National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2567

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

This Standard Reference Material (SRM) is specified for the use in the NIST modification to ASTM D 4742, Standard Test Method for Oxidation Stability of Gasoline Automobile Engine Oils by Thin-Film Oxygen Uptake (TFOUT). A unit of SRM 2567 consists of one ampoule each of the following: (1) an oxidized/nitrated fuel fraction; (2) a nitro-paraffin model compound; (3) a nitro-aromatic model compound; (4) a metal catalyst (naphthenate mixture) and; (5) distilled water. The metal naphthenate mixture has a 10:1 mass ratio for the elements lead and iron, respectively.

This SRM is intended for use in monitoring the batch variation of re-refined base oils or lubricants by simulating the chemical environment in an automotive engine, especially under the ASTM Sequence IIIE engine test conditions. To demonstrate that the chemicals in SRM 2567 correspond to the chemistry in the engine test, the oxidation induction time of eight IIIE oils were subjected to the modified thin-film oxygen uptake test (MTFOUT). The correlation established between MTFOUT oxidation time and IIIE hours demonstrates the validity of the simulation.

The certified correlation between the oxidation induction time by MTFOUT and IIIE hours to 375 % viscosity break point is shown in Figure 1. The MTFOUT induction time with precision data for each oil is listed in Table 1. The observed correlation coefficient is 0.91 and a 95 % confidence interval for it is (0.61, 0.99). (It should be noted that the interval is not symmetric.)

Expiration of Certification: The certification of SRM 2567 is valid, within the limits certified, for one year from the date of shipment from NIST.

Storage: The as-received sealed ampoules comprising the SRM unit, should be stored in the dark at a temperature between 10 °C and 25 °C.

Use: Each ampoule was flushed with argon before sealing. However, caution should be exercised when opening an ampoule (except for the ampoule containing distilled water) as its contents are very reactive when exposed to air, sunlight, and humidity. Before being opened, each ampoule should be shaken thoroughly and samples should be taken and used immediately in order to maintain sample integrity. *The certified correlation is not valid for ampoules that have been opened and resealed.*

The technical planning, coordination, and testing leading to this SRM were performed by J.X. Sun, Z.S. Hu, P. Pei, and S.M. Hsu of the NIST Ceramics Division.

Statistical analysis was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899
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Standard Reference Materials Program

PREPARATION

Fuel Fraction: The fuel fraction was produced by the oxidation/nitration of a high boiling VE gasoline fraction (ASTM VE engine test fuel) [2]. The neutralized product was used as the fuel fraction together with a nitro-paraffin and a nitro-aromatic. The total acid number of the fuel fraction of SRM 2567 was determined to be 6.5 mg KOH/g $\pm$ 0.5 mg KOH/g.

Fuel Catalyst (Nitro-paraffin and Nitro-aromatic Model Compounds): The nitro-paraffin and nitro-aromatic model compounds were purchased commercially. The stated purity of the as-received compounds is 99 %.

Metal Catalyst (Naphthenate): The metal naphthenate mixture in SRM 2567 is made from commercially available metal naphthenates. The mixture, which is provided for user convenience, is the mixture used at NIST to generate the correlation shown in Figure 1. The metal naphthenate mixture in SRM 2567 is from a single batch and each naphthenate has been characterized.

Test Conditions for Correlation: The MTFOUT conditions for the correlation shown in Table 1 and Figure 1 are as follows:

1. Fuel Components (in order of addition);
   a. Oil - 1.5 g
   b. Fuel Catalyst consisting of
      1) Fuel Fraction - 0.015 g
      2) Nitro-paraffin - 0.008 g
      3) Nitro-aromatic - 0.0075 g
   c. Metal Catalyst - 0.016 g
   d. Distilled Water - 0.03 g

2. 160 °C

3. 620 kPa (90 psig) Oxygen

An adjustable micropipetter may be used to obtain reproducible quantity of each component. Also, samples must be tested immediately after mixing and any delay may cause variations in the results obtained.

