File Note

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Date: 18 October 2013 File Ref: 1224-915537 Trail Support
Subject: **Environment Dustfall Particulate Characterization (Trail Operations)**
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**Summary**

Scanning electron microscopy and energy dispersive spectral (SEM-EDS) analysis was used to characterize the relative size and bulk composition of particulates in a group of 10 dust fall jar samples collected locally. The study showed that Pb occurred primarily in oxide form, and was associated with Fe, Cu, Sn, S, Zn, As, Sb and/or Si, lesser Ca, Mg and/or Mn, and rarely Au, Ag, Te, In, Se, Bi, Cd and /or Cl. Rare occurrences of Pb sulphide and metal were also found. Pb- bearing particulates ranged from 0.1 to 75 um in size, but the median particle size was ~3 µm or less. Pb-oxides containing As, Cd and (Se, In) were relatively coarser-grained than other Pb-oxide compositions, suggesting that they may have originated from fugitive dust emission, rather than as precipitates from gas stack emission.

The highest abundance of Pb-bearing particles was found at sites R1-D, R5, R8, R7 and R10 – all within sub 0.7 km distance from potential Pb-bearing particle sources. The higher numbers reflect the presence of fine particle agglomeration. The majority of particles from sites R-12 and Oasis were a relatively pure Pb-O phase. The largest total particle surface area (both agglomerated and liberated) or potential 'availability' to metal leaching of Pb-bearing phases was found at sites R1-D, R4, R6, R5 and R8. All of these sites are within sub 0.7 km distance from a potential Pb-bearing particle source, except for R-4.

**Background**

The Environment Group at Trail Operations is investigating the source of fugitive dust emissions into the surrounding community. The current set of particulate samples was collected using dust fall jars distributed across the Trail community in an effort to better understand the mechanism of dust deposition. Table 1 shows the sample name and elemental analysis. Figure 1 displays the location of sampling stations and distance between identified industrial particle source and sampling station.

**Sample Preparation**

The mixture of water, particulates and organic material (insects, pollen, other) in each dust fall jar was filtered using Whatman Grade 1 filter paper and the residue dried at room temperature.

Coarse organic material was removed from the filter residues using forceps. Particulate weights collected were each less than 0.1 g.

**Table 1**. Dustfall (jar) collection station location, particle distribution and assay data.

| Sample Name | Identity | Description | Northing | Easting | Elev. (meters) | EMS No. | Assay Code | T/Part (mg/Spl) | T/As (mg/Spl) | T/Cd (mg/Spl) | T/Pb (mg/Spl) | T/Zn (mg/Spl) | T/Tl (mg/Spl) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| R1 | Glenmerry | City Works yard inside main fence, east of buildings | 5438453 | 451089 | 453 | E260026 | PSD-EA67 | 64 | 0.01 | 0.01 | 0.25 | 1.1 | 0.001 |
| R1-D | Trail Hospital | Roof of Hospital next to new wing, go in behind admin wing. | 5439142 | 448761 | 476 | E260023 | PSD-EA68 | 20 | 0.02 | <0.01 | 0.25 | 1.1 | <0.001 |
| R4 | Birchbank | Old Teck farm near middle of field | 5447621 | 446877 | 544 | E260004 | PSD-EA61 | 42 | <0.01 | <0.01 | 0.02 | 0.09 | < 0.001 |
| R5 | Stoney Creek | Upper material laydown area, on south side of berm | 5439915 | 446516 | 501 | E207877 | PSD-EA73 | 24 | <0.01 | <0.01 | 0.17 | 0.97 | < 0.001 |
| R6 | Downtown | Roof of Amec Building on Cedar Avenue | 5438307 | 448299 | 433 | E260010 | PSD-EA65 | 28 | 0.01 | 0.01 | 0.28 | 1.4 | < 0.001 |
| R7 | On site | Combined 2 sampling station | 5438677 | 448147 | 434 | | | | | | | | |
| R8 | Tadanac | 303 Kootenay Avenue | 5439981 | 447263 | 463 | E259131 | PSD-EA75 | 28 | 0.03 | 0.02 | 0.59 | 1.4 | < 0.001 |
| R10 | Columbia Avenue | Back of 1428, go down road immediately right of Kiro, down alley near end of road | 5438693 | 448558 | 415 | E259128 | PSD-EA62 | 35 | 0.02 | 0.02 | 0.32 | 1.3 | < 0.001 |
| R12 | Warfield | At sample station | 5438323 | 445473 | 619 | E260011 | PSD-EA77 | 23 | <0.01 | <0.01 | 0.07 | 0.27 | < 0.001 |
| Oasis | Oasis | Very top of Oasis, turn left at fork and go to the end of pavement | 5442506 | 445573 | 548 | E260022 | PSD-EA71 | 13 | < 0.01 | <0.01 | 0.04 | 0.16 | < 0.001 |

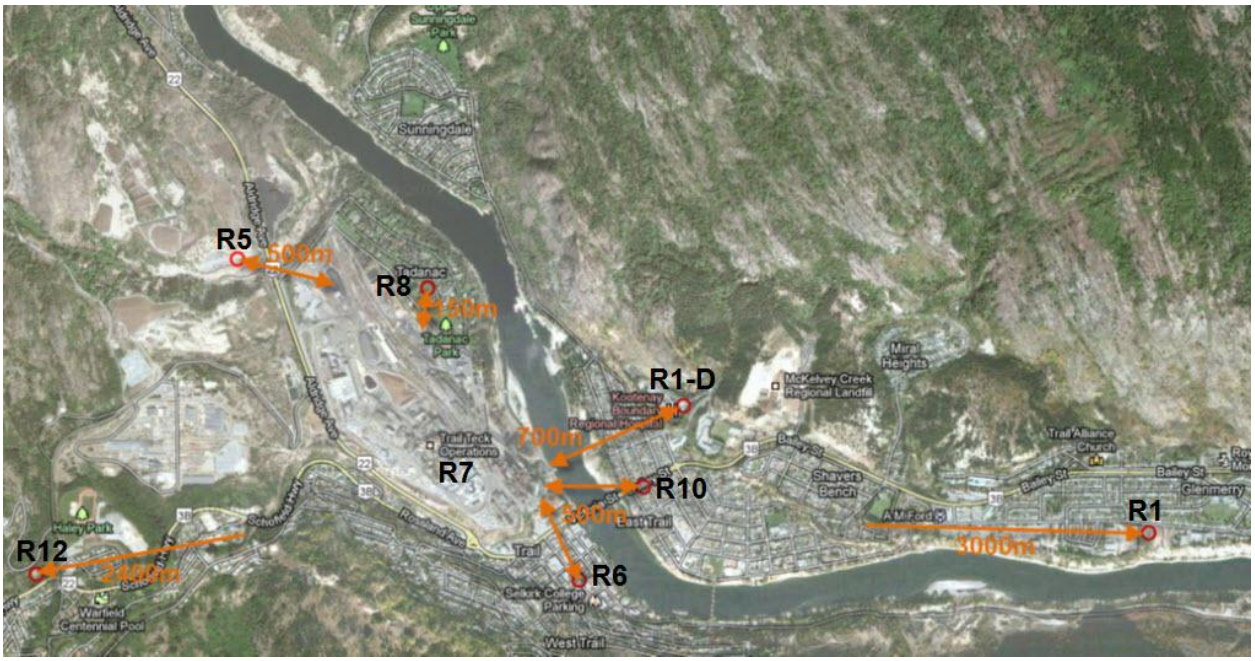


**Figure 1**. Dustfall (jar) collection station location.

**Sample Analysis**

Each sample was stub-mounted for SEM analysis. The mounts were examined using a FEI Quanta 600 SEM (tungsten filament, accelerating voltage 20kV) equipped with two EDSs for elemental X-ray detection. The EDS detection limit for this type of sample mounting is ~1 wt% detection limit. A minimum of 30 lead-bearing particles were located and measured for each sample in order to determine the relative abundance of phases.

Notes:

- The elemental information in this report is semi-quantitative.
- Compositional information is collected using a spot size of approximately 3 um, meaning that analyses may represent the combined spectra of several very fine particles.
- EDS does not accurately report the quantity of elements with an atomic number lower than fluorine.
- The presence of Cu in the particulates may be due to the addition of Cu sulphate to the dust fall jars to prevent or slow algal growth.
- Larger particles may have broken during mounting, which will affect particle size results.

