

Supporting Information

Formation pathway of norsethite dominated by solution chemistry under ambient conditions

Yi-Fan Zhang^{1, 2}, Qi-Zhi Yao³, Fei-Jin Qian^{1, 2}, Gen-Tao Zhou^{1, 2*}, Sheng-Quan Fu⁴

¹ CAS Key Laboratory of Crust-Mantle Materials and Environments, School of Earth and Space Sciences, University of Science and Technology of China, Hefei 230026, P. R. China.

² CAS Center for Excellence in Comparative Planetology, Hefei 230026, P. R. China.

³ School of Chemistry and Materials Science, University of Science and Technology of China, Hefei 230026, P. R. China.

⁴ Hefei National Laboratory for Physical Sciences at Microscale, University of Science and Technology of China, Hefei 230026, P. R. China.

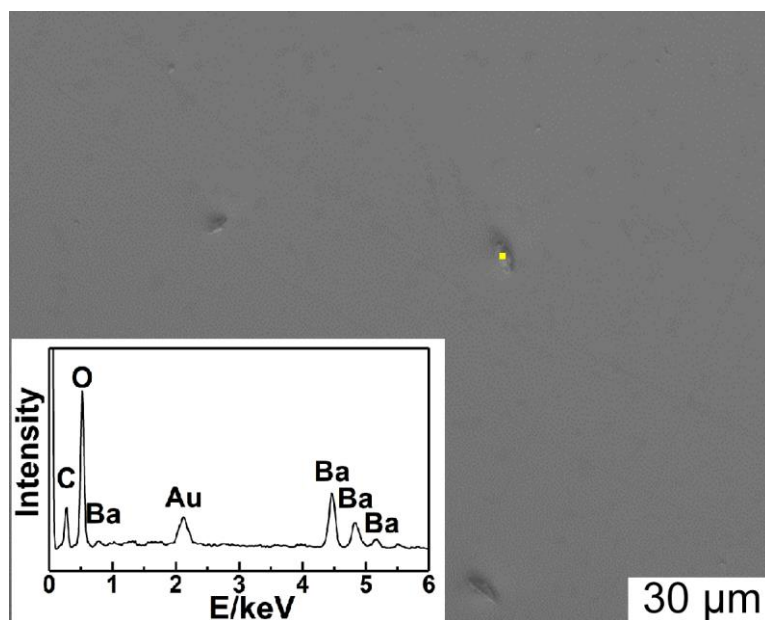


Figure S1. FESEM image and EDX analysis (inset) of the precipitate at Mg/Ba of 50/25 after mineralizing for 40 min.

