U. S. Department of Commerce
Malcolm Baldrige
Secretary
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National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1817a

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 1817a 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 1817a 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 1817a. 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 uncertainty is expressed as ± two standard deviations of the certified value. The correlation between the two methods is shown in Figure 1.

Notice and Warning to Users:

Expiration of Certificate: The certification of SRM 1817a 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 used without delay in order to maintain the integrity of the SRM sample. Certified values are not valid for ampoules that have been opened and resealed.

The technical planning and coordination leading to the certification of this SRM were performed by P.T. Pei and C.S. Ku, Ceramics Division, Institute for Materials Science and Engineering.

The analytical and oxidation tests were performed by L.S. Hsu, P.T. Pei, 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 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.

Gaithersburg, MD 20899
October 25, 1986

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 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 1817a was determined to be 2.2 ± 0.2. The infrared spectrum of the fraction is shown in Figure 2.

Metal Naphthenates

The metal naphthenate mixture in SRM 1817a is made of commercially available metal naphthenates and is based on used oil analyses. The total metal concentration of all 5 metals was 10.8% by weight. The remaining weight was comprised of naphthenic liquids and mineral spirits diluent. The mixture is provided for user convenience and is the mixture used at NBS to generate tables 1 and 2. In general, the metal content and the molecular weight distribution for any particular metal naphthenate may vary from batch to batch from the supplier. While no significant effect on the oxidation results caused by this variation has yet been observed at NBS, the metal naphthenates in this SRM are from a single batch, and each metal naphthenate has been carefully characterized. Each metal naphthanate has been filtered through an 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	IIID HR*	No. of Tests	Oxidation Induction Time, min.	
			Avg.	Std. Dev.
A	64	5	241	8
B	56	5	160	4
C**	48	5	96	2
D	40	5	144	4
E	40	5	108	2
F	24	5	80	1
G	16	5	41	2
H	16	5	51	1
I***	64	5	145	2
J***	56	5	151	3
K***	40	5	133	1

*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
Gold Pan

Oil	IIID HR*	No. of Tests	Oxidation Induction Time, min.	
			Avg.	Std. Dev.
A	64	10	31.5	3.6
B	56	5	21.1	1.3
C**	48	5	15.9	0.5
D	40	6	18.7	0.6
E	40	5	12.8	0.6
F	24	5	10.0	0.2
G	16	6	4.7	0.1
H	16	5	5.8	0.3
I***	64	6	20.0	0.6
J***	56	5	16.3	0.7
K***	40	6	18.9	1.1

*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.

