

Morphological and Chemical Characteristics of Airborne Tungsten Particles of Fallon, Nevada

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Abstract: Morphological and chemical characteristics were determined for airborne tungsten particles in Fallon, Nevada, a town that is distinguishable environmentally by elevated airborne tungsten and cobalt. From samples of airborne dust collected previously at six different places in Fallon, tungsten-rich dust particles were isolated and analyzed with automated electron microprobe and wavelength-dispersive spectrometry. Representative W particles were further analyzed using transmission electron microscopy. Morphologically, Fallon W particles are angular and small, with minimum and maximum sizes of $\leq 1 \mu\text{m}$ and $5.9 \mu\text{m}$ in diameter, respectively. The number and size of tungsten-rich particles decrease in Fallon with distance from a hard-metal facility located near the center of town. Chemically, Fallon airborne W particles include mixtures of tungsten with cobalt plus other metals such as chromium, iron, and copper. No W-rich particles were identifiable as CaWO_4 (scheelite) or MnWO_4 (huebnerite). From d-spacings, Fallon particles are most consistent with identification as tungsten carbide. Based on these multiple lines of evidence, airborne W particles in Fallon are anthropogenic in origin, not natural. The hard-metal facility in Fallon processes finely powdered W and W-Co, and further investigation using tracer particles is recommended to definitively identify the source of Fallon's airborne tungsten.

Key words: Fallon, Nevada, airborne particles, tungsten, cobalt, automated electron microprobe analysis, transmission electron microscopy

INTRODUCTION

Morphological and chemical characteristics were determined for airborne tungsten particles of Fallon, Nevada (Fig. 1a), for the purpose of determining their source as anthropogenic or natural. Located 100 km east-southeast of Reno, Fallon has experienced a cluster of childhood leukemia since 1997 (Expert Panel, 2004; Nevada State Health Division, 2004). This cluster, deemed “one of the most unique ever reported” (Steinmaus et al., 2004), has prompted extensive research in an effort to find a cause, including population mixing (Kinlen, 2004; Kinlen & Doll, 2004) and/or environmental factors (Moore et al., 2002; U.S. Agency for Toxic Substances and Disease Registry, 2002, 2003a, 2003b, 2003c, 2004; U.S. Centers for Disease Control and Prevention, 2003a, 2003b; Seiler, 2004). Among other findings, a distinguishing environmental feature of Fallon is

elevated airborne tungsten (W) and cobalt (Co). As measured within total suspended particulates filtered directly from air, airborne W and Co can be significantly elevated over Fallon (Sheppard et al., 2006). Elevated W and Co has been confirmed within Fallon relative to outlying desert areas using chemistry of lichens in and around Fallon (Sheppard et al., 2007b). The source area of airborne W and Co particles has been narrowed down to near the center of Fallon, northwest of the intersection of the main highways (Fig. 1b), using chemistry of surface dust collected within Fallon (Sheppard et al., 2007a).

The question arises: What exactly is the source of the airborne W and/or Co in Fallon? One possible answer is a natural source. Geologically, west central Nevada is rich in W minerals, especially scheelite (CaWO_4) and huebnerite (MnWO_4) (Stager & Tingley, 1988). Hydrologically, W is found in west central Nevada surface water (Johannesson et al., 2000) and groundwater (Seiler et al., 2005). Because of this natural and widespread occurrence of W throughout the region, elevated W in Fallon has been suggested to be an artifact of Nevada's natural environment (U.S. Centers for Disease Control and Prevention, 2003a).