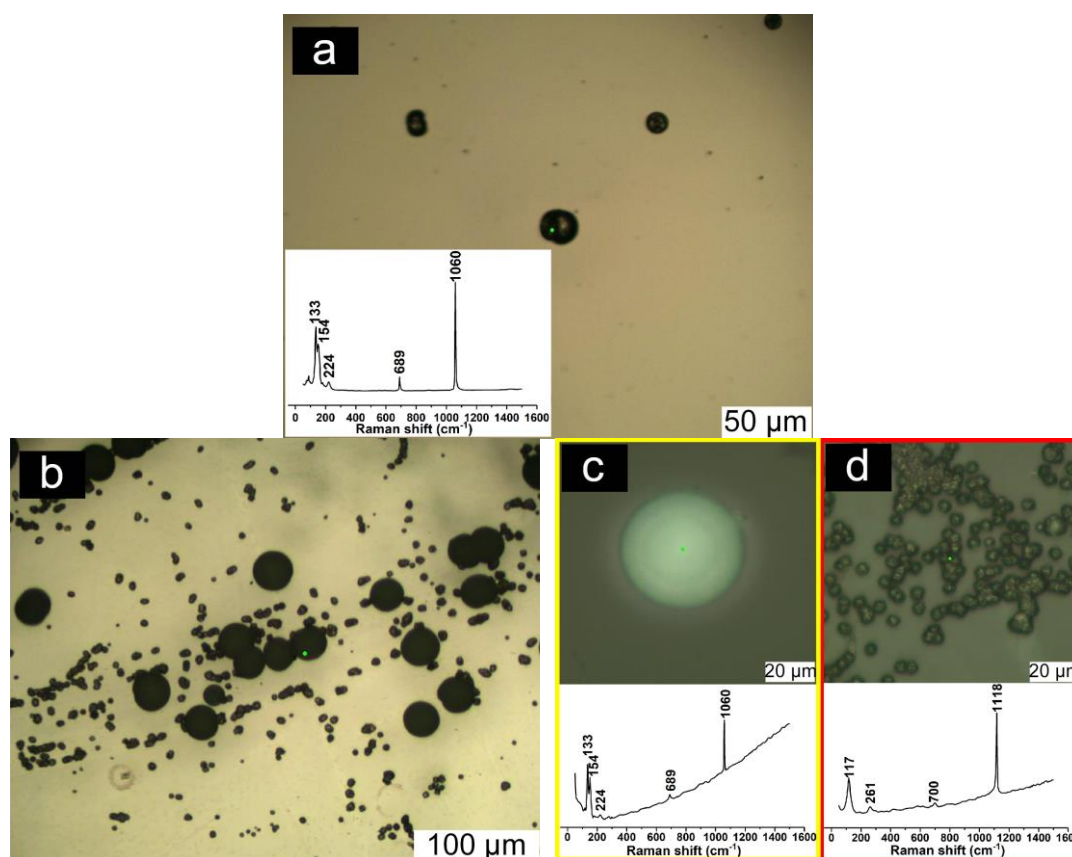


Figure S2. Micro-Raman spectra of the precipitate at Mg/Ba of 50/5 after mineralizing for 1 (a) and 2 h (b-d). As shown in Fig. S2a, few spherulites can be observed on the glass slide after 1 h of mineralization, being consistent with FESEM observations in Figure 1, panel c1. The corresponding Raman spectrum shows the vibrational bands at around 1060, 689, 224 and 133/154 cm^{-1} , which can be well assigned to the internal stretching (ν_1) and bending (ν_4) vibrations of the C-O bonds in the carbonate groups, and the external translational (T) and librational (rotatory) (L) modes of witherite, respectively (Buzgar and Apopei, 2009; Wang et al., 2009). It indicates that the mineralized products after 1 h of mineralization are witherite. After 2 h of mineralization, the products contain two different kinds of crystals, i.e. large spherulites and small rhombohedral particles (Fig. S2b). The magnified images and corresponding Raman spectra are depicted in Fig. S2c and d. The results reveal that the large spherulites are witherite (Fig. S2c). However, the vibrational bands at 1118, 701, 117 and 261 cm^{-1} on the spectrum of small rhombohedra (Fig. S2d) can be assigned to the internal stretching (ν_1) and bending (ν_4) vibrations of the C-O bonds in the carbonate groups, and the external translational (T) and librational (rotatory) (L) modes of norsethite (Böttcher et al., 1997), indicating they are norsethite.

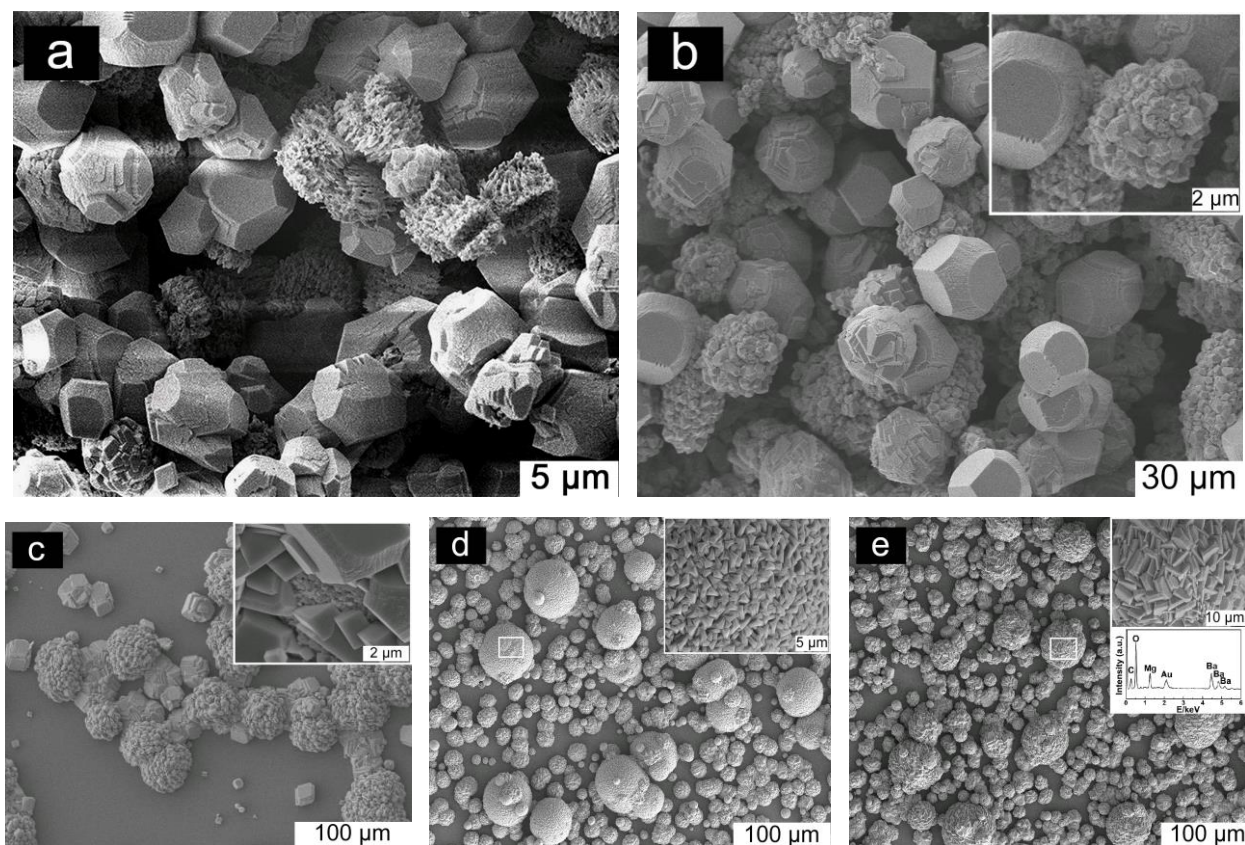


Figure S3. FESEM images of the products after mineralizing for 24 (a) and 48 (b) hours at Mg/Ba of 50/25, and the products after mineralizing for 12 (c), 24 (d) and 48 (e) hours at Mg/Ba of 50/5

