OR-CKP-10-1-S and OR-CKP-11-1-S

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Summary

This report summarizes the properties of particles collected on mounts OR–CKP–10–1–S,0 and OR-CKP–11–1–S,0.

Scanning electron microscopy (SEM) examination of mount OR–CKP–10–1–S,0 identified ~ 111 particles/particle groups ranging in size from ~0.5 – 65 μm in the longest direction. Five particle types were observed: C-bearing, Si-rich, metallic, salts and others (oxides). The most common particle type was C-bearing, defined as C present as a major[1] and/or minor element as detected by energy dispersive X-ray spectroscopy (EDX). C abundances ranged from ~2.1 – 24.2 wt. %. Composition of one carbonaceous particle was consistent with that of biogenic matter. Minor Ti was detected in 6 C-bearing particles. One particle, 3·4 μm^2 in size, contained minor Pb, with a composition similar to that of a brass screw previously analyzed by our group in February, 2016. Neither the maximum permissible C mass limit (1000 $ng \cdot cm^{-2}$) nor that for Pb (0.79 $ng \cdot cm^{-2}$) was approached. The second most common particle type was Si-rich. Other elements present in siliceous particles included O, Na, Mg, Al, S, Cl, K, and Fe. Four siliceous particles contained minor Zn, with detectable F in one particle. Metallic particles included those rich in Fe/Cr and Al. Salts included halite (NaCl), calcite and Fe-bearing sulfates.

SEM examination of the mount OR–CKP–11–1–S,0 identified ~314 particles/particle groups ranging in size from ~1–110 μm in the longest dimension. Two fibers, ~25·300 μm^2 and ~13·128 μm^2, were located near the center of the mount. Due to the high particle count, the mount was divided in two sub-equal sections designated as upper and lower. On the upper part of the mount, particles ≥ 0.05 μm were characterized; on the lower part, particles ≥ 5 μm were characterized. Five particle types were observed: Si-rich, C-bearing, metallic, salts and others (oxides). The most common particle type was Si-rich. The second most common particle type was carbonaceous with C abundances ranging from ~5–23 wt.%. Two Pb-containing particles, 8·3 μm^2 and 10·14 μm^2, were observed; the smaller particle contained minor O while the larger one contained major amounts of O, Fe and Zn, a composition that does not correspond to a single mineral, alloy or oxide phase. The maximum permissible C mass limit (1000 $ng \cdot cm^{-2}$) was not approached (335.1 $ng \cdot cm^{-2}$; see Calculations). Neither the maximum permissible limit for K (170

[1] For purposes herein, we define the major element range as ≥10%; minor as 1<10 %; and trace as <1%.

ng·cm^{-2}; see Calculations) nor Pb was approached. Sixteen particles with distinctive elements (Ba, Zn and Ti) were present. Metallic particles included those rich in Fe/Cr and Al. Salts included halite (NaCl), carbonates and sulfates.

Recommendation: No action needed. It was determined by MPS that during the time of the exposure of OR–CKP–10–1–S,0 and OR–CKP–11–1–S,0, the spacecraft was exposed only 1 day to the SSL, which these witness plates were located. The spacecraft was in the thermal vac chamber, if MSS understands correctly.

Procedures

The mounts were introduced into a SEM for particle characterization. Image mosaics were obtained for each Si wafer (OR–CKP–10–1–S,0 and OR–CKP–11–1–S,0) taken at 150X with each frame acquired for 30 s using low-angle backscatter electron (LABE) imaging to emphasize atomic weight variations. On OR–CKP–10–1–S ,0 all particles ≥ 0.05 μm in size were examined. Due to the high particle count on OR–CKP–11–1–S ,0, it was divided into two subequal sections, upper and lower. In the upper region, particles ≥ 0.05 μm in size were examined while on the lower region those particles ≥ 5 μm in size were examined. All EDX spectra were acquired for 60 s at 15 keV.

To reduce EDX spectra, we used the Quantitative Fit Method 'Filter without Standards' and the Correction Method 'Phi-Rho-Z'. The accuracy of standardless EDX analysis is unknown for the JSC spectrometer. As a test, one particle was analyzed which was composed only of Pb and O (spt CKP 11(13)) and so was thought to be a good match for Pb-oxide. Qualitative results showed this spectrum was indistinguishable from that of PbO as reported on a mineral database (see http://webmineral.com/; see Discussion). *Note: this suggests our standardless data reduction method may have a high degree of accuracy; however, this is speculative until the limits of the accuracy of our parameters are evaluated*.

