

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION 10 LABORATORY 7411 Beach Drive East Port Orchard, Washington 98366

MEMORANDUM

SUBJECT:Data Release for Asbestos Results from the U.S. EPA Region 10 LaboratoryPROJECT NAME:Storedahl Quarry Site Reconnaissance Sampling and Analysis
Yacolt, Washington

- PROJECT CODE: OCE-029A
- FROM: Gerald Dodo, Chemistry Supervisor U.S. EPA Region 10 Laboratory Laboratory Services & Applied Sciences Division
- TO: John Pavitt, Air Enforcement Specialist Air and Toxics Enforcement Section Enforcement and Compliance Assurance Division

Julie Wroble, Toxicologist Risk Evaluation Branch Laboratory Services and Applied Science Division

I have authorized release of this data package. Attached you will find the project narrative report concerning the analysis of samples from the Storedahl Quarry Site received on June 21, 2019. For additional information regarding the attached data, please contact Jed Januch at (360) 871-8731.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION 10 1200 Sixth Avenue, Suite 155 Seattle, WA 98101-3188

LABORATORY SERVICES & APPLIED SCIENCE DIVISION

MEMORANDUM

TO: John Pavitt, Air Enforcement Specialist Air and Toxics Enforcement Section Enforcement and Compliance Assurance Division U.S. EPA Region 10

> Julie Wroble, Toxicologist Risk Evaluation Branch Laboratory Services and Applied Science Division U.S. EPA Region 10

FROM: Jed Januch, Environmental Protection Specialist Laboratory Services Branch Laboratory Services and Applied Science Division U.S. EPA Region 10

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- REVIEW
- BY: Katie Adams, Chemist Laboratory Services Branch Laboratory Services and Applied Science Division U.S. EPA Region 10
- SUBJECT: Storedahl Quarry Site Reconnaissance Sampling and Analysis Yacolt, Washington

Project Code:OCE-029AAccount Code:20192020B10P000E50

This memorandum provides the results of analysis for samples collected at the Storedahl Quarry and submitted to the U.S. EPA Region 10 Laboratory on June 21, 2019. The samples consisted of six rock samples, one soil sample, three drill cuttings samples, and two conveyor dust samples plus a field duplicate. The purpose of the sample collection and analysis was to determine the mineralogy of the samples with specific focus on determining the presence of the zeolite minerals erionite and mordenite.

The samples were examined using a combination of methods including powder x-ray diffraction (XRD) and scanning electron microscopy (SEM) combined with energy dispersive spectroscopy (EDS), and screening-level analysis by stereomicroscopy and polarized light microscopy (PLM). Electron backscatter diffraction (EBSD) analysis, coupled with the SEM, was attempted on a couple of rock samples as a screening level analysis as well. The U.S. EPA Region



10 Laboratory has been accredited by A2LA and has Certificate Number 5027.01. For those tests for which the Laboratory has been accredited by A2LA, results in this report comply with ISO IEC 17025:2017 and the 2009 TNI Environmental Testing Laboratory Standard. Table 1 displays the sample numbers, locations where the samples were collected, the sample matrix, and the analysis performed. Field information was provided to the laboratory from other sources, such as Chain of Custody records.

Sample Number Location Longitude Latitude Description Analysis Performed 19254200 5Q1 122° 28' 53.622" W 45° 51' 39.702" N Soil Sample XRD 19254201 SQ2 Rock SM, PLM, SEM/EDS 19254202 SQ3 Rock SM, PLM, XRD, SEM/EDS 19254203 SQ4 Rock SM, PLM, SEM/EDS 19254204 SQ5 Rock SM, PLM, SEM/EDS 19254205 SO6 Rock SM, PLM, SEM/EDS 19254206 5Q7 122° 27' 10.758" W 45° 51' 36.06" N Drill Cuttings (Fines) XRD 19254207 **SQ8** 122° 27' 14.772" W 45° 51' 37.662" N Drill Cuttings (Fines) XRD 19254208 SQ9 122° 27' 14.928" W 45° 51' 36.87" N Drill Cuttings (Fines) XRD 19254209 SQ10 Rock SM, PLM SQ11 122° 27' 35.472" W 45° 50' 47.538" N 19254210 **Conveyor Dust** XRD 19254211 SQ12 122° 27' 35.472" W 45° 50' 47.538" N **Conveyor Dust** XRD SQ11 122° 27' 35.472" W 45° 50' 47.538" N 19254212 Conveyor Dust - field duplicate XRD

Table 1 - Sample identification for the thirteen samples submitted for analysis.