REFERENCES

[1] Sun, J.X., Pei, P.T., Hu, Z.S., and Hsu, S.M., "A New Catalyst Package for the Thin-Film Oxygen Uptake Test (TFOUT) in Simulating ASTM Sequence IIIE Test," Presented at STLE National Meeting, May, 1995, Chicago, IL.

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

Table 1. Test Repeatability of the Modified TFOUT for IIIE Simulation

IIIE-Oil	IIIE Hrs	SAE Viscosity Grade	MTFOUT	Oxidation Induction (min.) Time		
			No. of Exp	Avg	S.D.	RSD(%)
N	78.8	10W-30	4	170.8	5.4	3.2
E	73.5	10W-40	4	161.3	2.4	1.5
B	61.2	10W-30	4	146.0	8.4	5.8
A	56.0	10W-30	3	138.3	3.5	2.5
F	55.2	5W-30	4	155.3	1.3	0.8
D	49.5	5W-30	4	121.5	4.0	0.33
C	46.2	10W-30	4	120	2.8	2.3
G	39.8	10W-40	4	93.8	6.9	7.4

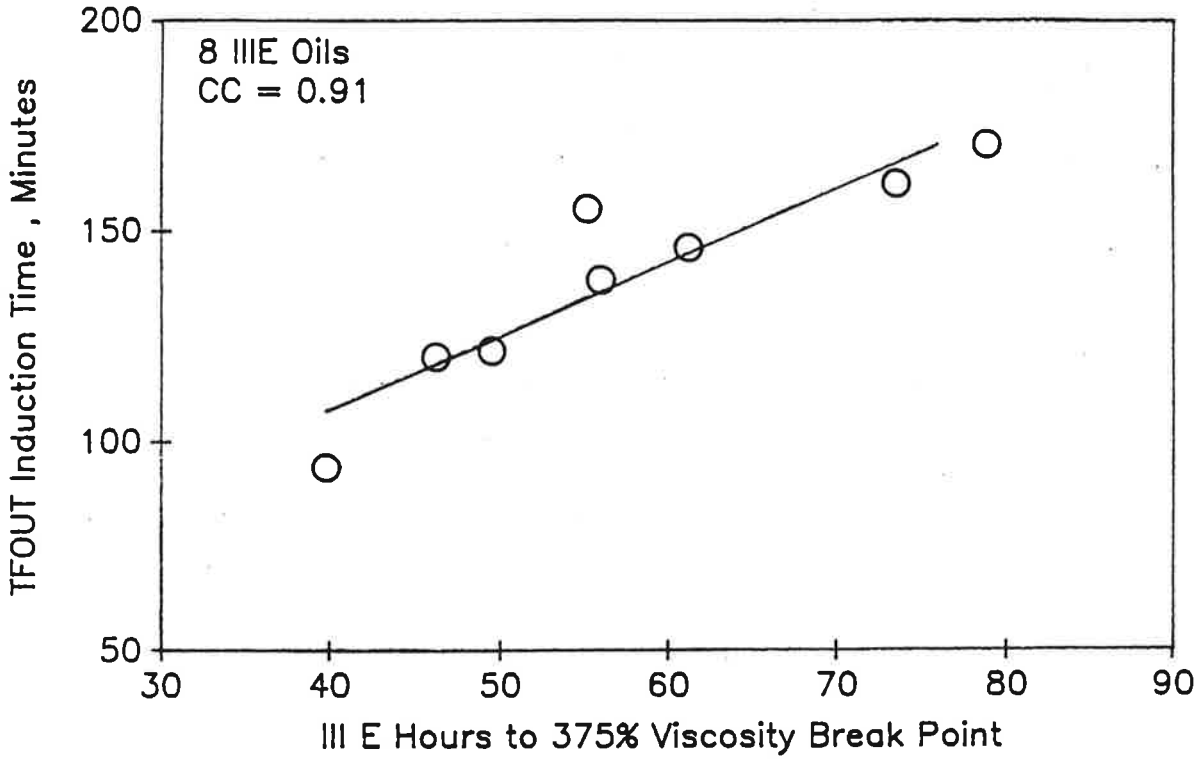

Figure 1: INTERCORRELATION BETWEEN MTFOUT AND IIIE HOURS TO 375% VISCOSITY BREAK POINT