 ALA | 6 | 0.98 ± 0.02 | 10.06 ± 0.09 | 0.0291 ± 0.0007 |
| V8 ALA | 13 | 2.04 ± 0.04 | 10.03 ± 0.10 | 0.0292 ± 0.0008 |
| V9 ALA** | 36 | 4.54 ± 0.09 | 9.73 ± 0.08 | 0.0292 ± 0.0005 |
| V10 ALA | 35 | 5.89 ± 0.11 | 9.76 ± 0.07 | 0.0288 ± 0.0004 |
| V11 ALA | 21 | 2.59 ± 0.05 | 9.77 ± 0.09 | 0.0287 ± 0.0007 |
| ESFa-5B vesicles (10 min) | | | | |
| V1 | 173 | 23.25 ± 0.26 | 9.97 ± 0.02 | 0.0289 ± 0.0002 |
| V2 | 19 | 2.95 ± 0.03 | 10.03 ± 0.04 | 0.0289 ± 0.0004 |
| V3 | 28 | 3.89 ± 0.04 | 9.96 ± 0.04 | 0.0290 ± 0.0005 |
| V4 | 18 | 2.62 ± 0.03 | 10.24 ± 0.05 | 0.0302 ± 0.0005 |
| V5 | 19 | 2.34 ± 0.03 | 10.02 ± 0.05 | 0.0284 ± 0.0005 |
| V6 | 28 | 3.57 ± 0.04 | 9.80 ± 0.03 | 0.0284 ± 0.0004 |
| V7 | 178 | 24.24 ± 0.27 | 9.81 ± 0.02 | 0.0293 ± 0.0001 |
| V10 | 20 | 2.99 ± 0.03 | 9.62 ± 0.04 | 0.0283 ± 0.0003 |
| V11 | 31 | 4.60 ± 0.05 | 9.44 ± 0.04 | 0.0296 ± 0.0002 |
| V12 | 23 | 3.62 ± 0.04 | 9.73 ± 0.05 | 0.0300 ± 0.0005 |
| ESFa-6B vesicles (10 min) | | | | |
| V1 | 9 | 1.45 ± 0.01 | 9.40 ± 0.08 | 0.0264 ± 0.0009 |
| V2 | 49 | 5.01 ± 0.05 | 9.56 ± 0.03 | 0.0297 ± 0.0005 |
| V3 | 9 | 1.49 ± 0.01 | 9.58 ± 0.07 | 0.0289 ± 0.0008 |
| <i>Air</i> | | | 9.8 | 0.0290 |
| <i>MFF_{Ne}</i> | | | 10.28 | 0.0297 |

Table S-9 Vesicle size distribution parameters of the experimental samples analysed by microtomography. The image treatment was done by using *ImageJ*. *Nb* represents the number of counted bubbles in one slice of the image stack obtained by the tomography; *Area* is the analysed surface of the glass; Bubble density corresponds to the number of vesicles by surface area; D_{max} and D_{mean} correspond to the maximum and mean diameter of the vesicles of the scanned slice; $Ln(n)$ is the density of the population of vesicles obtained by the linear regression of $Ln(n)$ vs D_{max} ; a is the slope of the trend line; R^2 is the correlation coefficient of the trend line; $D_{mean.th}$ is the theoretical mean diameter obtained by the inverse of the slope ($-1/a$); *Ves.* is the obtained vesicularity by VGSudioMax.

