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202

Thermal Stability of Hydrocarbon Fuels .

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AF Aero Propulsion Laboratory
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(Prepared under Contract No. AF 33(657)-10639
by Phillips Petroleum Company, Bartlesville,
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NOTICES

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FOREWORD

This report was prepared by Phillips Petroleum Company under USAF Contract No. AF 33(657)-10639. This contract was initiated under Project No. 3048, "Aviation Fuels", Task No. 304801, "Hydrocarbon Fuels". The work was administered under the direction of the AF Aero Propulsion Laboratory, Aeronautical Systems Division, with Mr. H. R. Lander acting as project engineer.

This report covers work carried out between June 1, 1963 and May 31, 1964.

The advice and guidance given by Robert M. Schirmer in this work, the contributions and assistance given by Harold C. Walters in assembling the report, engineering assistance by E. H. Fromm and statistical advice given by Meredith Goss is gratefully acknowledged.

ABSTRACT

This report covers the first year's effort by Phillips Petroleum Company, working under a three year Air Force Contract, AF 33(657)-10639, on factors that affect deterioration in thermal stability during storage of aviation turbine fuels and the development of a small-sample test method for the prediction of changes in thermal stability quality of hydrocarbon type fuels with time in storage.

A 5-ml Bomb test method, developed by Phillips Petroleum Company under a previous Air Force Contract, AF 33(616)-7241, was modified to improve its ability to measure small differences in thermal stability quality of fuels. This test method is based upon the loss in UV light transmittance experienced when a 5-ml sample of the fuel is heated to a given temperature for twenty minutes. A threshold failure temperature for a given fuel is determined by heating the bomb over a range of temperatures to establish the level of heating required to produce an arbitrary loss of 25 units in light transmittance at 350 millimicrons wavelength. A significant relationship was found between threshold failure temperatures determined for eleven non-additive fuels using the 5-ml Bomb test method and the ASTM-CRC Coker, but not for seven additive-containing fuels. A good relationship was found between the 5-ml Bomb and MINEX heat exchanger test rig, using seven fuels, three of which contained additives.

Several procedures were evaluated for accelerating the aging of fuels as possible test methods for predicting deterioration in thermal stability during storage. Procedures such as thermal stressing, ultraviolet irradiation, chemical initiation, and iron oxide catalysis, showed accelerated aging effects for a variety of fuels, as measured by deterioration in UV light transmittance. None of the accelerated aging procedures predicted the change in light transmittance or ASTM-CRC Coker performance of all fuels that occurred during 26 weeks 110°F hot room storage.

A storage program has been initiated with five fuels to study the environmental effects of temperature and oxygen content on the deterioration of thermal stability during storage.

This technical report has been reviewed and is approved.

Marc P. Dunnam
Mr. M. P. Dunnam
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I. INTRODUCTION

This report reviews experimental work carried out by Phillips Petroleum Company during the twelve month period from June 1, 1963 to May 31, 1964, which represents the first year's work under a three year contract with the Air Force. Earlier, related work completed by Phillips under a previous contract are summarized in References 1, 2, and 3. The major objectives of the present three year contract are described in detail in Appendix I of Progress Report #1 (Ref 4) and summarized briefly as follows:

I. Establish the reliability of the small scale jet fuel thermal stability test method (5-ml Bomb described in Ref 1).

II. Develop a small scale accelerated storage test method which will predict the normal storage life of JP-6 type fuels, based on deterioration in thermal stability quality.

III. Recommend corrective measures necessary to combat deterioration in thermal stability quality during storage of JP-6 type fuels. These will evolve from studies to:

  A. Determine the effect of various environmental factors.

  B. Determine the maximum concentration of various hydrocarbon components that can be tolerated without harmful effects.

  C. Determine the maximum concentration of various non-hydrocarbon contaminants that can be tolerated without harmful effects.

Experimental work during this first year period concentrated on objective I to establish the reliability of Phillips small scale jet fuel thermal stability test method with respect to (1) increasing the precision of the method, (2) establishing repeatability, (3) determining the effect of additives on precision and repeatability, and (4) developing possible correlations with other thermal stability test methods.

