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State of Washington

Quality Assurance Project Plan

Better Brakes Enforcement Study, 2022

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Quality Assurance Project Plan

Better Brakes Enforcement Study, 2022

by Ken Nelson and Amy Salamone

September 2022

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EAP: Environmental Assessment Program
HWTR: Hazardous Waste and Toxics Reduction Program

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2.0 Abstract

Under the Better Brakes Law, Chapter 70A.340 Revised Code of Washington (RCW), manufacturers of brake friction materials are required to report and certify brake friction products offered for sale in Washington State. The law, passed in 2010, set out to reduce the use of toxic material in brake friction materials, and beginning in 2015, restricted the use of asbestos, cadmium, chromium(VI), lead, and mercury.

The law also established a two-tier provision to phase out copper in brake friction materials by 2025. The first tier, beginning January 1, 2021, restricts the selling or offer for sale of brake friction materials in Washington State manufactured after January 1, 2021 and containing more than 5% copper by weight.

This study will (1) identify manufacturers that have not yet certified their products for sale in Washington in compliance with the 2021 metals restrictions, and (2) verify the accuracy of reported data submitted under the Washington State Brake self-certification program.

Metals analysis will be performed on drilled brake friction materials for total cadmium, copper, lead, mercury, antimony, nickel, and zinc. Testing for chromium(VI) will also be performed. Brake formulations may change as part of reducing the use of copper, so this study will also assess concentrations of antimony, nickel, and zinc.

3.0 Background

3.1 Introduction and problem statement

In 2010, Washington State passed the Better Brakes Law Chapter 70A.340 RCW[1], restricting the use of asbestos, cadmium, chromium(VI), lead, and mercury by 2015, and significantly phasing out copper by 2025, in brake friction materials. The law established a self-certification program for brake friction materials sold in Washington State.

To comply with the self-certification program (Chapter 173-901 Washington Administrative Code, WAC), manufacturers are required to test each brake friction formulation in accordance with SAE International (formerly Society of Automotive Engineers) method SAE J2975 (stabilized[2] in 2020). Summaries of this testing, along with signed self-certification documentation, are then submitted to the Washington State Department of Ecology (Ecology) through an independent industry-sponsored database hosted by NSF International (formerly the National Sanitation Foundation) and shared with Ecology on a quarterly basis. Quarterly brake friction material self-reporting has been ongoing since 2013.

Additionally, for brake products (including brake pads and shoes in which brake friction materials are used) manufactured after January 1, 2015, the product packaging must display one of the three levels of compliance of the brake friction material as shown in Figure 1. This LeafMark™ system, established by the Brake Manufacturers Council, meets the Better Brakes standards. The more shaded the leaf, the more levels of compliance the brake friction material fulfills. Compliance level A was in effect at the time of the 2017 Ecology consumer product study (Ecology, 2018). As of January 1, 2021, all brakes manufactured on or after that date must meet compliance level B. Compliance level N needs to be met by manufacturers as of January 1, 2025.

Level A: Limits levels of asbestos, chromium(VI), lead, and mercury to less than or equal to 0.1% and cadmium to 0.01% by weight.
Level B: Includes all the requirements of level A and copper levels must be less than or equal to 5% by weight.
Level N: Includes all requirements of level A and copper levels must be less than or equal to 0.5% by weight.

Figure 1: Product package markings indicating compliance level of brake friction materials.

[1] Chapter 70A.340 RCW and Chapter 173-901 WAC specifies prohibition of constituents in brake friction materials.

[2] Stabilized by the SAE Brake Linings Standards Committee and will no longer be subjected to periodic reviews for currency (from SAE J2975:2020).

The primary focus of this study is to (1) assess select metals (Table 10) in currently available brake friction materials, and (2) collect information provided on the edge code. The edge code is a unique identification (ID) code located directly on the brake friction material. Part of the edge code includes the specific environmental compliance level letter ("A", "B", or "N") and a two-digit abbreviation for the year of manufacture.

Discrepancies in product packaging are not a primary focus of this study, but markings indicating compliance will be noted and packaging photos will be made available for review by the Better Brakes Program (oversees the implementation of Washington's Better Brakes Law). The edge code marking is described in SAE J866 (stabilized in 2019) and in Ecology's publication, Guidance for Marking Brake Friction Material (Ecology, 2013), available at https://apps.ecology.wa.gov/publications/SummaryPages/1304011.html

In general, the Better Brakes Law applies to brake friction materials intended for all vehicles that travel on the highway. Notable exemptions to the law include brakes intended for use on motorcycles, military combat vehicles, race cars, collector vehicles and internally enclosed braking systems that emit no debris under normal operating conditions. Brake friction material from the exempted categories will not be tested.

3.2 Study area and surroundings

Products will be considered for study assessments if they are (1) sold in any physical location (e.g., brake retailers, discount stores, automotive parts stores, warehouse clubs) within Washington or (2) accessible for purchase online by Washington residents or businesses. This study will also include products available for purchase through Washington State Department of Enterprise Services (DES) contracts.

3.2.1 History of study area

See sections 3.1, as well as sections 3.2.2, 4.0, 4.3, 6.2.2.2, 7.1, 7.2.1, and 7.4, which outline the acquisition of available brake friction material in Washington stores and online.

3.2.2 Summary of previous studies and existing data

Ecology studies investigating the release of copper into rivers and streams in Washington State have been ongoing since 2011. These studies focused on copper and chemicals of concern in urban runoff and provided loading estimates of metals from brake products. These studies indicate that brakes are one of the sources of metals pollution through small releases of brake friction material from use over time. In 2017, Ecology performed a consumer product testing study of brake products sold in Washington State to confirm compliance with the first phase of the Better Brakes Law (Ecology, 2018).

Following is a list of several of these historical studies which includes the 2017 consumer product testing study:

- *Control of Toxic Chemicals in Puget Sound (Phase 3): Primary Sources of Selected Toxic Chemicals and Quantities Released in the Puget Sound Basin*, by Ecology (2011).
  This study provided baseline data on loading estimates of copper, lead, zinc, and cadmium

released from vehicle brake pads based on wear rates and metal concentrations reported in brake friction material.

- *Copper and Zinc in Urban Runoff (Phase 1) - Potential Pollutant Sources and Release Rates*, by Ecology (2017).
  This study assessed potential pollutant sources and release rates of copper and zinc. It found that an estimated 800 pounds of copper and 5,900 pounds of zinc are released into the environment each year on average in the study area in Thurston County. The main sources of copper and zinc included vehicle brake wear and roofing materials.
- *Better Brakes Enforcement Study 2017*, by Ecology (2018).
  This product testing study of brake pad friction materials assessed concentrations of total cadmium, copper, and lead in brakes sold in Washington State in 2017. Cadmium results for all 163 samples met the Better Brakes Law compliance criteria. Copper was present in 71 out of 163 brake samples at compliance level A. Compliance levels B or N for copper were met in 92 out of 163 brake samples. Lead was present in four out of 163 brake samples in levels that exceeded the Better Brakes Law compliance criteria.
- *Putting the Brakes on Water Pollution: A story of industry and government collaboration for copper-free brakes*, by EPA (2021).
  An evaluation was performed in September 2021 of current certified products through the independent industry-sponsored NSF International database. Of the self-reported registered friction materials, more than 75% of the products meet compliance level N for copper. Compliance level N for copper is required to be met by manufacturers by January 1, 2025.
- *Brake Pad Copper Reduction Status Report 2018*, by California Stormwater Quality Association (2019).
  This status report indicates there has been a 30 to 40% decrease in copper content of brake products on the market as of June 30, 2018 when compared to data sets collected before 2014.

Several other groups outside of Washington State have evaluated the release of toxic chemicals from brake friction material into the environment. In January 2015, the U.S. Environmental Protection Agency (EPA), Environmental Council of the States (ECOS), Motor Equipment Manufacturers Association (MEMA), and other industry stakeholders signed a national memorandum of understanding (MOU) on copper mitigation in watershed and waterways (Ecology, 2016).

Previous brake studies by Ecology, as well as reports from Ecology's previous product testing studies, can be reviewed by searching: https://ecology.wa.gov/pubs

Lab data and product information from Ecology's product testing studies are viewable by searching the online database: http://ecyapeem/PTDBPublicReporting

3.2.3 Parameters of interest and potential sources

This study will assess the concentration of the metals cadmium, lead, mercury, copper, antimony, nickel, zinc, and chromium(VI) on drilled brake friction materials (Section 7, Table 10). Brake friction material is the part of a motor vehicle brake designed to slow or stop movement through friction against a rotor. It does not include parking brakes or motor vehicles employing internal-closed-oil immersed motor vehicle brakes (WAC 173-901-040).

