Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1817

A Catalyst Package for Lubricant Oxidation

This Standard Reference Material is intended primarily for use in evaluating the oxidation stability of lubricating oils, i.e., automotive crankcase lubricants. SRM 1817 contains: (1) an oxidized/nitrated fuel fraction, (2) a metal naphthenate mixture, and (3) distilled water. The metal naphthenate mixture has the following weight percentages of metal naphthenates: lead-82, iron-7, copper-4, manganese-3.5, and tin-3.5.

SRM 1817 is used to simulate the chemical environment in an operating engine, specifically under the ASTM sequence IIID engine test conditions. Eight current ASTM sequence IIID engine test stand reference oils have been tested using SRM 1817. These tests used both the thin-film oxygen uptake test (TFOUT) [1] and the differential scanning calorimetry (DSC) test [2] 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 the standard deviation at 2 sigma. The intercorrelation between the two methods is shown in figure 2.

Notice and Warning to Users:

Expiration of Certificate: This certification 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 in the Inorganic Materials Division, Center for Materials Science by P.T. Pei, K.L. Jewett, R.G. Gates, and C.S. Ku.

The overall coordination leading to the certification of this SRM was performed by C.S. Ku and S.M. Hsu.

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 W.P. Reed.

Washington, D.C. 20234
May 10, 1983

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 of the fuel fraction of SRM 1817 was determined to be 2.3±0.3 (ASTM D644). The infrared spectrum of the fraction is shown in figure 1.

Metal Naphthenates

The metal naphthenate mixture in SRM 1817 is made of commercially available metal naphthenates according to the weight percentages described by Hotten and King [3]. The mixture is provided for user convenience and is the mixture used at NBS to generate tables 1 and 2. Depending on the individual metal, and the particular batch, the metal content and the molecular weight distribution of each metal naphthenate may vary significantly. However, no significant effects on oxidation results caused by this variance has yet been observed at NBS.

The metal naphthenates in this SRM have been carefully characterized and are from a single batch. 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," ASLE preprint No. 82-LC-ID-1, October, 1982.

[2] Hsu, S.M., Cummings, A.L., and Clark, D.B., "Evaluation of Automotive Crankcase Lubricants by Differential Scanning Calorimetry," SAE Paper No. 821252, SP-526, October, 1982.

[3] Hotten, B.W. and King, J.M., "Lubricating Oil Additive Composition," U.S. Patent No. 4,032,462 (June 28, 1977).

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

Test Condition: 1.5 g Oil
5 Wt.% Fuel Catalyst
5 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.
76A-1	64	5	281	±8
75B-1	56	5	206	±8
73B-1**	48	5	101	±2
79A	40	5	166	±2
81A	40	5	120	±4
77B-1	24	5	58	±1
77C	16	5	40	±2
72A-1	16	5	36	±1

* Viscosity Break Point Hour

** Passed the IIID engine test oxidation criterion (less than 400% viscosity increase at 40 hr), but failed the wear criterion.

Table 2. Induction Times of ASTM IIID Reference 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 Times, Min	
			Avg.	Std. Dev.
76A-1	64	9	31.0	2.5
75B-1	56	10	27.5	1.3
73B-1**	48	5	12.8	0.2
79A	40	8	21.5	1.5
81A	40	5	16.2	0.9
77B-1	24	5	9.6	0.7
77C	16	5	6.8	0.3
72A-1	16	5	6.1	0.2

* Viscosity Break Point Hour

** Passed the IIID engine test oxidation criterion (less than 400% viscosity increase at 40 hr), but failed the wear criterion.

