SURFACE VEHICLE RECOMMENDED PRACTICE

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Measurement of Copper and Other Elements in Brake Friction Materials

RATIONALE

The states of Washington and California passed laws in 2011 restricting the amount of cadmium, copper, hexavalent chromium, lead and mercury their compounds in brake friction materials. As part of the legislation, brake friction materials are required to be analyzed to confirm the levels of the restricted elements are below the values stated in the legislation. The SAE J2975 provides a method on how to generate samples of brake friction material for analysis, how to prepare the samples for analysis and how to analyze the samples.

We have expanded Section 4.3.2 for Hexavlent Chromium content due to some limitations discovered in recent testing.

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1. SCOPE

This procedure describes a method for generating, preparing and analyzing samples of new and unused brake friction materials for their chemical constituents.

1.1 Purpose

This recommended test practice is intended to establish a standardized and repeatable method for analyzing the amount of specific chemical elements in a new and unused brake friction material. Results from this test practice provide the basis to determine the appropriate environmental marking per SAE J866.

2. REFERENCES

2.1 Applicable Documents

The following publications form a part of this specification to the extent specified herein. Unless otherwise indicated, the latest issue of SAE publications shall apply.

2.1.1 EPA Publications

Available from the United States Environmental Protection Agency (EPA), http://www.epa.gov.

EPA Method 3050B Acid Digestion of Sediments, Sludges and Soils

EPA Method 3051A Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils

EPA Method 6010C Inductively Coupled Plasma - Atomic Emission Spectrometry

EPA Method 7471B Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)

EPA Method 3060A Alkaline Digestion for Hexavalent Chromium

EPA Method 7196A Chromium, Hexavalent (Colorimetric)

EPA Method 7199 Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography

EPA/600/R-93/116 Method for the Determination of Asbestos in Bulk Building Materials

SAE J866 Friction Coefficient Identification and Environmental Marking System for Brake Linings

3. DEFINITIONS

3.1 Symbols and Abbreviations

ICP-AES - Inductively Coupled Plasma - Atomic Emission Spectrometry

4. SAMPLE PREPARATION

4.1 Preparation of Brake Friction Material

4.1.1 Wipe entire friction assembly with lint-free wipe (i.e., Kimwipe®) to ensure assembly is free of any dust and foreign objects. Multiple wipes may be needed to accomplish this task. Alternatively, the assembly may be rinsed with de-ionized water and then dried with a lint free wipe.

4.1.2 Determine the thickness of the friction material. For assemblies without any steel back or shoe assembly, simply measure the thickness. For friction assemblies, the thickness of the friction material can be measured directly as shown in Figure 1 or the total assembly can be measured and then subtract the thickness of the pressure plate / shoe steel to obtain the friction material thickness.

FIGURE 1 - MEASUREMENT OF FRICTION MATERIAL THICKNESS

4.1.3 Place friction assembly in a heavy gage sealable plastic bag (to collect turnings). Place friction assembly on the table of the drill press in a safe manner that meets the desired safety requirements of the laboratory. Care should be taken to insure the plastic bag does not tear or become damaged during the clamping or drilling. (Some drill presses may have guarding that prohibits the effective use of a plastic bag. In this case, care should be taken to keep all grinding debris on the friction face.)

4.1.4 Set the depth stop on the drill press such that it is at the midpoint of the friction material thickness measured in 4.1.2 as shown in Figure 2.

FIGURE 2 - SETTING THE DRILL PRESS STOP AT ½ THE FRICTION MATERIAL THICKNESS

4.1.5 Set the speed of the drill such that it rotates approximately 400 ± 100 rpm at the spindle.

4.1.6 Using a 3mm diameter solid carbide drill bit, drill numerous borings (35-40 borings to a 5mm depth yields greater than 2 grams of sample) into the friction material uniformly around the entire area to generate enough fines (>2 g) for subsequent acid digestion. For larger samples of friction, a minimum of 1 hole per 2 cm^2 is recommended to ensure uniform sampling. To avoid any edge effects from the processing of the friction material, do not drill within 6 mm of the outer circumference of the friction material (Figure 3).

