

Analytical Report

Unknown Identification

Prepared by
Jackie Ranger

Prepared for
Oak Ridge Associated Universities (ORAU)
SwRI Project Number 21611.06.001
SwRI Task Order Number 150928-2

INTRODUCTION

One solid sample was received at SwRI on September 25, 2015 for unknown identification. The sample was logged into SwRI's Laboratory Information Management System (LIMS) under SRR #56224 and was assigned to task order #150928-2. Table 1 contains the sample information. A photo of the sample is provided in Figure 1.

Table 1 Sample Information

SwRI System ID	Customer Sample ID
583499	5184S0122

EXPERIMENTAL

Three analytical techniques were used to aid in the identification of the unknown sample. They were Fourier Transform Infrared Spectroscopy (FTIR), Energy Dispersive Spectrometry - X-ray Fluorescence (EDS-XRF), and X-ray Diffraction (XRD).

FTIR

A portion of the sample was extracted with heptane to solublize the black, asphalt/tar-like substance. Approximately 50 μ L of the extract was evaporated on a KBr Real Crystal® IR sample card. The infrared spectrum for the organic (heptane soluble) fraction of the sample was acquired from 650-4000 cm^{-1} using a Nicolet 560 Fourier Transform Infrared Spectrophotometer. The FTIR spectrum with the wavelengths of the major peaks identified are presented in Figure 2.

EDS-XRF

Heptane was added to another portion of the sample to separate the mineral, rock-like materials from the black substance. Optical photos are provided of the resulting solids in Figures 3, 4 and 5.

Solids that represent the major components were randomly selected, and subjected to EDS-XRF analysis using a scanning electron microscope (SEM). EDS-XRF identifies the elemental composition of a sample limited to the first 10 to 100 micron depth. EDS-XRF only quantitates elements of atomic numbers greater than 11 (sodium and higher), and is normalized to 100%. There are carbon and oxygen peaks identified on all spectrograms. However, carbon and oxygen, have atomic numbers of 6 and 8, respectively, and are not included in the quantitation reports.

The EDS spectra with the quantitation tables are presented in Figures 6, 7, and 8. The particles are identified as Particle 1 (ID: JFS1729 and JFS7129), Particle 2 (ID: JFS1730), and Clear Particle (ID: JFS1731). These particles are identified in the BSE image provided in Figure 9.

XRD

X-ray Diffraction was performed on the solids obtained after washing with heptane. The fraction analyzed consisted of the loose material and one of the larger pebbles. It was analyzed utilizing a random mount orientation. Figure 10 is the XRD spectra obtained, including the XRD library pattern matches.

CONCLUSION

Sample 5184S0122 was separated into two fractions in order to identify its composition. Heptane was used to isolate the organic constituents (black substance) from the inorganic fraction (rock-like material).

The FTIR analysis (refer to Figure 2) of the black substance identified peaks at $2850\text{--}3000\text{ cm}^{-1}$, $1400\text{--}1500\text{ cm}^{-1}$ and 725 cm^{-1} . This is indicative of a long-chained aliphatic hydrocarbon.

From the EDS-XRF spectrograms (refer to Figures 6, 7, and 8) of the rock-like materials, Particle 1 contains mainly aluminum and iron. Particle 2 consists mostly of iron, and the Clear Particle is mainly silicon. It appears that the mineral fraction is mostly river sand (quartz particles) with pieces of aluminum oxide and iron corrosion flakes. This is confirmed by the XRD results, which identified it as consisting of greater than 99% silica.

The piece of sample 5184S0122 was examined. It was determined that a layer of roofing felt material was visible on one side. An area of the roofing felt material was removed. The removed material was easily torn to expose the inner fibrous material. Optical photos are presented in Figures 11, 12, and 13. Based on the visual assessments, Sample 5184S0122 appears to be consistent with roofing material.