Observations

The majority of particles on OR–CKP–10–1–S,0 contain C as a major element. The particles are distributed relatively uniformly over the entire surface although the upper left and right regions have a slightly higher particle density compared with the center region of the mount. The SEM image mosaic, shown in Figure 1, shows particle locations, composition type, and EDX spectra names (designated as 'spt 10(1–20)'[2]). The most common type is C-bearing as shown by a representative spectrum in Figure 2A. One particle contains major C with N, O, Na, S, Cl, and K and is consistent with biogenic matter (*i.e.*, skin residue; see Fig. 2B). Trace/minor Ti (≤1 wt.%) was present in 6 C-bearing particles. Siliceous particles typically contain O, Na, Mg, Al, S, Cl, K, and/or Fe. In a few of these particles, Zn is present as a minor element. One unusual C-bearing particle contains Zn and F (Fig. 2C). One particle, 3·4 μm^2 in size, contained Pb. The EDX spectrum of this particle also showed the presence of Cu, Zn, C, O, and Ag (Figure 3A).

[2]The location for Spt 10(2), which shows composition of the substrate/mount, was not designated on Figure 1.

The composition of this Pb-bearing particle is similar to that of a brass screw analyzed by us in February, 2016 (Figure 3B). Permissible abundances for all key elements are within acceptable ranges.

The particle population on OR–CKP–11–1–S,0 was comprised of ~314 particles/particle groups ranging in size from ~1–110 μm in the longest dimension. Two fibers, ~25·300 μm^2 and ~13·128 μm^2, were located near the center of the mount. The SEM image mosaic, shown in Figure 4, shows particle locations, composition type, and EDX spectra names (designated as 'spt 11(1–39)'[3]). Due to the high particle count, the mount was divided in two sections, designated as upper and lower, as shown by the dashed line on the map in Figure 4. On the upper part of the mount, particles ≥ 0.05 μm were characterized; on the lower part, particles ≥ 5 μm were characterized. The most common particle type, numbering over 200, was Si-rich. Other elements present in siliceous particles include O, Na, Mg, Al, S, Cl, K, and/or Fe (Figures 5A, B). Twenty-two siliceous particles contained major K (~ 10 wt.%) (Figure 5C). Eleven of the siliceous particles also contained minor Zn, three contained Ba, and two contained minor Ti. Particles with major/minor amounts of C comprised the second most common particle type with abundances ranging from ~ 5–23 wt.% (Figure 5D). Two Pb-containing particles, 8·3 μm^2 and 10·14 μm^2, were observed (Figures 6A, B, resp.). The smaller particle contained minor O while the larger one contained major amounts of O, Fe and Zn. Thirty-four particles/particle groups were Fe-rich containing either Cr or O, fifteen particles were interpreted to be salts (*e.g.*, sulfate, carbonate, halite; see Figure 6C), and four were metallic Al. The majority of particles were single-phase with occasional accessory grains, typically ≤ 1 μm, on the surface (Figure 7). In some cases however particles were composed of two or more discrete phases (Figure 7).

Amount of C on OR–CKP–11–1–S,0 was estimated based on two models, one with an average particle volume of 864 μm^3 (12 ·12 · 6 μm^3) and the other with an average particle volume of 1728 μm^3 (12 · 12 · 12 μm^3) (see Calculations). Considering the presence of two large, C-rich fibers and the model with the largest average particle volume, the total C estimate for OR–CKP–11–1–S,0 does not approach the maximum permissible abundance limit for C. The total K estimate was also calculated for OR–CKP–11–1–S,0 and it does not approach the maximum abundance limit for K (see Calculations).

Discussion

Key diagnostic elements: The Contamination Knowledge effort is monitoring the abundances of the following diagnostic elements in collected particles: C, K, Ni, Sn, Nd, and Pb. Below is a summary for each of these elements:

OR–CKP–10–1–S,0

C: C-bearing particles were the most common particle type on the mount. C abundances ranged from ~2.1–24.2 wt. %. One particle had elemental signatures of a biological contaminant.

[3]The locations for Spt 11(18), 11(27), 11(28) are not shown on Figure 4. Location for spt 11(36&37) are not shown as the particle moved after analysis.

K: K was observed as a minor/trace element in ~10 particles, the majority of which were siliceous.

Ni: Not observed

Sn: Not observed

Nd: Not observed

Pb: One particle, 3x4 μm^2 in size, contained Pb (2.4 wt.%), Cu (15.3 wt.%), Zn (8.9 wt.%), Ag (2.0 wt.%), C (2.1 wt.%), O (41.6 wt.%), Al (0.3 wt.%) and Si (27.5 wt.%). With the exception of the presence of Ag, this particle had a similar composition to that of the brass screw analyzed by us in February 2016.

OR–CKP–11–1–S ,0

C: Approximately 51 particles, from ~1–49 μm in the longest dimension, contained major/minor amounts of C ranging from ~5–23 wt.%.

K: K was observed as a minor/trace element in ~70 particles, the majority of which were siliceous. Twenty-two particles had major K, each with ~ 10 wt.% K.

Ni: Not observed.