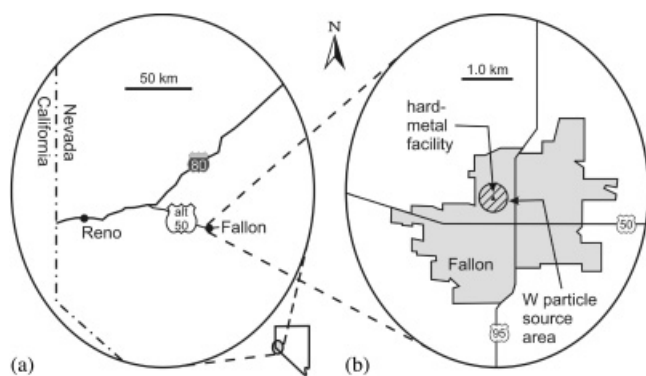


Figure 1. Maps of (a) west central Nevada showing the location of Fallon, and (b) Fallon (shaded polygon), showing the source area of airborne tungsten particles (hatched circle) and the location of the hard-metal facility (filled triangle).

An alternative explanation for the elevated airborne W in Fallon is an anthropogenic source. An industrial facility specializing in hard-metal metallurgy, which uses tungsten carbide and Co to produce tool materials (Harris & Humphreys, 1983), is located in the source area (Fig. 1b), and this hard-metal facility (HMF) has been named by the Nevada Division of Environmental Protection as a candidate source of W in Fallon (Mullen, 2003).

Morphological and chemical characteristics of airborne particulates can be used to distinguish natural particles from anthropogenic products. For example, automated electron microprobe analysis (AEMA) and transmission electron microscopy (TEM) can characterize individual particles by size and chemical makeup (Fletcher et al., 2001). Accordingly, the objective of this research was to characterize the morphology and chemical makeup of the airborne W particles of Fallon using microanalytical techniques, thereby answering the question of whether they are natural or anthropogenic (Seiler, 2006).

MATERIALS AND METHODS

Air Sampling

Airborne dust samples were collected within Fallon using portable, high-volume particulate air samplers (Sheppard et al., 2006). The filter type was glass-fiber, a common medium for high-volume sampling of airborne particulates (Eadie & Bernhardt, 1976; Lee & Mukund, 2001). Filters were 510 μm thick and had up to 99.99% retention for particles down to submicron in size (HI-Q Environmental Products Company, 2003).

For this study, six filters were selected from a sampling session that had 10 air samplers located throughout Fallon. The six filters were selected to optimize a transect of distance from the HMF in Fallon. The closest location was

Table 1. Electron Microprobe Analysis Conditions

Element and X-ray line	Wavelength dispersive spectrometer crystal	Standard
W-M α	Thallium acid phthalate	W metal ^a
Co-K α	Lithium fluoride	Co metal ^a
Mn-K α	Lithium fluoride	MnSiO ₃ ^b
Ca-K α	Pentaerythritol	CaCO ₃ ^b

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0.5 km away (hereafter known as “central Fallon”), and the most distant location was 13.4 km away (hereafter known as “Fallon outskirts”). The four intermediate locations were distributed between central Fallon and Fallon outskirts, with distances from the HMF of 1.31 km, 1.95 km, 3.79 km, and 6.68 km. The filters were submitted for particle microanalysis as blind samples.

Isolation of Tungsten Particles

To remove the collected particulate matter from the glass-fiber filters for microanalysis, a 2.0-cm \times 1.5-cm portion of each filter was placed into its own 50-ml plastic centrifuge tube with approximately 8 ml of ethanol (J.T. Baker, Phillipsburg, NJ). The tubes were sonicated for 20 min to dislodge the particles, and then the filter pieces were removed from the tubes and discarded.

Density separation was performed to concentrate heavy particles, including tungsten-rich materials, for microanalysis (Twenhofel & Tyler, 1941). Approximately 12 ml of methylene iodide, density = 3.3 g/ml (Geoliquids, Prospect Heights, IL) were added to each centrifuge tube, and the tubes were vortexed to mix the contents and then centrifuged for 10 min. The lighter ethanol fraction and a small portion from the surface of the heavier methylene iodide fraction were removed from each tube by pipette. The remaining heavy methylene iodide fraction was then filtered through 0.4- μm polycarbonate (PC) Nucleopore filters (Fisher Scientific, Hanover Park, IL), and the PC filters were mounted onto aluminum stubs and coated with \sim 20 nm of carbon. Preparation to this point was done in an ISO CL5 cleanroom. Blank glass-fiber and PC filters were similarly prepared for analysis.