| Sample | Nb | Area (mm ²) | Bubbles density (mm ⁻²) | D_{max} (μm) | D_{mean} (μm) | $Ln(n)$ (mm ⁻⁴) | a (mm ⁻¹) | R^2 | $D_{mean.th. (-1/a)}$ (μm) | Ves (%) |
|---|------|-------------------------|-------------------------------------|----------------|-----------------|-----------------------------|-------------------------|-------|----------------------------|---------|
| <i>CO₂-bearing glass</i> | | | | | | | | | | |
| ESFa-4 | 376 | 9.0 | 41.9 | 99.9 | 19.8 | 12.5 ± 0.3 | -66.9 ± 5.8 | 0.9 | 15.0 | 0.9 |
| ESFa-8 | 145 | 4.8 | 30.1 | 118.8 | 19.5 | 12.0 ± 0.4 | -63.7 ± 7.8 | 0.9 | 15.7 | 0.5 |
| ESFa-10 | 196 | 12.1 | 16.2 | 108.3 | 21.3 | 11.3 ± 0.4 | -62.9 ± 7.7 | 0.9 | 15.9 | 0.6 |
| ESFa-10E | 497 | 12.8 | 38.7 | 102.8 | 20.1 | 12.3 ± 0.3 | -63.6 ± 6.8 | 0.9 | 15.7 | 0.7 |
| ESFa-2D | 64 | 2.9 | 21.8 | 179.7 | 33.1 | 10.3 ± 0.6 | -36.4 ± 7.2 | 0.7 | 27.4 | 0.8 |
| ESFa-3D | 555 | 11.4 | 48.6 | 140.0 | 23.1 | 12.3 ± 0.2 | -56.1 ± 3.7 | 1.0 | 17.8 | 0.9 |
| ESFa-8D | 209 | 13.4 | 15.6 | 229.7 | 24.9 | 10.7 ± 0.4 | -46.0 ± 3.8 | 0.9 | 21.7 | 1.4 |
| ESFa-1C | 583 | 10.4 | 56.1 | 322.3 | 28.0 | 10.9 ± 0.5 | -30.2 ± 3.4 | 0.9 | 33.1 | 4.2 |
| ESFa-2C | 400 | 11.8 | 33.8 | 140.4 | 22.0 | 12.4 ± 0.5 | -63.2 ± 6.1 | 0.9 | 15.8 | 0.2 |
| ESFa-4C | 332 | 5.2 | 64.5 | 144.0 | 18.2 | 13.4 ± 0.5 | -80.6 ± 8.0 | 0.9 | 12.4 | 2.0 |
| ESFa-5C | 490 | 12.0 | 40.8 | 159.4 | 24.4 | 11.8 ± 0.4 | -49.5 ± 5.3 | 0.9 | 20.2 | 1.6 |
| ESFa-6C | 451 | 12.9 | 35.0 | 163.5 | 15.5 | 12.4 ± 0.4 | -73.7 ± 5.7 | 0.9 | 13.6 | 0.5 |
| ESFa-7C | 223 | 9.8 | 22.8 | 158.1 | 23.3 | 11.3 ± 0.5 | -52.3 ± 6.9 | 0.9 | 19.1 | 1.1 |
| ESFa-9C | 257 | 4.9 | 52.0 | 168.5 | 26.4 | 11.9 ± 0.5 | -45.7 ± 5.9 | 0.9 | 21.9 | 3.1 |
| ESFa-10C | 524 | 16.1 | 32.6 | 159.2 | 24.9 | 11.8 ± 0.3 | -52.5 ± 4.0 | 0.9 | 19.0 | 2.2 |
| ESFa-1B | 732 | 12.8 | 57.3 | 170.1 | 20.3 | 12.9 ± 0.5 | -67.4 ± 5.9 | 0.9 | 14.8 | 2.0 |
| <i>CO₂+Ne-bearing glass</i> | | | | | | | | | | |
| ESFa-1B | 384 | 9.5 | 40.5 | 119.8 | 18.0 | 12.6 ± 0.3 | -70.1 ± 4.9 | 1.0 | 14.3 | 1.5 |
| ESFa-2B | 1111 | 13.9 | 80.2 | 191.1 | 16.6 | 13.5 ± 0.4 | -77.1 ± 5.0 | 1.0 | 13.0 | 1.2 |