Sn: Not observed

Nd: Not observed

Pb: Two particles, 8·3 μm^2 (spt 11(13)) and 10·14 μm^2 (spt 11(15)) in size, contained Pb. Compositions are given below:

Spt 11(13)	
Element	Wt.%
Pb	92.8
O	7.2
Total	100.0

PbO (webmineral.com)	
Element	Wt.%
Pb	92.8
O	7.2
Total	100.0

Spt 11(15) + Si	
Element	Wt.%
O	30.7
Fe	49.0
Pb	8.4
Zn	5.1
Si	6.8

Total	100.0

For spt 11(13), the chemical data fits well with that of lead (II) oxide ($Pb^{+2}O$). One form of PbO is called Massicot, which is used as a paint pigment. Spt 11(15) has been reduced with and without the presence of Si. There was no corresponding single mineral, alloy, or oxide phase with similar composition to that of spt 11(15). This suggests this particle may be a mixture of two or more phases.

Spt 11(15) w/o Si	
Element	Wt.%
O	26.8
Fe	57.0
Pb	10.3
Zn	5.9
Total	100.0

Calculations

Estimate of C on OR–CKP–11–1– ,0

Model 1

Assumptions: Composed of 100% C
Based on 51 C-bearing particles
Average size 12 μm · 12 μm with an estimated depth of 6 μm

Model using cube:

C density ~ 1.5 g· cm^{-3} (ave density of polymer/graphite)
Surface area is estimated to be 1 cm · 1.5 cm = 1.5 cm^2

Volume/particle	= 864 μm^3
Total particles	= 51
Total volume C-rich particles:	=864 μm^3 · 51 = 44064 μm^3
	= $4.40 \cdot 10^{-8}$ cm^3
Total grams C	= $4.40 \cdot 10^{-8}$ cm^3 · 1.5 cm^{-2}
	= $6.60 \cdot 10^{-8}$ g or 66.0 ng
Total $C_{Particles}$	= 44.0 ng/cm^2

Model 2

Assumptions: Composed of 100% C
Based on 51 C-bearing particles
Average size 12 μm · 12 μm with an estimated depth of 12 μm

Model using cube:

C density ~ 1.5 g· cm^{-3} (ave density of polymer/graphite)
Surface area is estimated to be 1 cm · 1.5 cm = 1.5 cm^2

Volume/particle	= 12μm · 12μm · 12μm = 1728 μm^3
Total particles	= 51
Total volume C-rich particles:	=1728 μm^3 · 51 = 88128 μm^3
	= $8.81 \cdot 10^{-8}$ cm^3
Total grams C	= $8.81 \cdot 10^{-8}$ cm^3 · 1.5 cm^{-2}
	= $1.32 \cdot 10^{-7}$ g or 132 ng
Total $C_{Particles}$	= 88 ng/cm^2

Fibers
Assumptions: Composed of 100% C
Thickness of 25 μm

Model using cube:
C density ~ 1.5 g· cm^{-3} (ave density of polymer/graphite)

Fiber 1.

Volume =25μm · 300μm · 25μm
=187,500 um^3
=$1.88 \cdot 10^{-7}$ cm^3

Total C_{Fiber1} = $1.88 \cdot 10^{-7}$ cm^3 · 1.5g/cm^3
= $2.82 \cdot 10^{-7}$ g or 282 ng
= 282 ng · 1.5 cm^{-2}
= 188 ng · cm^{-2}

Fiber 2.

Volume = 13μm · 182μm · 25μm
= 59,150 um^3
= $5.91 \cdot 10^{-8}$ cm^3

Total C_{Fiber2} = $5.91 \cdot 10^{-8}$ cm^3 · 1.5g/cm^3
= $8.87 \cdot 10^{-8}$ g or 88.7 ng
= 88.7 ng · 1.5 cm^{-2}
= 59.1 ng · cm^{-2}

Total C_{Fibers} = 247.1 ng· cm^{-2}

Maximum C estimate on Mount 11 = $C_{Particles}$ + C_{Fibers}
= 88 ng · cm^{-2}+ 247.1 ng · cm^{-2}
= 335.1 ng · cm^{-2}

Estimate of K on OR–CKP–11–1–S,0

Assumptions: Based on 22 particles with an average of ~ 10 wt.% K.
Average size 10 μm · 10 μm with an estimated depth of 10 μm

Model using cube:
Surface area is estimated to be 1 cm · 1.5 cm = 1.5 cm^2
Estimated particle density (composition of these particles is consistent with that of sanidine ($(Na,K)AlSi_3O_8$)) = 2.52 g· cm^{-3}

Volume/particle	= 10 μm · 10 μm · 10 μm = 1000 μm^3
Total particles	= 22
Total volume particles:	=1000 μm^3 · 22 = 22,000 μm^3
	= $2.2 \cdot 10^{-8}$ cm^3
Total grams	= $2.2 \cdot 10^{-8}$ cm^3 · 2.52 g · cm^{-3}
	= $5.54 \cdot 10^{-8}$ g or 55.4 ng
Total grams K	= 5.5 ng · 1.5 cm^{-2}
Total $K_{Particles}$	= 3.7 ng · cm^{-2}

Figure 1. OR-CKP-10-1-S ,0 SEM montage, particle map. Spectra names are underlined.