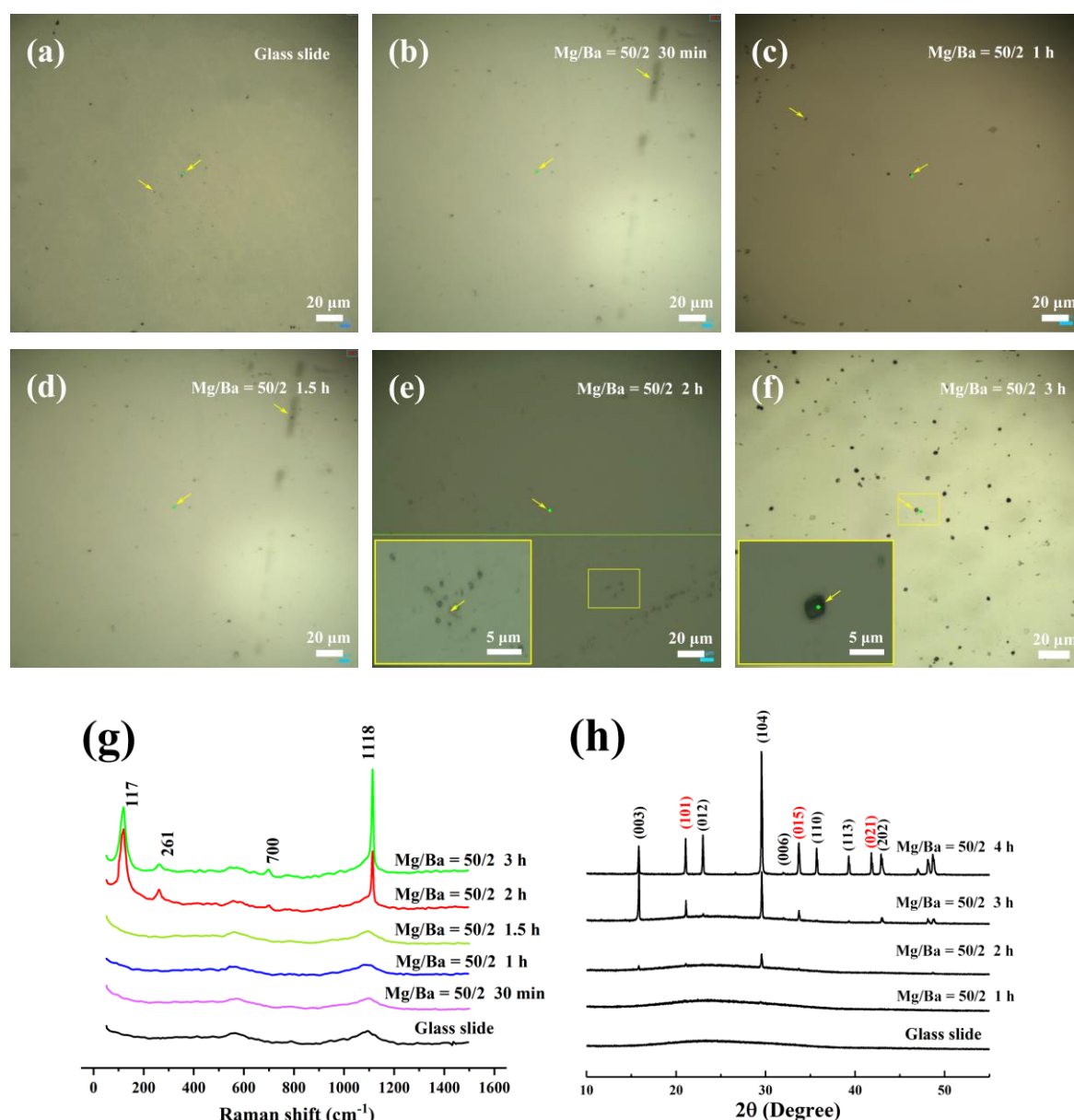


Figure S4. Micro-Raman observations of the glass slides before (a) and after 30 min (b), 1 (c), 1.5 (d), 2 (e), 3 h (f) of mineralization at initial Mg/Ba of 50/2, and corresponding representative Raman spectra (g) and XRD patterns (h). As a comparison, the powder XRD pattern of the sample collected at the mineralization for 4 h is also shown in panel h.

As shown in Figure S4g and h, the Raman spectrum of the glass slide (bottom in panel g) shows broad peaks around 1100 and 557 cm^{-1} , which are indicative of $\equiv\text{SiO}$ units and the vibrations of the bridging oxygen in Si-O-Si linkages in sodium silicate glasses, respectively (McMillan and Remmele, 1986), and the corresponding XRD spectrum (bottom in panel h) only donates a weak hump peak around $24^\circ 2\theta$, being consistent with the amorphous nature of the glass slide. The similar landscapes were also observed on the Raman and XRD spectra of the glass slide after 30 min, 1 and 1.5 h of mineralization, indicating that no precipitate was

detected at this time. However, after 2 and 3 h of mineralization, more particles scattered on the glass slides are visible, and the Raman analyses for the particles identify that they are norsethite (e.g., Fig. 7c and middle in panel g), i.e., the vibrational bands at 1118, 700, 117 and 261 cm^{-1} can be assigned to the internal stretching (ν_1) and bending (ν_4) vibrations of the C-O bonds in the carbonate groups, and the external translational (T) and librational (rotatory) (L) modes of norsethite (e.g., Böttcher et al., 1997). Further XRD analyses reveal that the glass slides contains norsethite, i.e., the (104) and (003) diffractions as well as superstructure diffractions (101) and (015) belonging to noresthite can be discerned on the XRD patterns (e.g., middle in panel h).