**Results and Discussion:**

Lead:

- The SEM-EDS study showed that lead occurred primarily in an oxide form. Clean Pb-oxide, with little to no other associated elements, was the most abundant Pb-phase in the R12 and Oasis samples.
- Pb-oxide was frequently associated with other elements including, and listed according to frequency, Fe, Cu, Sn, S, Zn, As, Sb, Si, (Ca, Mg, Mn), (Au, Ag, Te, In, Se, Bi), Cd and Cl.
- Rare occurrences of lead sulphide and lead metal also were identified.

Figure 2 shows the relative distribution of Pb-bearing phases in each sample based upon the number of occurrences recorded.

Higher prevalence of Pb-bearing particulates, listed in decreasing order, was found in R1-D, R5, R8, R7 and R10. The higher number of particles was attributed to the finer nature of the material and the presence of particle agglomerates, in comparison with the other samples. The finer material suggested particle excursions from a gas stack rather than fugitive dust emission. Samples with maximum total surface area of Pb-bearing phases, listed in decreasing order, were R1-D, R4, R6, R5 and R8. The higher calculated surface areas (based on either a rectangular or spherical particle) were primarily due to particle agglomerations.

Table 2 shows particle size relative to Pb-O composition for particles measured. The particle size range suggest that Pb-O particles associated with As, Cd and (Se, In) originate from fugitive dust emission, while other element-associated particles may have originated from either gas or fugitive dust emission.

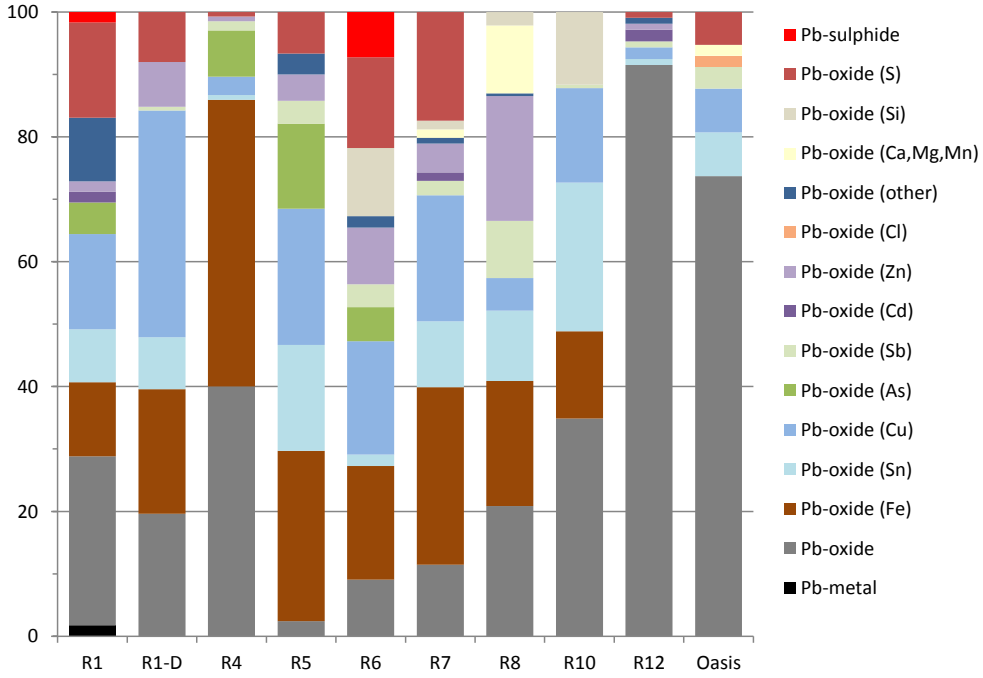


**Figure 2**. Relative abundance of Pb-particulate compositions in dust fall samples. Note: individual elements were associated with Pb-O phase.

**Table 2**. Observed particle size based on phase and associated element.

| Phase | Median Size | | Size Range (um) |
|---|---|---|---|
| | L (um) | W (um) | |
| Pb-O | 2.7 | 3.1 | 0.2 - 75 |
| Pb-O (Zn) | 2.3 | 2.3 | 0.6 - 32 |
| Pb-O (As) | 3.1 | 4 | **3.1 - 35** |
| Pb-O (Sb) | 1.7 | 2.1 | 0.6 - 18 |
| Pb-O (Cd) | 2.5 | 2.5 | **2 - 2.7** |
| Pb-O (Se, In) | 5.4 | 4.6 | **2.9 - 16** |
| Pb-O (S) | 3.5 | 4.5 | 0.2 - 67 |
| Pb-O (Fe) | 2.5 | 3 | 0.3 - 27 |
| Pb-O (Sn) | 3 | 4.7 | 0.1 - 20 |
| Pb-O (Si) | 3.2 | 4 | 0.3 - 31 |
| Pb-O (Cu) | 4.7 | 3.5 | 0.1 - 25 |
| Pb-O (Ca) | 1.8 | 1.8 | 0.4 - 14 |

Backscatter electron (BSE) images and associated elemental make-up of select particles can be found in Appendix 1. Images show that agglomerated particles were most often very small in size. A list of lead-bearing phase compositions is found in Appendix 2.

Zinc:

- Zinc typically occurred within the Pb-oxide phases. Also associated with this mixed composition were Fe and Cu, and lesser S, Sn, Si and Mn.
- Zinc was also present as an oxide, frequently containing minor Pb, Fe and/or S. The sulphur may be present as sulphate.

Other:

- As was mainly present within the Pb oxide, typically associated with Fe and/or Sn.
- Sb was mainly present within Pb-oxide and as Sb-oxide containing Pb, sometimes associated with Fe.
- Cd was a rare constituent of the samples. When present, it occurred as Pb-Cd-oxide, sometimes associated with S and/or Zn.
- Se and In were rare constituents in the samples. When present, they occurred within the Pb-oxide.
- S was mainly present as a Pb-S-oxide with strong associations to Cu and Fe. It is likely that S was present as sulphate. Pb-Cu-S-oxide commonly contained Fe or Sn.
- Fe was present with Pb-oxide, commonly associated with Sn and/or Cu. Pb-Fe oxide was also found associated with Mn and Si.
- Sn was mainly present in Pb-oxide, with rare associations to Cu and Mg.
- Si was mainly present in Pb-oxide, with rare associations to Cu and Mg.
- Cu was present in Pb-oxide.
- Ca was present in Pb-oxide; with rare association to Cl.

**Conclusions**

The SEM-EDS analysis was able to provide information on relative particulate size and overall composition in the dust fall jars. It had difficulty characterizing particles less than 1 - 2 µm in diameter.

- Pb-bearing phases are primarily oxides, which may be associated with Fe, Cu, Sn, S, Zn, As, Sb, Si, (Ca, Mg, Mn), (Au, Ag, Te, In, Se, Bi), Cd and Cl. Rare occurrences of Pb sulphide and metal were found.
- Pb particulates ranged from 0.1 to 75 µm in size, but the median particle size for Pb-oxide was ~3 µm. The particle size ranges suggest that the relatively coarse Pb-oxide phases associated with As, Cd and (Se, In) originated from fugitive dust emission – not gas stack emission.
- A higher prevalence of Pb-bearing particles was found at sites R1-D, R5, R8, R7 and R10 – all within sub 0.7 km distance from potential Pb-bearing particle source. The higher numbers reflect multiple finer particles agglomerated either before or in the collector.
- Majority of particles from sites R-12 and Oasis were a relatively pure Pb-O phase.
- The largest total particle surface areas of Pb-bearing phases were found at sites R1-D, R4, R6, R5 and R8. All sites are within sub 0.7 km distance from a potential Pb-bearing particle source, except for R-4.

**Recommendation**

It is recommended that sample collection protocols be reviewed/improved to concentrate particulates and which would be beneficial for SEM dust fingerprinting.

**Appendix 1.** Back-scatter electron (BSE) images from dustfall (jar) collectors: select particles.

Note: element in brackets refers to element being present in minor or trace amount.

