National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1817b

A Catalyst Package for Lubricant Oxidation

This Standard Reference Material (SRM) is intended primarily for use in evaluating the oxidation stability of lubricating oils, i.e., automotive crankcase lubricants. SRM 1817b contains: (1) an oxidized/nitrated fuel fraction, (2) a metal naphthenate mixture, and (3) distilled water. The metal naphthenate mixture has the following weight ratio of metal elements: 20:2:1:1:1 for lead, iron, copper, manganese, and tin, respectively.

SRM 1817b is used to simulate the chemical environment in an operating engine, specifically under the ASTM sequence IIID engine test conditions. Eleven IIID oils have been tested using SRM 1817b. Both the thin-film oxygen uptake test (TFOUT) [1] and the differential scanning calorimetry (DSC) test [2] were used to determine the oxidation induction times of these oils.

The certified values for oxidation induction times by TFOUT and DSC are given in tables 1 and 2, respectively. The uncertainties of the certified values are expressed as ± two standard deviations of the certified value. The correlation between the two methods is shown in figure 1, and a correlation plot between TFOUT induction times and the hours to 375% viscosity increase in ASTM sequence IIID engine testing is shown in figure 2. For reference, the SRM 1817a values for TFOUT oxidation induction times are also included and are summarized with the SRM 1817b values in table 3.

Notice and Warning to Users:

Expiration of Certificate: This certification of SRM 1817b is valid, within the limits certified, for one year from the date of purchase.

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 should be used without delay for the certified values to be valid. Certified values are not valid for ampoules that have been opened and resealed.

The analytical and oxidation tests were performed by P.T. Pei, L.H. Lum and K.L. Jewett, Ceramics Division, Institute for Materials Science and Engineering.

The overall coordination leading to the certification of this SRM was performed by C.S. Ku, R.G. Munro and S.M. Hsu, Ceramics Division, Institute for Materials Science and Engineering.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.L. McKenzie.

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Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(over)

PREPARATION

Fuel Fraction

The fuel fraction was produced by the oxidation/nitration of a high boiling point VD gasoline fraction (ASTM VD engine test fuel) [1]. The neutralized product was used as the fuel fraction. The total acid number (mg KOH/g) of the fuel fraction of SRM 1817b was determined to be 2.6 ± 0.3. The infrared spectrum of the fraction is shown in Figure 3.

Metal Naphthenates

The metal naphthenate mixture in SRM 1817b is made of commercially available metal naphthenates and is based on used oil analyses. The total metal concentration of all 5 metals by weight was 10.90%. The remaining weight was composed of naphthenic liquids and mineral spirits diluent. The mixture is provided for user convenience and is the mixture used at NIST to generate tables 1 and 2. In general, the metal content and the molecular weight distribution of any metal naphthenate may vary from batch to batch from the supplier. To avoid any potential effects on oxidation results caused by this variance, the metal naphthenates in the SRM are from a single batch, and each metal naphthenate has been carefully characterized. Each metal naphthenate has been filtered through a 0.2 µm filter. During a one-year period, some precipitates may be observed in the metal mixture, but oxidation test repeatability has not been found to be affected by the presence of such precipitates.

References

[1] Ku, C.S. and Hsu, S.M., "A Thin-Film Oxygen Uptake Test for the Evaluation of Automotive Crankcase Lubricants," Lubrication Engineering, 40, No. 2, pp. 75-83, 1984.

[2] Hsu, S.M., Cummings, A.L., and Clark, D.B., "Evaluation of Automotive Crankcase Lubricants by Differential Scanning Calorimetry," SAE SP-526, 127-138, Society of Automotive Engineers, Warrendale, PA, 1982.

Table 1. Induction Times of IIID Oils from Thin-Film Oxygen Uptake Test

Test Conditions:
1.5 g Oil
4 wt. % Fuel Catalyst
4 wt. % Metal Catalyst
2 % Water
620 kPa (90 psig) Oxygen
160 °C

Oil	III HR*	No. of Tests	Oxidation Induction Time. min. Avg.	Std. Dev.
A	64	5	250	5
B	56	5	163	4
C**	48	5	88	2
D	40	5	141	5
E	40	5	105	4
F	24	5	79	1
G	16	5	39	2
H	16	5	47	2
I***	64	5	136	2
J***	56	5	149	3
K***	40	5	135	2

* Viscosity Break Point Hour.

** Passed the IIID engine test oxidation criterion (less than 375% viscosity increase at 40 hr), but failed the wear criterion.