3.2.4 Regulatory criteria or standards

Washington State regulates the use of toxic metals and asbestiform fibers in brake friction material, and also mandates the phase out of copper by 2025, in accordance with the Better Brakes Law (Chapter 70A.340 RCW, formerly Chapter 70.285 RCW) and the Better Brakes Rule (Chapter 173-901 WAC). The standards for testing and certifying, as part of the law and rule, are:

- Testing brake friction materials using SAE J2975.
- Ensuring self-certification of compliance through an industry-sponsored registrar.
- Marking brake friction materials with an edge code per SAE J866.
- The LeafMark™ (Figure 1) certification mark for packaging.

California has a similar Brake Friction Material Law that includes a phase out of copper in brakes as well as a ban on other constituents.

In 2015, the EPA established a *Memorandum of Understanding (MOU) on Copper Mitigation in Watersheds and Waterways* (EPA, 2015) through the phase out of copper and other chemicals in brake friction material.

4.0 Project Description

During August-September 2022, Ecology will purchase up to 100 brake products from several Washington automotive retailers and online stores. Brake products manufactured as of January 1, 2021 will be prioritized for purchase. This manufacture date, as established by the Better Brakes Law for Washington State, is the first tier date limiting the amount of copper in brake friction material to no more than 5%. Brakes will be purchased from South Puget Sound area retail locations, online retailers, and through state DES contracts. All the brake products will be drilled by a contract lab, and the drilled turnings will be sent to Ecology's Manchester Environmental Laboratory (MEL) for metals analysis. Additional testing for chromium(VI) will be performed at a contract lab.

This study will provide data to help Ecology to (1) identify manufacturers that have not yet certified their products for sale in Washington State in compliance with the 2021 restrictions, and (2) verify the accuracy of self-reported certification data submitted by manufacturers.

4.1 Project goals

This study is being conducted with the following goals:

- Assess the concentration of metals in certified and uncertified brake friction materials (brake pads and shoes) for brake products manufactured as of January 1, 2021.
- Assess the concentration of select metals (Section 7, Table 10) in certified and uncertified brake friction materials (e.g., brake pads and shoes) for any brake products currently available in Washington.
- Provide data to support the assessment of concentrations of select metals (Section 7, Table 10) in brake friction materials to verify the accuracy of manufacturers' self-reported compliance information.
- Provide data to support the compliance and enforcement of the Better Brakes Law (Chapter 70A.340 RCW).

4.2 Project objectives

The following objectives will be carried out to meet the project goals:

- Identify up to 100 brake friction materials to analyze for this study, based on the following:
  - NSF International database of registered friction material from the most recent certifications for the year just prior to purchasing.
  - Data from previous Ecology studies or other brake friction material assessments.
  - Information regarding manufacture date and compliance level of products through in-store reconnaissance and internet research.
- Purchase up to 78 brake friction materials, including 16 considered "original equipment" and 62 considered "aftermarket parts" brake friction materials.

- Purchase an additional 22 brake friction materials, including 11 considered special use brake friction materials and 11 considered heavy duty and/or commercial vehicle use.
- Analyze up to 100 samples of brake products at MEL for the metals listed in Table 10, except for chromium(VI).
- Analyze up to 100 samples of brake products at a contract lab for chromium(VI).
- Provide metals data for the client to verify compliance and pursue enforcement of the Better Brakes Law as necessary.

4.3 Information needed and sources

The Better Brakes Program 2017 study, SAE International methods, and data from sources such as the EPA will be reviewed as applicable.

For product selection, the primary resource to review is the NSF International database of registered friction material. NSF International submits registration data of brake friction material to Ecology on a quarterly basis. The most recent quarters in the year prior to purchasing will be reviewed.

In 2020, the California Department of Toxic Substances Control (DTSC) also approved Automotive Manufacturers Equipment Compliance Agency Inc. (AMECA) to oversee registrations. If data are available from AMECA's website by the time of purchasing then it may also be reviewed. The Better Brakes Program may also be able to obtain data from AMECA if necessary for review.

To provide additional information for product selection, we will also review existing product testing data, supplemented with research of typical brake friction material offered for sale to Washington residents and through State contract.

Product research regarding its edge code and, in particular, the environmental compliance letter and year of manufacture should be identified if possible. The unique company ID listed in the edge code will be useful to cross reference the information provided by NSF International. Additional research may also include the country of manufacture, brake formulation (for example metallic, ceramic, semi-metallic, and organic), vehicle type, and if it is an original equipment or aftermarket brake product.

4.4 Tasks required

To meet study goals, the study will include the following tasks:

- During August-September 2022, purchase up to 100 brake products from several online automotive stores and Washington retailers. Brake products may be certified or uncertified. Purchasing of brake products and collection of data for compliance and enforcement for the Better Brakes Program will be conducted as follows:
    - Identify potential brake products from the categories described in Section 7, Table 8 and Table 9. Aftermarket brakes will be the bulk of brakes purchased for this study. Brakes will also be original equipment or for special use vehicles.

  - Consult latest NSF International and AMECA data of registered friction material to identify products that are currently certified.
  - Conduct in-store and online reconnaissance or investigation of any product information or edge codes to identify obvious compliance markings and year of manufacture.
  - Develop a purchasing plan to collect as many products that have a manufacture date of January 2021 or later based on certification data and in-store and online reconnaissance information.
  - The plan may be updated for subsequent purchasing events as additional evaluation of in-store packaging and online product information is gathered. The objective is to continuously target the purchase of brake products manufactured after January 1, 2021.
  - Transport brake products or receive brake products through shipping to Ecology's Product Testing Prep Room, utilizing appropriate personal protective equipment (PPE).
  - Document all purchasing information, brake product details, edge codes, environmental compliance levels, and product photos in Ecology's Product Testing Database (PTDB).
- Submit up to 100 brake friction material samples to contract lab for drilling in accordance with SAE J2975.
- Submit up to 100 drilled brake friction material samples to MEL for metals analysis.
- Submit up to 100 drilled brake friction material samples to a contract lab for analysis of chromium(VI).
- Perform internal Quality Assurance (QA) review on analytical data and PTDB entries.
- Average verified duplicate results for each analyzed metal and qualify as described in section 14.0.
- Perform internal QA on calculations for averaging of results.
- Review data validation report and enter individual results into the PTDB.
- Finalize PTDB data QA and write final report.
- The data will be provided to the client to verify compliance and pursue enforcement of the Better Brakes Law.
- Make lab data and product information from this study available to the public on Ecology's PTDB external website.

4.5 Systematic planning process

This QAPP represents comprehensive systematic planning for this study.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Table 1 shows the responsibilities of those who will be involved in this project.

Table 1. Organization of project staff and responsibilities.

Staff	Title	Responsibilities
Kathleen Gilligan Reducing Toxic Threats Unit, HWTR Phone: 360-407-6609	Client	Reviews project scope. Reviews the QAPP and approves the final QAPP.
Ken Nelson Product Testing Unit, EAP, SCS Phone: 360-522-2722	Project Manager	Writes the QAPP. Assists with product purchasing, processing, sample prioritization, and transportation of samples to the lab. Reviews the draft and final report.
Amy Salamone Product Testing Unit, EAP, SCS Phone: 360-407-6492	Principal Investigator	Reviews the QAPP. Coordinates with lab. Purchases and receives products; enters purchases and products into the PTDB, conducts QA review of these entries; processes samples, chain-of-custody, and sends samples to lab; enters drilling photos into PTDB. Calculates relative standard deviation (RSD). Conducts QA review of data, analyzes and interprets data, and enters data into PTDB. Writes the draft and final report.
Sara Sekerak Product Testing Unit, EAP, SCS, Phone: 360-407-6997	Unit Supervisor for Project Manager	Reviews the project scope and budget. Provides internal review of the QAPP, tracks progress, approves the budget, and approves the final QAPP. Reviews and approves the draft and final report.
Jessica Archer EAP, SCS Phone: 360-407-6997	Section Manager for Project Manager	Reviews the project scope and budget. Approves the final QAPP.
Lola Flores Reducing Toxic Threats Unit, HWTR Phone: 360-407-6876	Unit Supervisor for Client	Coordinates client project scope. Reviews and approves the final QAPP.
Richelle Perez P2RA Section, HWTR Phone: 360-407-6724	Section Manager for Client	Reviews the project scope and approves the final QAPP.
Alan Rue MEL Phone: 360-871-8801	Director	Provides internal review of the QAPP and approves the final QAPP. Coordinates drilling and analytical services with contract labs. Coordinates validation of contract lab's analytical data.
Arati Kaza Ecology Phone: 360-407-6964	Ecology Quality Assurance Officer	Reviews and approves the draft QAPP, approval to begin work, and the final QAPP.

EAP: Environmental Assessment Program
SCS: Statewide Coordination Section
HWTR: Hazardous Waste and Toxics Reduction Program
MEL: Manchester Environmental Laboratory
P2RA: Pollution Prevention and Regulatory Assistance
PTDB: Product Testing Database

5.2 Special training and certifications

Ecology staff conducting sample processing and data entry are trained according to Ecology's Standard Operating Procedure (SOP) PTP001 *Procedure for Product Collection and Sample Processing* (Wiseman, 2021) as well as SOP PTP002 *Data Entry and Data Entry Quality Assurance* (Wiseman, 2022). Staff who make purchases with an Ecology credit card must attend the online training program, *Ecology Curriculum – Credit Card Custodians and Purchase Coordinators – Ethics and Small Purchases*.