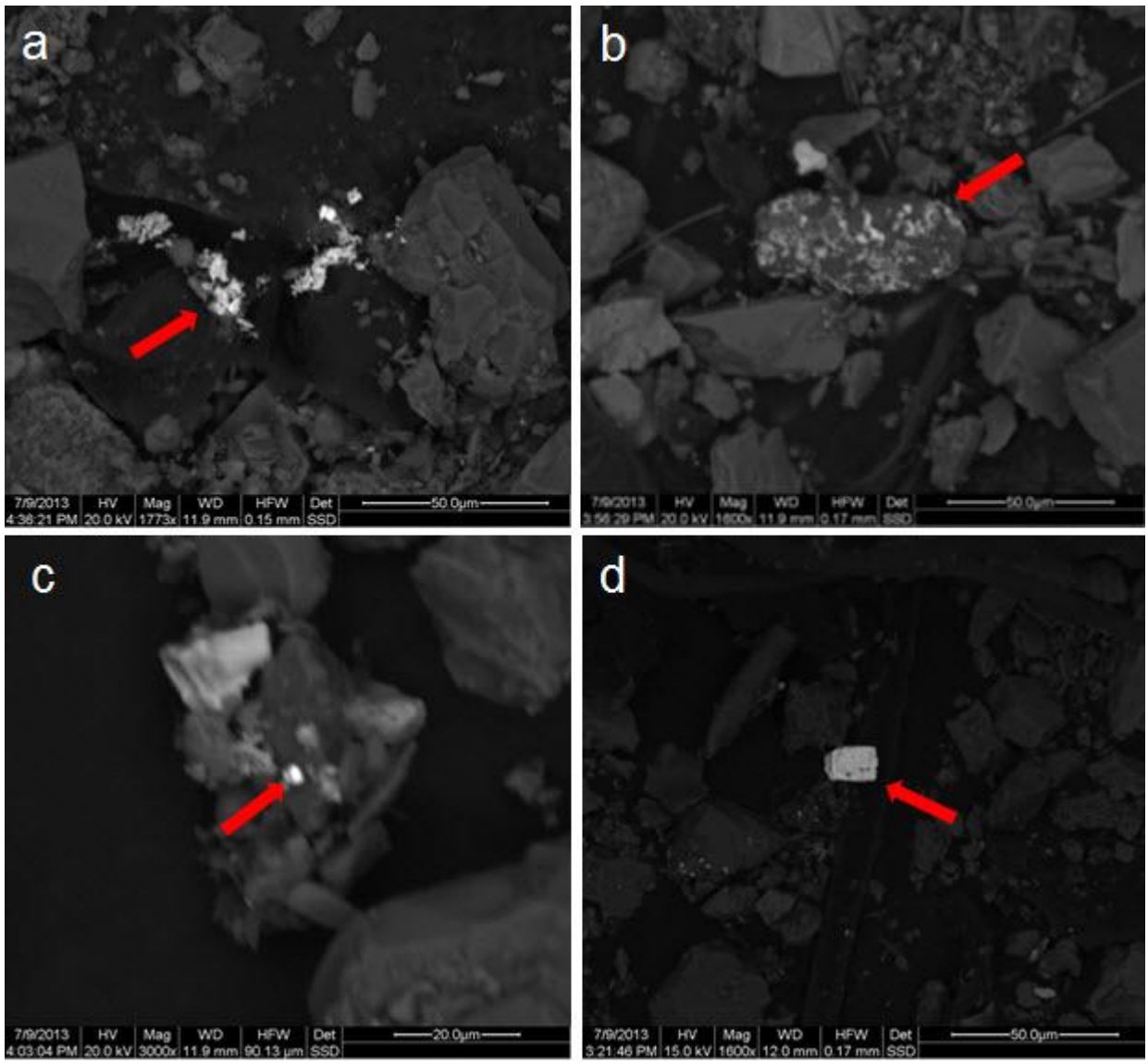


**Figure A1. R1 (Glenmerry)**: (a) Pb-O-(Sb) particles range in fineness and varying degrees of agglomeration; (b) rare Pb-O phase mixed with Si-O phase; (c) Pb-S particle; (d) Pb metal particle.

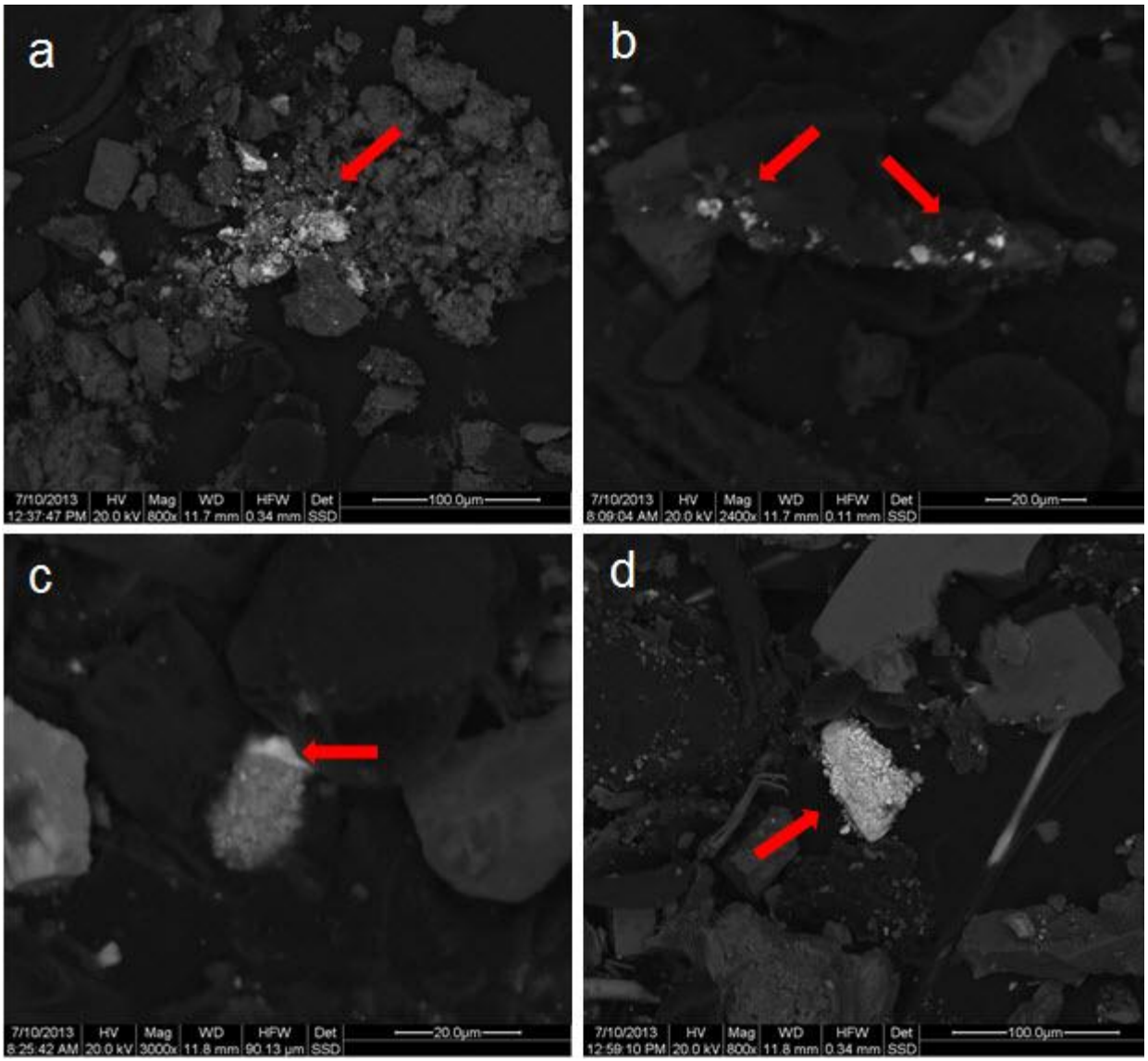


**Figure A2. R1-D (Trail Hospital)**: (a & b) Pb-O-(As, Fe) particles range in fineness and varying degrees of agglomeration; (c) rare occurrence of Pb-S-O phase associated with Cu-As-O-Mo-(Sn, Fe) phase; (d) Pb-Cu-S-O particle agglomerate.