Automated Electron Microprobe Analysis

An automated electron microprobe analysis was performed on the PC filters using a JEOL JXA-8200 Electron Microprobe and JEOL Automated Particle Analysis Program (JEOL Ltd., Tokyo, Japan). Analysis conditions for the microprobe included 20-kV accelerating voltage and 10-nA beam current. Selected fields on the filters were imaged using a backscattered electron detector to show atomic number

Table 2. Tungsten Particle Results from AEMA

Sample location (km from HMF ^a)	No. of particles analyzed using AEMA ^b	Tungsten particle count ^c	Maximum W particle size (μm) ^d	Tungsten particle types
0.50	965	337	5.9	W, W-Co, W-Co-Cr
1.31	1084	95	4.8	W, W-Co, W-Co-Cr
1.95	1000	47	2.7	W, W-Co, W-Co-Fe
3.79	1000	11	1.7	W, W-Co
6.68	1000	5	2.0	W, W-Co-Cr
13.4	1000	2	1.9	W
Total	6049	497		

^aHard metal facility.^bFor all samples, the same size of filter was analyzed, a rectangle 2.0×1.5 cm in dimension. Counts vary slightly due to the selection of analysis fields and times.^cTungsten particles are $>10\%$ tungsten (unnormalized) and have a tungsten mass fraction ≥ 0.7 .^dArea equivalent diameter.

contrast. Image resolution was 512×512 pixels, and image acquisition conditions and thresholds were set to detect high atomic number particles, including tungsten and high-Z minerals, down to sizes of $<1 \mu\text{m}$ in diameter. After image segmentation to define the particles present in the field, the electron beam was placed sequentially on each particle, and a 30-s wavelength-dispersive spectrometer (WDS) analysis was performed. Separate spectrometers were optimized and calibrated for measurement of tungsten, cobalt, calcium, and manganese (Table 1). AEMA was continued until approximately 1000 particles were evaluated for each Fallon sample.

After AEMA, W-rich particles identified in each sample were relocated in the electron microprobe and analyzed by energy-dispersive spectrometry (EDS) using a Thermo (Noran) Voyager EDS system. Manual EDS reanalysis was performed to confirm the results of WDS analysis, to check for additional elements in the particles, and to further identify the composition of the W-rich particles. The particles selected for manual reanalysis included those with the highest levels of Ca and Mn measured by AEMA, along with particles containing a range of Co contents.

Transmission Electron Microscopy

Several representative W particles found on the PC filters during AEMA were further analyzed using transmission electron microscopy. Additionally, material isolated from the air sampling filters was dispersed directly onto a carbon-coated copper grid, and W particles were located in the TEM, thereby allowing for analysis of somewhat smaller particles than those found by AEMA. This analysis was performed in a JEOL JEM-3010 TEM with a LaB_6 source operated at an accelerating voltage of 300 kV. Selected area electron diffraction (SAED) patterns were collected using a Gatan DualView $1.3 \text{ k} \times 1 \text{ k}$ anti-blooming CCD camera. Images were collected using a Gatan $2 \text{ k} \times 2 \text{ k}$ high resolution CCD camera.

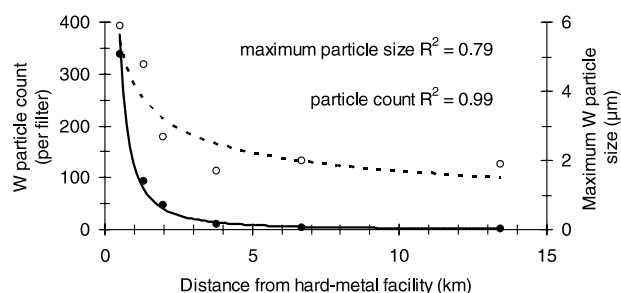


Figure 2. Tungsten particle counts per filter (filled circles) and maximum W particle size (open circles) within Fallon as a function of distance from the hard-metal facility. The same amount of filter was measured for each location. The power function models for particle count (solid line) and maximum particle size (dashed line) were calculated using all data points.