In an attempt to develop a small scale accelerated test method that will predict storage stability quality of jet fuels, exploratory work was conducted on the influence of ultraviolet irradiation, thermal stressing, chemical (free radical) initiation, and catalytic acceleration on changes in ultraviolet light transmittance. These results were compared to known changes in storage stability quality as measured by differential standard CRC Fuel Coker performance and differential light transmittance to determine if the accelerated procedure separated fuels in the proper order and magnitude.

Manuscript released by Lucien Bagnetto and Harold T. Quigg, 14 August 1964 for publication as an RTD Technical Documentary Report.

A storage program was initiated to study the environmental effects of time-temperature and dissolved oxygen content, in an effort to determine the mechanism that might be responsible for fuel deterioration during storage. The five fuels used in this program have been characterized with respect to physical properties and thermal stability as measured by the SSF Coker and Phillips Modified 5-ml Bomb.

II. PHILLIPS SMALL SCALE (5-ml Bomb) JET FUEL THERMAL STABILITY TEST METHOD

A. Background

A small sample (5-ml Bomb) test method previously developed for the Air Force by Phillips Petroleum Company(3) has been found useful for measuring gross differences in thermal stability quality of jet fuels. The Air Force requested that this method be reevaluated to determine if it could be made to recognize slight changes in thermal stability resulting from the addition of typical antioxidants in normal concentrations. Other objectives of the program are to evaluate the precision of the 5-ml Bomb procedure and to determine the extent of correlation between threshold failure temperature determined by the 5-ml Bomb and other test methods. During the second quarter of the contract year attention was focused on increasing the precision of the 5-ml Bomb while the third and fourth quarters were devoted to obtaining data for repeatability and correlation studies.

B. Original 5-ml Bomb Test Method

The 5-ml Bomb Thermal Stability Test Method as originally developed by Phillips is given in detail in Appendix I. In brief this method requires heating of eight to ten 5-ml volumes of fuel in a stainless steel bomb to various preselected temperatures, cooling and measuring the loss in light transmittance at 350 millimicron wave length. The loss in light transmittance is plotted against the maximum temperature to which the fuel sample is heated and a curve is fitted to the data points. Threshold failure temperature is arbitrarily defined as the temperature at which the curve intersects the 25 light-transmittance-loss level. The fuels that were used in the 5-ml Bomb precision study are shown in Table 24. To determine the precision of the test method three different base stocks were tested: (1) West Texas hydrotreated kerosine (2) Air Force Fuel SF6-6207 and (3) West Texas JP-6 blend. Tests were also made on separate portions of each of these fuels after blending with 100 and 500 parts per million of 2,6-ditertiary-butyl-4-methyl phenol (Ionol). The results of all runs by the 5-ml Bomb procedure using these fuels are tabulated in Table 25. Typical data for West Texas hydrotreated kerosine and Ionol blends are shown in Figure 1. This shows graphically the scattering and overlapping of the data points.

It is generally noted that certain antioxidants have a beneficial effect on thermal stability of JP-6 fuels. Specifically Kittredge(1) has shown that 500 ppm of Ionol added to a blended kerosine and alkylate base (K 500) reduced the average ASTM-CRC Fuel Coker preheater tube deposit code rating from 2.75 to 1.5 at 400/500°F test conditions. The same author(3) also showed that the addition of about 73 ppm Ionol to Air Force fuel SF6-6207 reduced the average coker deposit from 2.75