Sample Preparation Methods

Rock Samples

There was very little preparation needed for the stereomicroscope analysis and only light crushing with a mortar and pestle was needed to prepare rock samples for PLM analysis, specifically the amygdales found in cavities of the basalt rock. Samples analyzed by XRD were lightly crushed with a mortar and pestle using laboratory-grade isopropanol as a grinding medium. Samples analyzed by SEM/EDS were hand-picked and mounted on adhesive carbon tabs affixed to 9-mm aluminum cylinder stubs. The samples were coated with laboratory-grade carbon using a Cressington 108 Carbon/A carbon coater.

Soil, Drill Cuttings, and Conveyor Dust Samples

For XRD analysis, the samples were ground to a particle size less than 38-micrometers (μm) with the aid of a McCrone Micronizing Mill using agate grinding elements with laboratorygrade isopropanol as the grinding medium. The samples were top-loaded into 25-mm aluminum dishes with a 2-millimeter (mm) indent.

I used procedures from the USGS Laboratory Manual for X-Ray Powder Diffraction Open File Report 01-041 (2001) for thermal treatment and ethylene glycol treatment for confirming the presence of smectite clay.

Analytical Procedures

Powder X-ray Diffraction

The XRD analysis was performed according to the procedures in the EPA Region 10 Standard Operating Procedure for Analysis of Minerals by Powder X-ray Diffraction, ASB-005. 1 conducted the analysis with a Rigaku MiniFlex 600 X-ray diffractometer to acquire diffraction data using cobalt (Co) K α radiation at an average wavelength of 1.790207 angstroms (Å), generated at 15 milliamps (mA) and 40 kilovolts (kV). The Rigaku MiniFlex 600 X-ray diffractometer is equipped with a Rigaku D/teX Ultra, high-speed detector. The slits and filters installed were appropriate for analysis with cobalt radiation and the high-speed detector. Patterns were recorded at scan speeds of 5.0 degrees of theta/two-theta (° $\theta/2\theta$) units per minute, with a scan step of 0.02°, over a scan range of 5°-76° $\theta/2\theta$. The detection limit for XRD analysis is typically 1%-5%, though lower detections were possible for some phases.

The instrument control software used for this project was MiniFlex Guidance (version 1.4.0.4, Rigaku) and the data analysis software was PDXL-2 (version 2.3.1.0, MiniFlex, Rigaku). The mineral phase identification was made by comparison with the Powder Diffraction File (PDF) 2 maintained by the International Centre for Diffraction Data (ICDD, 2014). Interpretation of XRD data was based on information from mineralogy texts, USGS references, and journal publications.

SEM/EDS/EBSD

Analysis by SEM/EDS was done according to the U.S. EPA Region 10 SOP for sample analysis by SEM/EDS, MIN_001. The SOP for analysis by EBSD is currently in development and therefore the results acquired by this technique should be considered screening level. I conducted the analysis using a JEOL JSM6510LV SEM equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system and an integrated HKL Nordlys Nano EBSD camera system. Analysis was performed at an accelerating voltage of 15-20 kV with a working distance of 15-mm and varying spot size and magnifications. Analysis of the samples was done using high-vacuum mode with the secondary electron image detector (SEI).

Screening level analysis of a couple of suspected zeolite mineral particles (unpolished) was performed using the EBSD system with the SEM operating in low-vacuum mode with the back-scatter electron detector. The EBSD analysis did not appear to work on the zeolite minerals isolated for analysis. This was possibly due to the imperfect surface features and/or structure of the mineral and the potential interference from other materials adhering to the surface of the samples. The U.S. EPA Region 10 Laboratory does not currently have the technology to polish the surfaces of mineral samples.

Light Microscope Methods

In addition to XRD and SEM/EDS/EBSD analysis, samples were also screened with a PLM to aid in mineral identification by evaluation of optical properties. For the PLM analysis, I used a Carl Zeiss Axioskop 40 PLM and the stereomicroscope was a WILD Model M5 stereomicroscope

using relevant portions of the U.S. EPA Region 10 Standard Operating Procedure for Analysis of Bulk Asbestos Samples by Stereomicroscopy and Polarized Light Microscopy, ASB_001 (June 2018).