| Sample | Nb | Area (mm ²) | Bubbles density (mm ⁻²) | D _{max} (μm) | D _{mean} (μm) | Ln(n) (mm ⁻⁴) | a (mm ⁻¹) | R ² | D _{mean.th.} (-1/a) (μm) | Ves (%) |
|----------|-----|-------------------------|-------------------------------------|-----------------------|------------------------|---------------------------|-----------------------|----------------|-----------------------------------|---------|
| ESFa-3B | 953 | 10.6 | 89.7 | 161.8 | 18.5 | 12.9 ± .5 | -60.3 ± 7.0 | 0.9 | 16.6 | 1.7 |
| ESFa-5B | 133 | 11.3 | 11.8 | 196.6 | 37.9 | 9.5 ± 0.6 | -32.0 ± 6.2 | 0.7 | 31.3 | 0.9 |
| ESFa-6B | 39 | 5.6 | 6.9 | 90.6 | 29.5 | 10.1 ± 1.4 | -51.7 ± 11.6 | 0.7 | 19.4 | 0.4 |
| ESFa-7B | 494 | 13.3 | 37.1 | 166.2 | 22.6 | 11.6 ± 0.4 | -47.8 ± 5.5 | 0.9 | 20.9 | 4.0 |
| ESFa-10B | 157 | 4.7 | 33.3 | 91.4 | 19.8 | 12.1 ± 0.3 | -63.4 ± 7.2 | 0.9 | 15.8 | 0.8 |
| ESFa-2A | 702 | 10.3 | 68.0 | 160.9 | 17.3 | 12.5 ± 0.5 | -56.7 ± 5.9 | 0.9 | 17.6 | 0.5 |
| ESFa-5A | 308 | 8.1 | 37.9 | 273.7 | 30.3 | 10.5 ± 0.7 | -31.1 ± 5.2 | 0.8 | 32.1 | 2.5 |
| EN-E3 | - | - | - | - | - | - | - | - | - | - |

Table S-10 CO₂ diffusion in the melt (D_{CO_2}), bubble displacement (B_d) and bubble growth (G_r) at the time at which the experiments were quenched.

| Sample | time _{exp} min | D_{CO_2} (cm) | B_d (cm) | G_r (cm) |
|--|-------------------------|-----------------|------------|------------|
| <i>CO₂-bearing glass</i> | | | | |
| ESFa-4 | 30 | 1.3E-02 | 2.5E-03 | 2.5E-05 |
| ESFa-8 | 240 | 3.6E-02 | 3.1E-02 | 2.1E-04 |
| ESFa-10 | 240 | 3.6E-02 | 3.7E-02 | 2.1E-04 |
| ESFa-10E | 1140 | 7.9E-02 | 2.9E-01 | 1.0E-03 |
| ESFa-2D | 1140 | 7.9E-02 | 7.1E-01 | 1.7E-03 |
| ESFa-3D | 130 | 2.7E-02 | 1.5E-02 | 1.3E-04 |
| ESFa-8D | 360 | 4.4E-02 | 4.8E-02 | 4.3E-04 |
| ESFa-1C | 10 | 7.4E-03 | 1.7E-03 | 1.8E-05 |
| ESFa-2C | 10 | 7.4E-03 | 1.0E-03 | 8.8E-06 |
| ESFa-4C | 60 | 1.8E-02 | 4.2E-03 | 4.1E-05 |
| ESFa-5C | 60 | 1.8E-02 | 7.7E-03 | 6.7E-05 |
| ESFa-6C | 240 | 3.6E-02 | 1.2E-02 | 1.8E-04 |
| ESFa-7C | 240 | 3.6E-02 | 2.8E-02 | 2.5E-04 |
| ESFa-9C | 10 | 7.4E-03 | 2.9E-03 | 1.2E-05 |
| ESFa-10C | 240 | 3.6E-02 | 3.2E-02 | 2.5E-04 |
| ESFa-1B | 10 | 7.4E-03 | 1.8E-03 | 8.2E-06 |
| <i>CO₂+Ne-bearing glass</i> | | | | |
| ESFa-1B | 240 | 3.7E-02 | 1.6E-02 | 1.9E-04 |
| ESFa-2B | 240 | 3.7E-02 | 1.4E-02 | 1.7E-04 |
| ESFa-3B | 240 | 3.7E-02 | 1.7E-02 | 2.2E-04 |
| ESFa-5B | 10 | 7.5E-03 | 3.1E-03 | 1.7E-05 |
| ESFa-6B | 10 | 7.5E-03 | 4.3E-03 | 1.1E-05 |
| ESFa-7B | 240 | 3.6E-02 | 2.6E-02 | 2.8E-04 |
| ESFa-10B | 240 | 3.6E-02 | 2.0E-02 | 2.1E-04 |
| ESFa-2A | 240 | 3.6E-02 | 1.5E-02 | 2.4E-04 |
| ESFa-5A | 10 | 7.4E-03 | 2.0E-03 | 1.8E-05 |
| EN-E3 | 1800 | 9.9E-02 | - | - |