Staff will complete all required Ecology health and safety trainings. Brake friction material may contain asbestiform fibers; therefore, appropriate engineering controls and PPE should be used. All activities involving handling the brake products by Ecology staff should be performed in the fume hood with nitrile gloves and a lab coat (see section 9.2 for additional details). Staff will also complete all additional required purchasing and contracts trainings, as their role in this project requires.

5.3 Organization chart

Table 1 lists the key individuals and responsibilities.

5.4 Proposed project schedule

Tables 2 - 4 list key activities, due dates, and lead staff for this project.

Table 2. Schedule for product collection, data entry, laboratory work.

Task	Due date	Lead staff
Product purchasing	September 2022	Amy Salamone
Product receipt and data entry	September 2022	Amy Salamone
Internal data entry QA	October 2022	Amy Salamone
Drilling brake friction material for contract lab – up to 100 brake pad samples	November 2022	Alan Rue
Lab analyses for chromium(VI) samples – contract lab completed	February 2023	Alan Rue
Lab analyses for metals samples – MEL completed	April 2023	Alan Rue

Table 3. Schedule for data and study reviews.

Task	Due date	Lead staff
Lab data validation complete	May 2023	Alan Rue
All lab data QA reviewed	June 2023	Amy Salamone
All lab data loaded into PTDB	June 2023	Amy Salamone
PTDB study QA review	June 2023	Amy Salamone

Table 4. Schedule for final report.

Task	Due date	Lead staff
Draft to supervisor	July 2023	Amy Salamone
Draft to client/peer reviewer	July 2023	Amy Salamone
Final draft (all reviews done) to publications team	August 2023	Amy Salamone
Final report due on web	October 2023	Publications team

5.5 Budget and funding

This project is funded by Ecology's Environmental Assessment Program (EAP) Product Testing budget.

Table 5 presents the total estimated costs for this project, $129,333, which includes costs for product collection, lab budget, shipping, and contract fees.

Table 5. Project budget and funding.

Item	Cost ($)
Product Collection (up to 100)	15,000
Laboratory (see Table 6 for details)	102,085
Shipping	3,000
MEL Contract Fee for Drilling (10%)	1,313
MEL Contract Fee for Cr(VI) Analysis (30%)	7,935
Total	**129,333**

Table 6 presents lab budget costs for this project, estimated to be $102,085 broken down by sample type and number of samples. The number of quality control (QC) tests as part of the lab budget are only those tests that are not included in the cost of analysis (lab duplicates, matrix spikes, and matrix spike duplicates).

Table 6. Laboratory budget.

Parameter	Number of Samples	Number of QC Samples	Total Number of Samples	Cost Per Sample ($)	Lab Subtotal ($)
Total Metals: Cadmium, Lead, Mercury, Copper, Antimony, Nickel, Zinc	200	30[Ω]	230	225	51,750
Total Metals (Water Blanks): Cadmium, Lead, Copper, Antimony, Nickel, Zinc, and Chromium	4	0[+]	4	210	840
Total Mercury (Water Blanks)	4	0[+]	4	80	320
Total Metals: Digestion Vessels[‡]	24	0	24	400	9,600
Contract Lab Brake Friction Material Drilling[++]	100	25[◊◊]	125	105	13,125
Contract Lab Chromium(VI) Analysis[++]	200	30[Ω]	230	115	26,450
				Total	**102,085**

[Ω] Includes lab control samples, matrix spikes, and matrix spike duplicates.

[‡]Digestion vessels will need replaced after use during project.

[+]There will not be enough sample to run duplicates, matrix spikes, and matrix spike duplicates.

[++]It is anticipated that contract lab will ship drilled brake friction material directly to MEL. MEL will then ship samples for chromium(VI) analysis directly to contract lab.

[◊◊]These will be storage and processing blanks as defined in Section 9.

6.0 Quality Objectives

6.1 Data quality objectives

The main data quality objective (DQO) for this project is to collect brake friction material for a wide variety of vehicle types available for purchase in Washington from several online stores and Washington retailers. Products manufactured after January 1, 2021 will be targeted for purchase. Products will be purchased for a wide variety of vehicle types, including different passenger vehicles, heavy-duty vehicles, and special use vehicles like police cars, school buses, and garbage trucks (see Table 8 and Table 9). Brakes will be from a wide variety of manufacturers and represent all environmental certification levels.

Due to the complex, variable, and inhomogeneous nature of brake friction materials, a DQO will be duplicate digestion and testing of product samples during metals analysis. All analytical lab analyses will meet the corresponding EPA method's QA/QC and instruments' performance limits. The lab analysis will also meet the measurement quality objectives (MQOs) that are described below.

Ecology's product testing studies follow established Guidelines for Data Verification and Validation of Chemical Data from Ecology's QA Coordinator. Lab data used to evaluate compliance will undergo verification and validation following this QAPP, EPA National Functional Guidelines for Inorganic Data Review (EPA, 2020), and EPA Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA, 2009).

6.2 Measurement quality objectives

6.2.1 Targets for precision, bias, and sensitivity

The MQOs for the analytical metals results, expressed in terms of acceptable precision, bias, and sensitivity, are described in this section and summarized in Table 7.

Table 7. Measurement quality objectives for laboratory analysis.

Parameter	LCS, MS*, and Sample Duplicates (RPD)	LCS (% Recovery)	Matrix Spike* (% Recovery)	StorageΩΩ, Processing, and Method Blanks	Method Reporting Limit (ppm)**
Total Metals: Cadmium, Lead, Mercury, Copper, Antimony, Nickel, Zinc+	≤ 20%	85 – 115	75 – 125	<1 ppm	10
Chromium(VI)+	≤ 20%	80 – 120	75 – 125	<1 ppm total chromium∞∞∞	0.3

LCS = laboratory control sample; MS = matrix spike; RPD = relative percent difference; ppm = parts per million.
*Matrix spikes and matrix spike duplicates could have recovery issues due to unique product matrices.
ΩΩStorage and processing blanks are defined in Section 9.
**Individual method reporting limits vary based on specific matrix type. The listed method reporting limit is based on 0.25 gram sample size limit for analytical digestion. The reporting limit for chromium(VI) will be based on the amount of available drilled brake friction material.
+Duplicate sample relative standard deviation (RSD) as per SAE J2975 will be calculated and reported as part of the report and is discussed in 6.2.1.
∞∞∞There will be no storage and processing blanks analyzed; only the method blanks will be applicable. Total chromium will be analyzed at MEL on blanks; the contract lab will not analyze blanks for hexavalent chromium.

6.2.1.1 Precision

Precision is a measure of the variability in the results of replicate measurements due to random error. Per the recommendation in SAE J2975, and due to the complex, variable, and inhomogeneous nature of brake friction materials, the digestion and analysis for all samples will be performed in duplicate. The project manager will provide the average value of the duplicate results following the percent relative standard deviation (RSD) equation in the final report. Percent RSD (standard deviation / measurements average *100) should be less than 20% for the duplicate measurements. See Table 7 for MQOs. The RSD of duplicates will be calculated for the summary report, and RSDs that exceed 20% will be qualified with a "J" in the PTDB.

6.2.1.2 Bias

Bias is the difference between the analytical value and the true value. Assessments of laboratory bias will be determined by analysis of lab control samples (LCS) and matrix spike (MS) samples. Recovery of MS samples may be affected by potential interferences from inhomogeneous brake friction material and the presence of other metals and sulfides. The relatively-high expected levels of metals and sulfides present in the samples will also make it difficult to perform matrix spikes at an appropriate spike level. There are no standard reference materials for brake friction materials. See Table 7 for MQOs.

Assessment of potential bias by environmental contamination will be through collection of process blanks during sample drilling. One blank will be collected for each batch of product processing in order to assess potential environmental contamination. Three blanks will also be collected once, prior to any processing, for a storage container contamination check. The blanks will be treated as samples and will accompany samples during shipping and handling. Specifications for this contract service are described in the associated scope of work contract document which will be in an appendix of the final report.

6.2.1.3 Sensitivity

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the metals of interest. Lab sensitivity is conveyed by the method reporting limits for each analyte listed in Table 7.

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

In terms of product testing studies, comparability is the ability of data from this project to be compared to data generated by other similar projects or studies. Ecology's product testing studies follow standardized procedures for developing a QAPP (Sekerak, 2016) as well as sample collection, sampling processing, and data collection as described in SOP PTP001 (Wiseman, 2021) and SOP PTP002 (Wiseman, 2022).

Appropriate established methods (e.g. SAE J2975), procedures (SAE J866), and the SOP (PTP001) listed in Section 8.2 will be followed as applicable by matrix and analyte. The MEL methods corresponding to the EPA methods are listed in Section 9.2. The contract lab SOP for chromium(VI) analysis is usually provided for review as part of the contracting process. Data review for the metals analysis can be found in the MEL procedure as listed in Section 13.3.

