

|                                                                                                                                                                                                                                                                                                                                         |                                         |                                            |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------|--------------------------------------------|
| <b>NAVISTAR, INC.</b>                                                                                                                                                                                                                                                                                                                   |                                         |                                            |
| <b>Material, Parts, and Process Specifications<br/>(MPAPS)</b>                                                                                                                                                                                                                                                                          |                                         | <b>NUMBER:</b><br><b>MPAPS BT-49</b>       |
|                                                                                                                                                                                                                                                                                                                                         |                                         | Former Designation: <b>CEMS BT-49</b>      |
| <b>TITLE:</b><br><b>Determination Of Molybdenum Disulfide In Lubricants</b>                                                                                                                                                                                                                                                             |                                         | <b>CURRENT REV No.:</b><br><b>1802</b>     |
|                                                                                                                                                                                                                                                                                                                                         |                                         | <b>DATE:</b><br><b>Feb. 2018</b>           |
| <b>WRITTEN/EDITED BY:</b><br><b>W. Cook</b>                                                                                                                                                                                                                                                                                             | <b>APPROVED BY:</b><br><b>R. Goluch</b> | <b>SUPERSEDES:</b><br><b>Rev. Aug 2009</b> |
| <b>PRINTED COPIES OF THIS DOCUMENT MUST BE VERIFIED FOR CURRENT REVISION</b>                                                                                                                                                                                                                                                            |                                         |                                            |
| <i>This specification may involve hazardous materials, equipment, and procedures. This specification does not purport to address all of the safety issues associated with its use. The user is responsible to consult appropriate safety and health practices and to determine the applicability of regulatory limits prior to use.</i> |                                         |                                            |

## 1.0 SCOPE

This method covers a test procedure for determining molybdenum disulfide in lubricants by atomic absorption spectroscopy.

## 2.0 APPARATUS

- 2.1 Atomic-absorption Spectrophotometer, for use at 313.1 nm.
- 2.2 Molybdenum hollow-cathode lamp.
- 2.3 Pressure-reducing valves.
- 2.4 Analytical balance with a sensitivity of 0.1 mg.
- 2.5 Hot plate.
- 2.6 Fume hood.
- 2.7 Beaker, 250 ml, Pyrex or equivalent.
- 2.8 Watch glass, 90 mm diameter, Pyrex or equivalent.
- 2.9 Volumetric flask, glass, 100 ml.
- 2.10 Volumetric flask, glass, 1000 ml.
- 2.11 Spatula, stainless steel, semi-micro.
- 2.12 Funnel, glass, 75 mm diameter, 150 mm stem.
- 2.13 Graduated cylinder, glass, 50 ml.
- 2.14 Pipette, glass, volumetric, TD, 10 and 20 ml.

## 3.0 MATERIALS

- 3.1 Oxidant: Nitrous Oxide, Commercial Grade.
- 3.2 Fuel: Acetylene, Commercial Grade.
- 3.3 Distilled water per ASTM D 1193, Type II.
- 3.4 Sulfuric acid, ACS reagent grade, concentrated, H<sub>2</sub>SO<sub>4</sub>.
- 3.5 Nitric acid, ACS reagent grade, concentrated, HNO<sub>3</sub>.
- 3.6 Molybdenum disulfide, 98% MoS<sub>2</sub> minimum assay.
- 3.7 Pipette, Pasteur, glass, 153 mm.
- 3.8 Material Blank for Standards preparation: Material having a composition similar to the analysis sample but containing less than 0.001 mass percent molybdenum.

This document is restricted and may not be sent outside Navistar, Inc. or reproduced without permission from Corporate Technical Standards. Suppliers are required to assume all patent liability. ©2018 Navistar, Inc.

FEBRUARY 2018

|                               |                                                                                |                          |
|-------------------------------|--------------------------------------------------------------------------------|--------------------------|
| NUMBER:<br><b>MPAPS BT-49</b> | TITLE<br><b><i>Determination Of Molybdenum Disulfide<br/>In Lubricants</i></b> | REVISION:<br><b>1802</b> |
|-------------------------------|--------------------------------------------------------------------------------|--------------------------|

#### 4.0 PREPARATION OF STANDARDS

4.1 Weigh the following amounts of MoS<sub>2</sub> and the Material Blank into three 250 ml beakers.

| <u>Beaker</u> | <u>MoS<sub>2</sub>, grams</u> | <u>Material Blank, grams</u> |
|---------------|-------------------------------|------------------------------|
| 1             | 0.10 ± 0.001                  | 4.90                         |
| 2             | 0.15 ± 0.001                  | 4.85                         |
| 3             | 0.20 ± 0.001                  | 4.80                         |

4.2 Place the beakers containing the standards in a fume hood and add 20 ml of H<sub>2</sub>SO<sub>4</sub> to each beaker. Allow to stand at room temperature for ten minutes.

CAUTION: With some samples, there may be an immediate reaction with the H<sub>2</sub>SO<sub>4</sub>. Let any reaction subside before performing Step 4.3.

4.3 Add 20 ml of HNO<sub>3</sub> to each beaker, cover beakers with watch glasses and place samples on a cold hot plate.

4.4 Turn on hot plate and digest samples until the solution remains clear. Cautious periodical additions of 1 to 3 ml HNO<sub>3</sub> with a glass pipette will be necessary until the digestion is complete.

4.5 Quantitatively transfer the contents of each beaker to individual 1000 ml volumetric flasks and dilute to the mark with distilled water.

NOTE: Standards may be retained for up to three months for future use on similar materials.

#### 5.0 PROCEDURE

5.1 Weigh 5 ± 0.001 g of test sample into a 250 ml beaker.

5.2 Digest and dilute sample as described under "Preparation of Standards, Steps 4.2 thru 4.5.

5.3 Operate the atomic-absorption spectrophotometer per manufacturer's instructions for use at 313.3 nm, using a nitrous oxide/acetylene flame.

5.4 Pipette 20 ml aliquots from the flasks in Step 5.2 to 100 ml volumetric flasks and dilute to the mark with distilled water and record absorbance, at 313.3 nm, of the standard and test sample dilutions using a Nitrous Oxide flame.

NOTE: If the Spectrophotometer is equipped with a data processor, the concentration of MoS<sub>2</sub> may be determined directly. See the manufacturer's instructions for procedure.

#### 6.0 CALCULATION

6.1 Plot a graph of mass percent MoS<sub>2</sub> versus absorbency for the standards. Mass % MoS<sub>2</sub> in standards:

$$\text{Mass \%} = \frac{\text{wt. MoS}_2}{\text{Wt. Material Blank} + \text{wt. MoS}_2} \times \text{Purity of MoS}_2 \times 100$$

6.1.1 If the absorbency of the test sample does not fall on the calibration curve, adjust aliquot and final dilution volume as per Step 5.4, to make absorbency fall on calibration curve.

This document is restricted and may not be sent outside Navistar, Inc, or reproduced without permission from Corporate Technical Standards. Suppliers are required to assume all patent liability. ©2018 Navistar, Inc.