Supplementary Figures

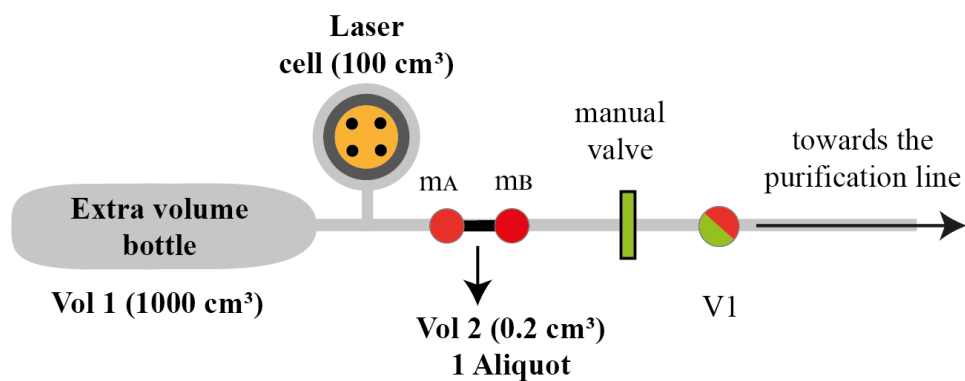


Figure S-1 Extraction and under-vacuum expansion volume attached to the purification line of the Noblesse mass spectrometer type (not at scale). The automatic valve, V1, is controlled by LabVIEW ©. The red circles show the manual valves for controlling Vol 2; the green rectangle valve isolates the expansion volume line from the purification line and V1, representing the automatic valve leading to the purification line. The extra volume bottle represents Vol 1 (1000 cm³). The laser cell holds the chunk of glass to be analysed and consists of a volume of 100 cm³ (Vol 3).

