

**MINERAL OIL CONTAMINATION OF BRAKE FLUID
BY THIN LAYER CHROMATOGRAPHY**

Application

This procedure describes a method for detecting and estimating mineral oil contaminants above 0.1 percent in brake fluids by thin layer chromatography.

Apparatus Required

Thin Layer Plates

Size 200 x 200 mm, Silica Gel coated, layer thickness 0.25 mm.

Source: Andermann & Co. Ltd. or Merck, Darmstadt

Syringe

20 microliter capacity.

Developing Tank with Sealing Lid

Of suitable size for processing the thin layer plates.

Spotting Guide

Aerosol Reagent Spraying Unit

Ultra-Violet Lamp

Having 254 and 336 micrometre wavelength.

The above apparatus is available from any reputable laboratory equipment supply house.

Materials Required

Developer Solution

Cyclohexane Reagent grade; CAUTION: Highly dangerous fire hazard.

Date	Action	Revisions
2001 01 30	Revised	Editorial – no technical change A. Cockman
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Solutions Required

Spray Reagent

Solution 0.1 % w/v sodium fluorescein in ethanol. CAUTION: Low flash point.

Standard Brake Fluid Solutions

Contaminated with 0.1 % v/v mineral oil. ASTM No. 3 to be used as contaminant.

Note: 0.05 % and 0.025 % v/v mineral oil contaminated standards are also used for new oil evaluations.

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 +/- 2 °C and 50 +/- 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Procedure

1. Using a pencil, mark a base line on the thin layer plate, 25 mm from the edge, the plate being later immersed with this edge in the developing solvent. Using the spotting guide for accurate location, apply on the base line, with the syringe, 2 individual spots, i.e. 10 microliters of the brake fluid under test and 10 microliters of the standard 0.1 % v/v mineral oil contaminated brake fluid.
2. Prepare the developing tank by lining with filter paper saturated with the developer solution, ensuring that there is approximately 15 mm depth of solution in the tank.

Note: Tank must be cleaned and solution changed once per two days minimum. The test fluid spots must not be immersed in the developing solution.
3. Immerse the plate with the edge, mentioned above, in the developing tank. Allow to stand for 1 h at 23 +/- 2 °C, ensuring that tank is sealed by means of the lid.
4. Remove the plate from the tank, and allow developer solution to volatilize.
5. When dry, place under a fume hood and spray the plate with the fluorescein spray reagent in such a way that the spraying unit is being traversed over the plate to produce a 50 % overlapping from one spray operation to the other. The operation must be repeated until uniform coat is achieved.