EDS analysis to confirm elemental identification of particles previously analyzed by AEMA and then relocated on TEM grids was performed using an Oxford INCA Energy system. EDS analysis was also done to identify the elemental composition of particles analyzed only in the TEM.

SAED patterns were collected from whole particles or thin edge areas and consisted of single or multiple crystal spot patterns. Atomic plane spacings (d-spacings) obtained by measurement of the diffraction patterns were matched using the PDF-4+ 2005 version of the crystallographic database maintained by the International Centre for Diffraction Data. Phases searched included tungsten metal, tungsten carbides, tungsten oxides, and hydrated phases. For any given phase, a database file representative of the majority for that phase was selected for final comparison. Database files with S or I designations, the two highest quality marks, were used for comparison.

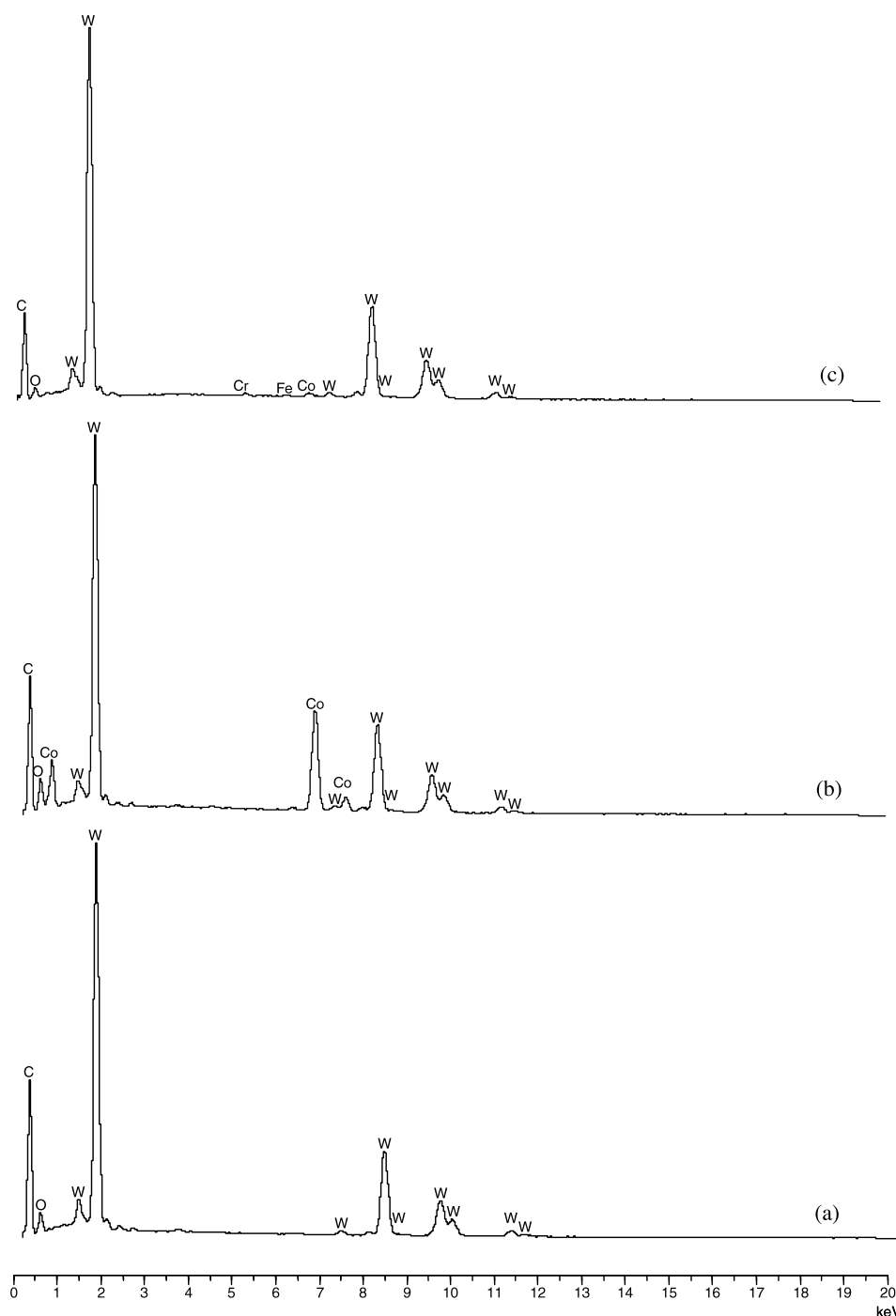


Figure 3. Representative EDS spectra of W particles found by AEMA and relocated for analysis. W particle (a), W-Co (b) particle, W-Co-Fe-Cr particle (c).

RESULTS AND DISCUSSION

AEMA

In this study, W-rich particles are defined as particles with >10% tungsten by weight (unnormalized) and a mass fraction of tungsten of ≥ 0.7 . By these criteria, 497 of the 6,049

particles analyzed are W rich (Table 2). Highly variable numbers of W-rich particles were found on the Fallon filters depending on their distance from the HMF. Using the HMF as a starting point, which is not to claim that it is the source of the W particles but rather just to use its location as a starting point, the numbers of W particles found per filter closely follow a power function of distance (Fig. 2),

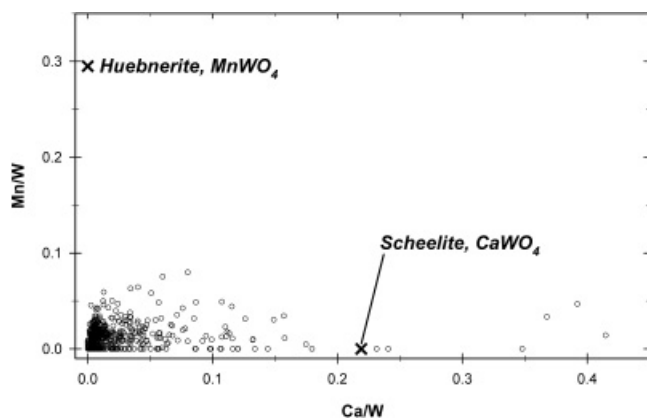