*** Oils I, J and K are from a different series of IIID oils.

Table 2. Induction Times of IIID Oils from Differential Scanning Calorimetry

Test Conditions:
3 vol. % Fuel Catalyst
3 vol. % Metal Catalyst
175 °C
3.62 MPa Oxygen
120 cc/min Flow
Gold Pan

Oil	IIID HR*	No. of Tests	Oxidation Induction Time, min. Avg.	Std.Dev.
A	64	8	31.8	2.5
B	56	6	20.3	1
C**	48	6	15.4	0.2
D	40	7	19.3	0.6
E	40	8	14.5	0.6
F	24	8	12.2	1
G	16	7	6.1	0.2
H	16	7	7.0	0.4
I***	64	5	19.5	0.7
J***	56	8	16.0	1
K***	40	9	18.4	1.2

* Viscosity Break Point Hour.

** Passed the IIID engine test oxidation criterion (less than 375% viscosity increase at 40 hr), but failed the wear criterion.

***Oils I, J and K are from a different series of IIID oils.

Table 3

Summary of TFOUT Oxidation Induction Times from SRM 1817a and SRM 1817b

Test Conditions:
- 1.5 g Oil
- 4 wt % Fuel Catalyst
- 4 wt % Metal Catalyst
- 2 % Water
- 620 kPa (90 psig) Oxygen
- 160 °C

Oil	IIID HR*	Oxidation Induction Time, min. SRM 1817a	SRM 1817b
A	64	241 ± 8	250 ± 5
B	56	160 ± 4	163 ± 4
C**	48	96 ± 2	88 ± 2
D	40	144 ± 4	141 ± 5
E	40	108 ± 2	105 ± 4
F	24	80 ± 1	79 ± 1
G	16	41 ± 2	39 ± 2
H	16	51 ± 1	47 ± 2
I***	64	145 ± 2	136 ± 2
J***	56	151 ± 3	149 ± 3
K***	40	133 ± 1	135 ± 2

* Viscosity Break Point Hour.

** Passed the IIID engine test oxidation criterion (less than 375% viscosity increase at 40 hr), but failed the wear criterion.