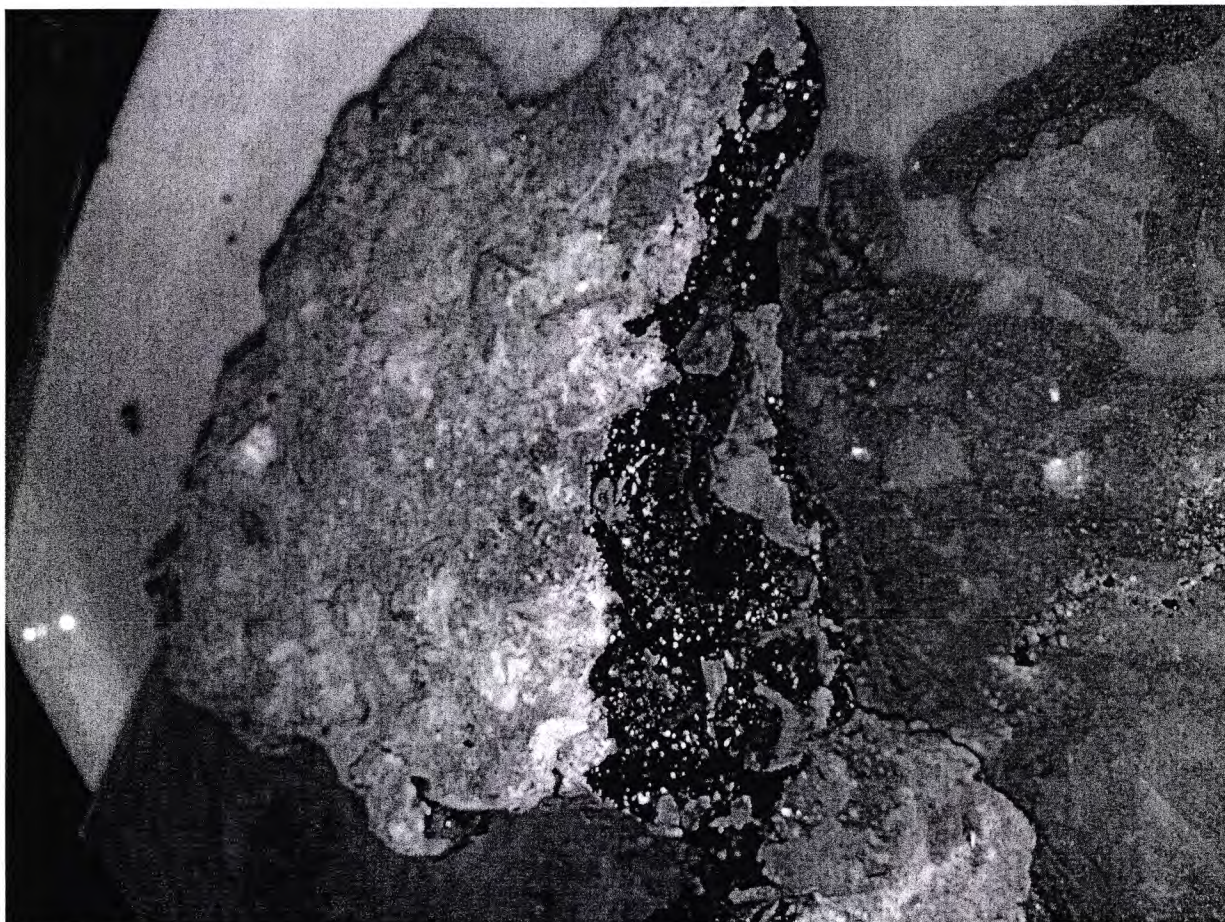


Figure 1. Photo of Sample 5184S0122

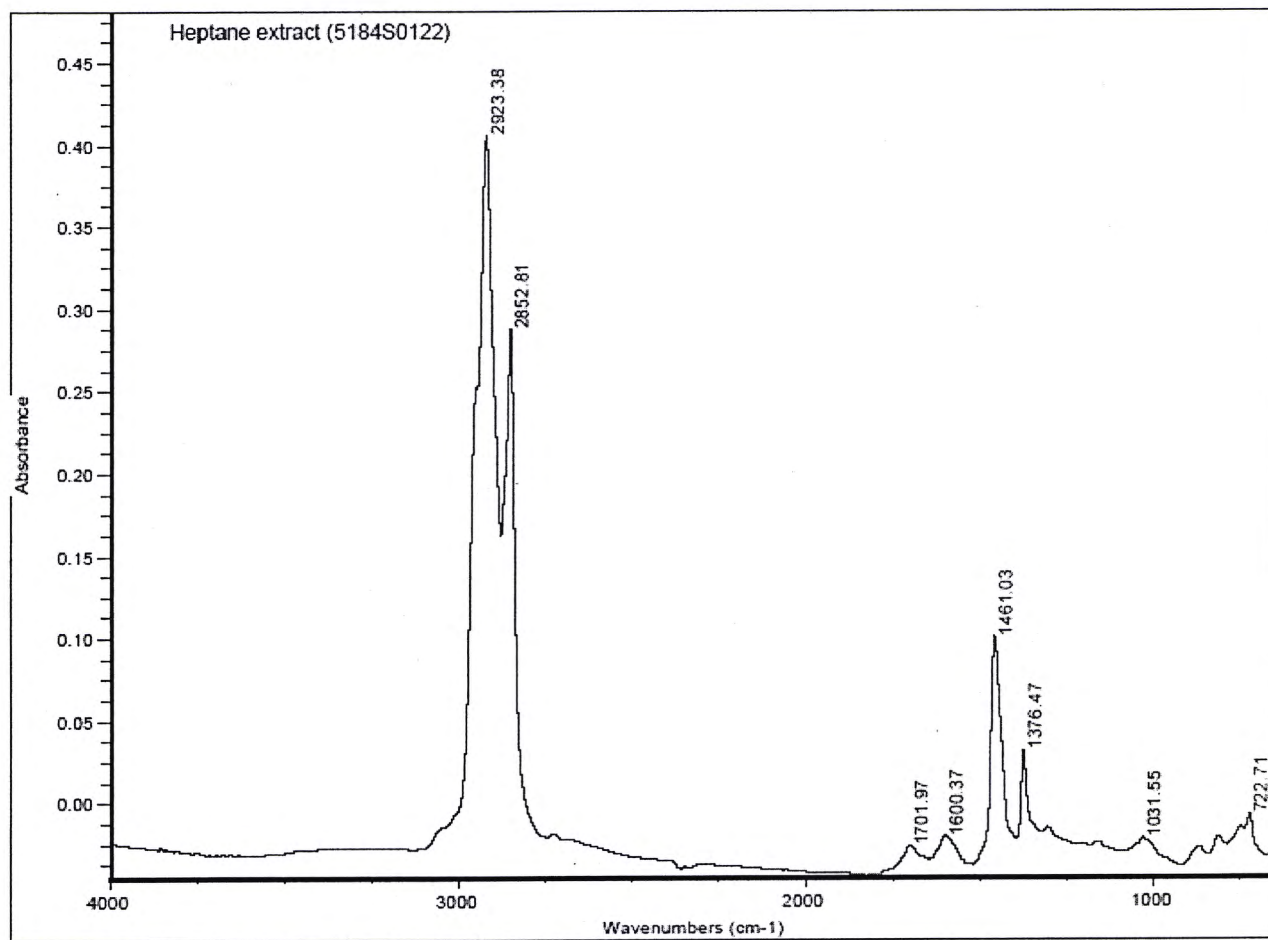
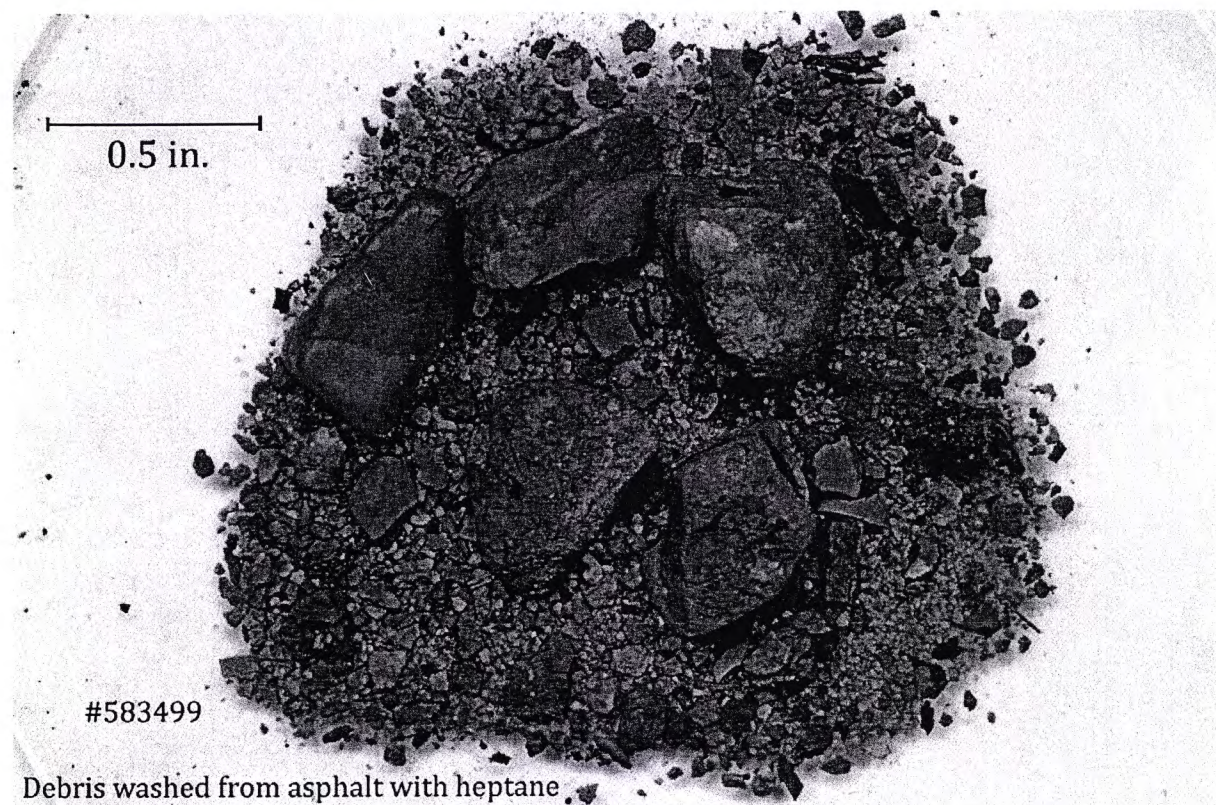