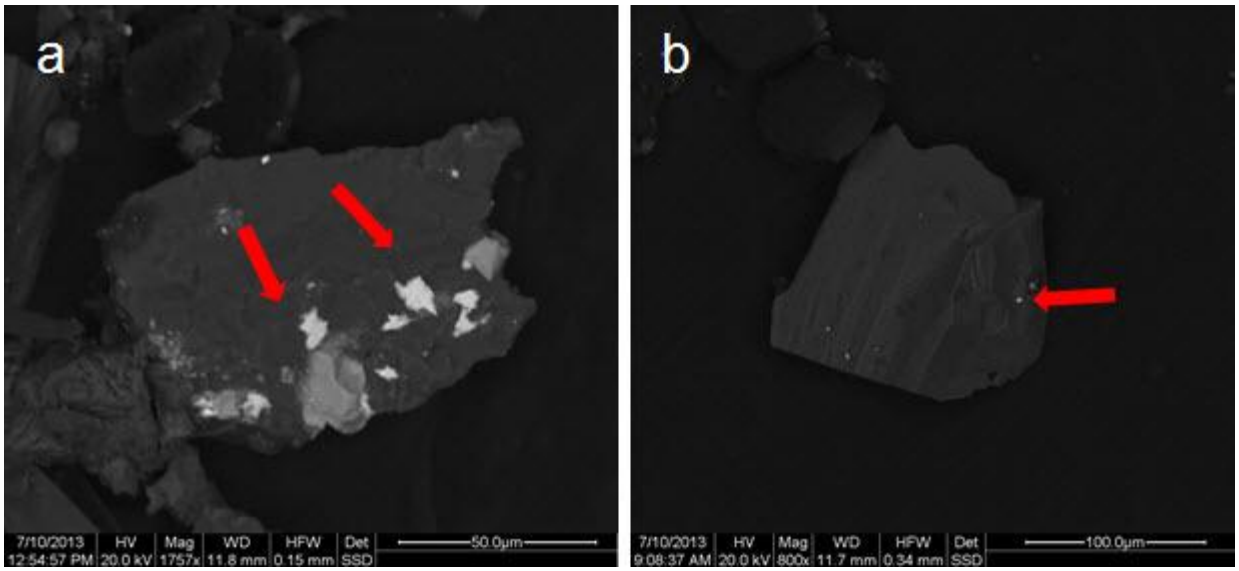


**Figure A3. R1-D (Trail Hospital)**: (a) several bright Pb-O phases and Zn-S (light gray) phases as part of Si-O particle (dark gray); (b) several bright Pb-O-(Sb) phases associated with Si-O particle (dark gray).

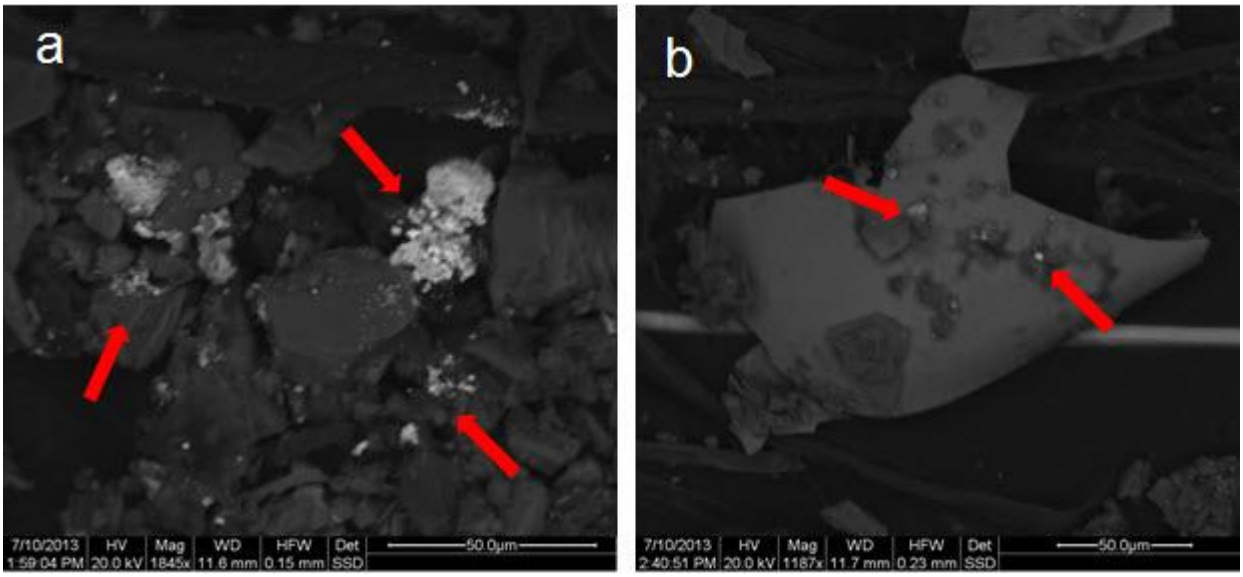


**Figure A4. R4 (Birchbank):** (a) multiple particles of Pb-O-(Fe), some agglomerated; (b) Pb-O-(Fe) particles trapped in slag.

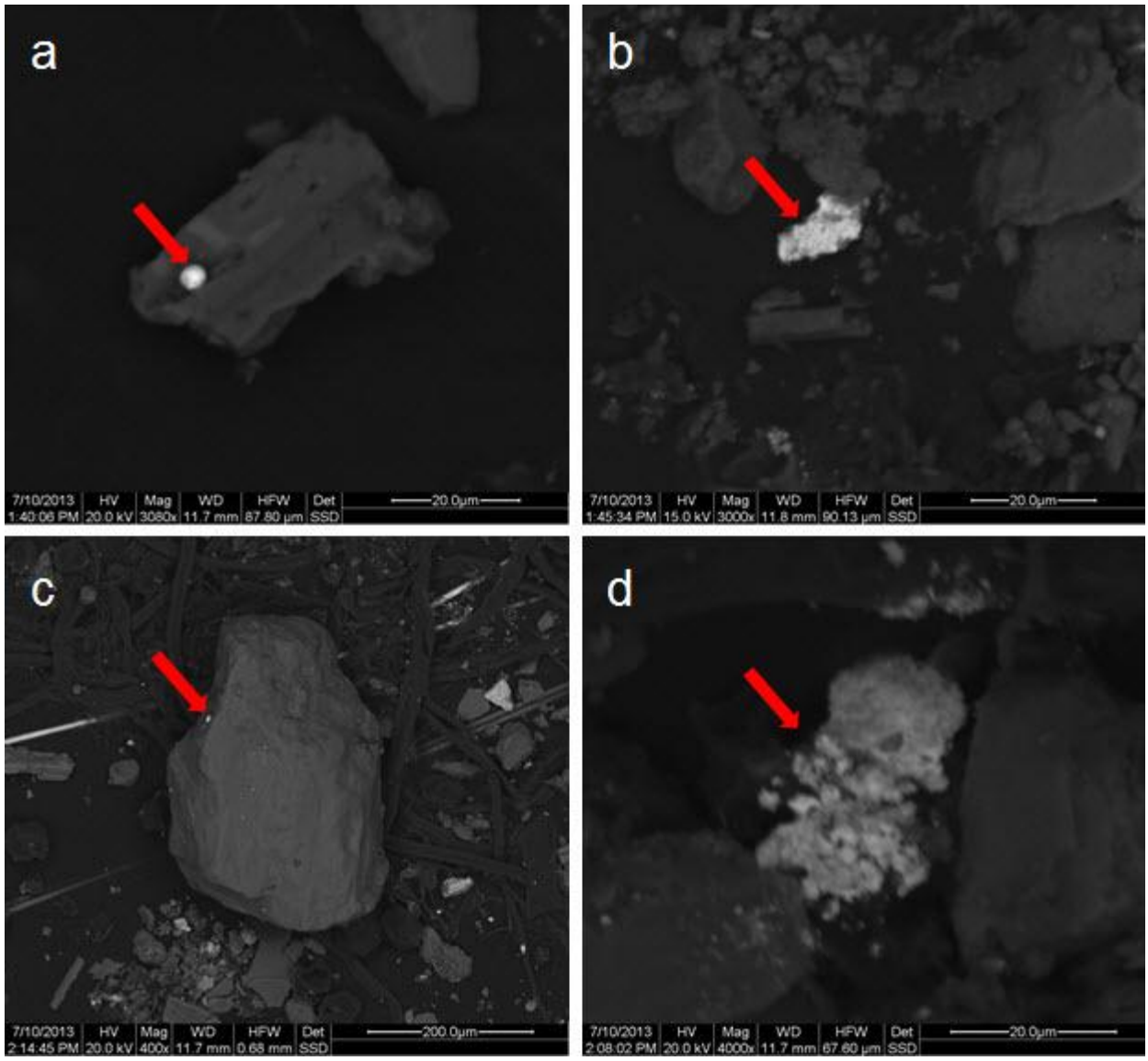


**Figure A5. R4 (Birchbank):** (a) Pb-O phase trapped in silicate; (b) multiple Pb-O particles agglomerated; (c) Pb-Cu-O phase trapped in large Fe-O phase particle; (d) multiple Pb-As-O-(Fe) particles, here many agglomerated.