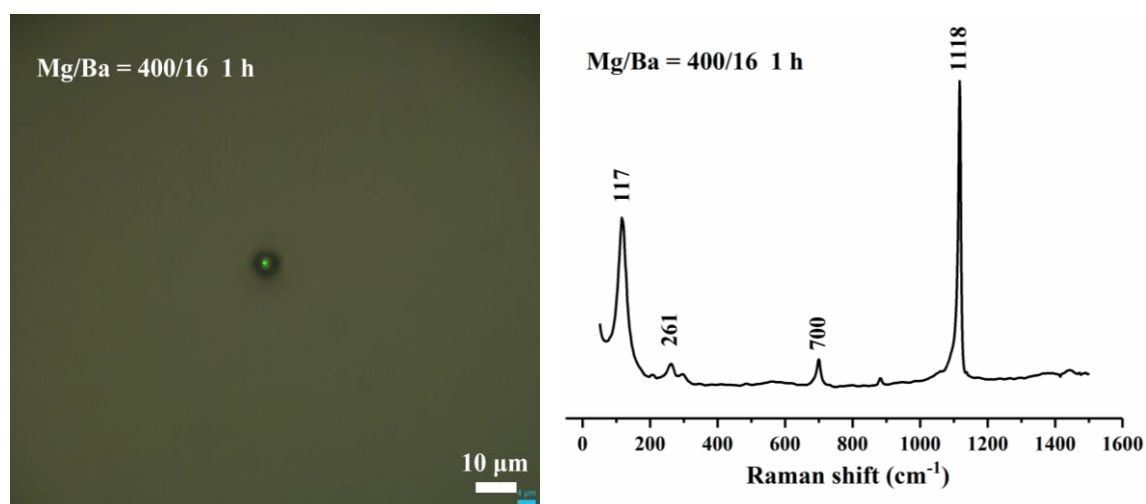


Figure S5: Typical micro-Raman spectrum of the product after 1 h of mineralization at initial Mg/Ba = 400/16.

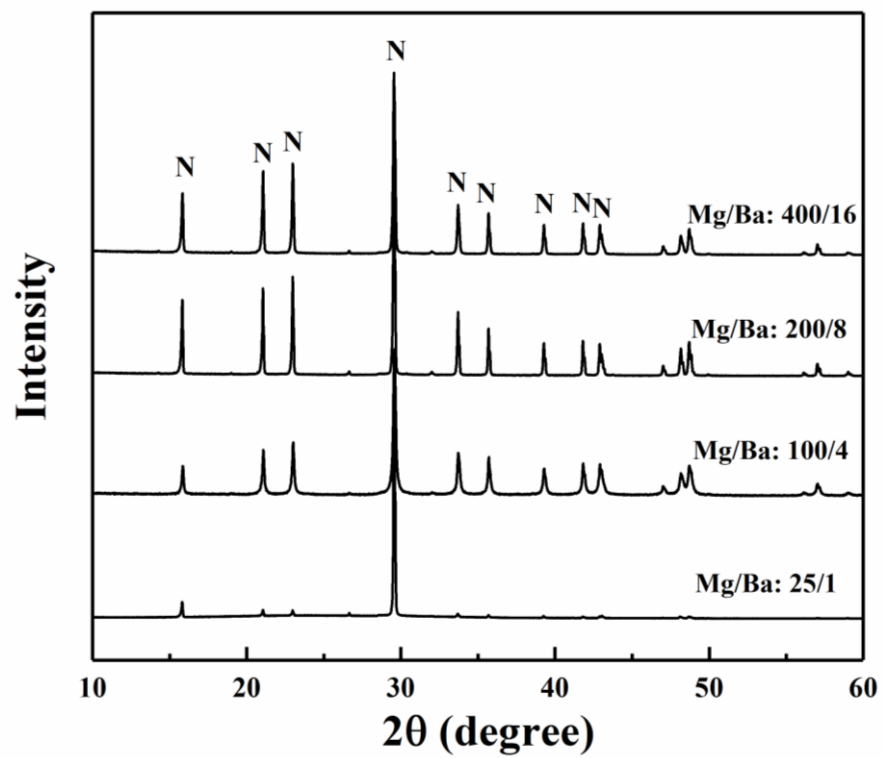


Figure S6: XRD patterns of the products in experiments with Mg/Ba of 25/1, 100/4, 200/8 and 400/16 after 4 h of mineralization. N: the characteristic diffraction of norsethite.