Figure 2. FTIR spectrum of heptane extract of Sample 5184S0122.



Debris washed from asphalt with heptane.
Figure 3. Optical photo of heptane washed solids from Sample 5184S0122.

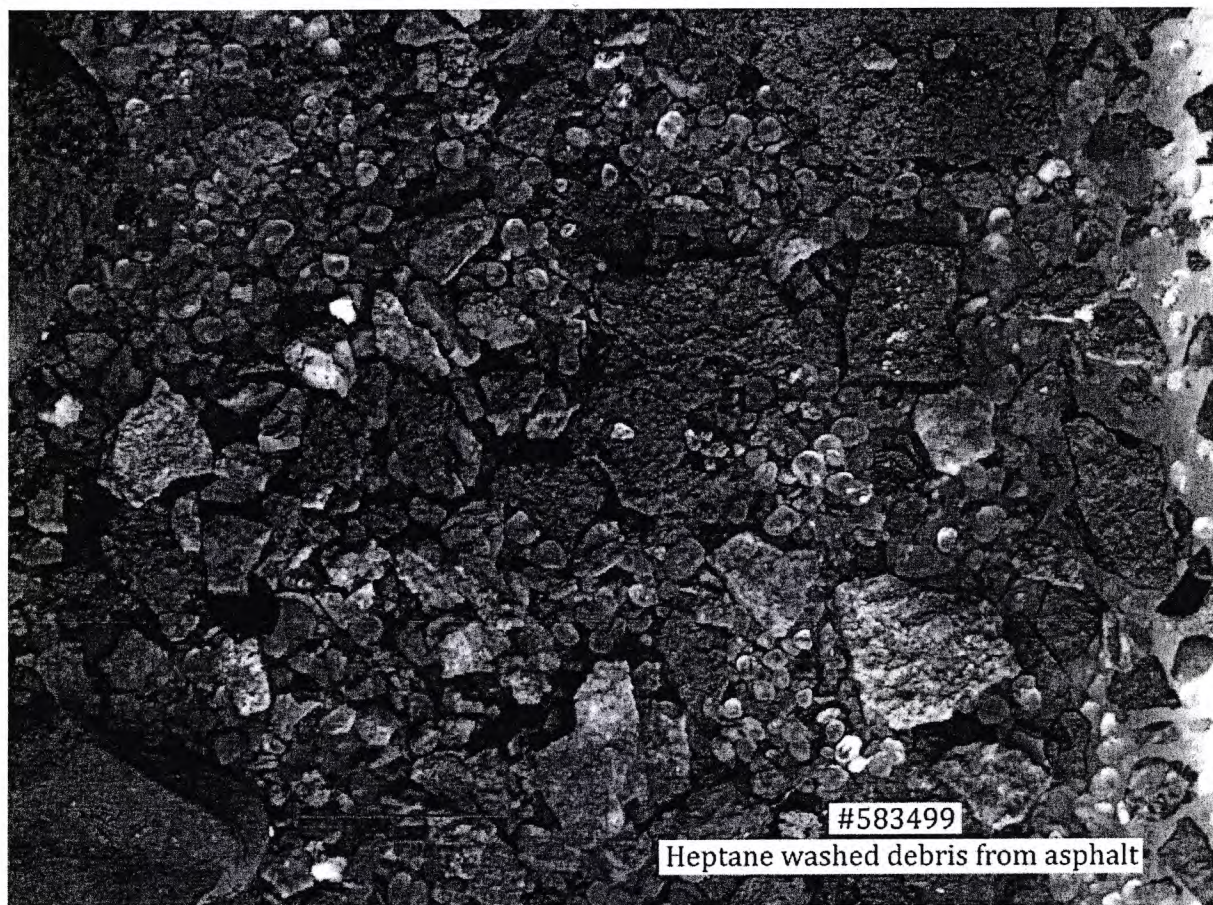


Figure 4. Optical photo of heptane washed solids from Sample 5184S0122.

