

**THERMOGRAVIMETRIC ANALYSIS (TGA)
OF GASOLINE GUMS**

Application

This test method describes the procedure for preparing gum residues from gasoline containing detergent additives and the method for measuring the thermal decomposition of the gum residues.

Significance and Use

The method was developed as an alternative for conducting engine tests to assess the tendency of fuels formulated with detergent fuel additives to buildup combustion chamber deposits in spark ignition engines. The gasoline gums consist of the polymeric detergent additives, carrier fluids and existent gums of gasoline components. The decomposition temperatures of the gum residues in the TGA test was found to give a reasonably good correlation with the buildup of combustion chamber deposits (CCD) in dynamometer tests.

The fuels that were used to establish the correlation included commercial fuels from service stations and specially blended fuels that were formulated using several base gasolines and various detergent additive chemistries and carrier fluids

Summary of Test Method

The unwashed gum of the gasoline sample is prepared per ASTM D 381 method³ and the sample is then heated in a TGA instrument at a constant rate of 5 °C/ minute from 150 °C to 500 °C in an air environment.

The thermal decomposition of the sample is analyzed as a function of weight loss versus temperature. The first derivative of the TGA curve identifies critical peak temperatures where the majority of gum residues will decompose.

Equipment Required

Thermogravimetric Analyzer: A system of related instruments that is capable of continuously weighing a sample under atmospheric control over a controlled temperature range for a defined period. The analyzer should have the capability to meet the conditions required for this method along with the software necessary to complete the required analyses.

Date	Action	Revisions
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1. Platinum sample pans of 100- μ L specimen size.
2. Methylene Chloride-ACS reagent grade
3. Dry Air, 99.9% purity

Sample Preparation

1. Prepare a minimum of 15 mg of unwashed gum by evaporating 50 ml of gasoline per ASTM D 381 method.
 - Two or more beakers of fuel sample may be required to generate sufficient gum quantity.
 - A maximum of 50 ml of gasoline shall be evaporated per beaker.
 - Report the weight of unwashed gum for each beaker in mg/50ml of gasoline.
2. Add sufficient volume of methylene chloride to the gum beaker(s) and swirl gently to dissolve the gum.
3. Transfer sample into a tared vial with a pipette.
4. Repeat steps 2 and 3 until the methylene chloride is clear.
5. Evaporate the methylene chloride from each vial by air evaporation, steam bath or oven set at 40 °C. Cover the vials with a paper towel or a loose fitting cap during evaporation.
6. Wash the inside wall of the vials with methylene chloride so that all gum residue collects on the bottom of the vial.
7. After solvent has evaporated completely, place the lids on the vials, and reweigh. The net increase in weight after adding the gum residue to the tared vial should not be significantly different from the total weight of unwashed gum collected in step 1 of this section.
8. Transfer the sample by adding an appropriate amount of methylene chloride to the vial.
9. Swirl sample in sealed vial for a few minutes to ensure that the sample is well mixed and all of the gum has dissolved. Note: The sample may collect on the vial wall if it is shaken too rapidly.

Procedure

1. Transfer 10 -15 mg of gum to the TGA sample pan using sufficient solvent and record the weight of gum placed on the sample pan.
2. Establish the air atmosphere with flow set at 55 +/- 5 mL/min.