Figure 4. Relationship of Ca/W and Mn/W weight ratios for W particles analyzed by AEMA. Reference points for the tungsten minerals scheelite (CaWO_4) and huebnerite (MnWO_4) are included. Data are presented as ratios of weight percents to reduce particle size effects on the measured X-ray intensities (Goldstein et al., 1992), (number of particles = 497).

confirming a similar power function with distance for loading of airborne W in Fallon (Sheppard et al., 2006).

Morphologically, these W particles are consistently small. The minimum W particle size on all filters is $\leq 1 \mu\text{m}$ in diameter, and the maximum W particle size is $5.9 \mu\text{m}$, found on the central Fallon filter (Table 2). Maximum W particle size on each filter decreases with distance from the HMF, again showing a power function of distance (Fig. 2). Given that large particles do not stay aloft as long as small particles (Pye, 1987), this pattern conforms to the suggestion that the area just northwest of the main crossroads of Fallon (Fig. 1b) is the source area of airborne W particles in Fallon (Sheppard et al., 2007a). In contrast to this consistent small size of the Fallon W particles, natural particles of scheelite and huebnerite can be much larger, up to $100 \mu\text{m}$ in length (McCrone et al., 1967; McCrone & Delly, 1973).

Chemically, EDS particle reanalysis shows that Fallon airborne W particles found by AEMA include mixtures of tungsten with cobalt plus other metals such as Cr, Fe, and Cu (Table 2; Fig. 3). A total of 77 of the 497 W-rich particles had major Co ($\text{Co/W} > 0.06$ by weight), with Co/W ratios in the W-rich particles ranging from 0 to 1.7. The association of W and Co in airborne particles could explain their temporal covariance within Fallon (Sheppard et al., 2006). In contrast to this co-occurrence of especially Co with Fallon W particles, natural particles of scheelite and huebnerite do not contain Co or Cr or Cu (McCrone & Delly, 1973).