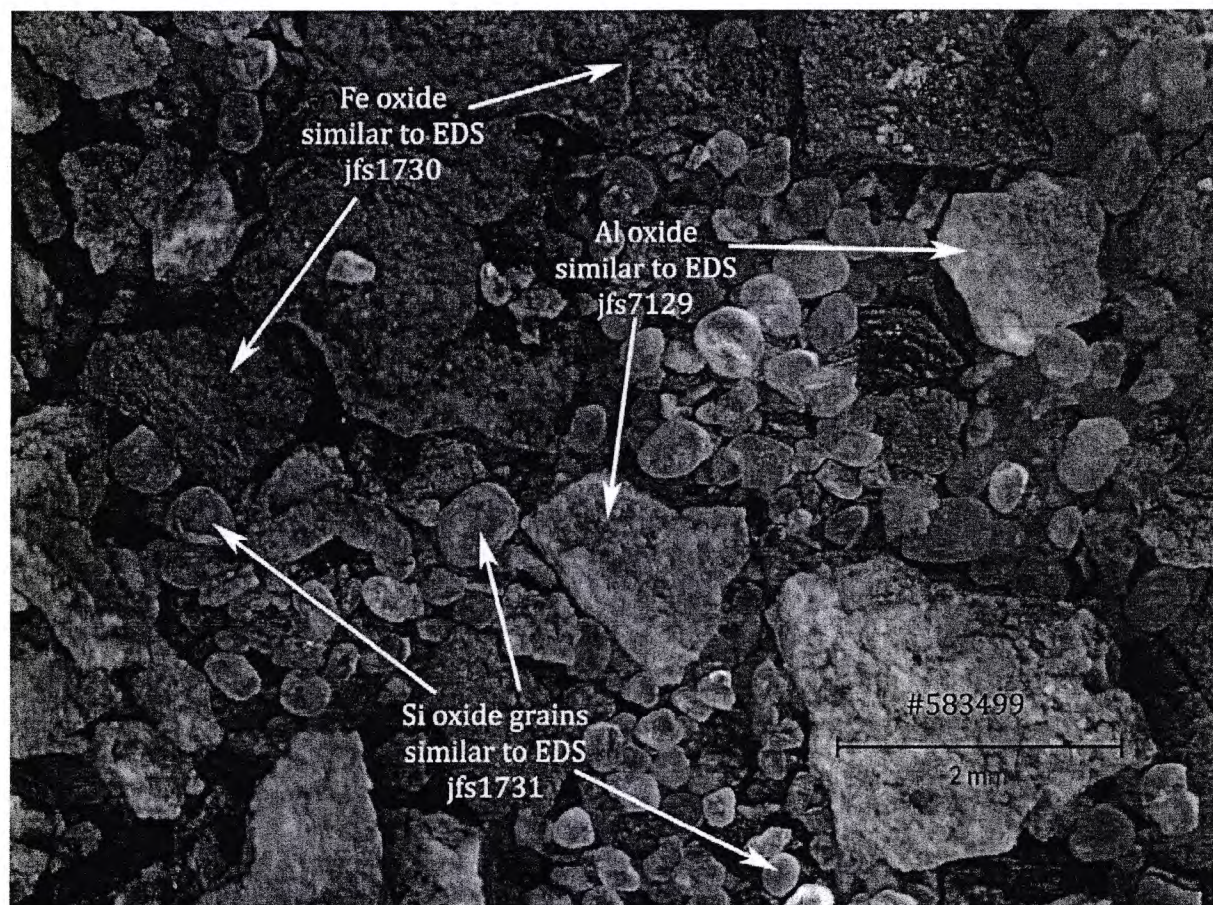
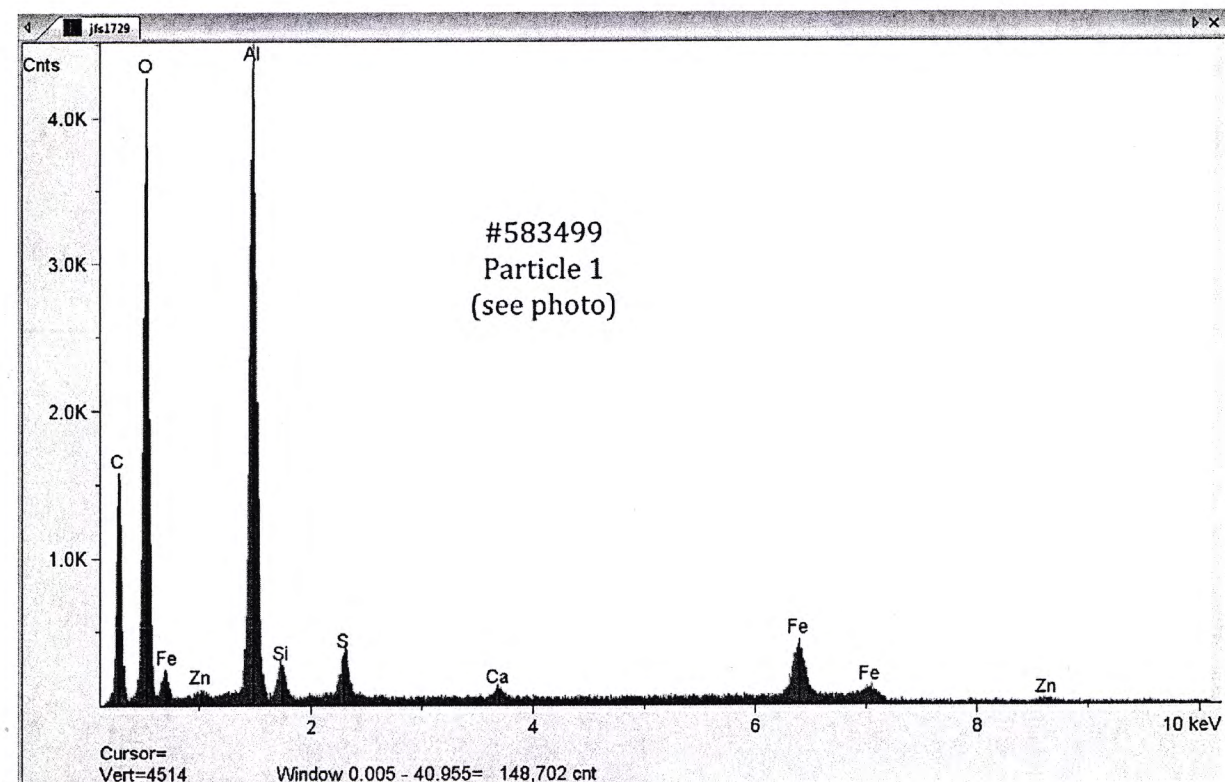