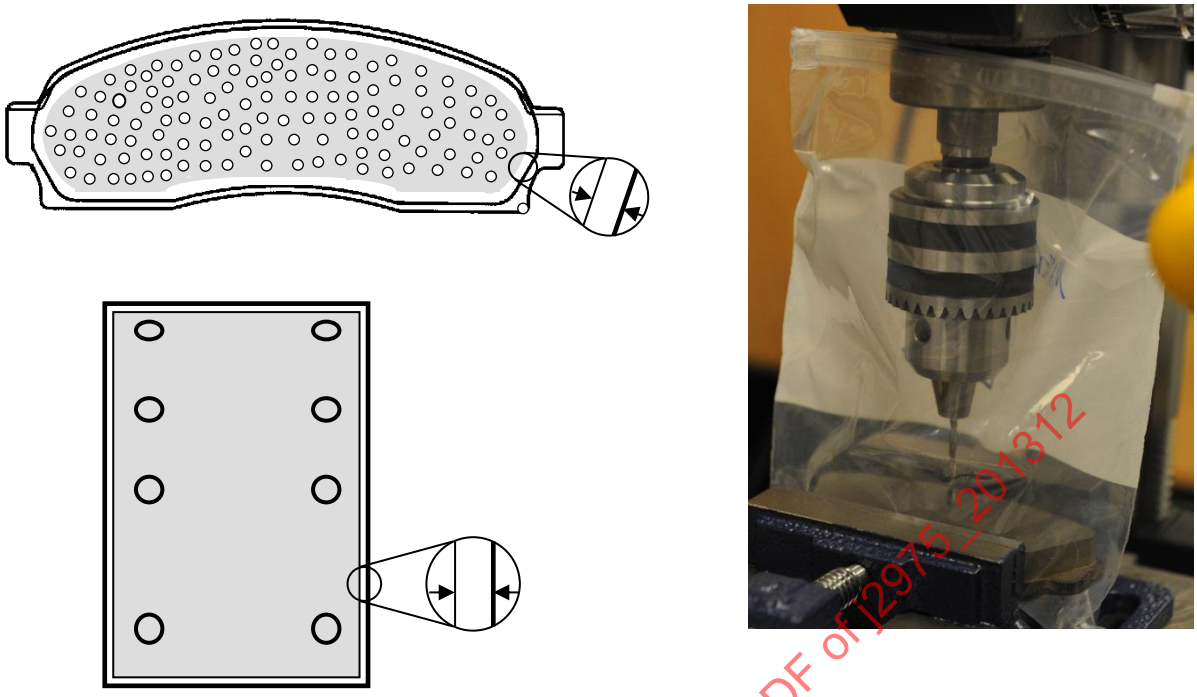

FIGURE 3 - A SCHEMATIC REPRESENTATION OF A DISC PAD AND LINING SHOWING THE AREA THAT CAN BE USED FOR SAMPLE COLLECTION (GRAY SHADED). ALSO SHOWN (RIGHT) IS A BRAKE FRICTION ASSEMBLY IN A SEALABLE PLASTIC BAG READY FOR THE START OF SAMPLE COLLECTION

4.1.7 The morphology of the drillings is dependent on the plunge rate and drill spindle speed which is specified in section 4.15 while drilling. A plunge rate of less than 0.33 mm/sec is recommended (~15 sec to drill a 5mm deep hole) to achieve a consistent and uniform granular particulate. Slower plunge rates yield a more powdery consistency. A faster plunge rate yields a mixture of coarser turnings and granules which results in an unacceptable measurement variation of the chemical content. The different morphologies resulting from different plunge rates are shown in Figure 4.ferred Morphology

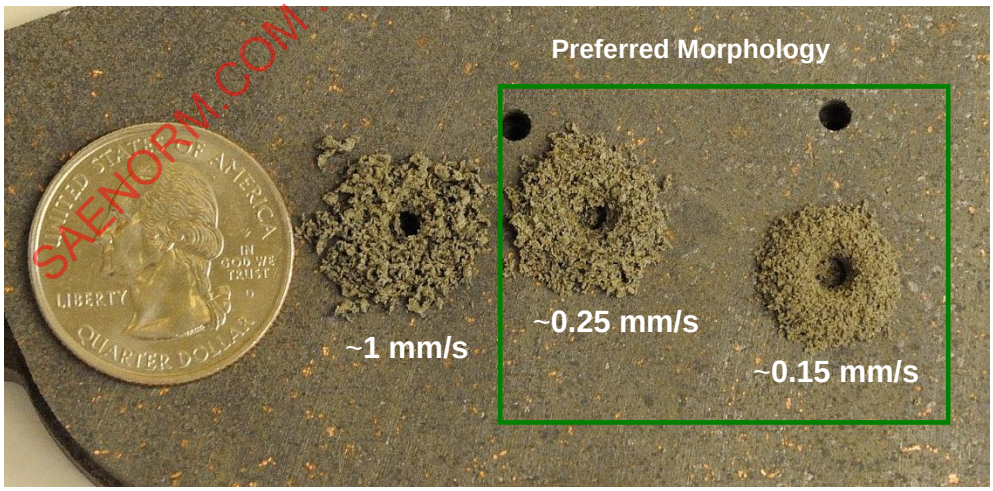

FIGURE 4 - THE DIFFERENCE IN DRILL TURNING MORPHOLOGIES THAT RESULTS FROM VARYING THE DRILL PLUNGE RATES

4.1.8 Collect drill turnings from the working face of the friction material and place in a clean sealable container. Label the sample accordingly.