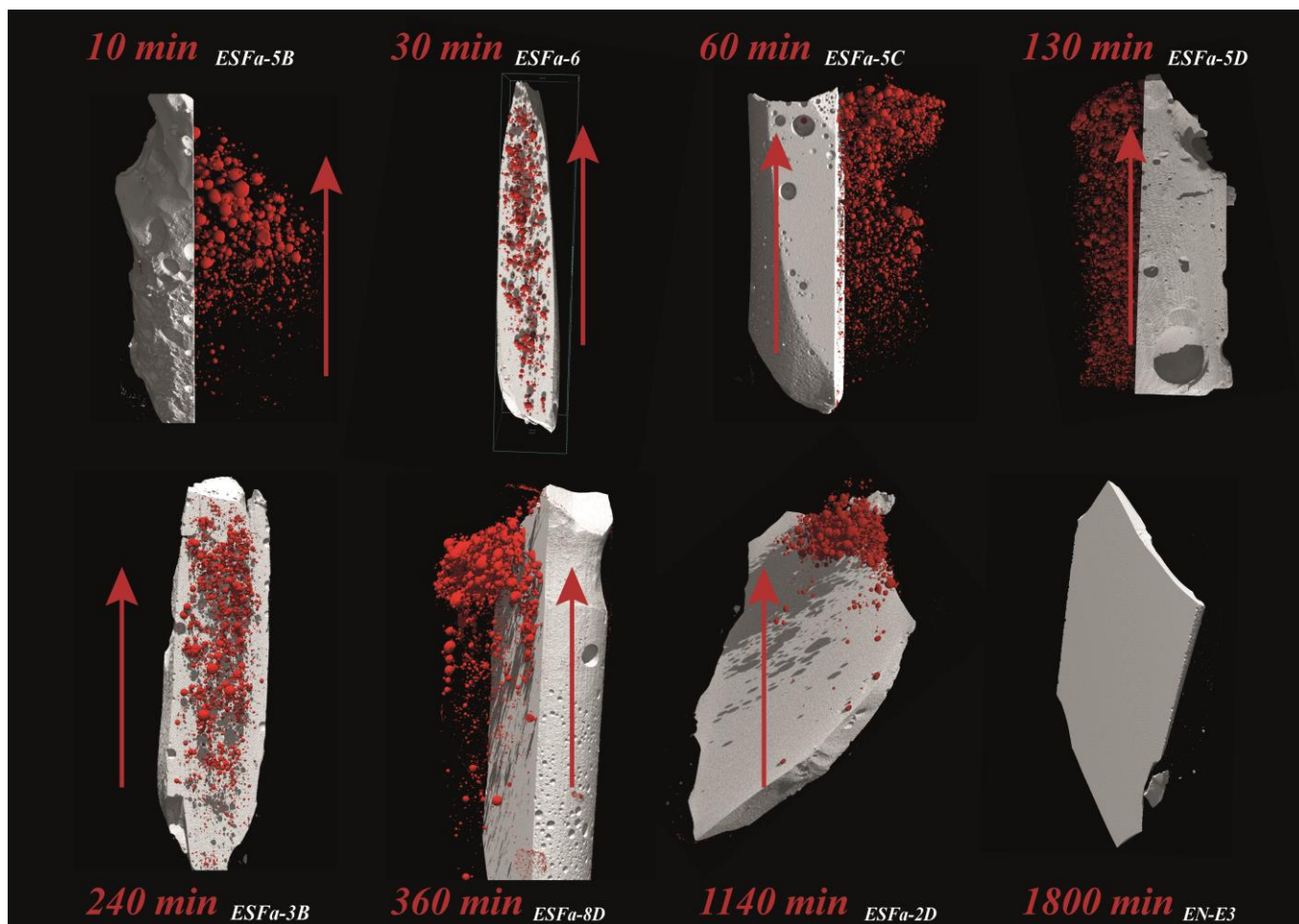


Figure S-2 3D Representative images for every experimental time. The experiment time increases from 10 to 1800 minutes, from top to bottom of the image. The accumulation of vesicles at one of the limits of the sample is evident when the experimental time increases until there are no more vesicles (at 1800 min of the experiment). The length of samples ranges from 1 to 1.2 cm. The red line marks the direction of bubble flow towards the top of the capsule.