Figure 5. Optical photo of heptane washed solids from Sample 5184S0122.



Elt.	Line	Intensity (c/s)	Atomic %	Conc	Units	
Al	Ka	119.05	71.59	61.70	wt.%	
Si	Ka	6.79	7.63	6.84	wt.%	
S	Ka	10.94	7.26	7.44	wt.%	
Ca	Ka	2.70	1.20	1.54	wt.%	
Fe	Ka	17.95	10.63	18.96	wt.%	
Zn	Ka	1.56	1.68	3.51	wt.%	
			100.00	100.00	wt.%	Total

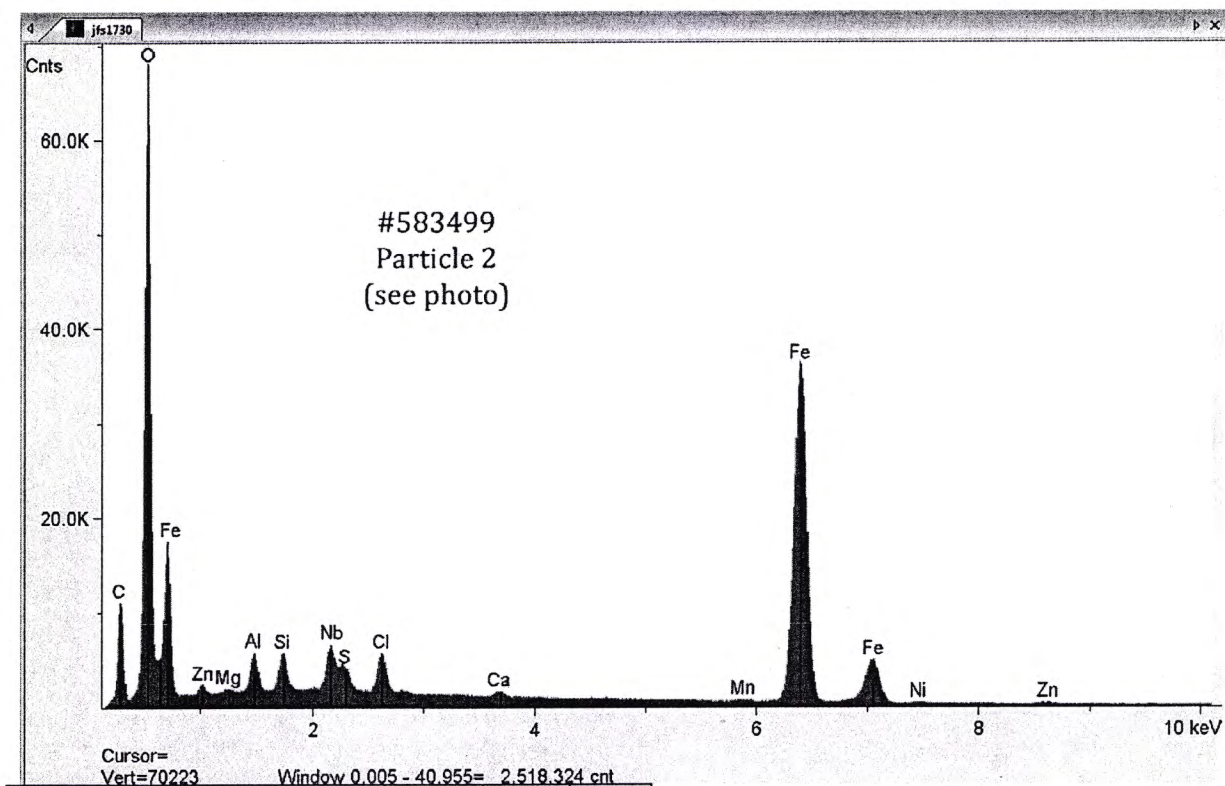
kV 20.0

Takeoff Angle 23.0°

Elapsed Livetime 300.0

Note: Results do not include elements with Z<11 (Na).

Figure 6. XRF spectrogram and analysis report for Particle 1 of heptane washed solids from Sample 5184S0122 (ID: JFS1729 and JFS7129, Refer to optical photo Figure 4 and BSE image Figure 8).



Elt.	Line	Intensity (c/s)	Atomic %	Conc	Units	
Mg	Ka	9.87	1.20	0.57	wt.%	
Al	Ka	108.54	8.67	4.60	wt.%	
Si	Ka	114.25	7.00	3.87	wt.%	
S	Ka	51.40	1.99	1.25	wt.%	
Cl	Ka	144.73	5.19	3.62	wt.%	
Ca	Ka	20.81	0.61	0.48	wt.%	
Mn	Ka	5.33	0.19	0.21	wt.%	
Fe	Ka	1,719.39	70.40	77.35	wt.%	
Ni	Ka	4.60	0.28	0.32	wt.%	
Zn	Ka	10.18	0.81	1.05	wt.%	
Nb	La	155.62	3.65	6.67	wt.%	
			100.00	100.00	wt.%	Total

kV 20.0

Takeoff Angle 23.0°

Elapsed Livetime 300.0

Note: Results do not include elements with Z<11 (Na).