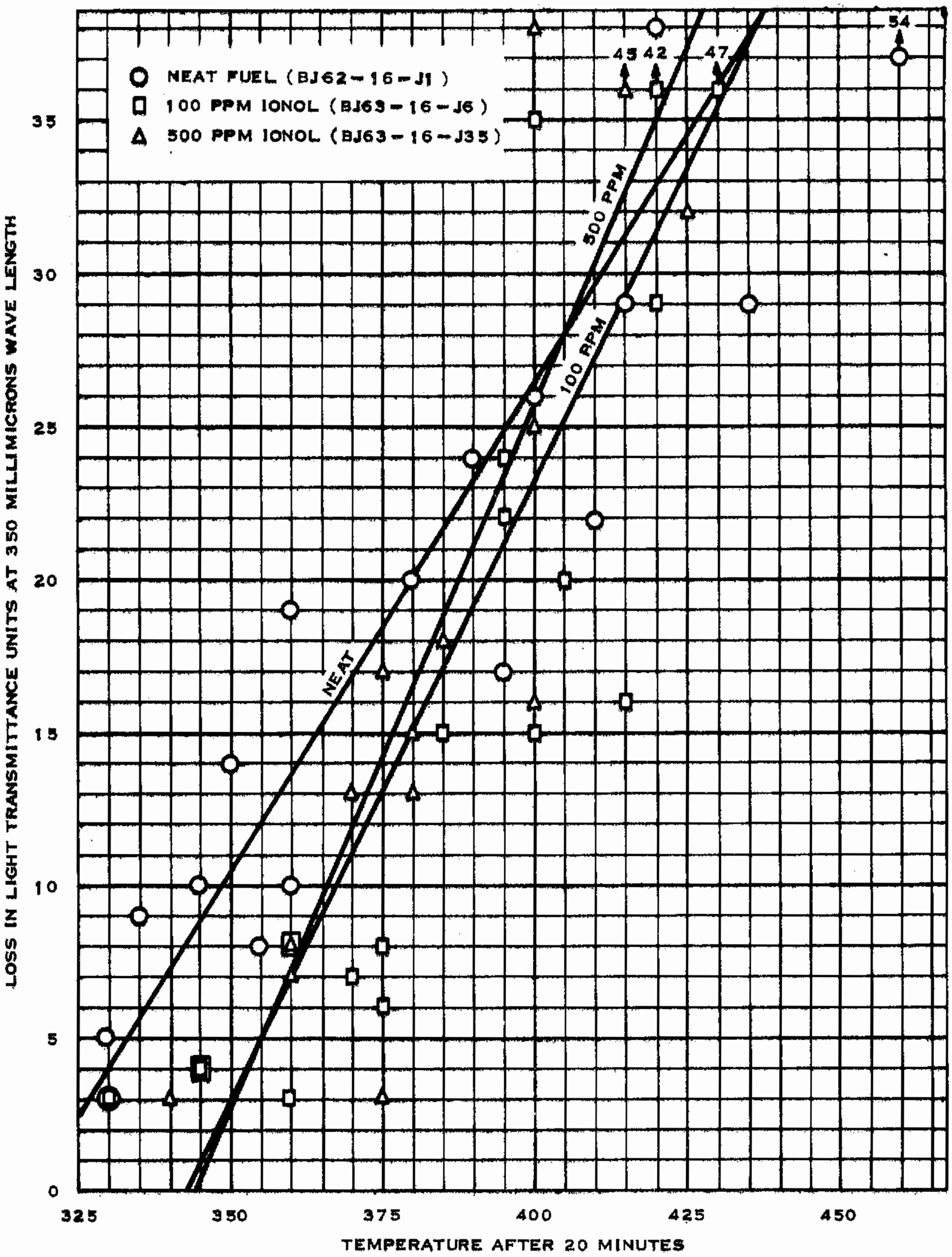


FIGURE 1 THERMAL STABILITY OF WEST TEXAS HYDROTREATED KEROSINE CONTAINING IONOL AS DETERMINED BY STANDARD 5-ML BOMB PROCEDURE

to 1.5 at 425/525°F conditions. It is apparent from the scatter of data in Figure 1, that the precision of the original 5-ml Bomb procedure is not sufficient to distinguish additive effects on thermal stability.

C. Studies to Improve Precision of 5-ml Bomb Test Method

Details of a study to improve the precision of the 5-ml Bomb are shown in Appendix II. From this study a Modified 5-ml Bomb Test Procedure was developed which resulted in improved precision. The improvement in precision is shown by the reduction in scatter of data points in Figures 2 and 3 compared to Figure 1. This procedure is shown in Appendix III. This procedure includes the following modifications.

1. Change in procedure for cleaning the bomb
2. Change in cooling technique after heating the bomb
3. Minimizing voltage input fluctuations to the furnace
4. Use of statistical methods for obtaining and interpreting the 5-ml Bomb data which include:
    - (a) Obtaining nine points over a specified light-transmittance-loss range
    - (b) Use of linear regression techniques for determining temperature for a given light-transmittance-loss
    - (c) Defining the maximum standard deviation from regression (4.0) for a satisfactory set of data.

Modifications which were investigated that showed no improvement in precision were:

1. Changing from a variable heating rate to a constant heating rate
2. Using a higher resolving Beckman spectrophotometer in place of the Bausch and Lomb Spectronic 20
3. Changing the wave length for measuring light transmittance from 350 to either 340 or 365 millimicrons.