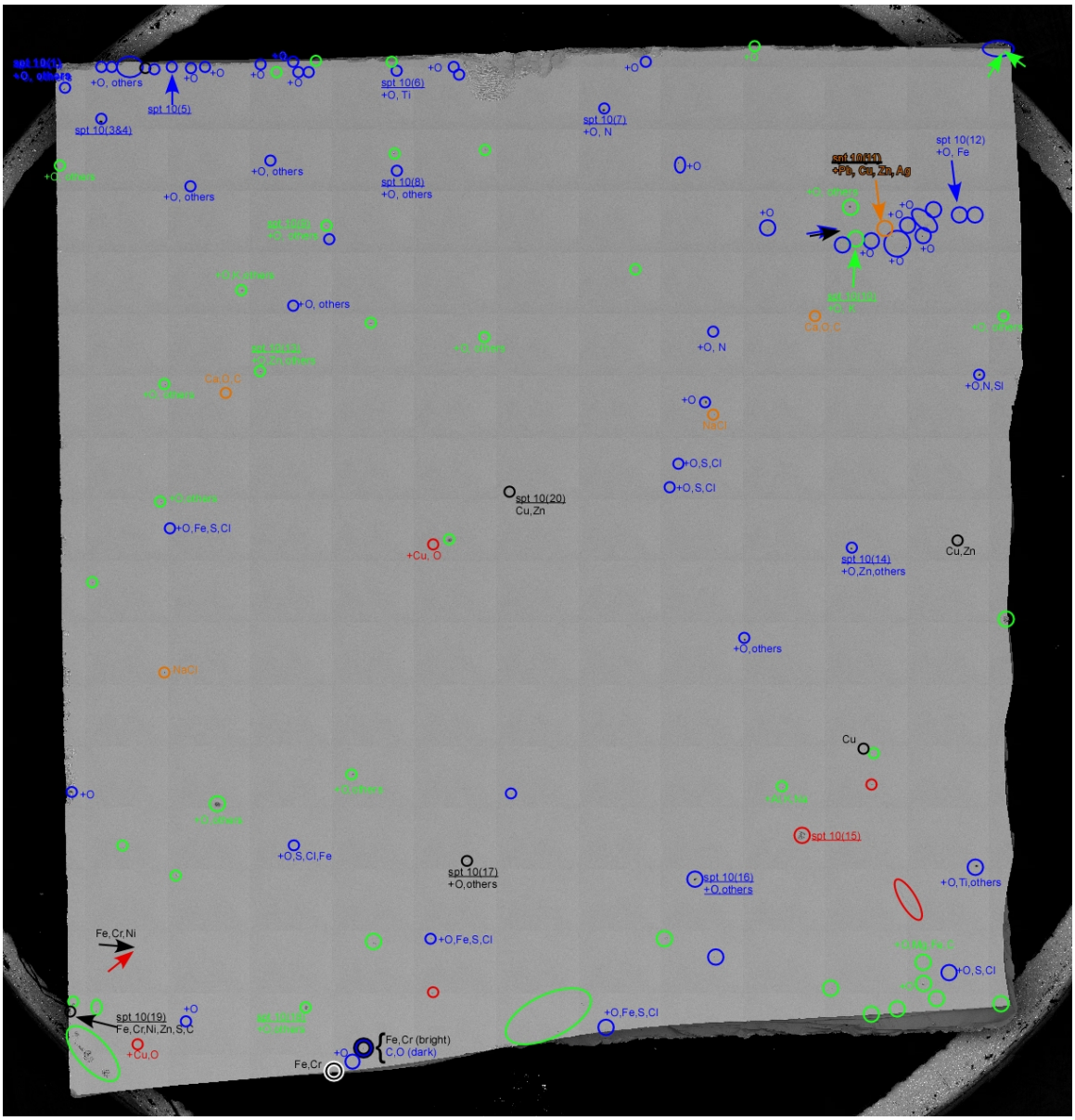

Figure 2. EDX spectra of three particles from OR-CKP-10-1-S,0. (A) Representative C-bearing spectrum for OR-CKP-10-1-S,0. (B) Particle, ~ 30 · 24 μm^2, composed of C, N, O, Na, S, Cl, and K. This element assemblage is consistent with that of biogenic matter (*i.e.*, skin residue, sweat). (C) One unusual C-bearing particle that contains Zn and F.