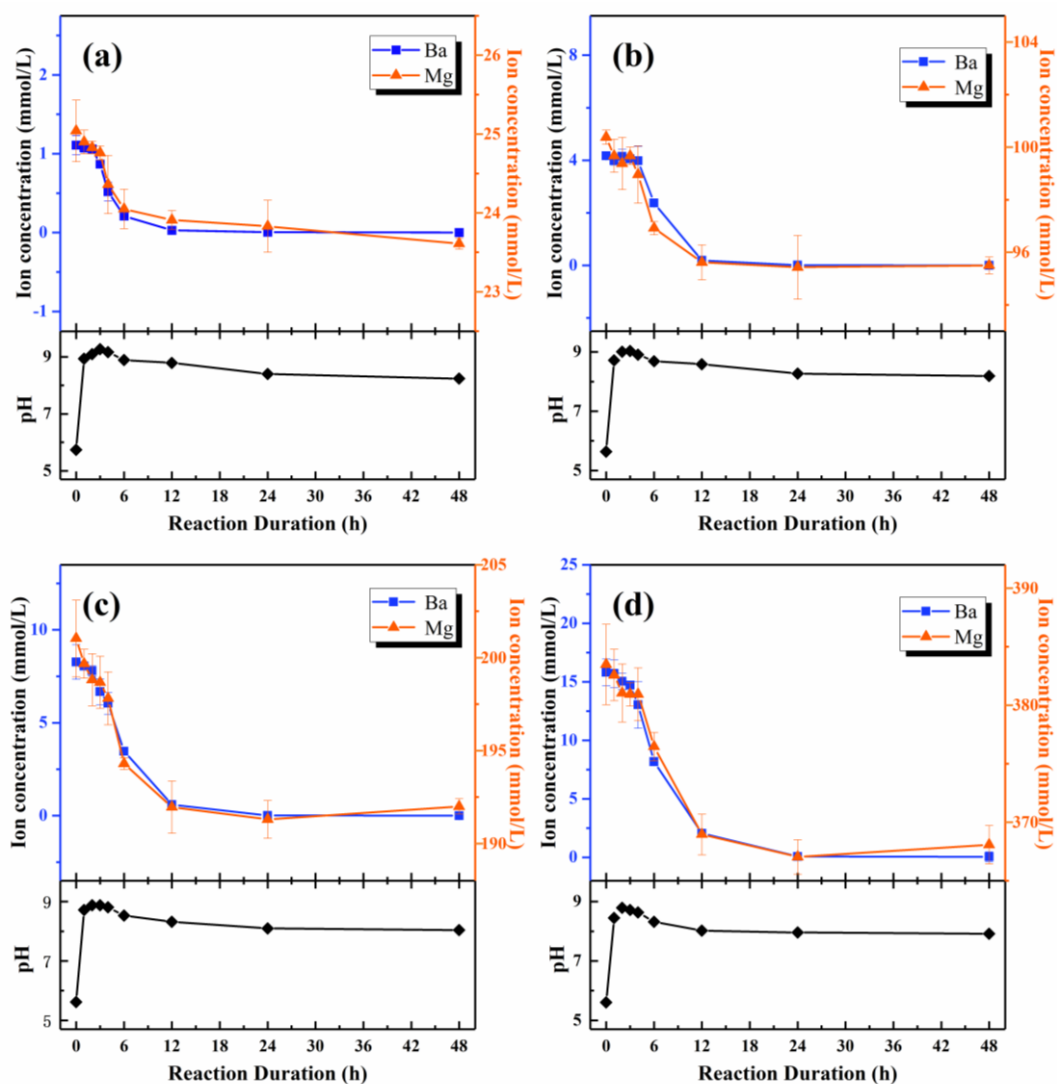


Figure S7: The time-dependent evolution of $[\text{Ba}^{2+}]$, $[\text{Mg}^{2+}]$ and pH in solution in the experiment with initial Mg/Ba of 25/1, 100/4, 200/8 and 400/16.

Table S1. Solution-chemical parameters (pH, concentrations of Ba^{2+} , Mg^{2+} , CO_3^{2-} and HCO_3^- , and resulting Mg/Ba ratios) and supersaturations Ω for witherite and norsethite after given mineralization durations. The weight percent of norsethite in solid products calculated by RIR method were also given.

| Exp. | Mineralization duration (hour) | $[\text{Mg}^{2+}]^*$ (mM) | $[\text{Ba}^{2+}]^*$ (mM) | Mg/Ba | pH | $[\text{CO}_3^{2-}]^a$ (mM) | $[\text{HCO}_3^-]^a$ (mM) | $[\text{CO}_3^{2-}]^b$ (mM) | $[\text{HCO}_3^-]^b$ (mM) | Ω^c Witherite | Ω^c Norsethite | Weight% of norsethite in products |
|-------|--------------------------------|---------------------------|---------------------------|---------|------|-----------------------------|---------------------------|-----------------------------|---------------------------|----------------------|-----------------------|-----------------------------------|
| 50/25 | 0 | 50.53 \pm 0.43 | 26.30 \pm 0.86 | 1.92 | 6.30 | - | - | - | - | - | - | - |
| 50/25 | 0.5 | 50.69 \pm 2.16 | 25.37 \pm 1.28 | 2.00 | 8.99 | - | - | - | - | - | - | 0 |
| 50/25 | 1 | 50.09 \pm 1.30 | 24.49 \pm 1.01 | 2.05 | 8.76 | 0.88 \pm 0.02 | 0.20 \pm 0.58 | - | - | 1.36 | 2.61 | 0 |
| 50/25 | 2 | 49.66 \pm 2.66 | 20.07 \pm 0.06 | 2.47 | 8.83 | 2.87 \pm 0.79 | 3.64 \pm 0.61 | 0.12 | 9.03 | 2.78 | 4.73 | 0 |
| 50/25 | 6 | 49.09 \pm 2.09 | 8.21 \pm 0.59 | 5.98 | 7.87 | 8.85 \pm 1.65 | 14.55 \pm 3.45 | 0.23 | 9.02 | 2.45 | 3.66 | 0 |
| 50/25 | 12 | 45.23 \pm 1.59 | 2.17 \pm 0.88 | 20.84 | 8.48 | 12.85 \pm 0.65 | 29.40 \pm 1.10 | 0.45 | 26.30 | 1.84 | 4.52 | 11.5 \pm 0.4 |
| 50/25 | 24 | 33.39 \pm 0.19 | 0.02 \pm 1.07E-4 | 1669.50 | 8.50 | 22.55 \pm 0.45 | 50.55 \pm 2.55 | 1.71 | 70.03 | 0.18 | 3.12 | 49.0 \pm 0.8 |
| 50/25 | 48 | 32.01 \pm 0.01 | 8.25E-3 \pm 2.55E-4 | 3880.00 | 8.45 | 77.90 \pm 0.30 | 172.30 \pm 0.10 | 1.01 | 37.54 | -0.12 | 2.87 | 63.7 \pm 0.8 |

* Cationic concentrations measured by ICP-AES.

^a Concentrations of CO_3^{2-} and HCO_3^- determined by titration method in the mineralizing solutions with different initial Mg/Ba ratios after given mineralization durations.

^b Concentrations of CO_3^{2-} and HCO_3^- calculated from the measured pH and TIC values in the mineralizing solutions with different initial Mg/Ba ratios after given mineralization durations.