The treatment of non-detects for averaging is outlined in Section 14.3. Unlike the 2017 study, all individual duplicate sample results uploaded to the PTDB that have RSD exceedances of 20% will be qualified "J", an estimate. During the 2017 study, no data were qualified because of RSD guideline exceedances. The samples that exceeded the 20% RSD guideline had results that were 10 times below the compliance limits, and those results will not be used for enforcement.

6.2.2.2 Representativeness

Ecology staff will purchase products representative of those available to Washington's diverse residents. The practice of purchasing products from larger chain stores and online is used to generally reflect merchandise available and sold to residents across Washington state (Sekerak, 2016). In-store purchases will be from large chain stores that have multiple locations in both small towns and large cities of Washington. Where possible, products will be those that are available to diverse racial, cultural, and economic groups in Washington.

6.2.2.3 Completeness

The project will be considered complete if 95% of the products are successfully collected and the analytical results of the corresponding samples meet the project MQOs in Table 7.

6.3 Acceptance criteria for quality of existing data

Not applicable.

6.4 Model quality objectives

Not applicable.

7.0 Study Design

7.1 Study boundaries

Ecology will collect up to 100 brake products (e.g., brake pads and shoes) from a wide range of vehicles from various manufacturers, within the scope of the Better Brakes Program for Washington State. Ecology's Product Testing staff will collect products either in person from South Puget Sound area retail locations or online through internet retailers. General reconnaissance will be conducted of available brake products in the stores where products are purchased. Knowing available products will be useful for subsequent purchasing events.

Products will be selected based on data reported quarterly under the WA State Better Brakes certification program through NSF International. Other sources, as presented in Section 4.3, will provide additional information to help in product selection. The study will prioritize testing brake friction material products that have not been certified. Additional brake pad products will be tested from certified brake friction materials to verify accuracy of reporting and to assess B or N level compliance (see Section 3).

The primary target for this study is brake friction materials from passenger vehicles (Table 8). This study will use the National Highway Traffic Safety Administration (NHTSA) Vehicle Classification subcategories to divide and select the number of original equipment parts and aftermarket parts. Aftermarket parts are parts that are used in the upkeep or enhancement of a motor vehicle. Passenger vehicles will include subcategories determined by vehicle weight and type, including mini, light, compact, medium, heavy, sport utility, pickup trucks, and vans, as listed in Table 9.

The study will also target the collection of 11 special use brake products from vehicles (those not exempted per WAC 173-901-130) such as buses, ambulances, police vehicles, garbage trucks, and high-end sports cars, as listed in Table 8. An additional 11 brake products will be collected from the heavy duty, commercial vehicle category (over 26,000 pounds per RCW 70A.340.040(3)), such as short-haul and long-haul trucks.

Table 8. Number of products to be collected by vehicle category classifications.

Primary Category	Sub-Category	Number of Products	Notes
Passenger Vehicles	Original Equipment	16	See Table 9 for additional details.
Passenger Vehicles	Aftermarket	62	Purchase two brake products for Ecology vehicles off a state contract, if available.
Special Use Vehicles*	Bus	3	Could also be over 26,000 lbs. At least one off state contract, if available.
Special Use Vehicles	Ambulance	2	At least one off state contract, if available.
Special Use Vehicles	Police Car	2	At least one off state contract, if available.
Special Use Vehicles	Refuse Truck	2	Could also be over 26,000 lbs. At least one off state contract, if available.
Special Use Vehicles	High-End Sports Cars	2	Original equipment for model year 2021 or newer.
Heavy Duty / Commercial Vehicles (Over 26,000 lbs)	Drum	10	For example, short- and long-haul trucks.
Heavy Duty / Commercial Vehicles (Over 26,000 lbs)	Disc	1	For example, short- and long-haul trucks.
Total # of samples	–	100	–

lbs = pounds

*Manufacturers of special use vehicles may apply for an exemption (WAC 173-901-130).

Table 9. Number of products to be collected by passenger vehicle sub-category classifications.

NHTSA Classification	Sub-Category	OE Products*	Aftermarket Products**
Passenger cars: mini	1,500 to 1,999 lbs (680–907 kg)	2	2
Passenger cars: light	2,000 to 2,499 lbs (907–1,134 kg)	2	3
Passenger cars: compact	2,500 to 2,999 lbs (1,134–1,360 kg)	2	11
Passenger cars: medium	3,000 to 3,499 lbs (1,361–1,587 kg)	2	11
Passenger cars: heavy	3,500 lbs (1,588 kg) and over	2	11
Sport utility vehicles	–	2	11
Pickup trucks	–	2	11
Vans	–	2	2
Total # of samples	–	16	62

OE = Original Equipment

lbs = pounds

kg = kilograms

*Each original equipment product will be from a different brand of vehicle of model year 2021 or newer.

**Aftermarket products will be selected from a variety of manufacturers.

7.2 Field data collection

7.2.1 Sampling locations and frequency

Products will be purchased from in-store and online retailers over several weeks starting in August 2022. Products will be purchased online or from large chain stores that have multiple locations in both small towns and large cities of Washington. Where possible, products will be those that are available to diverse racial, cultural, and economic groups in Washington. In-store and online purchase events will be planned based on in-store and online product information reconnaissance.

7.2.2 Field parameters and laboratory analytes to be measured

At a minimum, the following will be recorded in the PTDB for all products in this study: store name, street address, website address, purchase date, purchase price, brand name, manufacturer name, manufacture date, and distributer name, among other descriptive metadata.

Table 10 lists the lab analytes to be measured by matrix. MEL will analyze seven chemicals for the total metals analyte group . The hexavalent chromium analyte group (by EPA 7199) will be analyzed at a contract lab. There are no field parameters to be measured for this study.

Table 10. Laboratory analytes and sample matrices for each analyte.

Analyte Group	Chemical Name	Acronym	CAS Number	Matrix
Total Metals	Cadmium	Cd	7440-43-9	Drilled Brake Friction Material
Total Metals	Lead	Pb	7439-92-1	Drilled Brake Friction Material
Total Metals	Mercury	Hg	7439-97-6	Drilled Brake Friction Material
Total Metals	Copper	Cu	7440-50-8	Drilled Brake Friction Material
Total Metals	Antimony	Sb	7440-36-0	Drilled Brake Friction Material
Total Metals	Nickel	Ni	7440-02-0	Drilled Brake Friction Material
Total Metals	Zinc	Zn	7440-66-6	Drilled Brake Friction Material
Hexavalent Chromium	Chromium(VI)	Cr(VI)	18540-29-9	Drilled Brake Friction Material

CAS: Chemical Abstracts Service

7.3 Modeling and analysis design

Not applicable.

7.4 Assumptions underlying design

One assumption is that online purchasing of products is available to most people in Washington State and that retail chain stores sell similar products at various locations throughout Washington.

Another assumption is that products will have an edge code and be labeled as per SAE J866. Products without edge codes will most likely not be certified. However, if there is no edge code, identifying the year of manufacture will be a challenge.

A final assumption is that each brake product will have a unique edge code, especially if certified, which will be representative of a particular manufacturer batch.

7.5 Possible challenges and contingencies

As this study focuses on brake products manufactured after January 1, 2021, it will be necessary to confirm a manufacture date by the product's edge code. Some products may not have an edge code, which will require verification with the Better Brakes Program about any exemptions in place. In addition, with online purchases, it may be more challenging to verify the manufacture date prior to receipt in the product testing lab.

7.5.1 Logistical problems

A combination of online and in-store purchasing will be used for acquiring products efficiently. In-store and online purchases may not have a manufacture date that can be confirmed prior to receipt in the product testing lab. Verification of any exemptions in place for brake products without edge codes could delay the processing of those brake products. Verifying the edge code for in-store purchasing may require registrar's (NSF International and/or AMECA) Excel spreadsheets being available for use during the purchasing events.

7.5.2 Practical constraints

The limited availability of the Ecology credit card and the restrictions of its usage may place additional constraints, considering there could be multiple purchasing events for this study. Approximate dates of purchasing should be forwarded to the appropriate officer or manager, as outlined in the *Product Collection and Sample Processing SOP* (Wiseman, 2021), to minimize inefficient and unproductive outings.

7.5.3 Schedule limitations

This project is scheduled to be completed by June 2023. There are some limitations that may affect the project timeline in an unpredictable manner. Shipping and receiving of products and brake friction material may be subject to delay during transport for various reasons. Lab testing for the metals analytes that are not accredited at MEL will take an extended period of time if accreditation is not complete by the time samples arrive at MEL. Complex product matrices may require an extended period of time for lab analysis, especially if the contract lab has never analyzed consumer products in the past.

8.0 Field Procedures

8.1 Invasive species evaluation

Not applicable.