*** Oils I, J and K are from a different series of IIID oils.

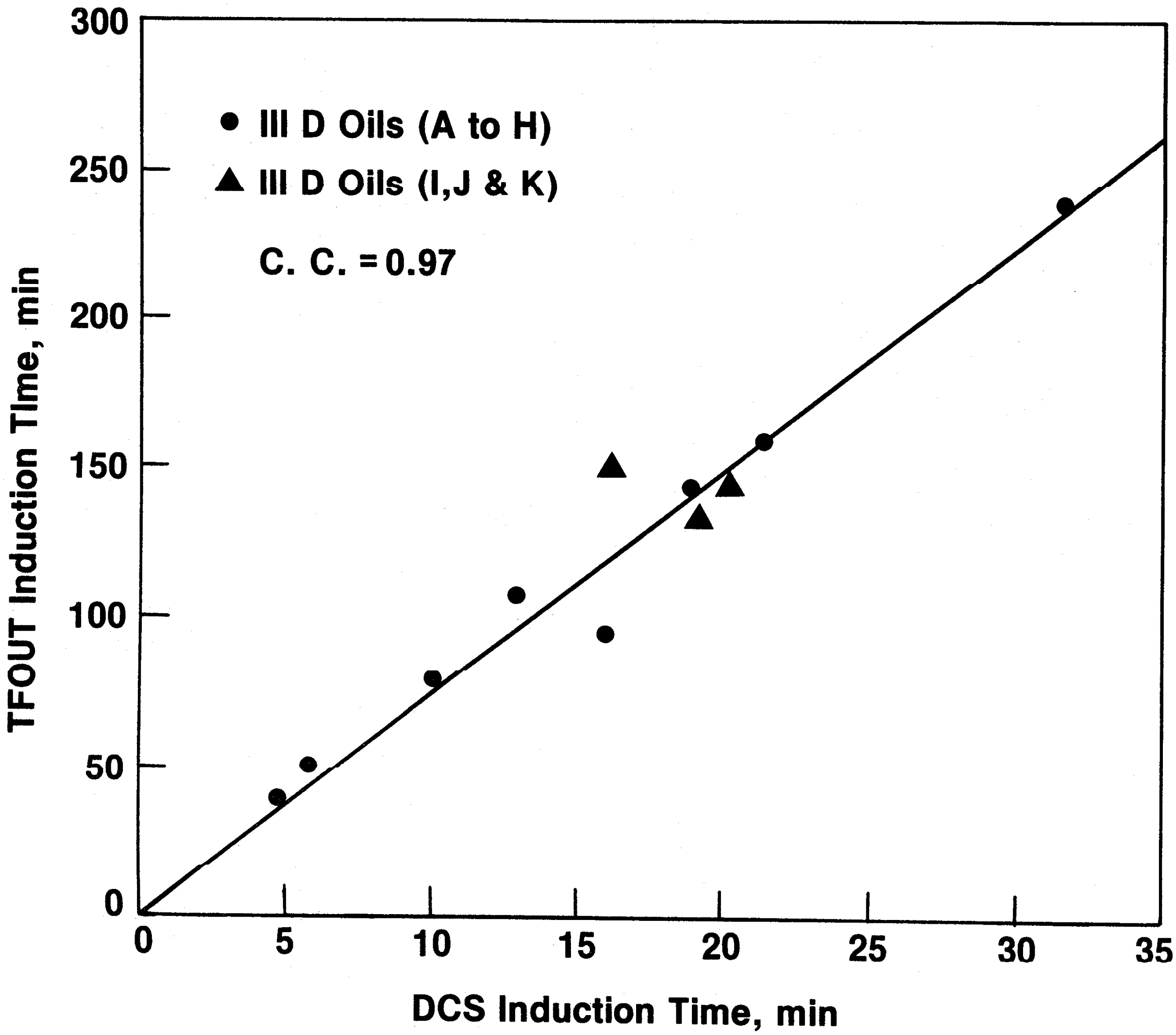

Fig. 1: Correlation between the Induction Times of TFOUT and DSC

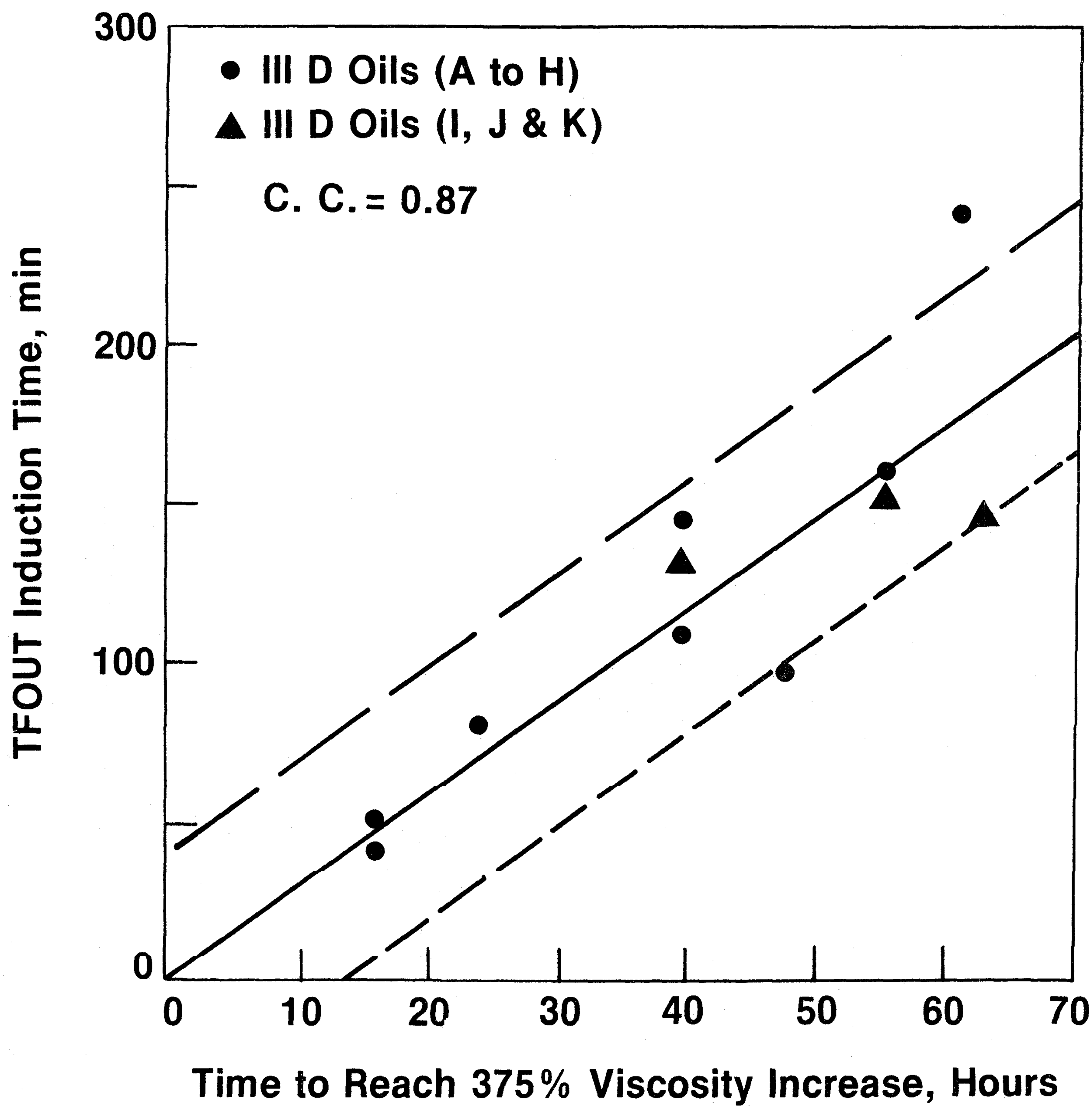

