National Institute of Standards & Technology

Reference Material 8501

Report of Investigation

A Catalyst Package for Lubricant Oxidation (ASTM Sequence IIIE Engine Test)

This Reference Material (RM) is released prior to the corresponding Standard Reference Material to provide an early access to the Catalyst Package for IIIE Oxidation. RM 8501 contains: (1) an oxidized/nitrated fuel fraction, (2) a nitro-paraffin model compound, (3) a metal naphthenates mixture, and (4) distilled water. The metal naphthenate mixture has the following weight ratio of elements: 20:2:1:1 for lead, iron, manganese, and tin, respectively.

RM 8501 is used to simulate the chemical environment in an operating automotive engine, specifically under the ASTM sequence IIIE engine test conditions. Seven IIIE oils have been tested using RM 8501. Both the modified thin-film oxygen uptake test (MTFOUT) and the differential scanning calorimetry (DSC) test were used to determine the oxidation induction times of these oils [1].

The correlation between the values for oxidation induction times by MTFOUT and DSC is shown in Figure 1.

Notice and Warning to Users:

Expiration of Report: The Report of Investigation for RM 8501 is valid, within the limits certified, for one year from the date of purchase and shipment from NIST.

Storage: Sealed ampoules, as received, should be stored in the dark at a temperature between 10-25 °C.

Use: Each ampoule should be shaken thoroughly before opening. Samples should be taken immediately after opening an ampoule and used without delay in order to maintain the integrity of the RM sample. Research report values are not valid for ampoules that have been opened and resealed.

The technical planning, coordination, and testing leading to the research report were performed by C.S. Ku, P.T. Pei, and S.M. Hsu, Ceramic Division, Materials Science and Engineering Laboratory.

The technical and support aspects involved in the preparation, data analysis, and issuance of this Reference Material were coordinated through the Standard Reference Materials Program by T.E. Gills.

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Gaithersburg, MD 20899

William P. Reed, Chief
Standard Reference Materials Program

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Preparation

Fuel Fraction

The fuel fraction was produced by the oxidation/nitration of a high boiling VE gasoline fraction (ASTM VE engine test fuel) [1]. The neutralized product was used as the fuel fraction together with nitro-paraffin. The total acid number of the fuel fraction of RM 8501 was determined to be 6.5 ± 0.5 mg KOH/gm.

Nitro-paraffin Model Compound

The nitro-paraffin model compound is purchased commercially. The stated purity of the nitro-paraffin is 99.1% of 1-nitro paraffin.

Metal Naphthenates

The metal naphthenate mixture in RM 8501 is made from commercially available metal naphthenates. The total metal concentration is 10.9% by weight. The mixture is provided for user convenience and is the mixture used at NIST to generate correlation shown in Figure 1. The metal naphthenates mixture in this RM 8501 is from a single bath and each naphthenate has been characterized and filtered through an 0.2 μm filter.

Catalysts Use

Each ampoule was sealed in argon. Caution should be exercised not to expose the catalyst materials to air, sunlight, and humidity as these materials contain very reactive compounds. Once opened, the shelf life is very limited.

The test conditions for the correlation shown in Figure 1 are:

MTFOUT Test Conditions:
- 1.5 gm Oil
- 2 wt. % Fuel Catalyst
- 2 wt. % Nitro-paraffin
- 3 wt. % Metal Catalyst
- 2 wt. % Water
- 620 kPa (90 psig) Oxygen
- 160 °C

DSC Test Conditions:
- 2 vol. % Fuel Catalyst
- 2 vol. % Nitro-paraffin
- 4 vol. % Metal Catalyst
- 1450 kPa (210 psig) Oxygen at 120 ml/minute flow rate
- 170 °C
- NIST designed and sealed aluminum pan (2)
- 0.6-0.7 mg sample size for each DSC test.

Care must be taken to ensure that adequate mixing has occurred and test the sample(s) immediately after mixing; delay may result in variation of results.

References

[1] Ku, C.S., Pei, P.T., and Hsu, S.M., "A Modified Thin-Film Oxygen Uptake Test (TFOUT) for the Evaluation of Lubricant Stability in ASTM Sequence IIIE Test". SAE Paper No. 902121, 1990.