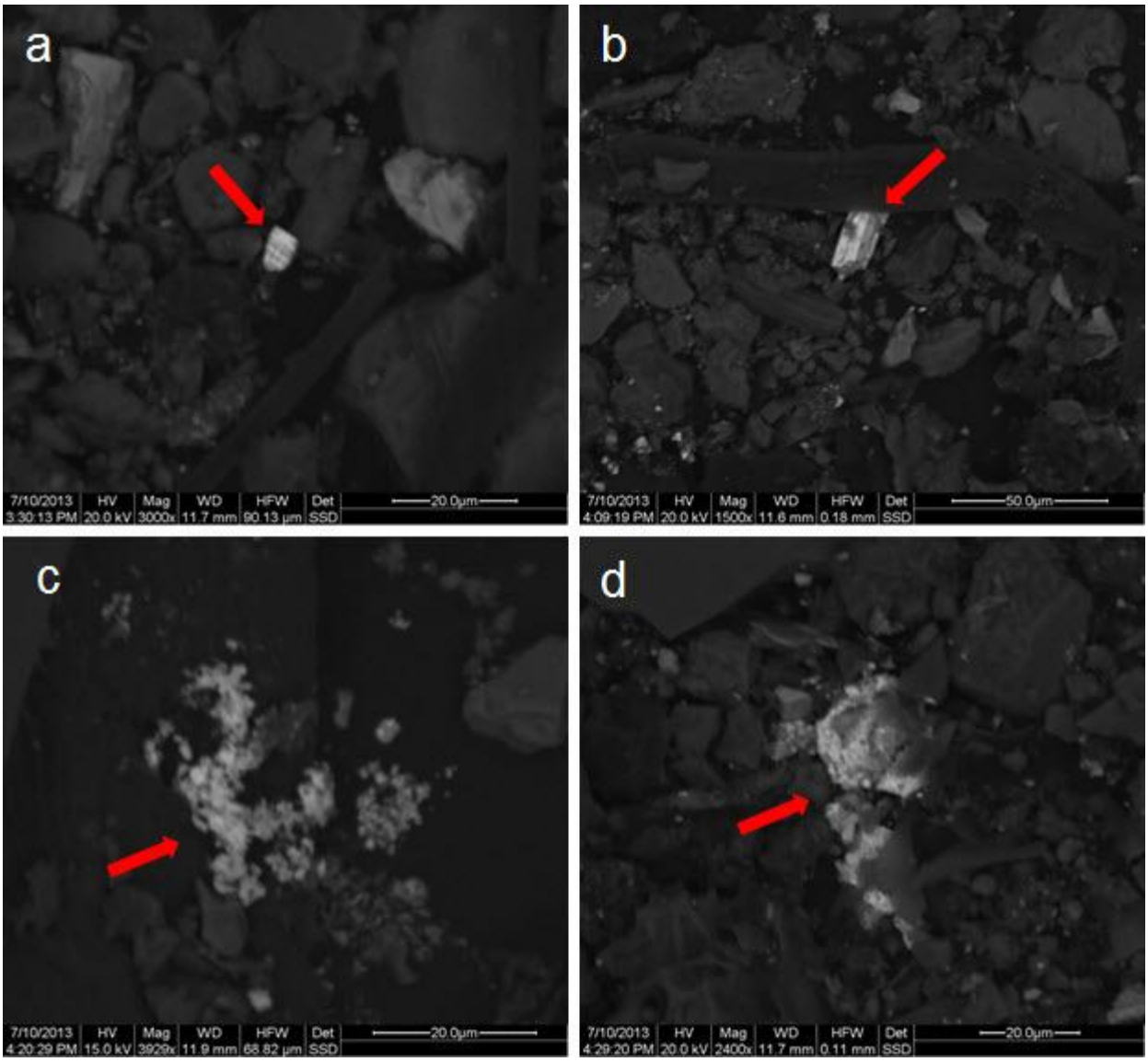


**Figure A6. R5 (Stoney Creek)**: (a) Pb-O-Cu particle; (b) Pb-O-(Cu) particle; (c) multiple Pb-O-(Cu) particles, mostly agglomerated; (d) particles of phase Pb-O-S-(Cu), mostly agglomerated.

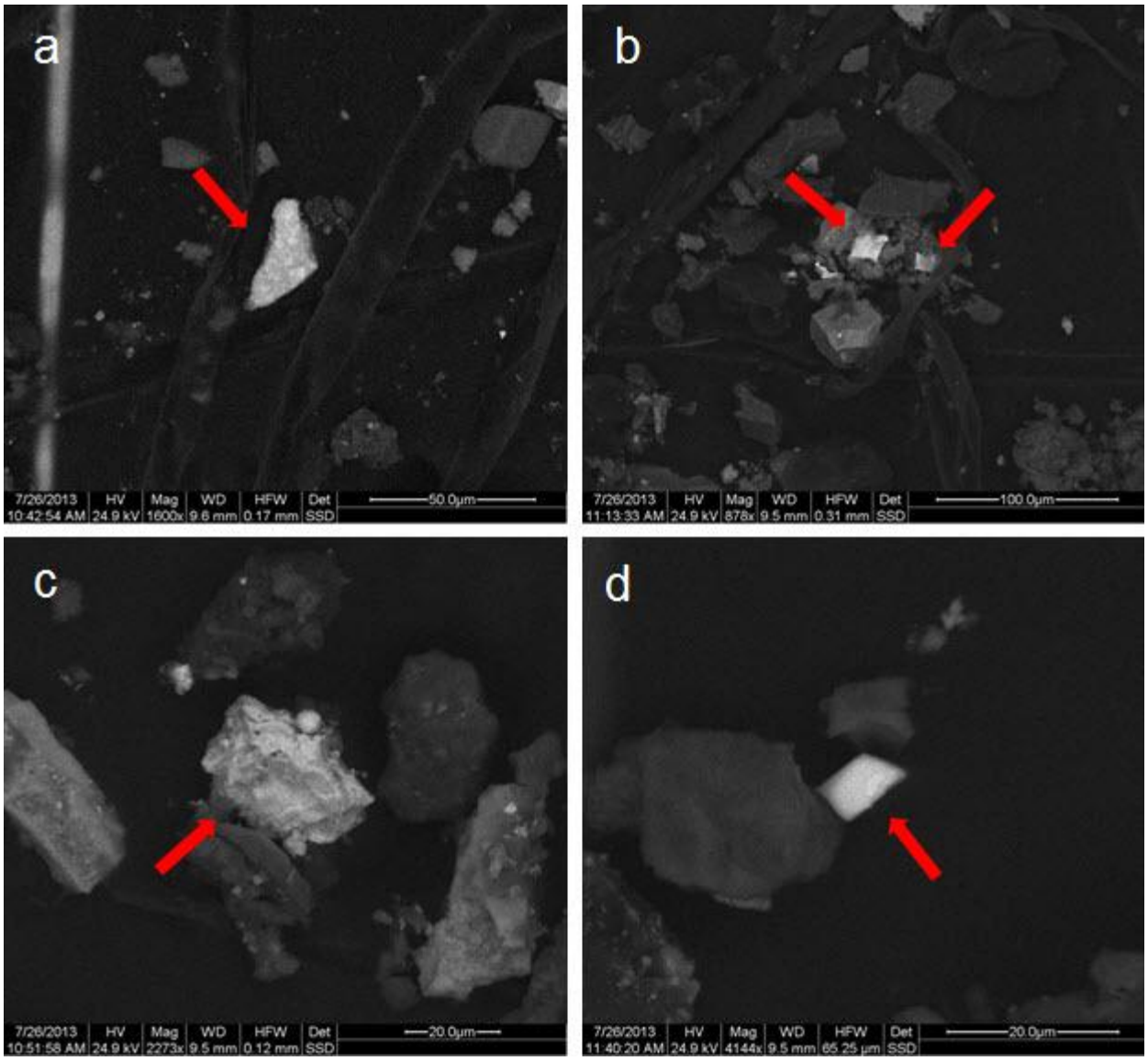


**Figure A7. R6 (Downtown)**: (a) blended particle phase Cu-Pb-O-S-Zn; (b) particles of Pb-S; (c & d) particles of Pb-As-Sb-O.