^c Supersaturations calculated based on $[\text{CO}_3^{2-}]^a$.

| Exp. | Mineralization duration (hour) | [Mg ²⁺]* (mM) | [Ba ²⁺]* (mM) | Mg/Ba | pH | [CO ₃ ²⁻] ^a (mM) | [HCO ₃ ⁻] ^a (mM) | [CO ₃ ²⁻] ^b (mM) | [HCO ₃ ⁻] ^b (mM) | Ω ^c Witherite | Ω ^c Norsethite | Weight% of norsethite in products |
|-------|--------------------------------------|------------------------------|------------------------------|----------|------|---|---|---|---|-----------------------------|------------------------------|---|
| 50/10 | 0 | 50.08 ± 0.12 | 10.30 ± 0.24 | 4.86 | 5.50 | - | - | - | - | - | - | - |
| 50/10 | 0.5 | 49.95 ± 0.32 | 10.29 ± 0.64 | 4.85 | 8.97 | - | - | - | - | - | - | - |
| 50/10 | 1 | 49.85 ± 0.78 | 10.19 ± 0.91 | 4.89 | 8.84 | 4.44 ± 0.39 | - | - | - | 1.91 | 4.06 | 0 |
| 50/10 | 2 | 49.51 ± 0.81 | 5.64 ± 0.17 | 8.78 | 8.60 | 4.78 ± 0.03 | 14.64 ± 1.09 | 1.03 | 29.72 | 2.11 | 4.70 | 0 |
| 50/10 | 6 | 47.59 ± 0.24 | 1.30 ± 0.08 | 36.61 | 8.78 | 18.15 ± 6.05 | 13.55 ± 4.95 | 0.78 | 25.35 | 1.84 | 4.76 | 5.1 ± 0.1 |
| 50/10 | 12 | 46.39 ± 0.30 | 0.09 ± 0.03 | 515.44 | 8.78 | 32.40 ± 0.40 | 46.20 ± 0.70 | 1.13 | 47.14 | 0.98 | 4.17 | 44.4 ± 0.5 |
| 50/10 | 24 | 44.79 ± 0.47 | 2.10E-3 ± 5.20E-4 | 21328.57 | 8.70 | 37.55 ± 0.53 | 61.15 ± 0.65 | 4.67 | 144.98 | -0.61 | 2.61 | 50.9 ± 0.6 |
| 50/10 | 48 | 43.61 ± 1.97 | 8.15E-4 ± 3.60E-4 | 53509.20 | 8.53 | 70.10 ± 0.10 | 213.40 ± 1.20 | 2.37 | 94.74 | -0.91 | 2.41 | 67.0 ± 0.1 |
| 50/5 | 0 | 50.07 ± 0.33 | 5.14 ± 0.12 | 9.74 | 5.60 | - | - | - | - | - | - | - |
| 50/5 | 0.5 | 49.98 ± 0.18 | 5.10 ± 0.20 | 9.80 | 8.88 | - | - | - | - | - | - | - |
| 50/5 | 1 | 49.56 ± 0.21 | 4.92 ± 0.15 | 10.07 | 8.62 | 4.55 ± 0.39 | - | - | - | 1.45 | 3.46 | 0 |
| 50/5 | 2 | 49.10 ± 0.10 | 1.88 ± 0.56 | 26.12 | 8.60 | 8.02 ± 3.53 | 3.51 ± 1.66 | 0.70 | 18.98 | 1.43 | 3.82 | 16.5 ± 1.1 |
| 50/5 | 6 | 48.10 ± 0.62 | 0.46 ± 8.67E-4 | 104.57 | 8.71 | 20.35 ± 1.35 | 12.05 ± 1.45 | 0.80 | 46.68 | 1.34 | 4.23 | 72.9 ± 0.6 |
| 50/5 | 12 | 46.56 ± 0.60 | 0.05 ± 0.02 | 931.20 | 8.40 | 24.40 ± 1.90 | 54.45 ± 2.55 | 2.23 | 33.32 | 0.48 | 3.45 | 87.3 ± 0.5 |
| 50/5 | 24 | 45.95 ± 0.13 | 3.66E-3 ± 1.36E-3 | 12550.14 | 8.45 | 42.55 ± 1.50 | 64.30 ± 1.30 | 4.22 | 130.97 | -0.55 | 2.52 | 92.8 ± 0.6 |
| 50/5 | 48 | 45.50 ± 0.34 | 1.59E-3 ± 8.67E-5 | 28616.35 | 8.43 | 76.50 ± 0.30 | 226.90 ± 1.70 | 1.33 | 47.23 | -0.68 | 2.59 | 95.0 ± 0.6 |