None of the W-rich particles contains a Mn/W ratio indicative of huebnerite, and only a small number of W-rich particles have a Ca/W ratio near or exceeding that of scheelite (Fig. 4). A total of 29 of the W-rich particles have Ca/W ratios above 0.1, but approximately half of these particles also contain cobalt. Manual EDS checks of W-rich,

Ca-containing particles after AEMA revealed that the Ca was found in secondary material attached to or near the W-rich particles; no W-rich particles of Fallon were identifiable as CaWO_4 (scheelite) or MnWO_4 (huebnerite).

Based on these counts and size patterns of the airborne W particles of Fallon as well as their chemical compositions, they are anthropogenic. Fallon airborne W particles are primarily W metal or compounds, mixed with Co. Tungsten does not occur naturally as a pure metal (U.S. Agency for Toxic Substances and Disease Registry, 2005), and W-Co particles are a product of hard-metal metallurgy (Harris & Humphreys, 1983). None of these particles relocated and manually analyzed by EDS can be confirmed as natural W minerals.

TEM

TEM images of representative tungsten-containing particles show them to be angular as opposed to round (Fig. 5). By reference comparison, metal grindings can be irregular and rough shaped with sharp edges (McCrone et al., 1967). Corresponding EDS spectra from these particles confirm that they contain Fe, Co, or Cr as associating metals, with no evidence of Ca or Mn to indicate scheelite or huebnerite (Fig. 5). Fe, Co, and Cr were not found in EDS spectra collected from the carbon support film directly adjacent to the particles, indicating that the metals are associated with the W particles and the metal peaks do not result from X-ray scatter elsewhere on the sample grid.

D-spacings obtained from measurement of SAED patterns are consistent with identification as tungsten carbide, WC (Table 3), which is an anthropogenic product of hard-metal metallurgy (Harris & Humphreys, 1983). D-spacings for these and other particles not matched by WC could be matched by tungsten oxide, WO_3 (PDF No. 01-089-1287), or by another tungsten carbide phase, W_2C (PDF No. 01-089-2371). These phases also have d-spacings that are very close to those for WC, and it is probable that small amounts of other phases are present in the particles. However, spacing tables for WO_3 and W_2C contain many more d-spacings than those for WC. Overall, the experimental data are most consistent with identification as WC, both in terms of the number of d-spacings and the match to high intensity spacings in the database files.

CONCLUSIONS

Airborne W particles of Fallon, which can be significantly elevated in central Fallon relative to comparison towns (Sheppard et al., 2006) as well to outlying desert areas (Sheppard et al., 2007b) and the outskirts of Fallon (Sheppard et al., 2007a), are anthropogenic in origin, not natural. An industrial facility exists in Fallon that processes finely powdered W and W-Co, and it has been considered a candidate source of the elevated W in Fallon (Mullen, 2003;