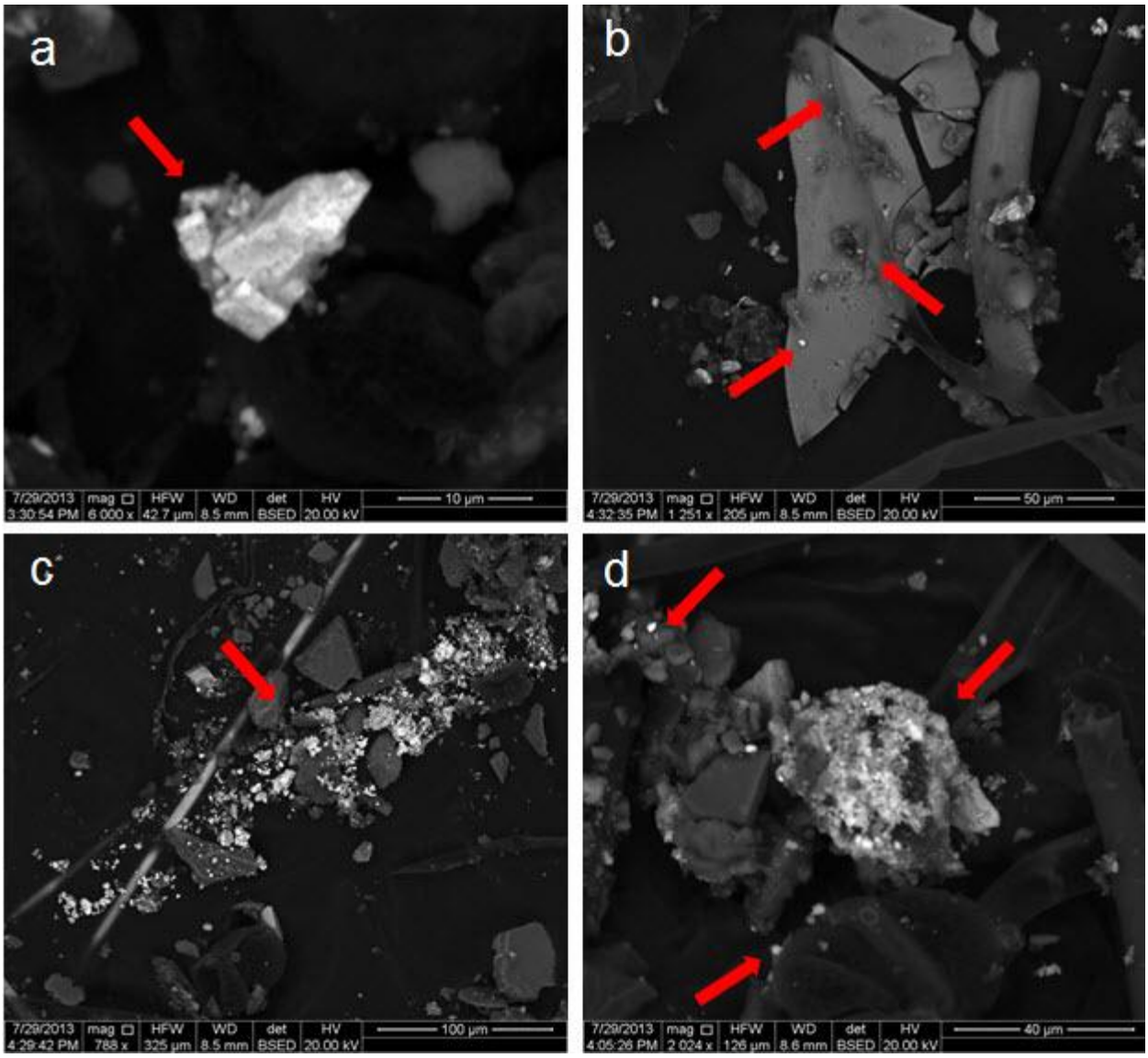


**Figure A8. R7 (On site)**: (a) particle of phase Pb-S-O-(Cu); (b) Pb-S-O-(Cu,Fe) phase trapped in slag; (c) Zn-Cu-Pb-Fe-S-O particles, some agglomerated; (d) multiple-phase agglomerate as well as liberated particles: single Pb-O-(Cu,Mn,Sb) phase particles associated with Ca-C-O particles, multiple Pb-O-(Sn,Fe) and Pb-O-(Cu,Fe) phase particles agglomerated.

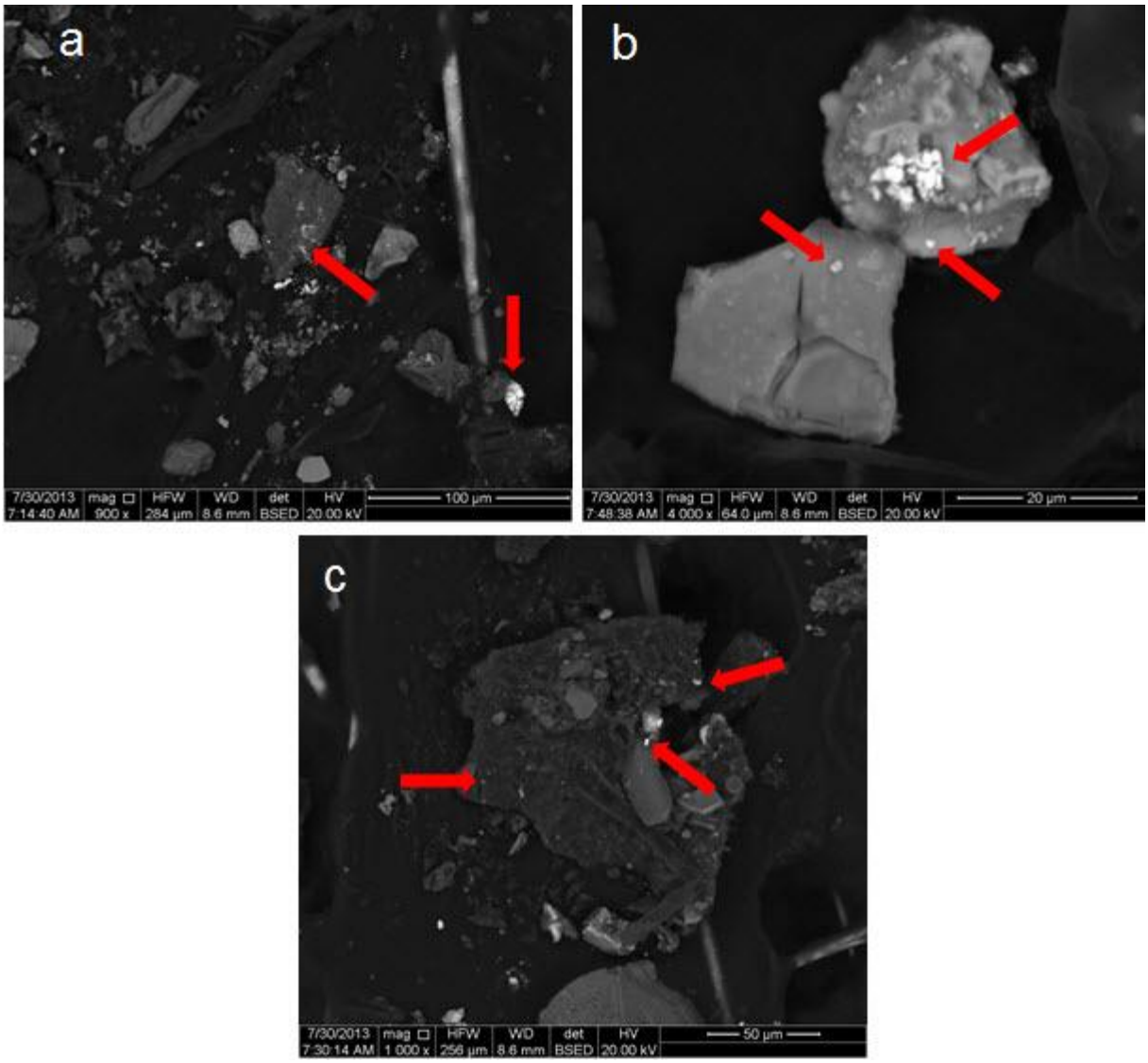


**Figure A9. R8 (Tadanac)**: (a) multiple particles of Pb-O-(Cu) trapped in Ca-Al-Si-O particle and larger Fe-Zn-O-(Cu,Pb) phase (lower right); (b) agglomerated and single Pb-Zn-O-(Fe) particles associated with Zn-O particle (arrows on right) and Pb-O-(Zn) phase trapped in slag; (c) bright Pb-O-(Cu) particle trapped in slag (center arrow) and multiple fine Sb-O-(Pb) particle phases trapped in slag (a few indicated).