| Exp. | Mineralization duration (hour) | [Mg ²⁺]* (mM) | [Ba ²⁺]* (mM) | Mg/Ba | pH | [CO ₃ ²⁻] ^a (mM) | [HCO ₃ ⁻] ^a (mM) | [CO ₃ ²⁻] ^b (mM) | [HCO ₃ ⁻] ^b (mM) | Ω ^c Witherite | Ω ^c Norsethite | Weight% of norsethite in products |
|------|--------------------------------------|------------------------------|------------------------------|---------|------|---|---|---|---|-----------------------------|------------------------------|---|
| 50/2 | 0 | 50.73 ± 0.23 | 2.25 ± 0.11 | 22.55 | 5.54 | - | - | - | - | - | - | - |
| 50/2 | 1 | 50.85 ± 0.30 | 2.24 ± 0.02 | 22.70 | 8.69 | 4.67 ± 0.70 | - | - | - | 1.18 | 3.28 | - |
| 50/2 | 2 | 50.83 ± 0.31 | 2.23 ± 0.38 | 22.80 | 9.32 | 4.93 ± 0.39 | 13.29 ± 0.45 | 0.62 | 3.49 | 2.18 | 5.22 | - |
| 50/2 | 3 | 50.63 ± 0.43 | 2.15 ± 0.21 | 23.55 | 9.22 | 7.75 ± 0.25 | 13.80 ± 0.30 | 0.82 | 4.65 | 2.18 | 5.25 | - |
| 50/2 | 4 | 50.29 ± 0.30 | 1.91 ± 0.10 | 26.33 | 8.58 | 16.60 ± 0.40 | 13.70 ± 1.10 | 0.80 | 37.67 | 1.82 | 4.59 | 100.0 |
| 50/2 | 6 | 49.44 ± 0.34 | 1.12 ± 0.04 | 44.14 | 8.43 | 24.35 ± 0.75 | 12.62 ± 0.84 | 1.23 | 81.34 | 1.54 | 4.26 | 100.0 |
| 50/2 | 12 | 48.88 ± 0.34 | 0.36 ± 0.04 | 135.78 | 8.63 | 38.40 ± 1.20 | 55.35 ± 1.65 | 2.32 | 97.29 | 1.55 | 4.74 | 100.0 |
| 50/2 | 24 | 48.62 ± 0.15 | 0.09 ± 0.01 | 540.22 | 8.74 | 55.55 ± 0.45 | 67.60 ± 0.70 | 4.73 | 157.15 | 1.11 | 4.45 | 100.0 |
| 50/2 | 48 | 48.42 ± 0.23 | 0.01 ± 1.10E-3 | 4842.00 | 8.43 | 82.40 ± 2.40 | 232.40 ± 4.60 | 1.73 | 115.04 | 0.12 | 3.43 | 100.0 |
| 25/1 | 0 | 25.04 ± 0.39 | 1.11 ± 0.12 | 22.56 | 5.74 | - | - | - | - | - | - | - |
| 25/1 | 1 | 24.90 ± 0.15 | 1.08 ± 0.06 | 23.06 | 8.94 | 5.41 ± 0.11 | - | - | - | 1.27 | 3.46 | - |
| 25/1 | 2 | 24.83 ± 0.08 | 1.06 ± 0.04 | 23.42 | 9.10 | 5.23 ± 0.35 | 10.24 ± 0.81 | - | - | 1.82 | 4.53 | - |
| 25/1 | 3 | 24.76 ± 0.09 | 0.87 ± 0.02 | 28.46 | 9.27 | 6.05 ± 0.19 | 11.09 ± 0.71 | - | - | 1.89 | 4.74 | - |
| 25/1 | 4 | 24.36 ± 0.37 | 0.52 ± 0.12 | 46.85 | 9.17 | 9.06 ± 0.27 | 11.96 ± 0.02 | - | - | 1.68 | 4.55 | 100.0 |
| 25/1 | 6 | 24.05 ± 0.25 | 0.21 ± 0.01 | 114.52 | 8.89 | 29.8 ± 0.60 | 16.00 ± 0.30 | - | - | 1.37 | 4.30 | 100.0 |
| 25/1 | 12 | 23.91 ± 0.12 | 0.03 ± 1.87E-3 | 797.00 | 8.79 | 40.60 ± 1.40 | 58.40 ± 0.30 | - | - | 0.67 | 3.73 | 100.0 |
| 25/1 | 24 | 23.83 ± 0.33 | 5.29E-3 ± 3.13E-4 | 4504.73 | 8.40 | 56.10 ± 3.10 | 68.15 ± 2.35 | - | - | -0.35 | 2.48 | 100.0 |