Quality Assurance and Quality Control

At the beginning of each day of data collection, the instrument alignment and stability were checked by measuring the position and peak heights of the 3.1355 Å (111), 1.9201 Å (220), and 1.6375 Å (311) peaks of SRM 640c - silicon powder. The silicon powder was mounted in a quartz dish. The results of the daily alignment checks made during the period of August 6, 2019 to September 16, 2019 are displayed is Figure 1. Table 2 shows the peak positions fall within the +/- 0.1° acceptance criteria established in the SOP for the XRD and indicate acceptable alignment and instrument stability over the course of the project.





Table 2 – Daily XRD alignment and stability checks compared with peak positions in the SRM 640c Certificate (calculated for $CoK\alpha$ radiation).

Silicon Cert	8/6/2019	,	8/7/2019		8/8/2019	1	8/9/2019		8/12/2019		8/19/2019	1	8/20/2019	· .	8/22/2019)	9/12/2019)	9/16/2019	
2-theta	20	Δ	20	Δ	20	Δ	20	Δ	20	Δ	20	Δ	28	Δ	20	Δ	20	Δ	20	Δ
33.1491	33.1308	0.02	33.0936	0.06	33.11	0.04	33.1074	0.04	33.1188	0.03	33.1121	0.04	33.1278	0.02	33.1357	0.01	33.1295	0.02	33.1307	0.02
55.5283	55.5132	0.02	55.4789	0.05	55.4924	0.04	55.4887	0,04	55.499	0.03	55.4977	0.03	55.5116	0.02	55.5178	0.01	55.5117	0.02	55.5141	0.01
66.2185	66.1982	0.02	66.1672	0.05	66.18	0.04	66.1754	0.04	66.1587	0.06	66.1872	0.03	66.1997	0.02	66.2047	0.01	66.1989	0.02	66.2015	0.02

The wide-range instrument alignment checks of the goniometer were made on August 6, 2019 and September 16, 2019, using SRM 1976, a flat corundum plate. Data from the line positions used to check goniometer alignment are displayed in Figure 3 and show that the peak positions are within $\pm 0.1^{\circ} \theta/2\theta$ angle listed in the Certificate of Analysis for SRM 1976 (certificate issue date March 14, 2007). Figure 4 shows the peak intensities below 76° $\theta/2\theta$ are within 70-130% of the values in the certificate. Therefore, the wide-range instrument alignment

was acceptable for the project since the diagnostic peaks for the minerals identified fall below 76° $\theta/2\theta$. The SEM only required daily manual checks of electron gun and aperture alignment. The EDS has a silicon drift detector and is only calibrated during preventative service visits under the service contract. I did periodic checks to monitor the position and check intensity of the copper peak at 8.040 electron volts (eV) using a X-Checker EDS calibration standard (the copper pin). The Carl Zeiss Axioskop 40 PLM was checked daily for microscope alignment and Köhler Illumination. I verified the refractive index liquids used for this project were accurate on June 19, 2019.

Figure 2 - Wide-range instrument alignment checks on $\frac{8}{6}$ and $\frac{9}{16}$, using SRM 1976 – a corundum plate.



Figure 3 – Wide range alignment check for peak positions on 8/6/19 and 9/16/19.





Figure 4 - Wide range alignment check for peak intensity on 8/6/19 and 9/16/19.

Results of Analysis

For analysis of the rock samples, I concentrated my efforts to identify the white colored amygdales found in cavities of the basalt rock samples collected. Sample 19254202 had the largest quantity of this mineral which is shown in Figure 5. Samples 19254203, 19254204, and 19254205 also had similar mineral presenting in either in a radiating pattern on the surface or in cavities of the basalt host rock. Analysis by PLM revealed the material has a low refractive index in the 1.480 – 1.500 range. XRD analysis of the mineral isolated from sample 19254202 revealed the presence of a zeolite identified as stilbite based on the crystal structure and resulting diffraction pattern (Figure 6). The refractive index evaluated by PLM and chemistry determined with SEM/EDS (Figure 7) support the identification of stilbite. Sample 19254201 also has a white to clear mineral in the surface of a host rock, though the optical properties, with the refractive index being close to 1.5500, matches quartz.

Figure 5 – Stereomicroscope image of white radiating crystals on a basalt rock from sample 19254202 (left) and a polarized light microscope image of the subsample of the crystals mounted in 1.4800 refractive index liquid (right).