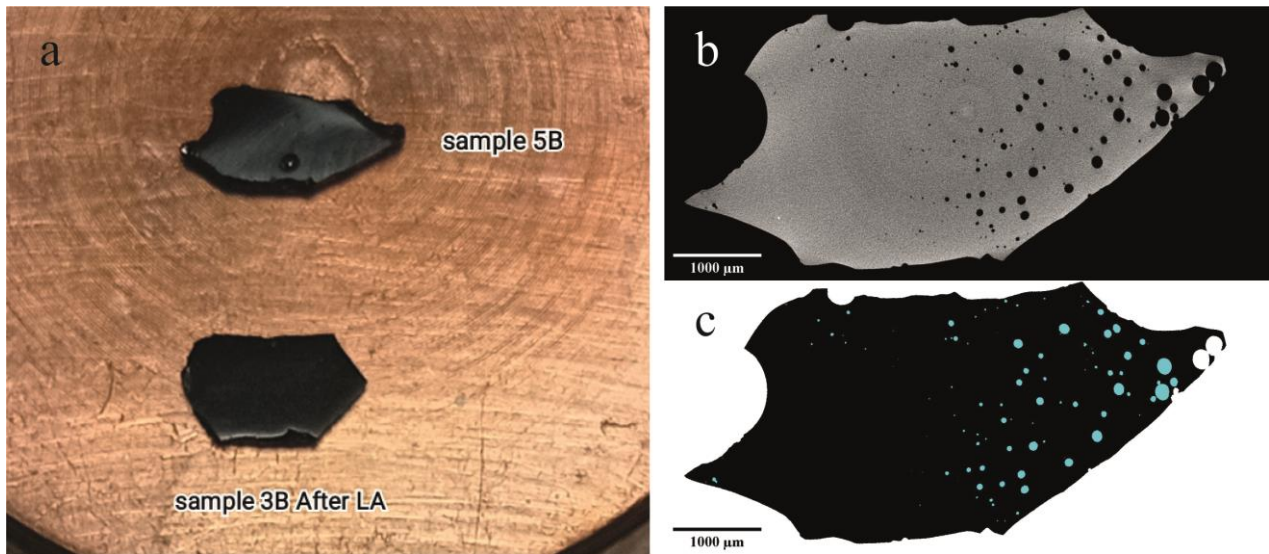


Figure S-3 (a) *ESFa-3B-240min* (approximately 5 mm in length) and *ESFa-5B-10min* samples (approximately 7 mm in length) placed inside the laser cell. The images on the right display the *ESFa-5B-10min* sample, with the image obtained through tomography (b) and further processed using *ImageJ* (c) to determine the vesicle size distribution.

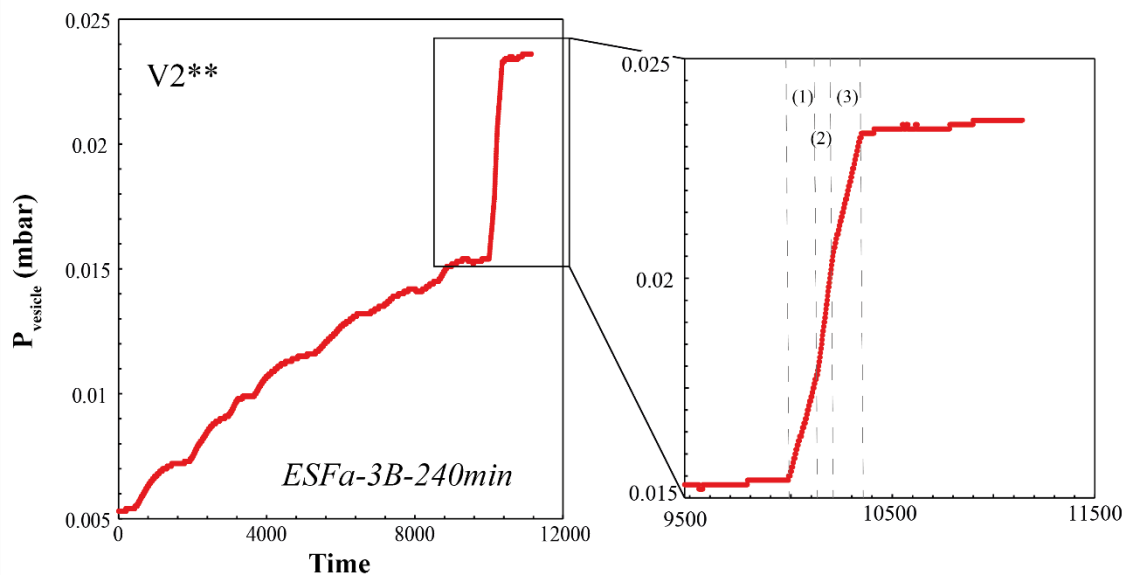


Figure S-4 Partial pressure in mbar, as recorded by the manometer, during laser ablation of three vesicles from sample *ESFa-3B-240min*. The blow-up on the right reveals three changes in the slope of the pressure curve. Dashed lines mark the representative pressure for each pierced vesicle, labelled (1), (2), and (3) in the right-hand plot. The time is not represented in specific units but is instead recorded by the software.

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