4.2 Friction Material Sample Processing for Chemical Analysis

4.2.1 Either EPA Method 3050B (via hot block) or EPA Method 3051A may be used for the digesting the friction material powder collected in 4.1. If analysis of antimony is desired, follow the optional digest listed in the EPA Method 3050B section 7.5 or EPA Method 3051A section 11.3.2.

4.3 Chemical Analysis of Brake Friction Material Sample

4.3.1 Using the digested sample generated in section 4.2.1, perform an analysis for the desired chemical elements following EPA Method 6010C – Inductively Coupled Plasma – Atomic Emission Spectrometry. Because of the complex and heterogeneous nature of brake friction materials, it is recommended that digestion and analysis for each pad be performed in triplicate (as a minimum) and the average value be reported. Percent Relative Standard Deviation (standard deviation / measurements average *100) should be less than 20% for the triplicate measurements. Depending on the amount of specific components in the friction materials, additional dilutions during the analysis may be necessary.

4.3.2 Chromium detection

4.3.2.1 If chromium is identified in section 4.3.1, at a level exceeding the criteria of 0.1% by weight, use EPA Method 3060A, followed by either EPA Method 7196A or EPA Method 7199, to determine the hexavalent chromium content.

4.3.2.2 If the sample spike recovery during EPA Method 3060A is below 80%, it may indicate the presence of common sulfide-based compounds (S^{2-}) or raw materials, such as antimony trisulfide, copper sulfide, molybdenum disulfide, metal alloys or others. Some of these materials and their reactions with other constituents may mask the total amount of hexavalent chromium present in the formulation. In this event, conduct tests in triplicate for hexavalent chromium on all the individual chromium-based raw materials as declared by the manufacturer.

4.3.2.3 If the content of hexavalent chromium in all raw materials is below 0.1% by weight, the friction material composition can be certified as containing less than 0.1% of Cr(VI). If hexavalent chromium was at level exceeding 0.1% in at least one raw material proceed to point 4.3.2.4

4.3.2.4 In order to calculate the total hexavalent chromium present in a given formulation, the supplier needs to identify the corresponding material content in percent-by-weight in the total formulation being tested.

NOTE: As the actual contribution of hexavalent chromium is a function of the friction material formulation, it is the responsibility of the manufacturer to demonstrate (through the product registration process) the actual contribution of hexavalent chromium to the total formulation, and to demonstrate compliance to any specific rule, regulation, or requirement applicable to the product.

4.3.3 If chromium is identified in section 4.3.1 at a level exceeding the criteria of 0.1% by weight, then use either EPA Method 7196A - Chromium, Hexavalent (Colorimetric) or EPA Method 3060A - Alkaline Digestion for Hexavalent Chromium followed by EPA Method 7199 - Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography to determine the hexavalent chromium content.

4.3.4 To determine the mercury content, follow EPA Method 7471B – Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique) or EPA Method 245.5.

4.3.5 For asbestos fiber analysis, follow EPA/600/R-93/116 – Method for the Determination of Asbestos in Bulk Building Materials.

4.4 Presentation of Results

4.4.1 At a minimum, the presentation of the results is to contain:

- Client
- Project
- Project Number
- Laboratory Name
- Sample Identification - Manufacturer, Edge Code, etc.
- Sample Type - friction pad, friction block, etc.
- Laboratory Sample Number
- Matrix
- Sample Date / Time
- Sample Preparation Date / Time
- Analysis Data / Time
- Batch ID
- Preparation Method Code
- Preparation Method Name
- Analysis Method Number Code
- Analyte Name
- Result
- Detection Limit
- Reporting Limit
- Reporting Units
- Result Qualifier
- Basis (dry/wet)
- Dilution
- Spike Level
- % Recovery
- Upper Control Limit
- Lower Control Limit
- RPD Analyst
- Sample Comments
- Result Comments