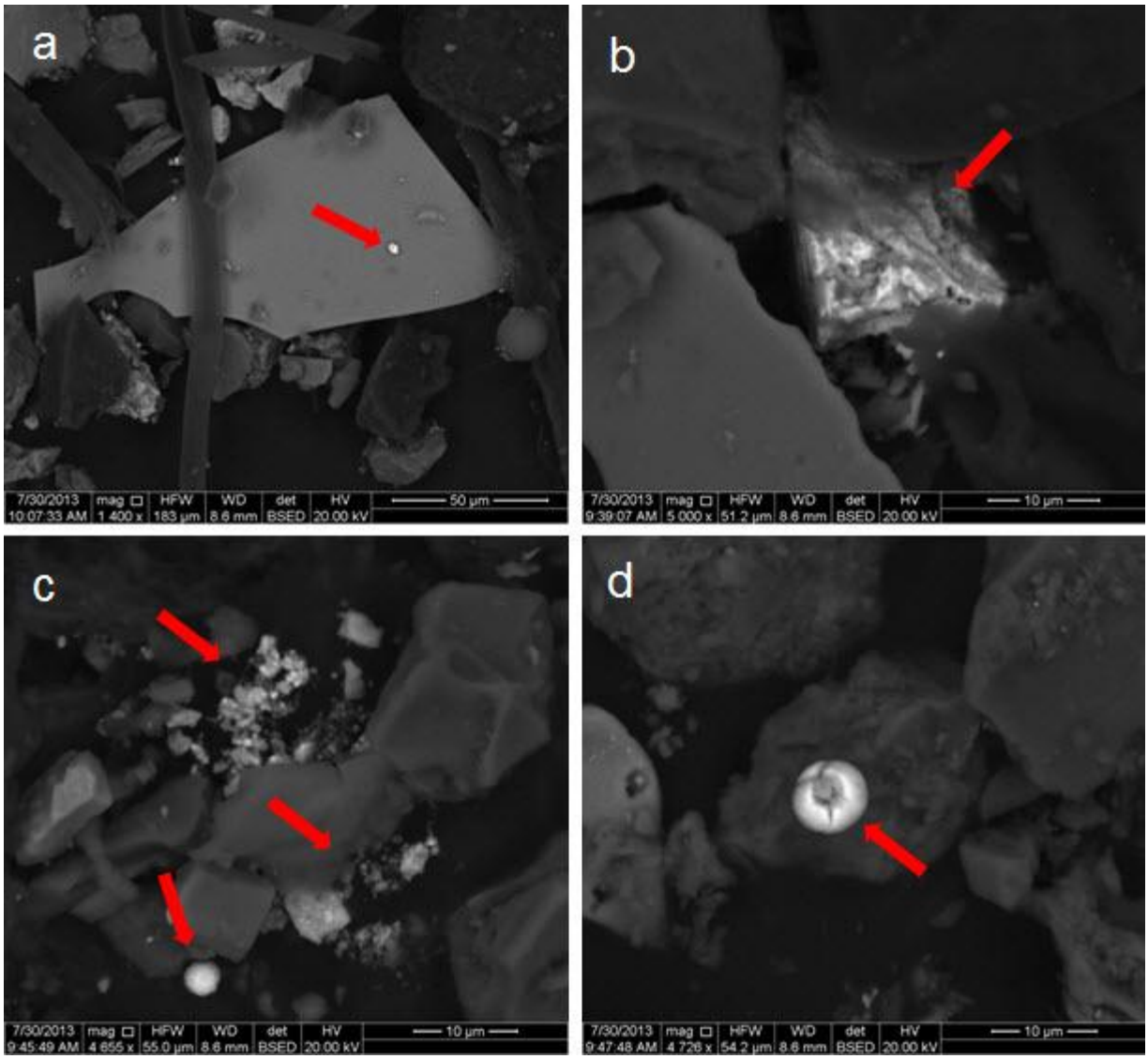


**Figure A10. R10 (Columbia Avenue)**: (a) Pb-O-(Sn) phase trapped in slag; (b) liberated Pb-Cu-O particle; (c) Pb-O particles of varying size, some agglomerated; (d) Pb-O particle associated with Si-O particle.

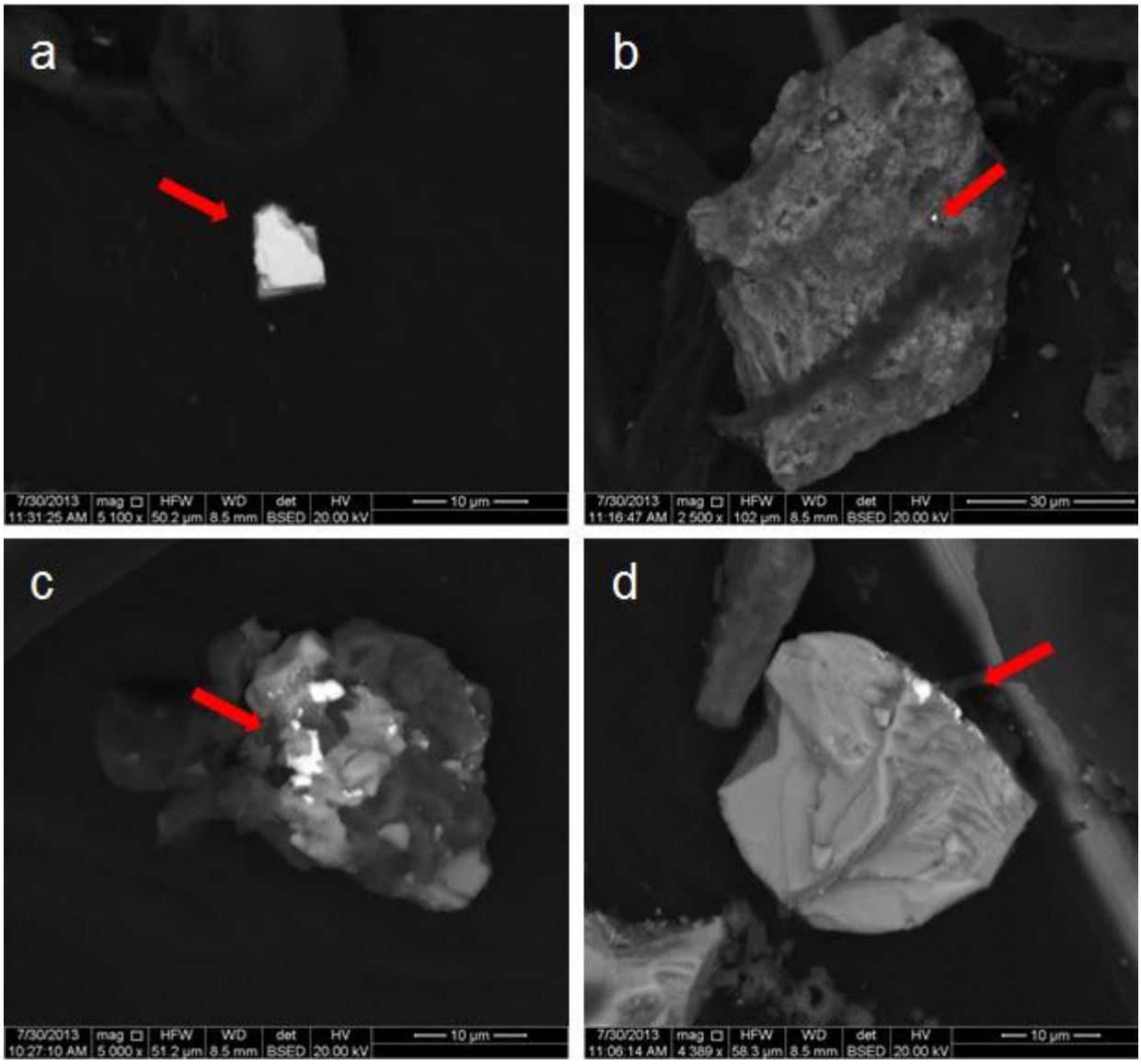


**Figure A11. R12 (Warfield)**: (a & b) Pb-O particle-phase can be liberated or trapped in such as Fe-O particle; (c) multiple (bright) Pb-O phases associated with Zn-S-O (light gray) and Si-O (dark gray) phases; (d) Pb-Zn-O phase associated with Zn-S-O particle.