| Exp. | Mineralization duration (hour) | [Mg ²⁺]* (mM) | [Ba ²⁺]* (mM) | Mg/Ba | pH | [CO ₃ ²⁻] ^a (mM) | [HCO ₃ ⁻] ^a (mM) | [CO ₃ ²⁻] ^b (mM) | [HCO ₃ ⁻] ^b (mM) | Ω ^c Witherite | Ω ^c Norsethite | Weight% of norsethite in products |
|-------|--------------------------------------|------------------------------|------------------------------|----------|------|---|---|---|---|-----------------------------|------------------------------|---|
| 25/1 | 48 | 23.61 ± 0.07 | 1.64E-3 ± 2.08E-4 | 14396.34 | 8.24 | 81.20 ± 2.80 | 208.60 ± 1.20 | - | - | -0.82 | 2.03 | 100.0 |
| 100/4 | 0 | 100.38 ± 0.27 | 4.17 ± 0.04 | 24.07 | 5.64 | - | - | - | - | - | - | - |
| 100/4 | 1 | 99.67 ± 0.63 | 3.99 ± 0.17 | 24.98 | 8.72 | 4.46 ± 0.12 | - | - | - | 1.27 | 3.44 | - |
| 100/4 | 2 | 99.38 ± 0.99 | 4.15 ± 0.27 | 23.95 | 9.01 | 5.38 ± 0.21 | 9.94 ± 1.05 | - | - | 2.00 | 4.88 | - |
| 100/4 | 3 | 99.67 ± 0.33 | 4.05 ± 0.08 | 24.61 | 9.03 | 6.66 ± 0.30 | 10.68 ± 1.04 | - | - | 2.06 | 5.00 | - |
| 100/4 | 4 | 98.96 ± 1.09 | 3.98 ± 0.55 | 24.86 | 8.91 | 10.96 ± 0.51 | 15.11 ± 0.73 | - | - | 2.16 | 5.20 | 100.0 |
| 100/4 | 6 | 96.92 ± 0.25 | 2.37 ± 0.005 | 40.89 | 8.69 | 24.45 ± 0.75 | 23.45 ± 1.35 | - | - | 2.04 | 5.17 | 100.0 |
| 100/4 | 12 | 95.62 ± 0.66 | 0.19 ± 0.01 | 503.26 | 8.59 | 38.10 ± 1.10 | 64.05 ± 0.45 | - | - | 1.16 | 4.49 | 100.0 |
| 100/4 | 24 | 95.43 ± 1.21 | 8.76E-3 ± 8.33E-4 | 10893.84 | 8.27 | 50.00 ± 0.80 | 80.35 ± 5.45 | - | - | -0.37 | 2.79 | 100.0 |
| 100/4 | 48 | 95.50 ± 0.32 | 1.46E-3 ± 1.25E-3 | 65410.96 | 8.19 | 76.40 ± 1.80 | 212.02 ± 4.38 | - | - | -0.98 | 2.34 | 100.0 |
| 200/8 | 0 | 201.04 ± 2.06 | 8.27 ± 0.93 | 24.31 | 5.62 | - | - | - | - | - | - | - |
| 200/8 | 1 | 199.68 ± 0.78 | 8.04 ± 0.21 | 24.83 | 8.72 | 4.32 ± 0.20 | - | - | - | 1.40 | 3.65 | - |
| 200/8 | 2 | 198.81 ± 1.40 | 7.79 ± 0.27 | 25.51 | 8.88 | 4.94 ± 0.12 | 9.94 ± 1.02 | - | - | 2.01 | 4.89 | - |
| 200/8 | 3 | 198.68 ± 1.40 | 6.67 ± 0.70 | 29.77 | 8.88 | 6.46 ± 0.30 | 12.60 ± 0.84 | - | - | 2.06 | 5.04 | - |
| 200/8 | 4 | 197.81 ± 1.42 | 6.04 ± 0.58 | 32.73 | 8.81 | 9.32 ± 0.56 | 17.24 ± 0.75 | - | - | 2.12 | 5.21 | 100.0 |
| 200/8 | 6 | 194.30 ± 0.31 | 3.46 ± 6.25E-3 | 56.09 | 8.53 | 20.40 ± 0.80 | 14.20 ± 0.50 | - | - | 1.82 | 4.84 | 100.0 |
| 200/8 | 12 | 191.96 ± 1.41 | 0.59 ± 0.04 | 322.68 | 8.32 | 36.05 ± 1.25 | 48.25 ± 1.75 | - | - | 1.26 | 4.48 | 100.0 |

| Exp. | Mineralization duration (hour) | [Mg ²⁺]* (mM) | [Ba ²⁺]* (mM) | Mg/Ba | pH | [CO ₃ ²⁻] ^a (mM) | [HCO ₃ ⁻] ^a (mM) | [CO ₃ ²⁻] ^b (mM) | [HCO ₃ ⁻] ^b (mM) | Ω ^c Witherite | Ω ^c Norsethite | Weight% of norsethite in products |
|--------|--------------------------------------|------------------------------|------------------------------|-----------|------|---|---|---|---|-----------------------------|------------------------------|---|
| 200/8 | 24 | 191.30 ± 1.01 | 0.01 ± 1.04E-3 | 17472.26 | 8.10 | 42.55 ± 0.53 | 61.15 ± 0.65 | - | - | -0.62 | 2.50 | 100.0 |
| 200/8 | 48 | 192.00 ± 0.41 | 1.82E-3 ± 1.56E-3 | 105216.00 | 8.04 | 74.50 ± 0.50 | 235.50 ± 1.40 | - | - | -1.04 | 2.41 | 100.0 |
| 400/16 | 0 | 383.48 ± 3.44 | 15.80 ± 1.15 | 24.27 | 5.61 | - | - | - | - | - | - | - |
| 400/16 | 1 | 382.58 ± 2.19 | 15.70 ± 1.17 | 24.37 | 8.45 | 2.38 ± 0.14 | - | - | - | 1.15 | 3.07 | - |
| 400/16 | 2 | 381.04 ± 2.48 | 15.03 ± 0.72 | 25.35 | 8.79 | 3.76 ± 0.53 | 7.42 ± 0.28 | - | - | 1.99 | 4.76 | - |
| 400/16 | 3 | 380.94 ± 1.00 | 14.71 ± 0.33 | 25.90 | 8.72 | 4.92 ± 0.30 | 13.18 ± 0.22 | - | - | 2.16 | 5.11 | - |
| 400/16 | 4 | 380.94 ± 2.26 | 13.03 ± 2.00 | 29.24 | 8.64 | 7.63 ± 0.31 | 17.15 ± 0.63 | - | - | 2.20 | 5.25 | 100.0 |
| 400/16 | 6 | 376.46 ± 1.21 | 8.17 ± 0.11 | 46.08 | 8.32 | 9.55 ± 0.45 | 15.75 ± 1.05 | - | - | 1.81 | 4.67 | 100.0 |
| 400/16 | 12 | 368.96 ± 1.74 | 2.05 ± 0.23 | 179.98 | 8.02 | 17.80 ± 0.40 | 34.40 ± 1.00 | - | - | 1.29 | 4.22 | 100.0 |
| 400/16 | 24 | 367.04 ± 1.47 | 0.07 ± 4.00E-3 | 5243.43 | 7.96 | 26.60 ± 1.20 | 56.70 ± 1.90 | - | - | -0.03 | 3.04 | 100.0 |
| 400/16 | 48 | 368.08 ± 1.64 | 0.05 ± 0.41 | 7361.60 | 7.92 | 72.80 ± 0.80 | 196.40 ± 3.80 | - | - | 0.23 | 3.72 | 100.0 |

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