Figure 2

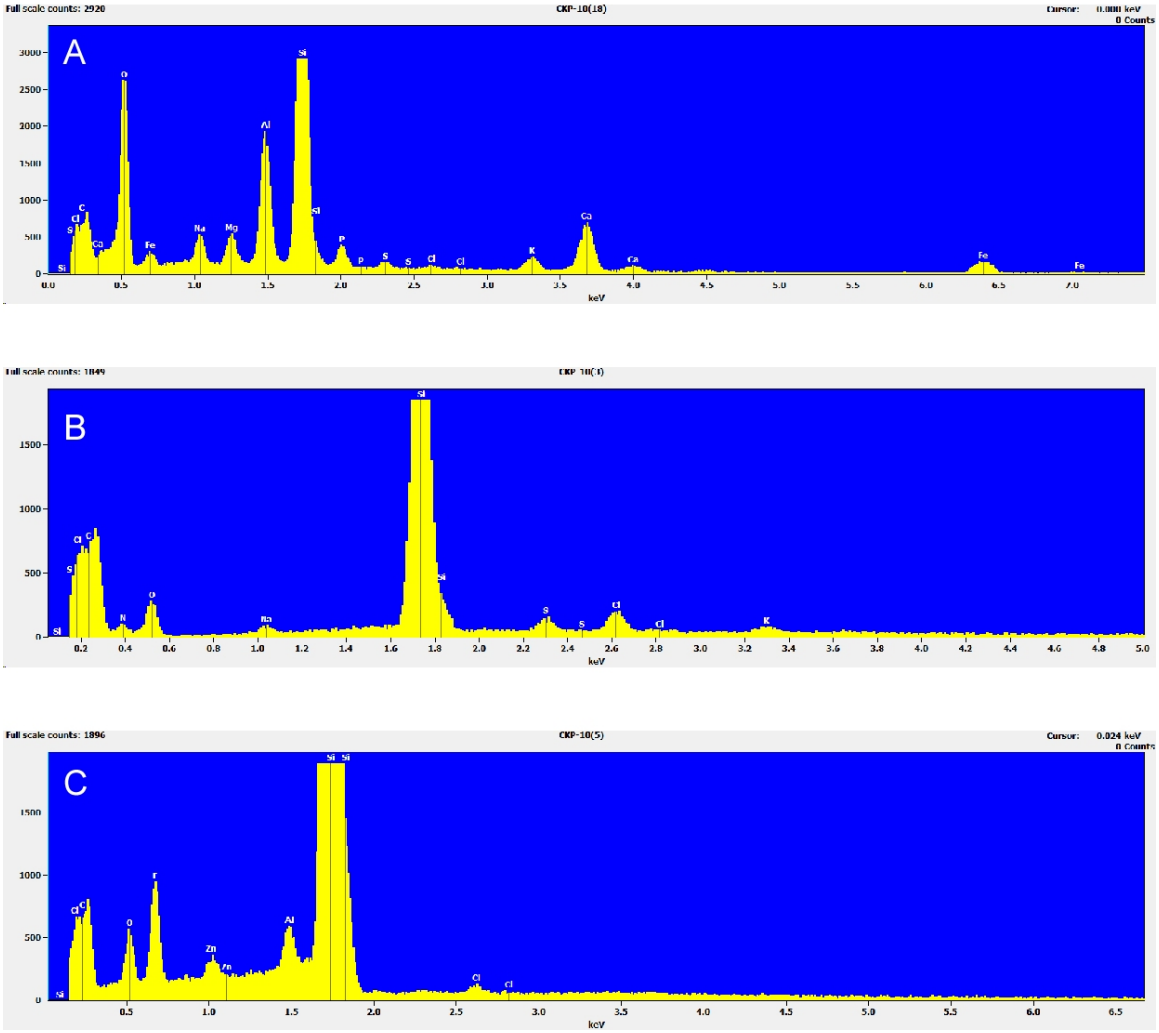

Figure 3. EDX spectra of a Pb-bearing particle from OR-CKP-10-1-S,0 and a brass screw. (A) Pb-bearing particle, ~ 3·4 μm^2, composed of Cu, Zn, C, O, Pb and Ag. (B) Two overlapping spectra showing similarities in composition of the Pb-bearing particle in (A) (yellow) and a brass screw (red), analyzed by us using the same experimental conditions. The spectra are similar with the exception of Ag in the particle.