Figure 7. XRF spectrogram and analysis report for Particle 2 of heptane washed solids from Sample 5184S0122 (ID: JFS1730, Refer to optical photo Figure 4 and BSE image Figure 8).

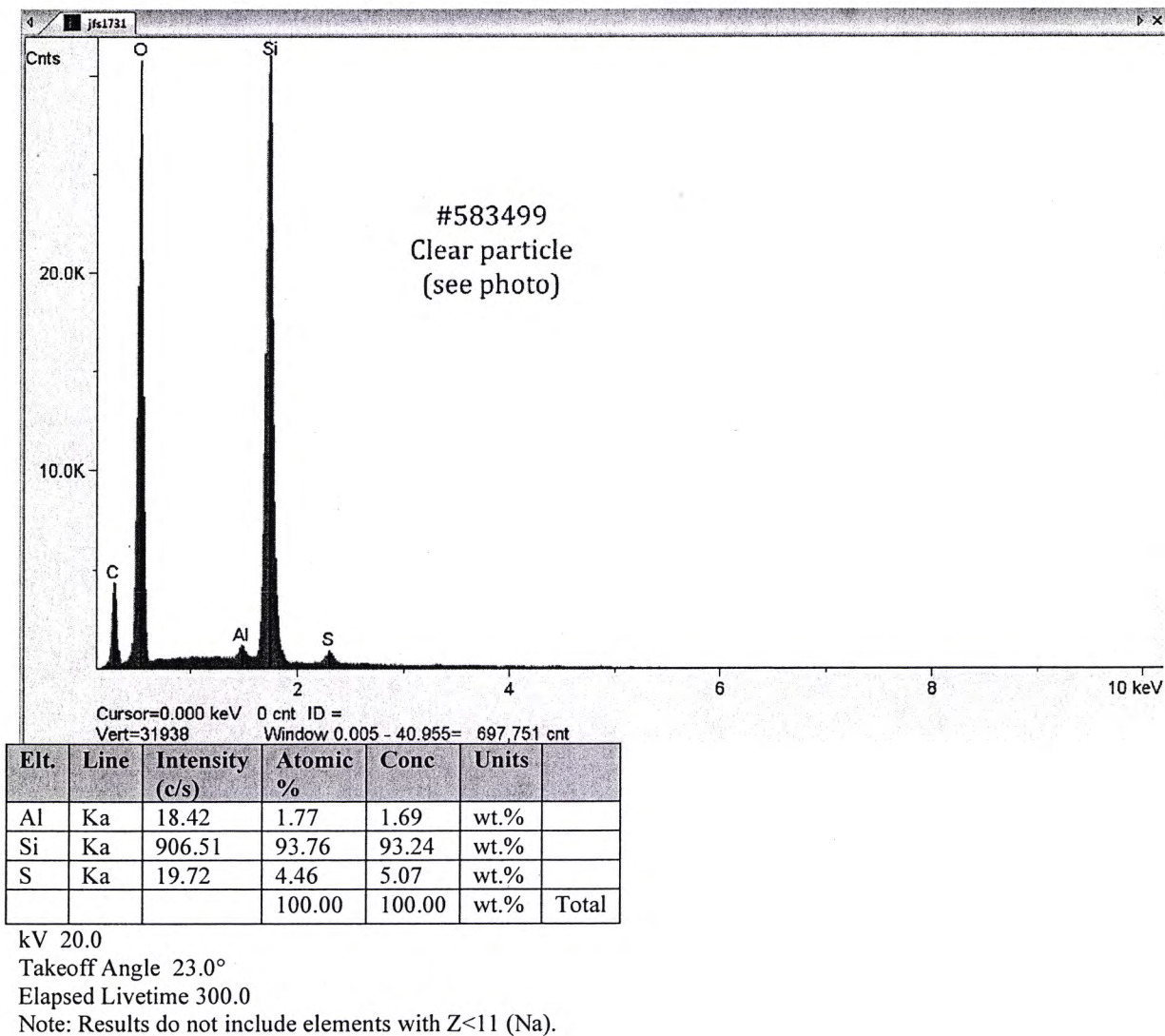


Figure 8. XRF spectrogram and analysis report for Clear Particle of heptane washed solids from Sample 5184S0122 (ID: JFS1731, Refer to optical photo Figure 4 and BSE image Figure 8).