Figure 6 - Annotated XRD pattern of specimen taken from sample 19254202.



Figure 7 - SEM image (left) and EDS data (right) for a mineral particle isolated from sample 19254202.



Another rock sample that was analyzed was 19254209. This sample consisted of a very small quantity of mineral fragments extracted from a hole in a rock. The small fragments were mounted in 1.4800 refractive index liquid and appear to have a much higher refractive index than the stilbite identified in other samples from this group. There was not enough sample to continue to analysis for a more definitive identification.

Soil, Drill Cutting, and Conveyor Dust Samples

The composition of the soil sample, three drill cuttings samples, and two conveyor dust samples submitted for analysis were relatively similar with some variation in presence or abundance of the minerals identified. I did not detect erionite or mordenite by XRD analysis. Figure 8 displays XRD patterns resulting from analysis of the samples. The analysis revealed that all contained quartz, mica, feldspar, pyroxene, iron oxide, zeolite (stilbite), and smectite. The identification of smectite was confirmed using a combination of thermal treatment and ethylene glycol treatment.

Figure 8 – XRD patterns for soil, drill cuttings, and conveyor dust samples.



Figure 9 shows the response of the broad peak at approximately $6.5^{\circ} \theta/2\theta$ when exposed to thermal treatment at 550° C for 30-minutes and ethylene glycol treatment. Heating smectite evaporates interlayer water that results in collapse of the structural layers and a shifting the d-spacing from about 15 Å to approximately 9.5 - 10 Å. The ethylene glycol treatment was done by placing the sample in a sealed glass canister containing ethylene glycol. The sample was heated in the canister for 4-hours allowing the glycol vapors to permeate the sample. The ethylene glycol is absorbed by smectite resulting in swelling of the structural layers to approximately 17Å.

Figure 10 shows an XRD pattern for an aliquot of material isolated from sample 19254207 which was taken with the aid of a hand-magnet. The XRD analysis confirms the presence of iron oxide tentatively identified as magnesioferrite.

Figure 9 - Sample 19254210, comparing aliquots in ambient condition to thermal treatment and ethylene glycol treatment.



Figure 10 - XRD analysis of a sub-sample isolated with a hand-magnet from sample 19254207.



Summary

I did not detect the zeolites erionite or mordenite in the samples submitted for analysis for this project. The analysis did reveal the presence of a zeolite mineral called stilbite, as well as other minerals including quartz, smectite, mica, amphibole, feldspar, pyroxene, and iron oxide. Table 3 displays a summary of the minerals identified in the samples analyzed for this project.

Table 3 – Summary of	f results of analysis
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Sample Number Location		Description	Minerals Identified							
19254200	SQ1	Soil Sample	smectite, mica, zeolite (stilbite), amphibole, quartz, feldspar, pyroxene, iron oxide							
19254201	SQ2	Rock	quartz							
19254202	SQ3	Rock	zeolite (stilbite)							
19254203	SQ4	Rock	zeolite (stilbite)							
19254204	SQ5	Rock	zeolite (stilbite)							
19254205	SQ6	Rock	zeolite (stilbite)							
19254206	SQ7	Drill Cuttings (Fines)	smectite, mica, zeolite (stilbite), amphibole, quartz, feldspar, pyroxene, iron oxide							
19254207	SQ8	Drill Cuttings (Fines)	smectite, mica, zeolite (stilbite), amphibole, quartz, feldspar, pyroxene, iron oxide							
19254208	5Q9	Drill Cuttings (Fines)	smectite, mica, zeolite (stilbite), amphibole, quartz, feldspar, pyroxene, iron oxide							
19254209	SQ10	Rock	Small sample size - ruled out zeolite based on refractive index							
19254210	SQ11	Conveyor Dust	smectite, mica, zeolite (stilbite), amphibole, quartz, feldspar, pyroxene, iron oxide							
19254211	SQ12	Conveyor Dust	smectite, mica, zeolite (stilbite), amphibole, quartz, feldspar, pyroxene, iron oxide							
19254212	SQ11	Conveyor Dust - field duplicate	smectite, mica, zeolite (stilbite), amphibole, quartz, feldspar, pyroxene, iron oxide							

References

ICDD, Powder Diffraction File-2, release 2014: Newton Square, Pennsylvania, International Centre for Diffraction Data, cd-rom.