Figure 3

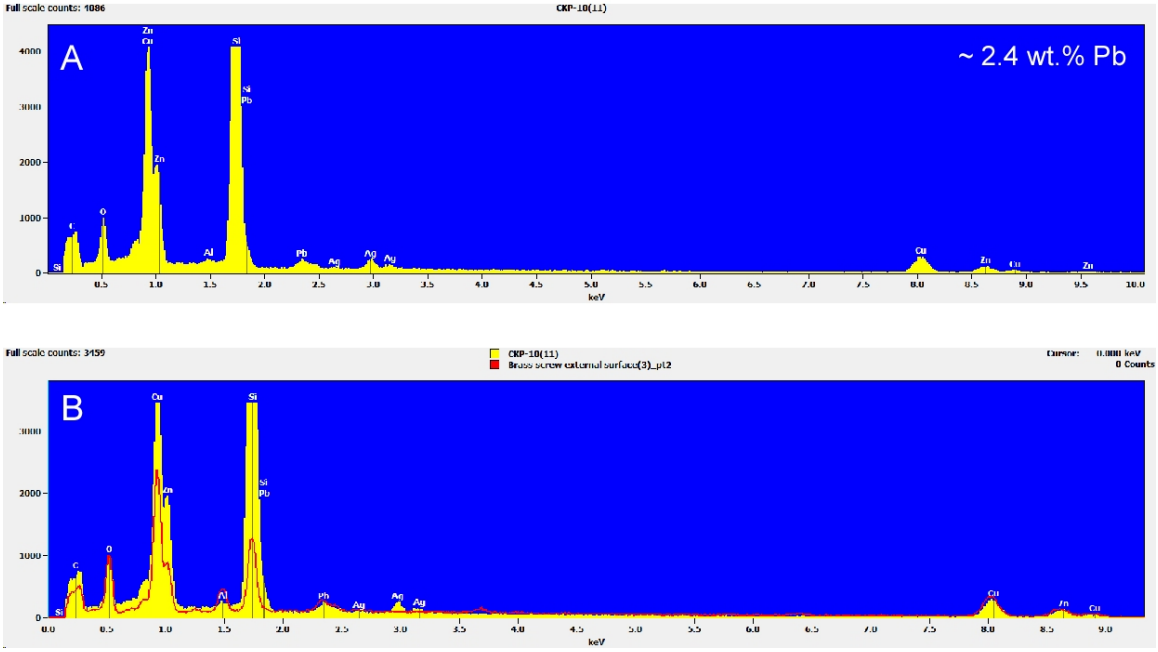

Figure 4. OR-CKP-11-1-S,0 SEM montage, particle map. Spectra names are underlined.