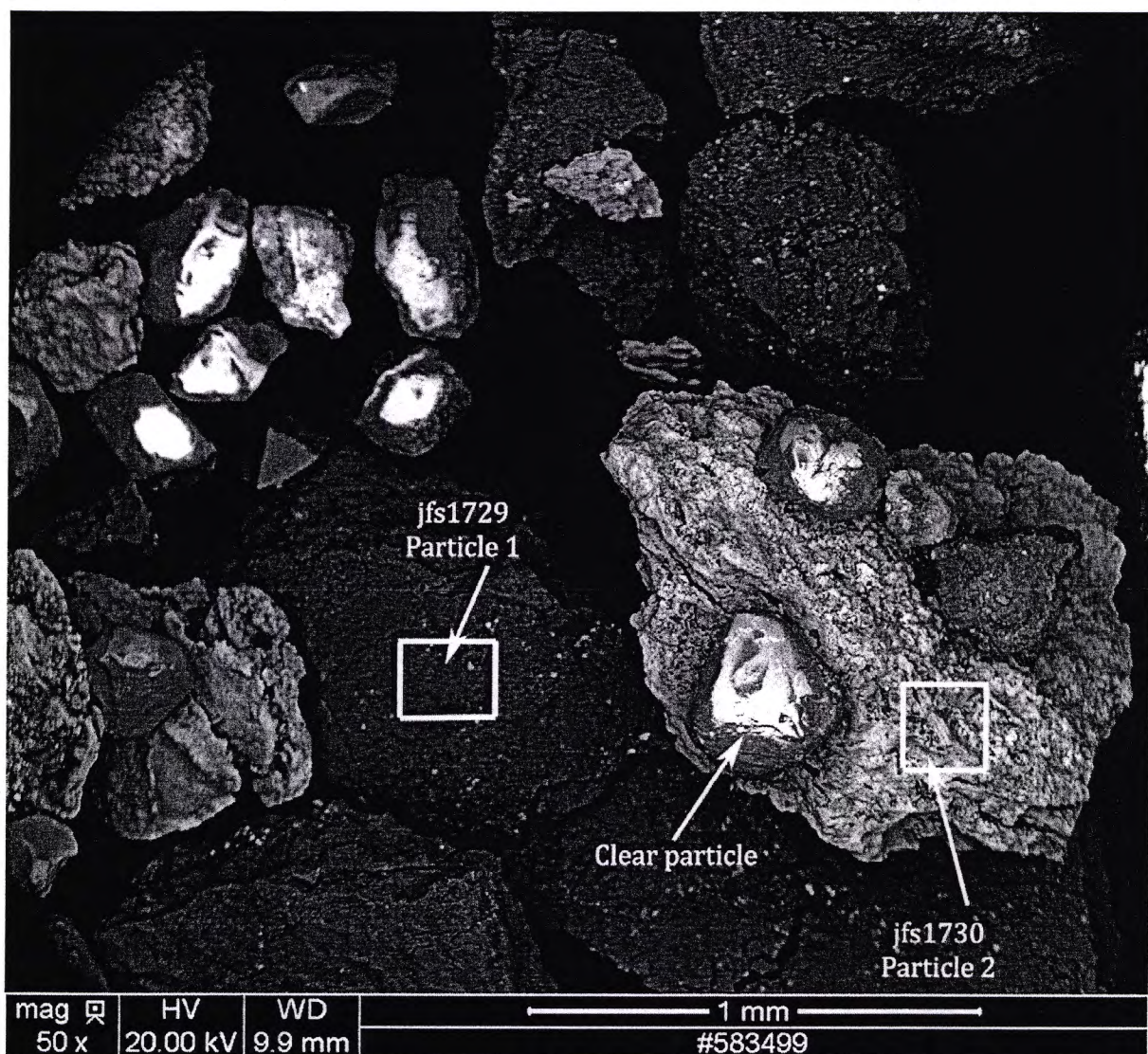
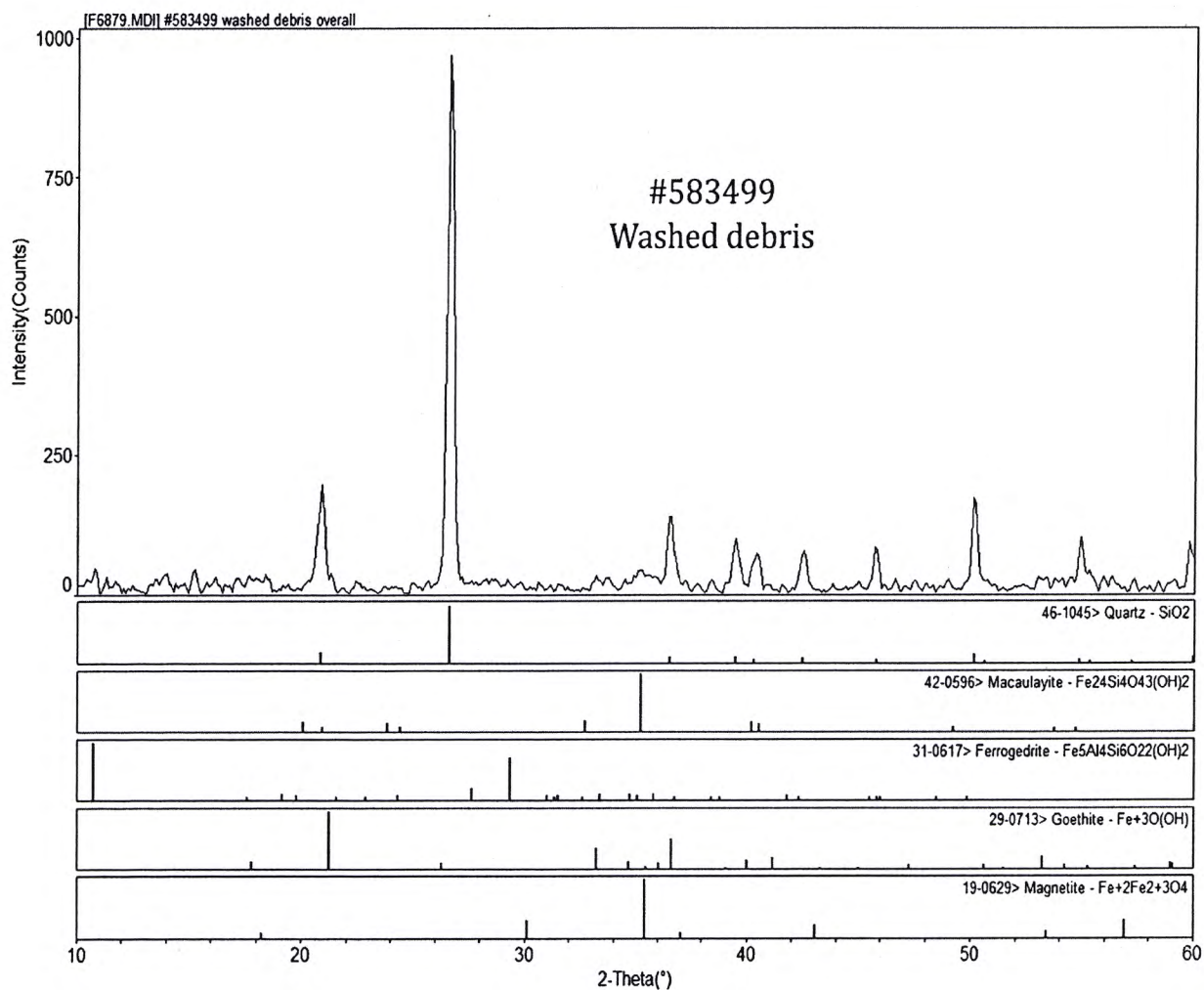


Figure 9. BSE image of the heptane washed solids from Sample 5184S0122.