D. Additive Effects by the Modified 5-ml Bomb Procedure

One purpose of this investigation was to determine if the 5-ml Bomb procedure could be modified to the point where it could recognize the small improvements that some additives impart to the CRC Coker thermal stability of some fuels. As an example in Figure 3 and in detail in Appendix II it is shown that the modified 5-ml Bomb recognized the effect of 30, 100 or 500 ppm Ionol at 0, 15 or 25 light-transmittance-loss levels. Data are also shown in Appendix II for blends of AN 701

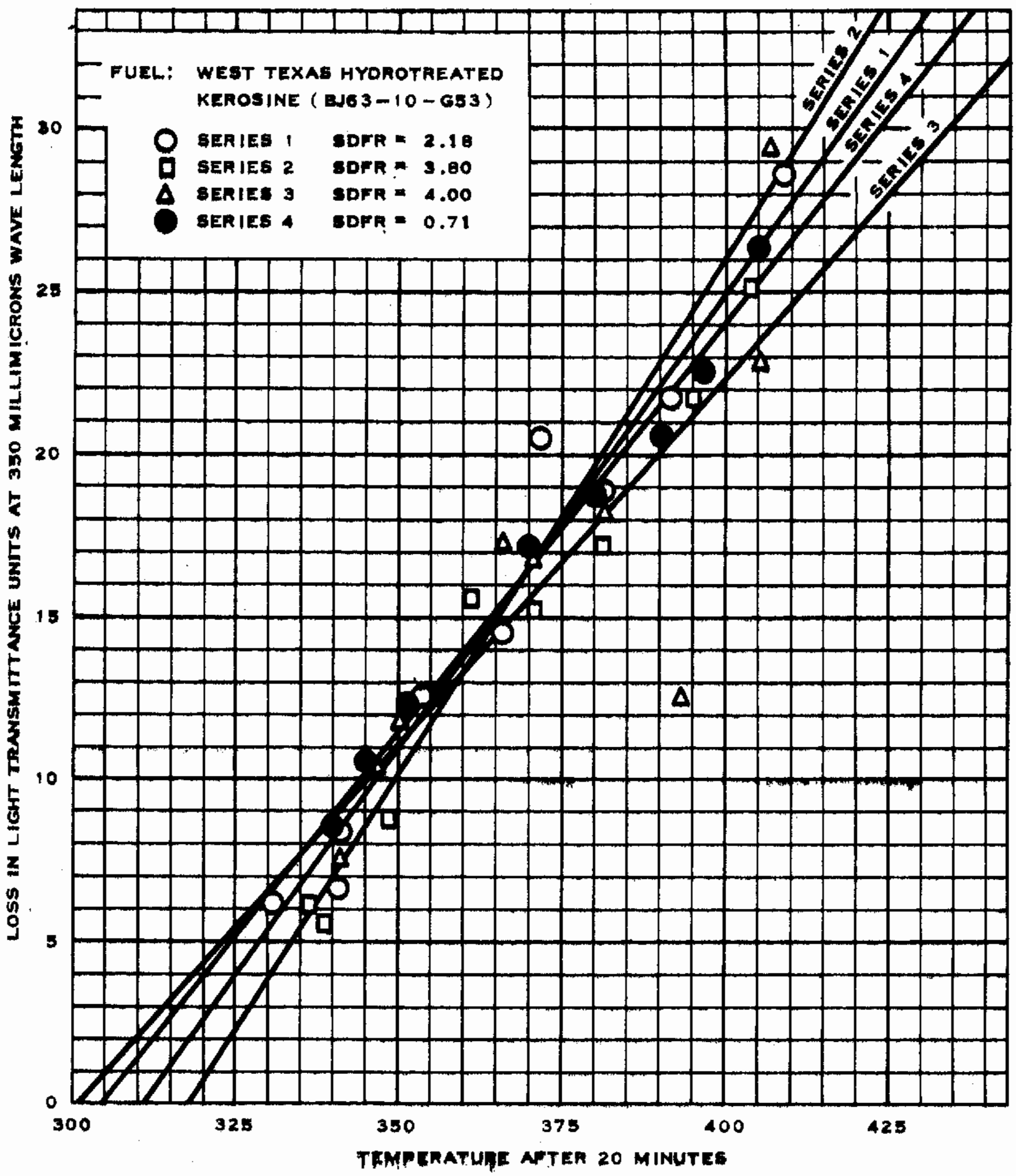


FIGURE 2 MODIFIED 5-ML BOMB REPEATABILITY FOR FOUR SERIES OF RUNS WITH WEST TEXAS HYDROTREATED KEROSINE

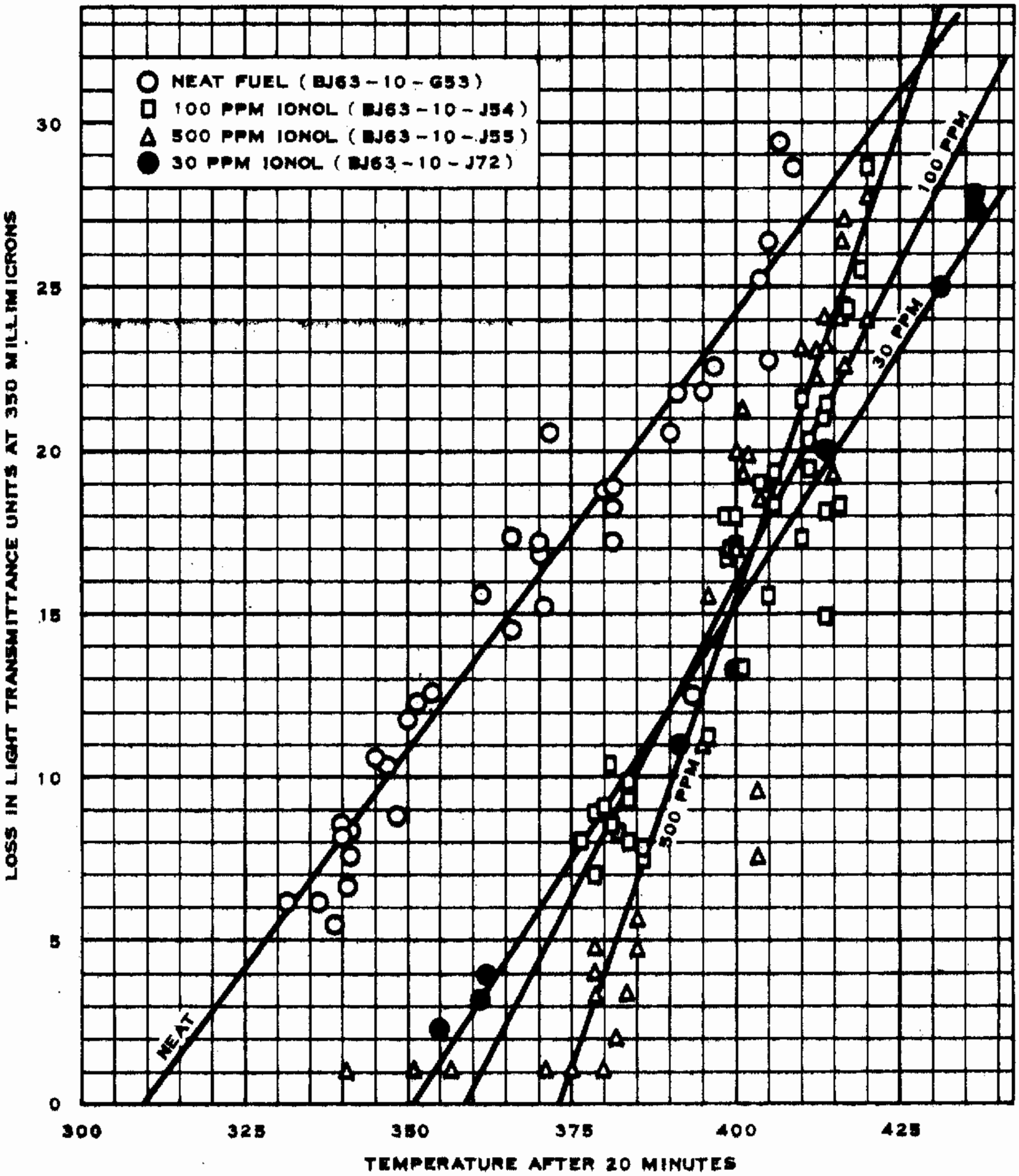


FIGURE 3 THERMAL STABILITY OF WEST TEXAS HYDROTREATED KEROSINE CONTAINING IONOL AS DETERMINED BY A MODIFIED 5-ML BOMB PROCEDURE

and du Pont 22. From these data it is concluded that the Modified 5-ml Bomb procedure recognized at least directionally, improvements in thermal stability imparted by antioxidants.