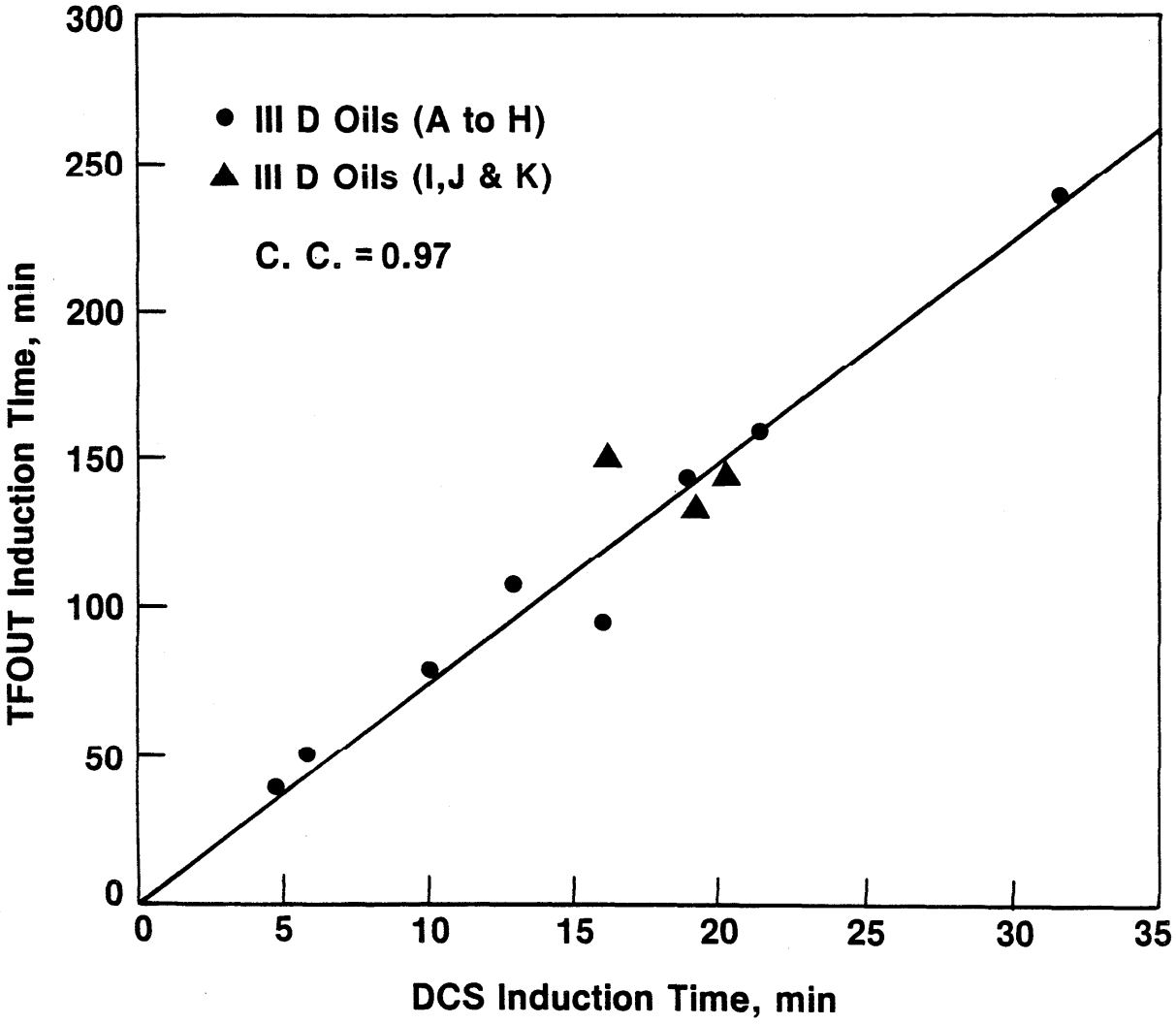

Fig. 1: Intercorrelation between the Induction Times of TFOUT and DSC

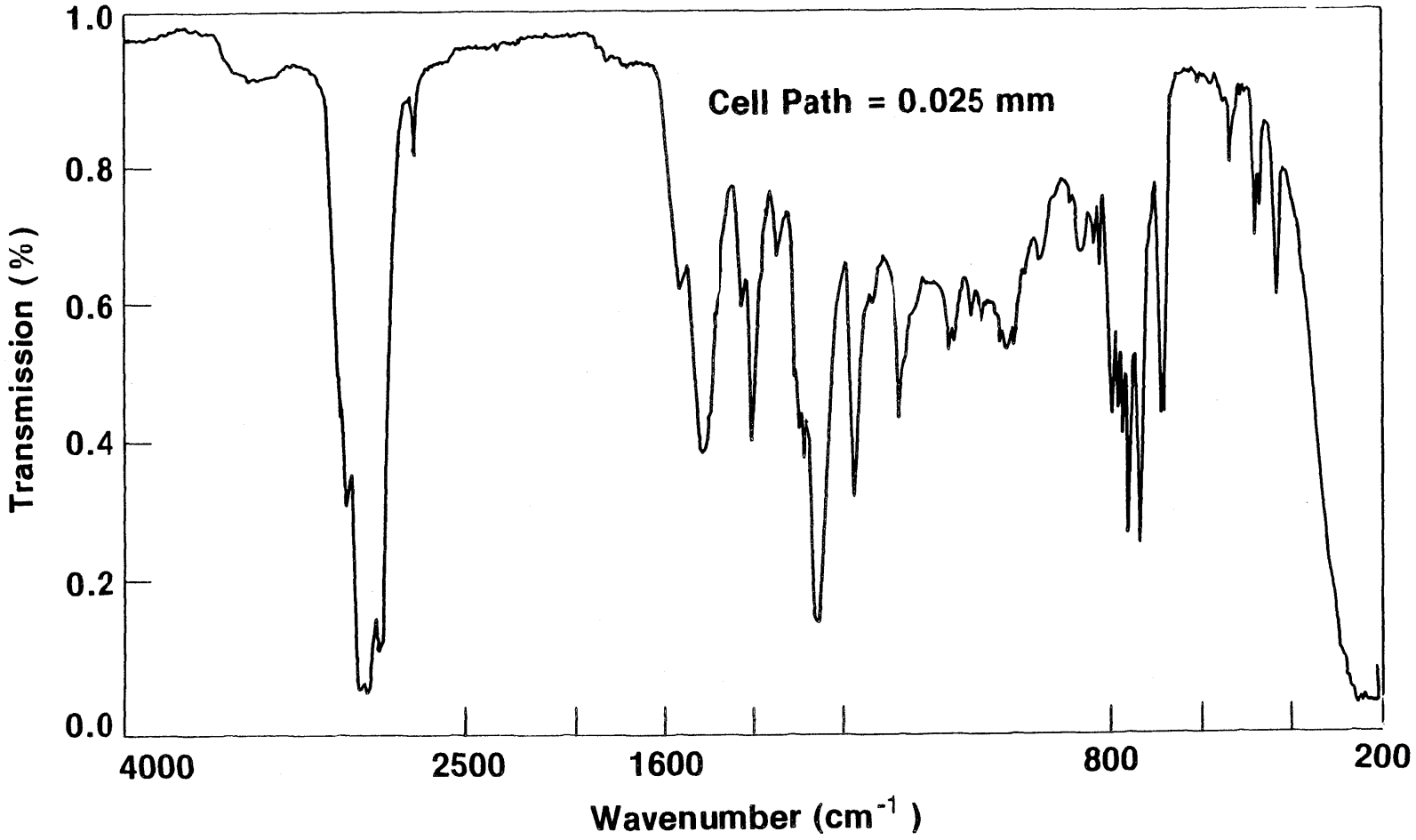

Fig. 2: Infrared Spectrum of Fuel Fraction