

NAVISTAR, INC.		
Material, Parts, and Process Specifications (MPAPS)		NUMBER: MPAPS BT-4
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Saponifiable Matter		CURRENT REV No.: 1408
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<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>		
Change Notice: Updated to new MPAPS format; editorial changes.		

1.0 SCOPE

This method covers a procedure for determining the quantity of saponifiable matter in lubricants.

2.0 APPARATUS

- (1) Erlenmeyer flask, 500 ml.
- (2) Reflux air condenser, 480 mm in length.
- (3) Hot plate.
- (4) Burettes and graduated cylinder, 10, 50 and 100 ml.
- (5) Separatory funnels, 500 ml.
- (6) Beakers, 600 and 250 ml.
- (7) Steam bath.
- (8) Analytical balance with a sensitivity of 0.01 g.

3.0 MATERIAL

- (1) n-Hexane (per ASTM D128).
- (2) Reagent alcohol.
- (3) Potassium hydroxide, alcoholic, 0.5 N (KOH).
- (4) Hydrochloric acid, 0.5 N (HCl).
- (5) Hydrochloric acid, concentrated (HCl).
- (6) Ethyl ether, ACS.
- (7) Acetone, ACS.
- (8) Phenolphthalein, reagent grade.
- (9) Distilled water.

4.0 SOLUTION PREPARATION

PHENOLPHTHALEIN INDICATOR

Dissolve 1.0 g phenolphthalein in 50 ml reagent alcohol then add 50 ml distilled water.

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5.0 PROCEDURE

5.1 Weigh into an Erlenmeyer flask a sample sufficient to produce a fatty acid residue of approximately 2 g (approximately 5 g for Steering Gear Lubricant). Record sample weight to the nearest 0.1 g. Add 50 ml n-Hexane, 50 ml reagent alcohol and 30 ml KOH solution. Assemble the apparatus as shown on Page 2 and reflux gently for 1.5 hours. Remove the flask from the hotplate and allow to cool to room temperature. Add 3 drops of indicator solution and titrate the contents of the flask with HCl solution. At least 7 ml excess 0.5 N KOH must have been present to assure complete saponification. If less than this amount was present, add 15 ml KOH solution and resaponify the contents of the flask for 30 minutes. Recheck for excess KOH.

5.2 Add approximately 5 ml KOH solution to the flask so the contents are decidedly alkaline. Quantitatively transfer to a clean separatory funnel using sufficient distilled water to reduce the alcohol concentration to approximately 60%. Add sufficient n-Hexane to bring the volume in the funnel to 150 to 200 ml. Shake the funnel and contents vigorously and allow the layers to separate. Draw off the bottom alcohol layer into a second clean separatory funnel. Add 50 ml of 60% alcohol wash solution to the n-Hexane and shake the funnel and contents vigorously. Allow the layers to separate and draw off the alcohol layer combining with alcohol solution in the second funnel. Repeat the alcohol wash and discard the n-Hexane layer.

5.3 Add 50 ml n-Hexane to the funnel containing the alcohol washings and shake funnel and contents vigorously. After the layers have separated, draw off the alcohol layer into a clean separatory funnel. Wash the n-Hexane layer with 60% alcohol solution combining the alcohol washes. Discard the n-Hexane layer. Repeat the n-Hexane and alcohol washes. Quantitatively transfer the alcohol solution into a 600 ml beaker using 60% alcohol and evaporate on a steam bath to a syrupy consistency.

5.4 Dissolve the syrupy material in about 60 ml hot distilled water and quantitatively transfer to a separatory funnel using approximately 100 ml hot distilled water. Add 10 ml concentrated HCl and cool the funnel and contents to room temperature.

5.5 Add 100 ml ethyl ether to the funnel to dissolve liberated fatty acids. Shake funnel and contents vigorously (CAUTION: The pressure built-up in shaking must be cautiously relieved by bleeding through the inverted stopcock). Allow the layers to separate. Draw off the water layer into a clean separatory funnel. Wash the ether layer twice with 50 ml portions of acidic water. Combine the water washes with the water layer in the separatory funnel. Quantitatively transfer the ether layer into a weighed 250 ml beaker.

5.6 Wash the water solution twice with 50 ml portions of ethyl ether. Discard the water solution. Wash the ether washings with 50 ml of acidic water. Combine the ether layer with ether in the 250 ml beaker. Evaporate the ether on a steam bath to remove solvents. Cool and weigh beaker and contents to the nearest 0.01 g. Add 5 ml acetone and evaporate on the steam bath. Weigh to the nearest 0.01 g. Repeat acetone additions and weighings to constant weight. Record the weight of fatty acids.