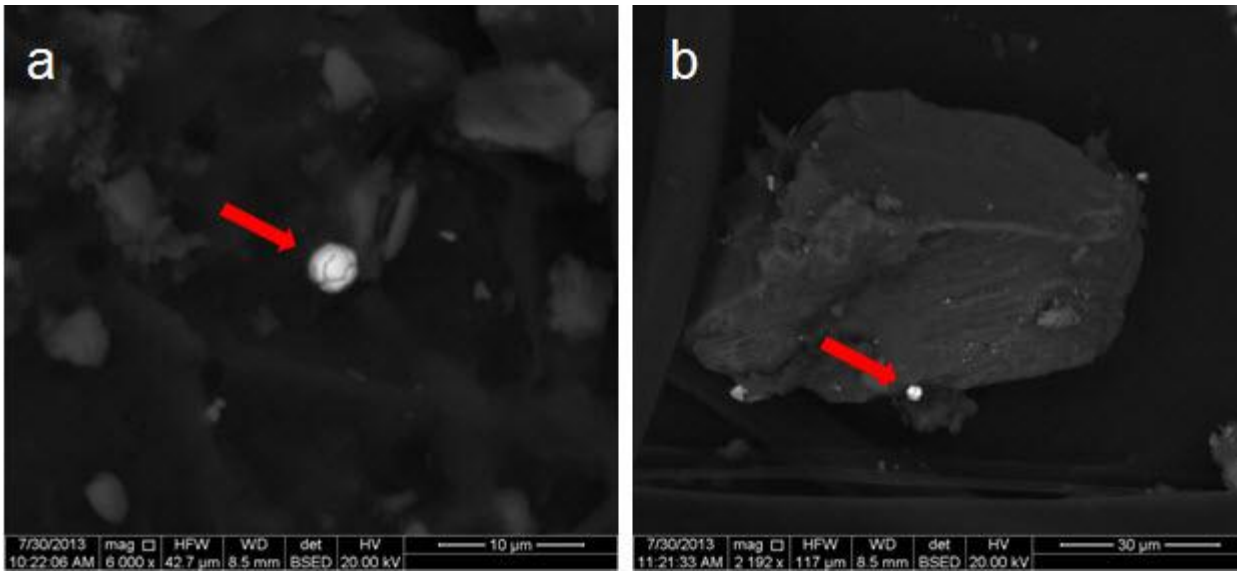


**Figure A12. R12 (Warfield)**: (a & b) Pb-Cd-O particle-phase liberated and associated with another phase, such as Si-O.

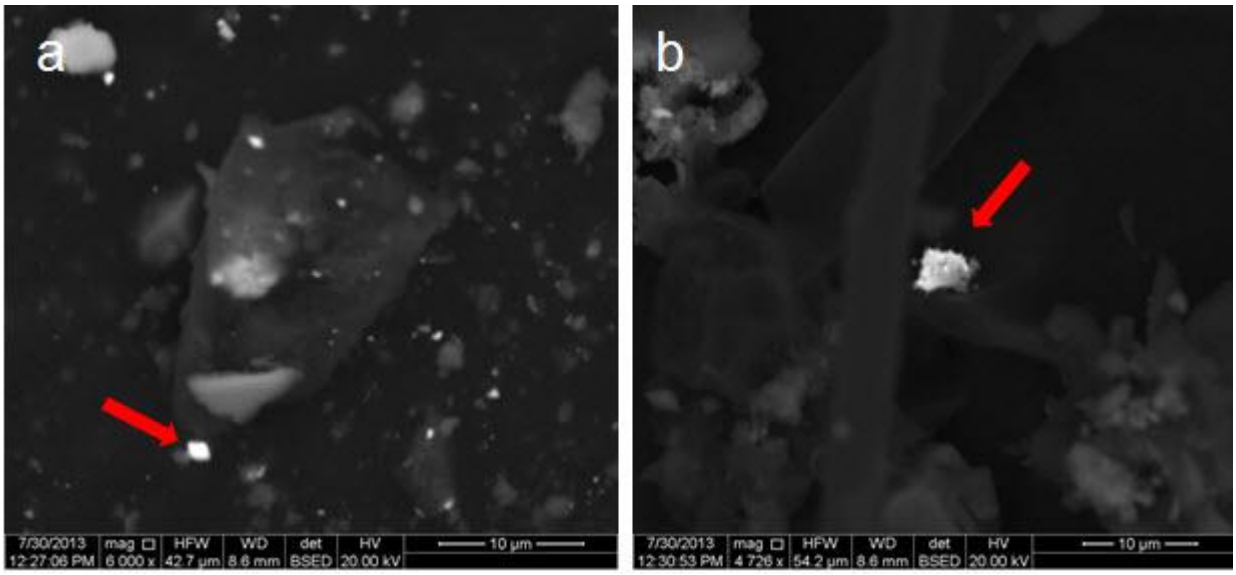


**Figure A13. R12 (Oasis)**: (a) rare Pb-Cl-O-(Ca) particle; (b) liberated Pb-O-(Sn) particle.

**Appendix 2.** Phases located in material from dustfall (jar) collectors.

Note: There were many particles in each sample. All occurrences of Pb-bearing particle-phases were not recorded (due to time considerations). Elemental compositions challenged the EDS due to incorrect take-off angle for elemental determinations. Elements in brackets refer to minor or trace presence.

| Element associated with Pb-O phase | Phase |
|---|---|
| Pure | Pb-O |
| Zn | Pb-Zn-O, Pb-O-(Zn), Pb-Zn-O-(Fe), Zn-Pb-Fe-O, Fe-Pb-Zn-O, Pb-O-(Zn, Fe), Pb-Zn-O-(Cu, Fe), Pb-O-(Zn, Cu, Fe), Pb-Fe-Zn-Cu-Sn-O, Pb-O-(Zn, Sn, Fe), Pb-Zn-O(Fe, Mn), O-Pb-Fe-Zn-Cu-S, Zn-Cu-Pb-Fe-S-O, Cu-Pb-S-Zn-O, Pb-Si-S-Cu-Fe-Zn-O, Pb-Si-Zn-Fe-O |
| As | Pb-O-(As), Pb-As-Sb-O, Pb-As-O-(Fe), Pb-Sn-O-(As), Pb-O-As-Sn-(Fe), Pb-As-Fe-Sn-Si-O |
| Sb | Pb-Sb-O, Sb-Pb-O, Pb-O-(Cu, Sb), Pb-O-(Sb), Sb-O-(Pb), Pb-Sb-Fe-O, Pb-Sb-O-(Fe), Pb-O-(Fe, Sb), Pb-O-(Fe, Cu, Sb), Pb-O-(Cu, Mn, Sb), Pb-Bi-Fe-O-(Zn, Sb), Pb-Au-Te-S-O-(Sb, Cu, Zn), Sb-Pb-O-(Fe, Si), Pb-Sn-O-(Sb, Fe) |
| Cd | Pb-Cd-O, Cd-Pb-O, Pb-Cd-S-O, Pb-Cd-Zn-S-O |
| Se, In | Pb-Cu-Se-O, Pb-In-O, Pb-O-(In) |
| S | Pb-S, Pb-S-O, Pb-O-(S), Pb-Cu-S-O, Pb-O-(S, Cu), Pb-Ag-Cu-S-O, Pb-Cu-O-(S), Pb-S-O-(Cu), Cu-S-O-(Pb), Pb-O-(Cu, S), Pb-O-S-(Fe), Pb-S-O-(Cu, Fe), Pb-O-(Fe, Cu, S), Cu-S-O-(Pb, Sn), Pb-Cu-S |
| Fe | Pb-Fe-O, Pb-O-(Fe), Fe-O-(Pb), Pb-Cu-Fe-O, Pb-Cu-O-(Fe), Pb-Fe-O-(Zn, Cu), Pb-O-(Cu, Fe), Pb-Sn-O-(Fe), Pb-Sn-Fe-O, Pb-O-(Sn, Fe), Fe-Pb-Sn-O, Pb-Sn-O-(Cu, Fe), Pb-O-(Sn, Cu, Fe), Pb-Fe-Mn-O, Pb-Fe-Si-O |
| Sn | Pb-Sn-O, Pb-O-(Sn), Pb-O-(Sn, Cu), Pb-O-(Mg, Cu, Sn) |
| Si | Pb-Si-O, Pb-O-(Si), Pb-Cu-Si-O, Pb-Mg-O-(Cu, Si), Pb-O-(Cu, Mg, Si) |
| Cu | Pb-Cu-O, Pb-O-(Cu), Cu-Pb-O, Cu-O-(Pb) |
| Other | Pb, Pb-O-(Ca), Pb-Cl-O-(Ca) |