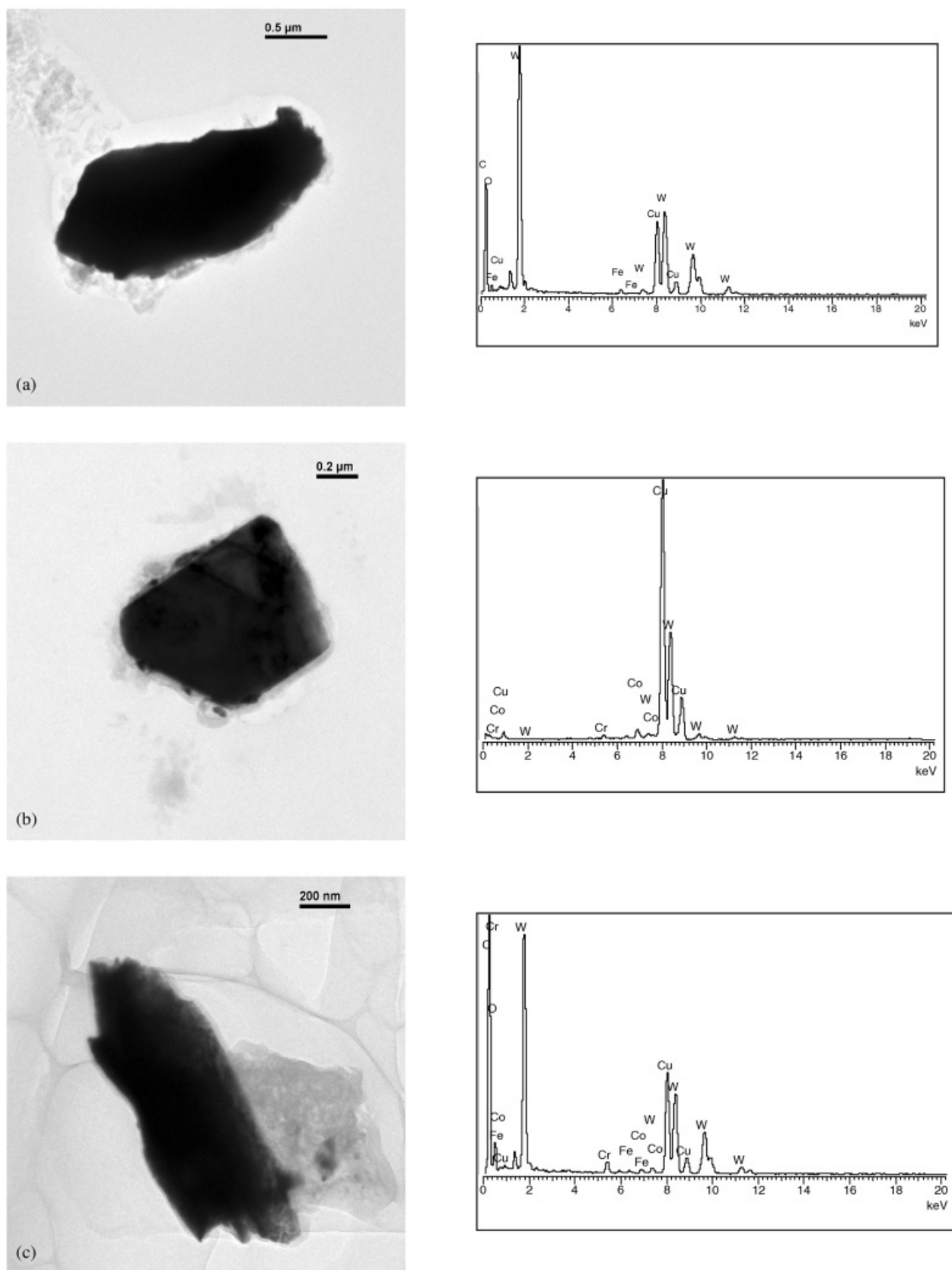


Figure 5. Representative TEM images and corresponding EDS spectra of W particles. Cu peaks arise from TEM grid. W-Fe particle (a), W-Co-Cr particle (b) (located near grid bar; absence of peaks below 2 keV due to absorption of low energy x-rays), W-Co-Fe-Cr particle (c).

Table 3. TEM SAED Results for Selected Representative Tungsten Particles

Particle measured d-spacings (Å)			ICDD database spacing table for WC (PDF No. 04-002-2679)	
A	B	C	d-Spacing (Å)	Intensity
			2.8365	36
2.51	2.52	2.47	2.5167	89
1.90		1.88	1.8825	100
		1.79 ^a		
1.46	1.45	1.45	1.4530	25
			1.4183	8
1.29		1.28	1.2932	28
1.25	1.26		1.2583	14
	1.25	1.25	1.2356	25
1.14		1.14	1.1502	22
	0.95	0.95	0.9512	12

^aMatched by WO₃, PDF No. 01-089-1287.

Sheppard et al., 2006). However, the facility claims zero external emissions (Goodale, 2005). Further investigation, for example an environmental analysis using tracer particles (Heiken, 1986), is recommended to resolve this question.

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