8.2 Measurement and sampling procedures

Product purchasing and processing will follow SOP PTP001 *Procedure for Product Collection and Sample Processing* (Wiseman, 2021). Data entry and data quality assurance will follow SOP PTP002 *Data Entry and Data Entry Quality Assurance* (Wiseman, 2022). Products collected for this study will be shipped to, or transported to, Ecology's product testing preparation room where they will be processed by staff using appropriate engineering controls and PPE (see Section 9.2 for details). Photos will be taken of each product's packaging, any informational inserts, the brake pad or shoe itself, and the edge code if present. To minimize contact with potentially hazardous material, product meta data entry should be performed using pictures taken of the products to the maximum extent possible.

8.3 Containers, preservation methods, holding times

Table 11 presents sample matrices, sample minimum quantities, container specifications, preservation methods, and holding times. Brake products collected will be packed in sealed, certified metals-free, plastic bags when received at the product testing preparation room. All brake friction material products and drilled friction material samples will be labeled according to Section 8.5 and transported in Ecology-supplied coolers. The drilled friction material samples are not required to be chilled or packed with ice during transportation. These samples will be collected in a heavy gage sealable plastic bag (certified metals-free) provided by the contract lab, before being transferred to two 50 ml vials (certified metals-free, with tightly-fitting screw caps).

Table 11. Sample containers, preservation, and holding times.

Parameter	Matrix	Minimum Quantity Required	Container	Preservative	Holding Time
Total Metals: Cadmium, Lead, Mercury, Copper, Antimony, Nickel, Zinc, and Chromium	drilled friction material	1-2 g[+]	certified metals-free 50 ml plastic tube and screw cap	room temperature	1 year[Ω]
Chromium(VI)	drilled friction material	1-2 g[+]	certified metals-free 50 ml plastic tube and screw cap	room temperature	1 year[Ω]
Total Metals: Cadmium, Lead, Copper, Antimony, Nickel, Zinc, and Chromium	certified metals-free water blanks	50 ml	certified metals-free 50 ml plastic tube and screw cap	pH<2 with nitric acid[Ω+], ≤6°C	6 months
Total Mercury[++]	certified metals-free water blanks	50 ml	certified metals-free 50 ml plastic tube and screw cap	pH<2 with nitric acid[Ω+], ≤6°C	28 days

[+]Minimum quantity does not include sample duplicates, matrix spikes, and matrix spike duplicates, but does account for lab duplicates.
[Ω]This is an approximate holding time for product testing samples received at MEL. Storage may not be standard across all labs.
[++]Mercury will need to be performed on a different blank than the metals because each container can hold only 50 ml.
[Ω+] MEL will preserve blanks upon receipt. Total chromium will be analyzed at MEL on blanks, as the contract lab will not be analyzing blanks for hexavalent chromium.

8.4 Equipment decontamination

To obtain reliable data, it is essential that Product Testing staff employ effective decontamination processes. These procedures should follow protocols outlined in the *Product Testing SOP for Product Collection and Sample Processing* (Wiseman, 2021). Brake friction material may contain asbestos fibers; therefore; staff will open packages and handle brake products in a fume hood and while wearing appropriate PPE. Staff will clean the fume hood with a 1% Liquinox solution followed by 24% ethanol spray prior to use, between processing each product, and after use. Staff will place a new sheet of aluminum soil, dull side up, lined with Kim wipes onto the fume hood working space to sit each new brake product on. No two brake products will be present in the fume hood together at any time. Once a product is sealed into a labeled plastic bag, the bag will remain sealed until processing by the drilling laboratory.

8.5 Sample ID

Individual product component identifications (IDs) are auto-generated by the PTDB during product and component login, as described in the *Product Testing Database Standard Operating Procedure for Data Entry and Data Entry Quality Assurance* (Wiseman, 2022). Product IDs convey information about the store of purchase, the purchase event, product number, and component number (e.g., "AZ-1-3-1" means Auto Zone store, purchase event 1, product number 3, component number 1 of the product tested (Sekerak, 2016)).

MEL will generate a seven-digit work order number (WO#; e.g., 1601027) for each sample set(s) for an individual study. During sample processing, the addition of a two-digit suffix to the WO# will result in a lab sample ID number (e.g., 1601027-01, 1601027-02) for each sample (Sekerak, 2016). There will be two subsamples per sample for the duplicate analysis of the eight selected metals, resulting in 16 individual results per sample.

Staff will record lab sample ID numbers and their corresponding product component IDs on both the sample containers and the chain of custody form. Lab sample ID numbers are generated the same for both MEL and subcontract analysis. The same MEL sample ID will be retained for the subcontract analysis of chromium(VI), as the remaining sample from total metals analysis will be sent for testing.

Product Testing staff will label each bag containing a brake product with the product component ID number and the three associated MEL-assigned lab numbers. Product Testing staff will send labels to the contract lab for them to affix to each 50 ml tube containing the final drilled friction material samples.

8.6 Chain of custody

Chain of custody will be maintained for all samples throughout the study. Specific protocols are outlined in the *Product Collection and Sample Processing SOP* (Wiseman, 2021) for storage of products, samples, and shipment of product component samples to the lab. Refer to Section 9.2 for safe handling of brake products.

The contract lab will use the MEL chain of custody form for all samples throughout the contracted process, unless the contract lab specifies the use of their chain of custody form. MEL will make a comment about pretreatment on the chain of custody used for shipping samples to the contract lab for chromium(VI) analysis. The comment should indicate the lab pretreatment method code as SAEJ2975-Drill.

8.7 Field log requirements

In addition to the specific protocols outlined in the *Product Collection and Sample Processing SOP* (Wiseman, 2021), the information listed below will be recorded for each product purchased, unless the information is unavailable. This information will be logged into the PTDB when entering the product information:

- Vehicle make, model, and year that the brake is purchased for.
- Brake edge code.
- The environmental compliance level letter and the brake manufacture year to be determined from the edge code.
- Notation that the product packaging does not have the LeafMark™ symbol.
- Notation that the LeafMark™ environmental compliance level letter does not match the compliance code on the brake product.

8.8 Other activities

Ecology staff will pre-screen some existing retention samples from the 2017 study. The pre-screening results will not be uploaded to the PTDB. MEL will use the pre-screening data to verify the suitability of the method for analyzing brake products (see Section 9.1 for details). Retention samples may be kept for drilled friction material if there is material left over after analysis. Since drilled brake friction material is not readily available, storing some retention samples may be beneficial for future brake product studies. Also refer to the *Product Collection and Sample Processing SOP* (Wiseman, 2021) for guidance on retaining samples.

Brake products will be sent to the contract lab to be processed by drilling to create small particle sized friction material for analysis, in accordance with SAE J2975. Chapter 173-901 WAC references SAE J2975:2011. The current version is SAE J2975:2020, which may be relevant for the drilling lab.

The contract lab will send the drilled brake friction material samples directly to MEL in two labeled 50 ml plastic tubes, as stated in Table 11. As part of the contracting process, the tubes will be certified metals-free. The drilling contract lab will perform a screen for asbestos and will not process any brakes in which asbestos is a concern. After observing their standard holding time, the drilling contract lab will dispose of, or recycle, the drilled brake products.

The drilling contract lab will collect three 50 ml vial blanks and processing drill blanks for each drilling batch, as outlined in Sections 6.2.1.2 and 9.3. Deionized water will be certified metals-free as part of the contracted process. Since the blanks are in an aqueous state, they will be packed in a small cooler with cold packs, alongside the sealed brake friction material samples. After MEL receives the blanks, they will preserve them to a pH less than 2.0 with nitric acid, as required.

9.0 Laboratory Procedures

9.1 Lab procedures table

This study plans for MEL to conduct analyses for selected metals listed in Table 10, with the exception of chromium(VI) which will be tested at a contract lab. MEL will attempt to obtain the target reporting limits outlined in Table 12. MEL will use digestion method EPA 3051A (MEL SOP# 720015) and analysis method EPA 6010D (MEL SOP# 720029) by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES). Because method EPA 3050B was used on the 2017 study, MEL will use this method to analyze retention samples from the 2017 study. Analyzing the retention samples before performing analysis for this 2022 study will have four purposes:

- Verification that accredited metals can be analyzed for drilled brake friction material by comparing results to previous testing, including evaluation of recovery of matrix spikes and lab control samples.
- Verification of digestion efficiency for the reconfiguration of microwaves during sample preparation (refer to Section 9.2 for details.
- Verification of the feasibility for analyzing mercury using EPA 6010D, based on recovery of matrix spikes and lab control samples.
- Obtaining accreditation for antimony, copper, nickel, zinc, and mercury.

A contract lab will be used for the drilling of the brake friction material so uniform granular particulate is produced for subsequent metals digestion. MEL will manage all contracts for drilling and lab analysis.

MEL will receive two vials of brake friction material from the contract drilling lab. One of those vials will be send directly to the contract lab for chromium(VI) analysis by EPA 7199. The chain-of-custody form will also include a comment about pretreatment (see Section 8.6).