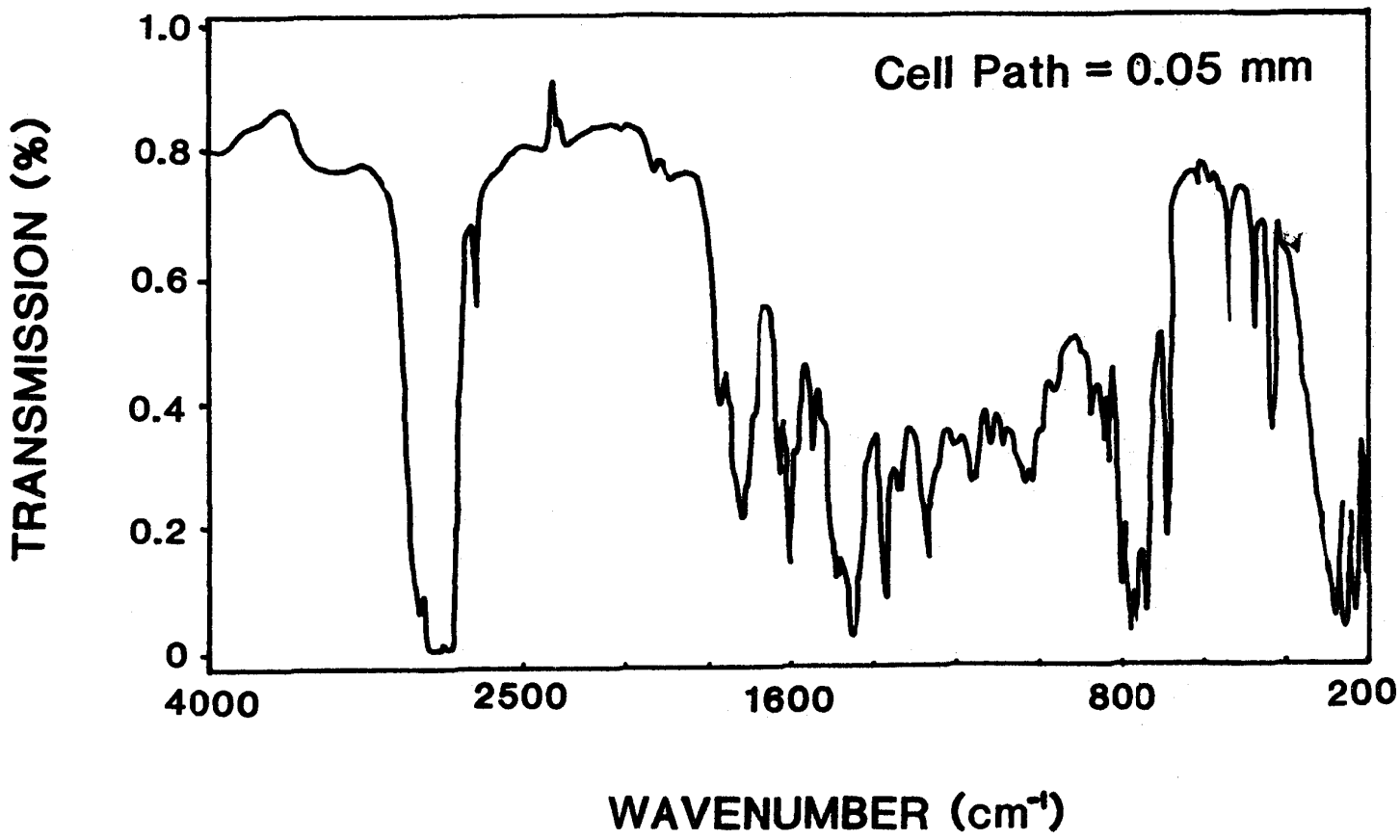

Fig. 1 IR Spectrum of Fuel Fraction

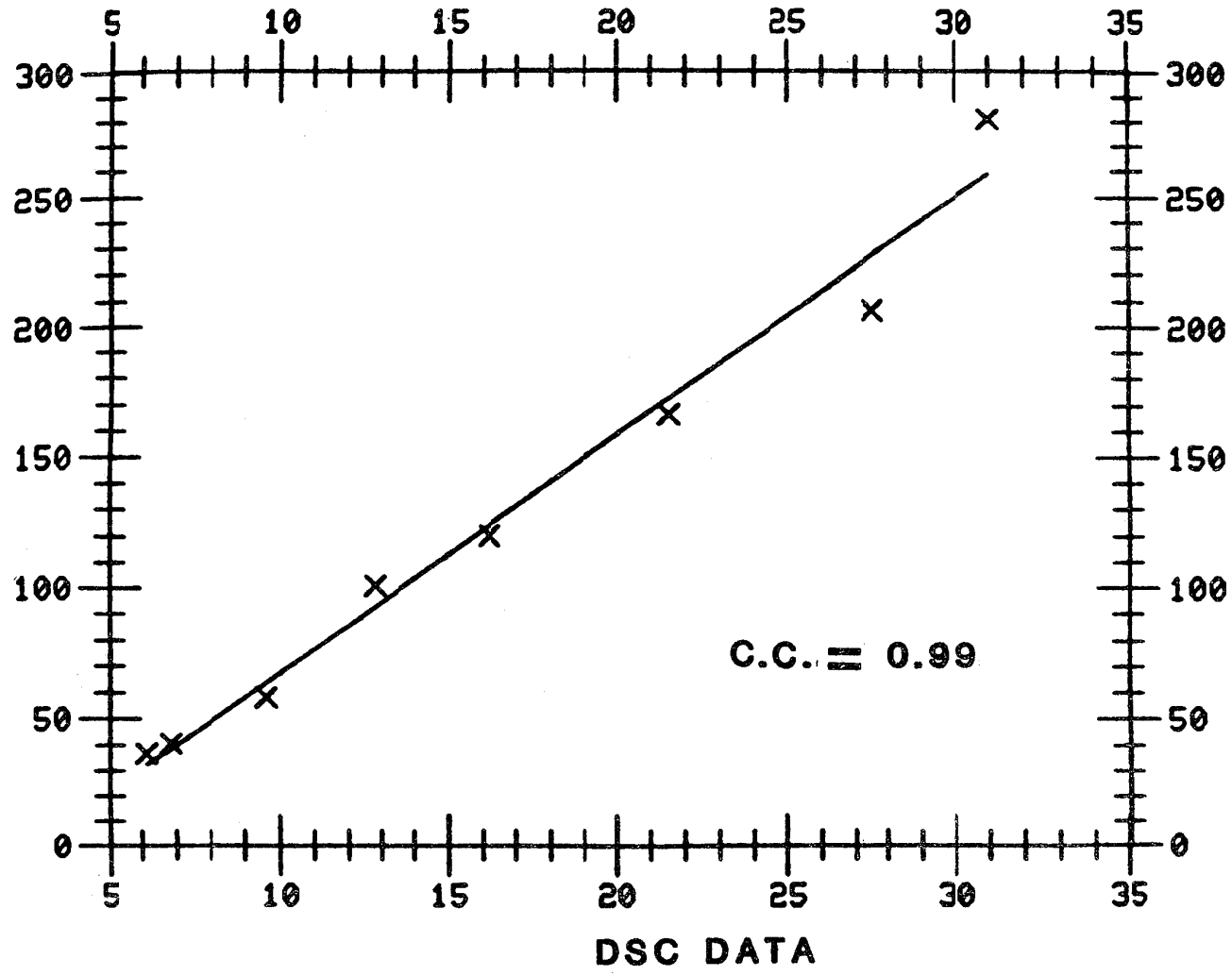

FIG. 2 Intercorrelation between TFOUT and DSC