Fig. 2: Correlation between TFOUT Induction Time and IIID Time Required to Reach 375 Viscosity Increase

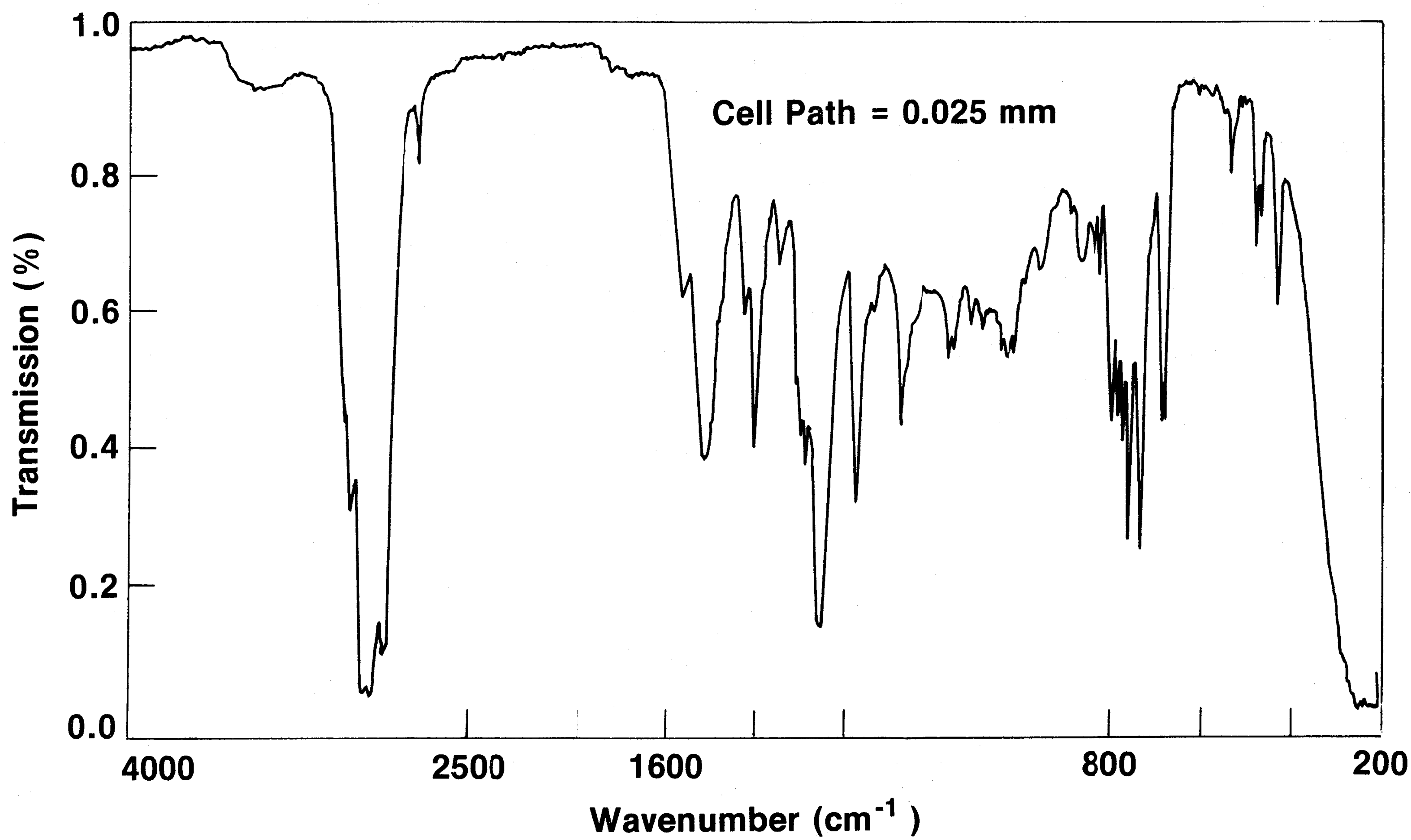

Fig. 3: Infrared Spectrum of the Fuel Fraction