[2] Zhang, Y., Pei, P.T., Perez, J.M. and Hsu, S.M., "A New Method to Evaluate Deposit Forming Tendency of Liquid Lubricants by Differential Scanning Calorimetry," Presented at the STLE National Meeting, April, 1991, Montreal, Canada.

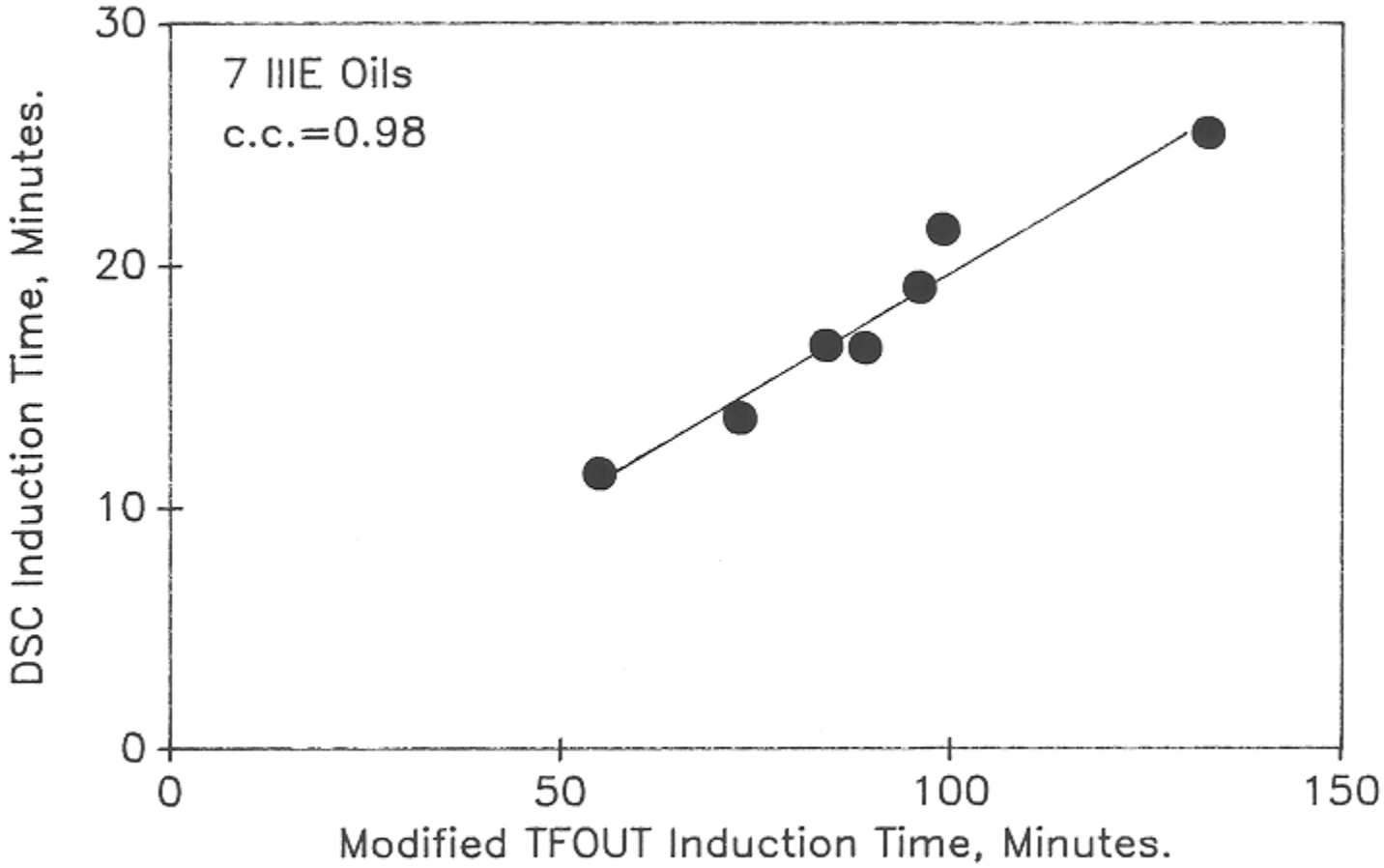

Fig. 1. INTERCORRELATION BETWEEN MTFOUT AND DSC