Table 12. Measurement methods (laboratory).

Analyte	Sample Matrix	Number of Samples	Estimated Arrival Date	Expected Range of Results (%)	Target Reporting Limit (ppm)[◊]	Analytical Method
Total Metals: Cadmium, Lead, Mercury, Copper, Antimony, Nickel, Zinc[∞]	Drilled Brake Friction Material	Up to 100	November 2022	<0.1 - 20	10	EPA 6010D[ΩΩ] ICP-OES[Ω]
Total Metals: Cadmium, Lead, Copper, Antimony, Nickel, Zinc, Chromium	Water Blanks	Up to 4	November 2022	<RL	0.5	EPA 200.8[++] ICP-MS[+]
Total Mercury	Water Blanks	Up to 4	November 2022	<RL	0.5	EPA 7470A[∞∞] Cold Vapor
Chromium(VI)	Drilled Brake Friction Material	Up to 100	November 2022	<0.1 - 10	0.3	EPA 7199[◊◊] Ion Chromatography
Drilled Brake Friction Material	Brake Friction Material	Up to 100	October 2022	<0.1 (asbestos)[**]	N/A	Contract Lab SAE J2975[*]

[◊]Individual lab reporting limits may vary based upon specific matrix type.
[∞]The drilled brake friction materials will be sent directly to MEL by contract lab.
[ΩΩ]EPA = U.S. Environmental Protection Agency.
[Ω]ICP-OES = Inductively Coupled Plasma-Optical Emission Spectrometry.
[++]EPA 200.2 (MEL SOP# 720002) will be used as the preparation method. For EPA 200.8 the MEL SOP# is 720035.
[+]ICP-MS = Inductively Coupled Plasma-Mass Spectrometry.
[∞∞]For EPA 7470A the MEL SOP# is 720009.
[◊◊]EPA 3060A will be used as the preparation method. MEL will send the drilled brake friction materials directly to the contract lab if total chromium result exceeds 0.1% by weight.
[**]Brake friction material may contain asbestiform fibers; therefore, staff should use appropriate engineering controls and/or PPE (see Section 9.2).
[*]A computer numerical control (CNC) drill will be used.

9.2 Sample preparation methods

Sample processing and preparation done by Product Testing staff will follow procedures outlined in the *Product Collection and Sample Processing SOP* (Wiseman, 2021). Brake products may contain asbestiform fibers; therefore, staff should use appropriate engineering controls and PPE. All activities involving handling brake products should be performed in the fume hood while wearing a lab coat and nitrile gloves. Product data entry should be performed using the pictures taken, to the maximum extent possible.

MEL will send brake products to the contract lab to be processed by drilling in accordance with SAE J2975. The contract lab will send the drilled brake friction material to MEL for metals analysis. MEL will send each sample duplicate to another contract lab for chromium(VI) analysis, as described in Section 9.1.

MEL staff should use appropriate PPE when receiving the drilled brake friction material. Due to potentially hazardous materials in brake dust, the brake friction material should be handled in a fume hood.

MEL will perform microwave-assisted acid digestion by EPA 3051A. The microwave digestion is a different preparation method than performed in 2017, in which EPA 3050B (hot plate digestion) was used. Before this 2022 study, MEL will perform microwave digestions on retention samples to verify compatibility with drilled brake friction material. In addition to comparing results from the 2017 study, MEL will evaluate matrix spikes on the retention samples as well as recoveries from lab control samples. As part of this process, MEL will verify if the digestion is suitable for the analysis of mercury and obtain accreditation for mercury analysis using EPA 6010D (see section 9.4).

9.3 Special method requirements

The drilling contractor will be required to prepare storage and drill processing blanks. Before beginning any drilling, a storage blank will be prepared by filling a clean empty plastic vial (same as used for the drilled brake friction material samples, refer to Section 8.3), with a minimum of 50 ml deionized water (DI). Three storage blank vials will be collected at the beginning, middle, and end of the drilling process for all the samples submitted. The storage blanks should be stored and shipped to MEL with their associated samples. MEL will preserve these blanks to a pH <2 with nitric acid, as required.

The contract lab will prepare drill processing blanks for each batch of brake friction material drilled or at the end of a drilling session. The contract lab will define their meaning of a batch (or a drilling session) and what drill friction material samples are associated with each drill processing blank. It is anticipated there will be at least 10 processing blanks collected for this 2022 study. This information will be recorded on the chain of custody form and/or written lab narrative report that will accompany the samples sent to MEL.

Each processing blank will be prepared by opening a clean empty vial (same as used for the drilled brake friction material) filled with deionized water, and setting the vial near the drilling operations. At the end of the drilling for the selected sample of the batch, the vial will be capped. The drill processing blanks are treated like the storage blanks. They will be stored, shipped, and chilled, along with the associated samples. Some of the blanks will be analyzed to assist in evaluating any potential sources of contamination (see Section 10.1). The deionized water used by the contract lab should be specified metals-free as part of the contracting process (see Section 8.3).

MEL and the contract lab carrying out the analysis for chromium(VI) must meet the acceptance criteria in Section 6.2 and also demonstrate the ability to achieve the reporting limits outlined in Section 9.1. There are no reference standards for brake friction materials.

9.4 Labs accredited for methods

Ecology's MEL is currently accredited for analysis of cadmium, chromium, and lead, by EPA Method 6010D. MEL is scheduled to have accreditation for antimony, copper, mercury, nickel, and zinc by EPA 6010D before analyzing brake samples for this 2022 project. EPA 6010D is an approved method in SAE J2975 for testing of chromium, copper, mercury, and other metals present in brake friction material. The contract lab is accredited for analysis of chromium(VI) by EPA 7199.

10.0 Quality Control Procedures

10.1 Table of field and laboratory quality control

Table 13 presents the lab QC procedures for this study. Lab QC tests will consist of method blanks, lab control samples, lab control sample duplicates, sample duplicates, matrix spikes, matrix spike duplicates, drill processing blanks, storage blanks, and field replicates.

Table 13. Quality control samples, types, and frequency.

Parameter	LCS/LCSD	Method Blanks	Sample Duplicates	Matrix Spikes/MSD	Field Replicates	Field Drill Processing Blanks	Field Storage Blanks
Metals Analysis	1/batch	1/batch	1/batch	1/batch	Up to 2	1/batch	3◊◊

LCS = laboratory control samples
LCSD = laboratory control sample duplicates
MSD = matrix spike duplicates
◊◊One blank will be for total metals and one will be for total mercury.
Batch = 20 samples or fewer of analytical samples

10.2 Corrective action processes

The project manager will work closely with the laboratories, appropriate QA representatives, and any independent reviewers conducting data validation to examine data that fall outside of QC criteria. Product Testing staff will also adhere to appropriate SOPs and study-specific processing and preparation protocols. When QC criteria are not met, or if the integrity of the processing and preparation processes are in question, the project manager will determine if samples should be re-sampled, re-analyzed, rejected, or used with appropriate qualifications.

11.0 Data Management Procedures

11.1 Data recording and reporting requirements

Product login will follow the Product Testing Program SOP

- PTP002 *Product Testing Database Standard Operating Procedure For Data Entry and Data Entry Quality Assurance, Version 2.1* (Wiseman, 2022).

Study data will be stored in Ecology's Product Testing Database (PTDB). The database stores product descriptions, purchase receipts, photos of products, drilling photos, lab data, and case narratives.

Lab data will be transferred electronically from MEL's Laboratory Information Management System (LIMS) into the PTDB or arrive as an electronic data deliverable (EDD) package (.CSV).

After verification by the project manager, the data from duplicate analysis of each brake friction material will be entered into the PTDB. Therefore, each component will have three different sets of results for the parameters tested. Averaging and reporting will be conducted using the protocol described in Section 6.2.1.

Before loading any data into the PTDB, the project manager will perform a QA review. After completing the QA review, the project manager or designated staff will upload the final QA-reviewed data to the internal PTDB (Sekerak, 2016). The QC samples sent to the lab (storage blanks and processing blanks), to be analyzed with the study samples, can be uploaded as part of the EDD into the internal PTDB, but will not be transferred to the public database.

Documentation for contract drilling brake friction material

The contract lab will provide documentation including: before and after photos of the drilled brake products, before and after photos of the labelled collection tubes, and correlation between the blanks and the associated MEL ID numbers.

The project manager will verify the documentation for the drilling of brake friction material for completeness and accuracy (per SAE J2975). The before and after photos provided by the contract lab will be uploaded to the internal PTDB after verification. All project photos will be stored in the Product Testing F Drive, in study-specific folders.

11.2 Laboratory data package requirements

Labs performing analyses for this project will provide an EDD package (i.e., .csv or .xlsx files) after completing their work.

Case narratives will be included to discuss any problems encountered with the analyses, corrective action taken, changes to the requested analytical method, and a glossary for data flags and qualifiers. All sample results and QC data will be included with the package (Sekerak, 2016).