Welton, Joann E., (2003) <u>SEM Petrology Atlas, Methods in Exploration Series No. 4</u>, The American Association of Petroleum Geologists.

Deer, W.A., Howie, R.A., and Zussman, J., (1992) <u>An introduction to The Rock-Forming</u> <u>Minerals.</u> Harlow, England: Pearson Education Limited.

Appendices

- 1. Chain of Custody Records
- 2. Instrument QC Data
- 3. XRD Data
- 4. SEM/EDS Data
- 5. SM/PLM Images

Stereomicroscope image taken with a Nikon Coolpix camera and processed with Image J digital imaging software.



Quartz fragments – PLM image taken with a Zeiss AxioCam HRc digital camera and processed with Zen 2 Core imaging software.



Stilbite fragments – stereomicroscope image taken with a Nikon Coolpix camera and processed with Image J digital imaging software.



Stilbite fragments – PLM image taken with a Zeiss AxioCam HRc digital camera and processed with Zen 2 Core imaging software.





Stilbite fragments – PLM image taken with a Zeiss AxioCam HRc digital camera and processed with Zen 2 Core imaging software.





Storedahl Quarry, OCE-029A

Sample 19254204

Stilbite fragments – PLM image taken with a Zeiss AxioCam HRc digital camera and processed with Zen 2 Core imaging software.





Stilbite fragments – PLM image taken with a Zeiss AxioCam HRc digital camera and processed with Zen 2 Core imaging software.



PLM image taken with a Zeiss AxioCam HRc digital camera and processed with Zen 2 Core imaging software. Sample was very small quantity.



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.3 mm, and spot size 68. The magnification was 100x.







Figure 2 - EDS data for structure isolated from sample 19254202.



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.8 mm, and spot size 68. The magnification was 160x.



Figure 1- Elongated structure isolated from sample 19254202.







Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.6 mm, and spot size 68. The magnification was 330x.







Figure 2 - EDS data for structure isolated from sample 19254202.



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.4 mm, and spot size 68. The magnification was 450x.



100µm









Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.4 mm, and spot size 68. The magnification was _350x.



Figure 1 - Elongated structures isolated from sample 19254203.



Figure 2 - EDS data for structure isolated fram sample 19254203.



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.7 mm, and spot size 68. The magnification was 450x.







Figure 2 - EDS data for structure isolated from sample 19254203.



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.7 mm, and spot size 68. The magnification was 170x.









Figure 2 - EDS data for structure isolated from sample 19254204



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.7 mm, and spot size 68. The magnification was 90x.







Figure 2 - EDS data for structure isolated from sample 19254204



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.7 mm, and spot size 68. The magnification was 200x.







Figure 2 - EDS data for structure isolated from sample 19254204



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.7 mm, and spot size 68. The magnification was 150x.



Electron Image 13

Figure 1 - Structures isolated from sample 19254205.



Figure 2 - EDS data for structure isolated from somple 19254205.



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.7 mm, and spot size 68. The magnification was 600x.



Electron Image 14





Figure 2 - EDS data for particle isolated from sample 19254205



Image collected with a JEOL JSM6510LV Scanning Electron Microscope equipped with an Oxford Instruments AztecEnergy X-Max-50 EDS system. The secondary electron detector (SE1) was used at 20 kV, working distance 14.7 mm, and spot size 68. The magnification was 170x.



Electron Image 15





Figure 2 - EDS data for particle isolated from sample 19254205





Site Visit 6/20/19 Storedahl Quarry Yacolt, Washington





<-SQ1

SQ2->



Latitude: 45° 51' 39.702" N Longitude: 122° 28' 53.622" W

Sample (SQ3) of fan-shaped mass of crystals from rock







Rock samples from Quarry (SQ4, SQ5, SQ6)







Larger rock samples to be analyzed





SQ7 sample of fines at base of rock pile

Latitude: 45° 51' 36.06" N Longitude: 122° 27' 10.758" W

SQ8 and SQ9



Latitude: 45° 51' 37.662" N Longitude: 122° 27' 14.772" W

Latitude: 45° 51' 36.87" N Longitude: 122° 27' 14.928" W





SQ10 – sample of crystal from inside this hole in the rock



Sample SQ11 and SQ12





Latitude: 45° 50′ 47.538″ N Longitude: 122° 27′ 35.472″ W