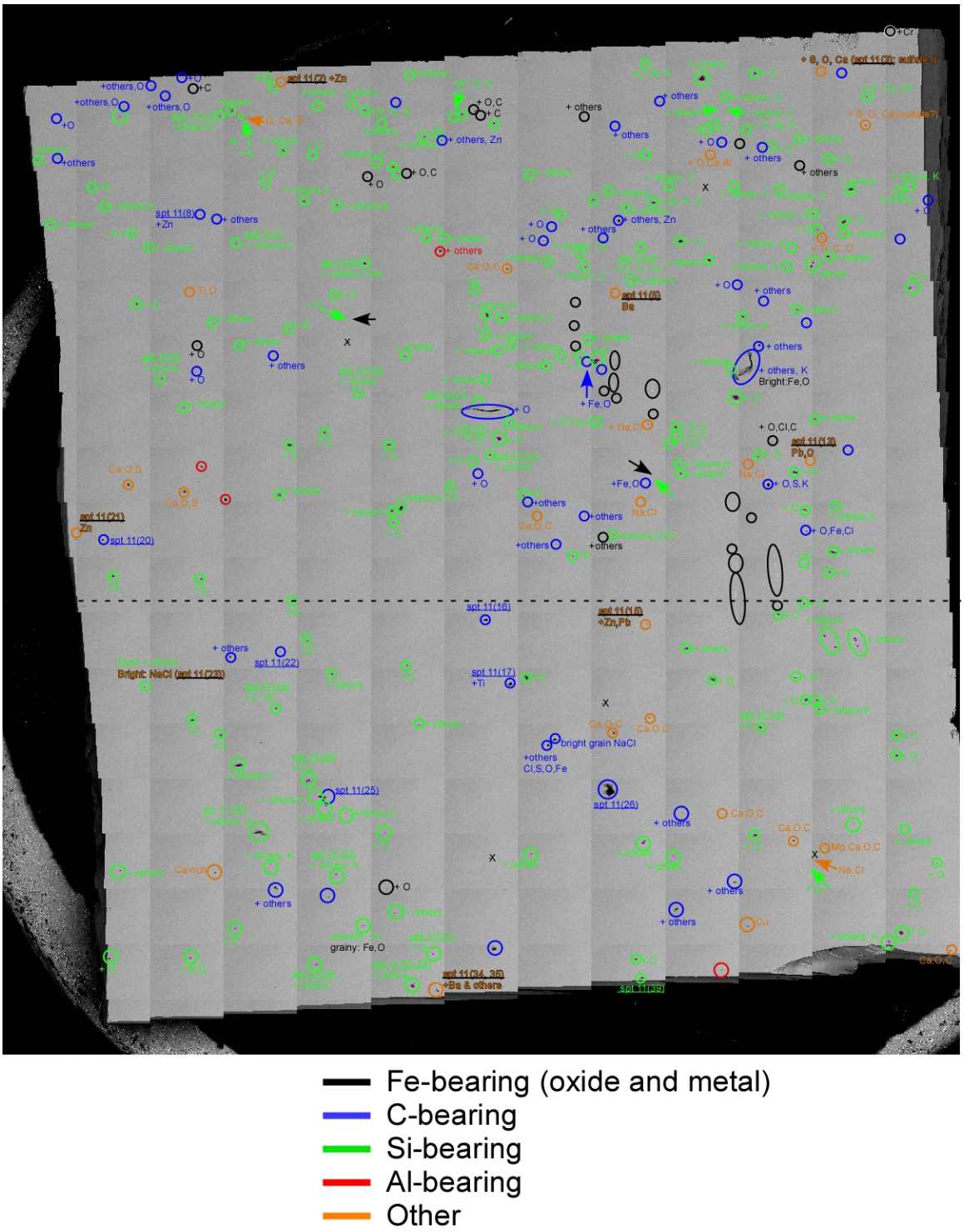

Figure 5. EDX spectra of four particles from OR-CKP-11-1-S,0. (A, B) Representative Si-rich spectra for OR-CKP-11-1-S,0. (C) Particle, ~ 21·21 μm^2, composed of Si, Al, O, K, Na. K is a major element and is present at ~ 10 wt.%. The spectrum is nearly identical to that of sanidine ($(Na,K)AlSi_3O_8$). (D) Representative C-rich spectrum showing C, Si, Al, Na, S, Cl, Ti, Mg, and Ca.

Figure 5

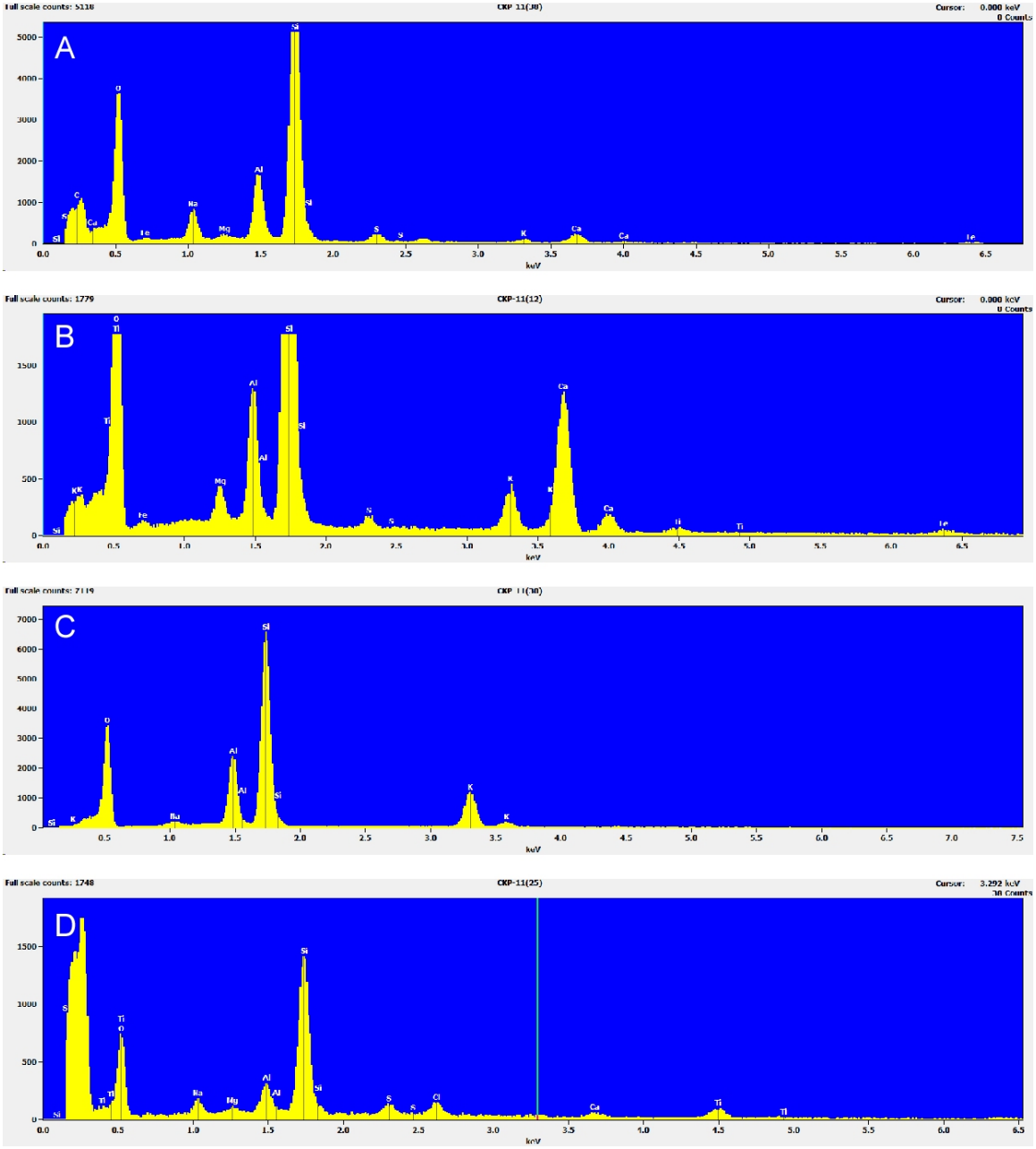

Figure 6. EDX spectra of three particles from OR-CKP-11-1-S,0. (A) Pb-rich particle, ~8·3 μm^2, with minor O (see Discussion for EDX data) (B) Pb-bearing particle, ~10·14 μm^2, with Fe, O, Zn, Si, and minor/trace Al (see Discussion for EDX data). (C) Spectrum of a particle, ~50 ·21 μm^2, showing Si, Ba, S, O, Cl, C and minor/trace Mg, Al, consistent with a barium sulfate – $BaSO_4$ – also known as barite.