When data validation is required, study-specific contract lab requirements will be discussed more thoroughly in contract documents (e.g., Request for Laboratory Services). For chromium(VI) analysis, the contract lab will deliver a Tier 4 Level data package to MEL with the complete raw laboratory dataset. For MEL-generated data, case narratives and supporting analytical data will fulfill the requirements in Section 13.2. An independent validation will be performed as described in Section 13.3.

11.3 Electronic transfer requirements

Case narratives will be in Adobe Acrobat (.PDF) format, and EDDs will be in a .CSV spreadsheet format.

For data generated by MEL, case narratives will be sent to the project manager via email, and electronic data will be delivered through LIMS into the internal PTDB.

Contract labs will submit data to MEL as a fully paginated and bookmarked comprehensive .PDF file, with all contract-specified content, along with the EDD in the case of chromium(VI) analysis (.CSV). Smaller files may be sent through email, while larger files may be required to be submitted on compact disk. MEL will provide the contract lab for chromium(VI) analysis with the EDD template and EDD Help documents at the time of the request of services.

MEL will email the following to the project manager: case narratives in PDF format, as well as final EDDs with MEL-amended results and MEL-amended qualifier columns in an Excel spreadsheet format

11.4 EIM/STORET data upload procedures

Not applicable. Section 11.1 describes the database where data will be stored for this project.

11.5 Model information management

Not applicable.

12.0 Audits and Reports

12.1 Field, laboratory, and other audits

The analytical lab must participate in performance and system audits of their routine procedures.

The product testing process conducted at Ecology will be audited at least once a year.

12.2 Responsible personnel

Ecology's QA Officer or a designee will conduct the product testing process audit. The processes can include: product acquisition, product documentation and data entry in the PTDB, sample screening, sample processing, chain-of-custody, and adherence to product testing QAPPs and SOPs.

12.3 Frequency and distribution of reports

A final published report summarizing the data and findings will include, at a minimum:

- An overview of the study.
- Clear and concise goals and objectives of the study.
- General descriptions of products purchased.
- Summary of the contracted drilling documentation.
- Summary of lab results.
- Discussion of lab results and data quality.
- Conclusions.

12.4 Responsibility for reports

The principal investigator will be responsible for the final report.

13.0 Data Verification

13.1 Field data verification, requirements, and responsibilities

The project manager, or assigned and qualified designee, will conduct a final review of product entry and drilling documentation generated within a project.

Product Testing staff will review all data entered into the PTDB at several stages during each study according to the *Product Testing Database SOP for Data Entry and Data Entry Quality Assurance* (Wiseman, 2022).

13.2 Laboratory data verification

Lab data verification is a review process to assess the quality and completeness of analytical data. A detailed examination of all lab data sets includes a review for errors, omissions, interpretations, calculations, qualifications, and compliance with all appropriate QC acceptance criteria and contract requirements. MEL's QA Coordinator will assess contract lab Tier 4 level data packages, following MEL's SOPs and the EPA *National Functional Guidelines for Inorganic Data Review* (EPA, 2020).The contract lab will provide a Tier 4 level data package, but MEL will perform only a Stage 3 EPA validation, as outlined in Section 13.3. MEL's SOPs for data reduction, review, and reporting will meet the needs of the project. The specific MEL SOP# for review of inorganics data and package completion is 720017 (MEL, 2020). An independent Stage 3 validation will be performed as described in Section 13.3.

Lab staff will generate case narratives and submit them, along with the lab data, to the project manager. The narratives will include a discussion describing if (1) MQOs are met, (2) proper analytical methods and protocols were followed, (3) calibrations and controls were within limits, and (4) data were consistent, correct, and complete, without errors or omissions (Sekerak, 2016).

The principal investigator, with guidance from Ecology's QA Officer as necessary, will be responsible for the final acceptance of the lab data. The contract lab case narratives and electronic data deliverable, along with MEL's written report, will be assessed for completeness and reasonableness. Based on these assessments, the data will be accepted, accepted with qualifications, or rejected and re-analysis considered.

13.3 Validation requirements, if necessary

Lab data validation is an analyte-specific and sample-specific process that extends the evaluation of data beyond data verification in order to determine the analytical quality of a specific data set. Data validation requirements for Ecology's product testing studies follows established Ecology Guidelines for Data Verification and Validation of Chemical Data. MEL will conduct an EPA Stage 3 validation for MEL-generated data and contract lab data, using the recommended validation checks described in EPA Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA, 2009), or equivalent. During Stage 3 validation, recalculations include:

- Initial calibration (ICAL)
- Continuing calibration(s) (CCAL)
- QC sample results [LCS/LCSD/OPR (Ongoing Precision and Recovery), MS/MSD LD (Laboratory Duplicate), as appropriate]
- Field sample result for one sample and duplicate, if analyzed
- For chromium(VI)
    - Method or analysis type/technology
    - Matrix

13.4 Model quality assessment

Not applicable.

14.0 Data Quality (Usability) Assessment

14.1 Process for determining if project objectives were met

The project manager will assess the quality of the data based on case narratives and data packages. Lab QC tests will be examined to determine if the labs met MQOs for method blanks, LCSs, duplicates, matrix spike samples, duplicate sample analysis, and drilling blanks when applicable. The project manager will examine reporting limits to ensure that they are met as described in Sections 6 and 9.

Further assessments of duplicates, spike performance, and sample duplicates will be used to evaluate any effects of sample matrix on the data quality.

Evaluating blanks will aid in determining contamination, interferences, and precision for samples with low concentrations near analytical detection limits.

This project's final report will discuss data quality and whether the project objectives were met. If limitations in the data are identified, these limitations will be noted.

14.2 Treatment of non-detects

Lab data will be reported down to the reporting limit, with an associated "U" or "UJ" qualifier for samples below the reporting limit (Sekerak, 2016).

For computing RSD, non-detects will be averaged using the reporting limit. If all three replicates are non-detects, the average result will be reported as less than the reporting limit.

14.3 Data analysis and presentation methods

The final report will include a summary of the results. Data and simple summary statistics will be presented in tables and graphs. Example summary statistics may include minimum, maximum, and frequencies of detection.

The final report will also include a link to the study data available on the external database: https://apps.ecology.wa.gov/ptdbpublicreporting/

14.4 Sampling design evaluation

The number and type of collected samples will be sufficient to meet the objectives of this study.

14.5 Documentation of assessment

The final report will include documentation of assessment.

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16.0 Appendix: Acronyms, Abbreviations, and Glossary

Acronyms and Abbreviations

AMECA	Automotive Manufacturers Equipment Compliance Agency Inc.
CAS	Chemical Abstracts Service
CCAL	Continuing Calibration
CNC	Computer Numerical Control
e.g.	For example
DES	Washington State Department of Enterprise Services
DI	Deionized
DQO	Data Quality Objective
DTSC	California State Department of Toxic Substances Control
EAP	Environmental Assessment Program
Ecology	Washington State Department of Ecology
ECOS	Environmental Council of the States
EDD	Electronic Data Deliverable
EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency
HWTR	Hazardous Waste and Toxics Reduction
ICAL	Initial Calibration
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectrometry
ID	Identification
ISO	International Organization for Standardization
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LD	Laboratory Duplicate
LIMS	Laboratory Information Management System
MEL	Manchester Environmental Laboratory
MEMA	Motor Equipment Manufacturers Association
MOU	Memorandum Of Understanding
MQO	Measurement Quality Objective
MS	Matrix Spike
MSD	Matrix Spike Duplicate
N/A	Not Applicable
NHTSA	National Highway Traffic Safety Administration
NSF	National Sanitation Foundation
OE	Original Equipment
OPR	Ongoing Precision and Recovery
P2RA	Pollution Prevention and Regulatory Assistance
PTDB	Product Testing Database
PPE	Personal protective equipment

QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCW	Revised Code of Washington
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SCS	Statewide Coordination Section
SOP	Standard Operating Procedure
TM	Trademark
WAC	Washington Administrative Code
WO	Work Order

Units of Measurement

°C	degrees centigrade
g	gram, a unit of mass
kg	kilograms, a unit of mass equal to 1,000 grams
lbs	pounds
mg/kg	milligrams per kilogram (parts per million)
ml	milliliter

Quality Assurance Glossary

Accreditation: A certification process for laboratories, designed to evaluate and document a lab's ability to perform analytical methods and produce acceptable data. For Ecology, it is "Formal recognition by (Ecology)…that an environmental laboratory is capable of producing accurate analytical data." [WAC 173-50-040] (Kammin, 2010)

Accuracy: The degree to which a measured value agrees with the true value of the measured property. USEPA recommends that this term not be used, and that the terms *precision* and *bias* be used to convey the information associated with the term *accuracy* (USGS, 1998).

Analyte: An element, ion, compound, or chemical moiety (pH, alkalinity) which is to be determined. The definition can be expanded to include organisms, e.g., fecal coliform, Klebsiella (Kammin, 2010).