Southwest Research Institute

[JSPENCER7]jspencer[C:\Users\jspencer\Documents\SwRI data\XRD\data> Thursday, Oct 01, 2015 03:22p (MDI/JADE5)

Figure 10. XRD of the heptane washed solids from Sample 5184S0122.



Figure 11. Optical photo of a piece of Sample 5184S0122.

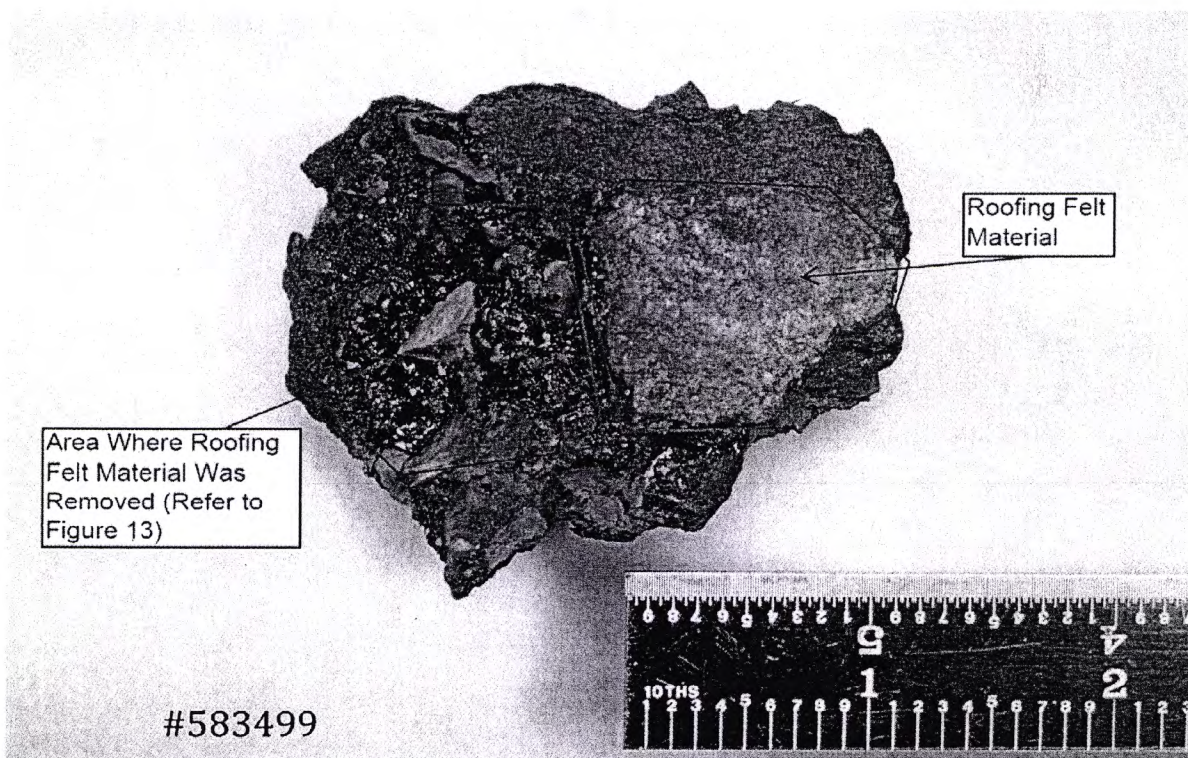


Figure 12. Optical photo of a piece of Sample 5184S0122 with identification.

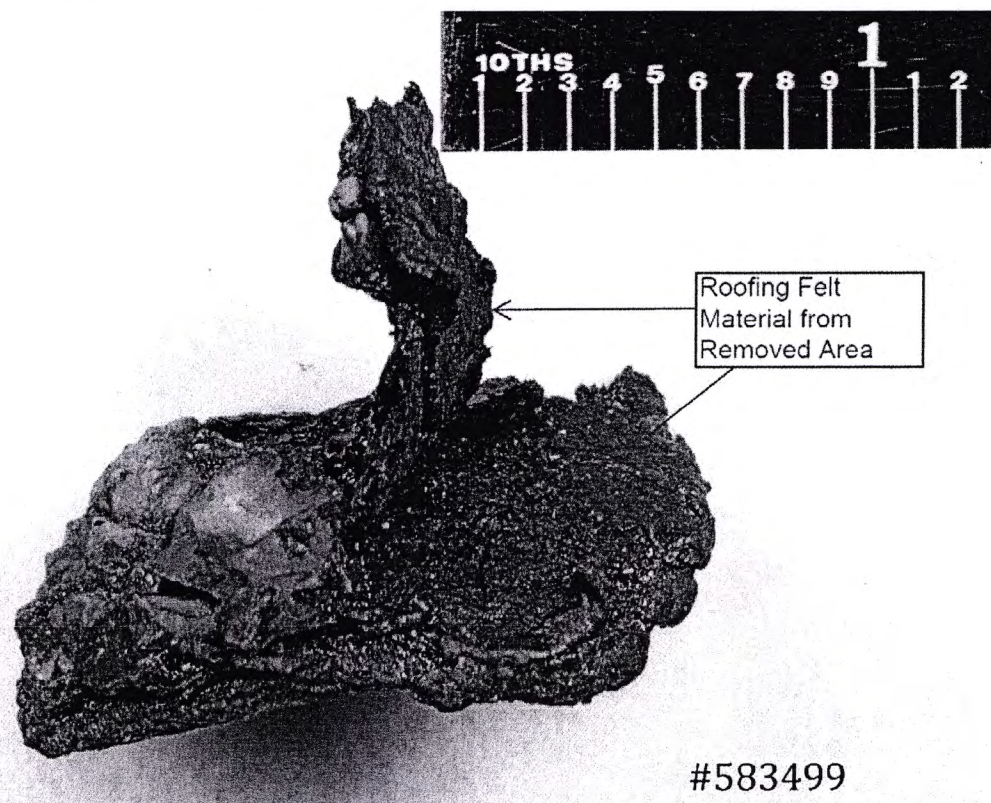


Figure 13. Optical photo of torn roofing felt material removed from a piece of Sample 5184S0122.