Figure 6

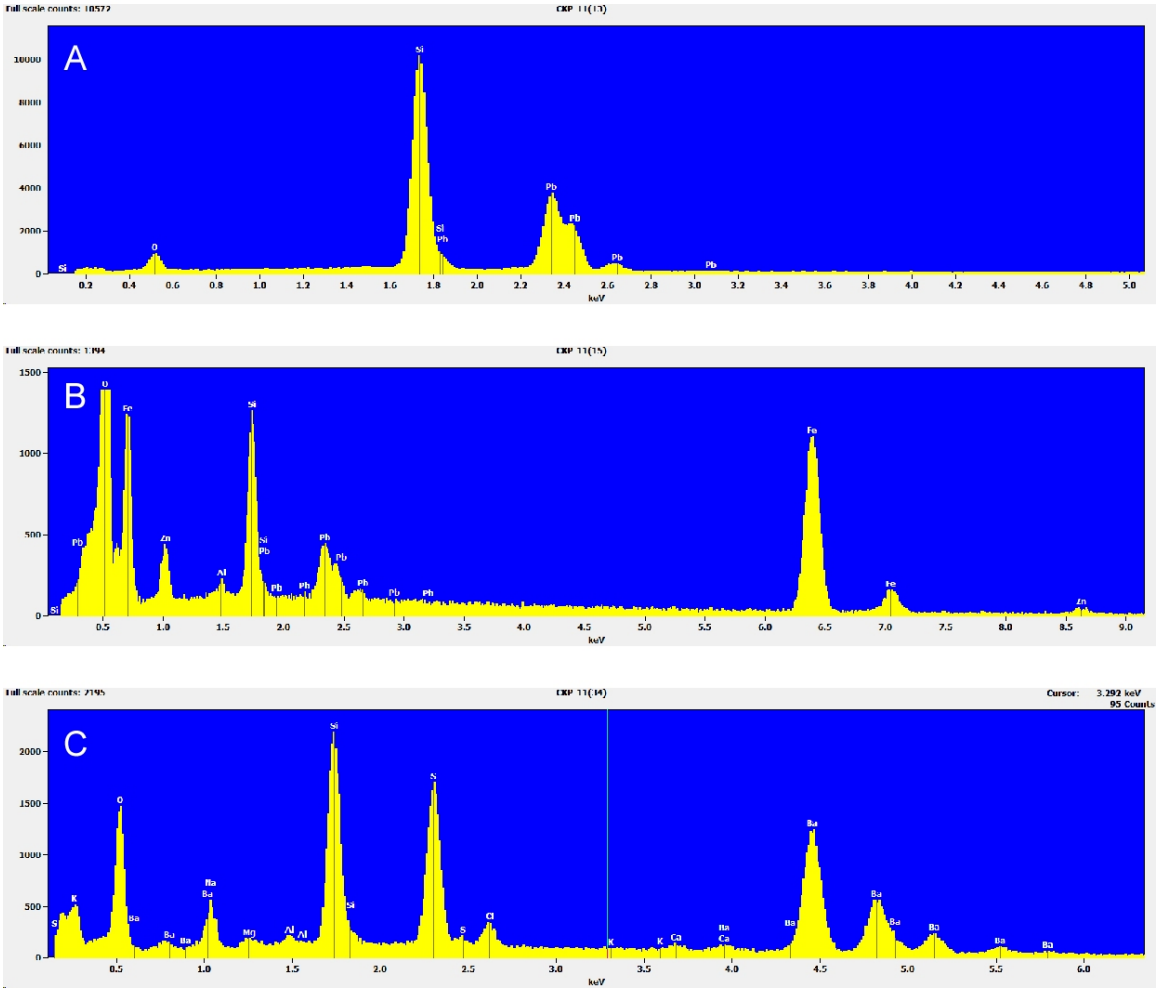

Figure 7. FESEM images and EDX spectra of two particles from OR-CKP-11-1-S,0.
Left image: Particle composed of two chemically distinct phases, shown as dark and light regions using LABE.
Left spectrum: Two overlying spectra of the particle described above showing the dark phase is C-rich (yellow) while the light phase is composed of halite (red).
Right image: Single-phase particle with few accessory grains.
Right spectrum: Composition of the single-phase particle showing it is Si-rich with O, C, Na, S, Ca, and K. Excluding the C, this composition is similar to that of albite ($NaAlSi_3O_8$).

Figure 7