Bias: The difference between the sample mean and the true value. Bias usually describes a systematic difference reproducible over time and is characteristic of both the measurement system and the analyte(s) being measured. Bias is a commonly used data quality indicator (DQI) (Kammin, 2010; Ecology, 2004).

Blank: A synthetic sample, free of the analyte(s) of interest. For example, in water analysis, pure water is used for the blank. In chemical analysis, a blank is used to estimate the analytical response to all factors other than the analyte in the sample. In general, blanks are used to assess possible contamination or inadvertent introduction of analyte during various stages of the sampling and analytical process (USGS, 1998).

Calibration: The process of establishing the relationship between the response of a measurement system and the concentration of the parameter being measured (Ecology, 2004).

Check standard: A substance or reference material obtained from a source independent from the source of the calibration standard; used to assess bias for an analytical method. This is an obsolete term, and its use is highly discouraged. See Calibration Verification Standards, Lab Control Samples (LCS), Certified Reference Materials (CRM), and/or spiked blanks. These are all check standards but should be referred to by their actual designator, e.g., CRM, LCS (Kammin, 2010; Ecology, 2004).

Comparability: The degree to which different methods, data sets and/or decisions agree or can be represented as similar; a data quality indicator (USEPA, 1997).

Completeness: The amount of valid data obtained from a project compared to the planned amount. Usually expressed as a percentage. A data quality indicator (USEPA, 1997).

Continuing Calibration Verification Standard (CCV): A quality control (QC) sample analyzed with samples to check for acceptable bias in the measurement system. The CCV is usually a midpoint calibration standard that is re-run at an established frequency during the course of an analytical run (Kammin, 2010).

Control chart: A graphical representation of quality control results demonstrating the performance of an aspect of a measurement system (Kammin, 2010; Ecology 2004).

Control limits: Statistical warning and action limits calculated based on control charts. Warning limits are generally set at +/- 2 standard deviations from the mean, action limits at +/- 3 standard deviations from the mean (Kammin, 2010).

Data integrity: A qualitative DQI that evaluates the extent to which a data set contains data that is misrepresented, falsified, or deliberately misleading (Kammin, 2010).

Data quality indicators (DQI): Commonly used measures of acceptability for environmental data. The principal DQIs are precision, bias, representativeness, comparability, completeness, sensitivity, and integrity (USEPA, 2006).

Data quality objectives (DQO): Qualitative and quantitative statements derived from systematic planning processes that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions (USEPA, 2006).

Data set: A grouping of samples organized by date, time, analyte, etc. (Kammin, 2010).

Data validation: An analyte-specific and sample-specific process that extends the evaluation of data beyond data verification to determine the usability of a specific data set. It involves a detailed examination of the data package, using both professional judgment and objective criteria, to determine whether the MQOs for precision, bias, and sensitivity have been met. It may also include an assessment of completeness, representativeness, comparability, and

integrity, as these criteria relate to the usability of the data set. Ecology considers four key criteria to determine if data validation has actually occurred. These are:

- Use of raw or instrument data for evaluation.
- Use of third-party assessors.
- Data set is complex.
- Use of EPA Functional Guidelines or equivalent for review.

Examples of data types commonly validated would be:

- Gas Chromatography (GC).
- Gas Chromatography-Mass Spectrometry (GC-MS).
- Inductively Coupled Plasma (ICP).

The end result of a formal validation process is a determination of usability that assigns qualifiers to indicate usability status for every measurement result. These qualifiers include:

- No qualifier – data are usable for intended purposes.
- J (or a J variant) – data are estimated, may be usable, may be biased high or low.
- REJ – data are rejected, cannot be used for intended purposes.
  (Kammin, 2010; Ecology, 2004).

Data verification: Examination of a data set for errors or omissions, and assessment of the Data Quality Indicators related to that data set for compliance with acceptance criteria (MQOs). Verification is a detailed quality review of a data set (Ecology, 2004).

Detection limit (limit of detection)**:** The concentration or amount of an analyte which can be determined to a specified level of certainty to be greater than zero (Ecology, 2004).

Duplicate samples: Two samples taken from and representative of the same population, and carried through and steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variability of all method activities including sampling and analysis (USEPA, 1997).

Field blank: A blank used to obtain information on contamination introduced during sample collection, storage, and transport (Ecology, 2004).

Initial Calibration Verification Standard (ICV): A QC sample prepared independently of calibration standards and analyzed along with the samples to check for acceptable bias in the measurement system. The ICV is analyzed prior to the analysis of any samples (Kammin, 2010).

Laboratory Control Sample (LCS): A sample of known composition prepared using contaminant-free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is prepared and analyzed in the same batch of regular samples using the same sample preparation method, reagents, and analytical methods employed for regular samples (USEPA, 1997).

Matrix spike: A QC sample prepared by adding a known amount of the target analyte(s) to an aliquot of a sample to check for bias due to interference or matrix effects (Ecology, 2004).

Measurement Quality Objectives (MQOs)**:** Performance or acceptance criteria for individual data quality indicators, usually including precision, bias, sensitivity, completeness, comparability, and representativeness (USEPA, 2006).

Measurement result: A value obtained by performing the procedure described in a method (Ecology, 2004).

Method: A formalized group of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, data analysis), systematically presented in the order in which they are to be executed (EPA, 1997).

Method blank: A blank prepared to represent the sample matrix, prepared and analyzed with a batch of samples. A method blank will contain all reagents used in the preparation of a sample, and the same preparation process is used for the method blank and samples (Ecology, 2004; Kammin, 2010).

Method Detection Limit (MDL): This definition for detection was first formally advanced in 40CFR 136, October 26, 1984 edition. MDL is defined there as the minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, and reported to be greater than zero (Federal Register, October 26, 1984).

Percent Relative Standard Deviation (%RSD): A statistic used to evaluate precision in environmental analysis. It is determined in the following manner:

$$\%RSD = (100 * s)/x$$

where s is the sample standard deviation and x is the mean of results from more than two replicate samples (Kammin, 2010).

Parameter: A specified characteristic of a population or sample. Also, an analyte or grouping of analytes. Benzene and nitrate + nitrite are all parameters (Kammin, 2010; Ecology, 2004).

Population: The hypothetical set of all possible observations of the type being investigated (Ecology, 2004).

Precision: The extent of random variability among replicate measurements of the same property; a data quality indicator (USGS, 1998).

Quality assurance (QA): A set of activities designed to establish and document the reliability and usability of measurement data (Kammin, 2010).

Quality Assurance Project Plan (QAPP): A document that describes the objectives of a project, and the processes and activities necessary to develop data that will support those objectives (Kammin, 2010; Ecology, 2004).

Quality control (QC): The routine application of measurement and statistical procedures to assess the accuracy of measurement data (Ecology, 2004).

Relative Percent Difference (RPD): RPD is commonly used to evaluate precision. The following formula is used:

[Abs(a-b)/((a + b)/2)] * 100

where "Abs()" is absolute value and a and b are results for the two replicate samples. RPD can be used only with 2 values. Percent Relative Standard Deviation is (%RSD) is used if there are results for more than 2 replicate samples (Ecology, 2004).

Replicate samples: Two or more samples taken from the environment at the same time and place, using the same protocols. Replicates are used to estimate the random variability of the material sampled (USGS, 1998).

Representativeness: The degree to which a sample reflects the population from which it is taken; a data quality indicator (USGS, 1998).

Sample (field): A portion of a population (environmental entity) that is measured and assumed to represent the entire population (USGS, 1998).

Sample (statistical): A finite part or subset of a statistical population (USEPA, 1997).

Sensitivity: In general, denotes the rate at which the analytical response (e.g., absorbance, volume, meter reading) varies with the concentration of the parameter being determined. In a specialized sense, it has the same meaning as the detection limit (Ecology, 2004).

Spiked blank: A specified amount of reagent blank fortified with a known mass of the target analyte(s); usually used to assess the recovery efficiency of the method (USEPA, 1997).

Spiked sample: A sample prepared by adding a known mass of target analyte(s) to a specified amount of matrix sample for which an independent estimate of target analyte(s) concentration is available. Spiked samples can be used to determine the effect of the matrix on a method's recovery efficiency (USEPA, 1997).

Split sample: A discrete sample subdivided into portions, usually duplicates (Kammin, 2010).

Standard Operating Procedure (SOP): A document which describes in detail a reproducible and repeatable organized activity (Kammin, 2010).

Surrogate: For environmental chemistry, a surrogate is a substance with properties similar to those of the target analyte(s). Surrogates are unlikely to be native to environmental samples. They are added to environmental samples for quality control purposes, to track extraction efficiency and/or measure analyte recovery. Deuterated organic compounds are examples of surrogates commonly used in organic compound analysis (Kammin, 2010).

Systematic planning: A step-wise process which develops a clear description of the goals and objectives of a project, and produces decisions on the type, quantity, and quality of data that will be needed to meet those goals and objectives. The DQO process is a specialized type of systematic planning (USEPA, 2006).

References for QA Glossary

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