E. Repeatability and Correlations

After demonstrating that the Modified 5-ml Bomb procedure could recognize the small effects of antioxidants on thermal stability quality, a second phase of the program with the 5-ml Bomb was initiated. Details of this program are shown in Appendix IV. Briefly this consisted of multiple evaluations of a wide range of fuels over a considerable period of time using the Modified 5-ml Bomb. This series included a number of fuels furnished by the Air Force to Phillips Petroleum on which threshold failure temperatures had been or would be established by the ASTM-CRC Coker, Research Coker with ambient reservoir or the MINEX Rig. Other fuels which Phillips had evaluated in the ASTM-CRC Coker or were part of the storage stability program were also included.

1. Repeatability Program

From these data it can be concluded that overall repeatability of threshold failure temperature was poor when a wide variety of fuels and additives were tested. An examination of repeatability data for individual fuels shows that repeatability varied from very good to poor. The previous antioxidant study which showed good repeatability was limited to one base fuel and the same base plus two concentrations of an antioxidant.

An examination of data on one base fuel which was included in both test programs showed no change in repeatability from program to program; however, there was a shift in test severity as indicated by an increase in threshold failure temperature for the second program over the antioxidant program.

A thorough examination of the data has not shown an explanation of these variations.

While it is recognized that repeatability of the Modified 5-ml Bomb data is poorer than desired and that reproducibility of Coker data supplied by more than one laboratory may influence the results, a study was made of the relationships that may exist between the 5-ml Bomb and other thermal stability test methods.

2. Relationship Between Modified 5-ml Bomb and ASTM-CRC Coker

As shown in Appendix IV relationships were established between the Modified 5-ml Bomb at 10, 15 and 25 light-transmittance-loss levels and the ASTM-CRC Coker for non-additive fuels. A light-transmittance-loss level of 25 provided the best relationship between the Modified 5-ml Bomb and the Coker. Coker threshold failure temperatures plotted against temperature for a light-transmittance-loss of 25 are shown in Figure 4. The line shown represents the calculated relationship for the non-additive fuels (designated by "x"). Fuels containing antioxidants are shown as open points while those containing antioxidants plus metal deactivator are shown as closed points. It should be noted that the four fuels containing antioxidants only fall to the right of the line for non-additive fuels and with one exception all of the fuels containing metal deactivator in addition to antioxidant fall to the left of the line. This suggests that the Modified 5-ml Bomb and Coker have different responses to additives.

3. Relationship Between Modified 5-ml Bomb and MINEX

Another small-scale method for measuring fuel thermal stability is the MINEX test rig(9) which uses heat transfer loss in a single tube heat exchanger as a measure of fuel thermal stability quality.

As shown in Appendix IV statistically significant relationships were established between MINEX ratings and Modified 5-ml Bomb data at 10, 15 and 25 loss-levels with a 25 loss-level providing the best relationship. MINEX threshold failure temperatures plotted against temperatures for a light-transmittance-loss of 25 are shown in Figure 5. It should be noted that the MINEX and the Modified 5-ml Bomb appear to recognize the presence of additives and additive types more nearly the same than do the 5-ml Bomb and the Coker.

4. Relationship Between the Modified 5-ml Bomb and the SSF Coker

The SSF Coker is being used in a program to evaluate changes in storage stability quality of five JP-6 type fuels as part of this investigation. In Figure 6 are shown SSF threshold failure temperatures plotted against temperatures for a 25 loss-level in the 5-ml Bomb. It is apparent that there is a linear relationship for the three non-additive fuels while the two fuels containing metal deactivator in combination with an antioxidant fall to the left of the curve as with the ASTM-CRC Coker.

Comparing Figures 4 and 6 it can be seen that on the basis of 5-ml Bomb data the SSF Coker is more severe than the ASTM-CRC Coker at low levels of thermal stability quality and milder at the upper limit of rating ability of the ASTM-CRC Coker.

III. EXPLORATORY STUDIES TO DEVELOP A PROCEDURE TO PREDICT STORAGE STABILITY QUALITY

In anticipation of the large volumes of jet fuels that will have to be stored to meet the demands of present and future subsonic and supersonic flights, purchasers would like assurance that fuels will continue to be thermally stable after at least eighteen months storage. One of the objectives of the present study is to develop a small scale and relatively simple test procedure to predict the effect of long term ambient storage on thermal stability quality.

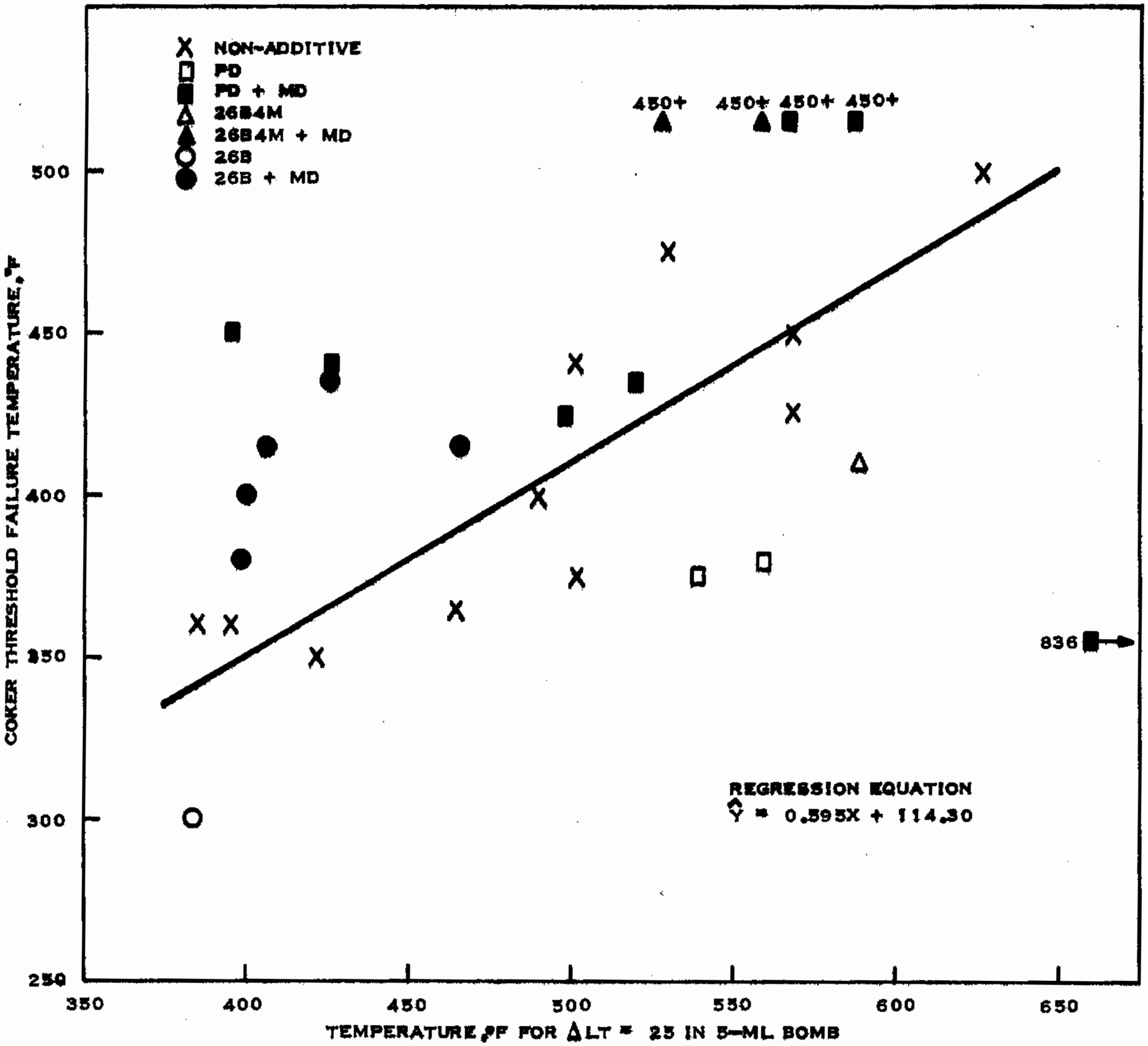


FIGURE 4 RELATIONSHIP BETWEEN 5-ML BOMB AND ASTM-CRC FUEL COKER RATINGS

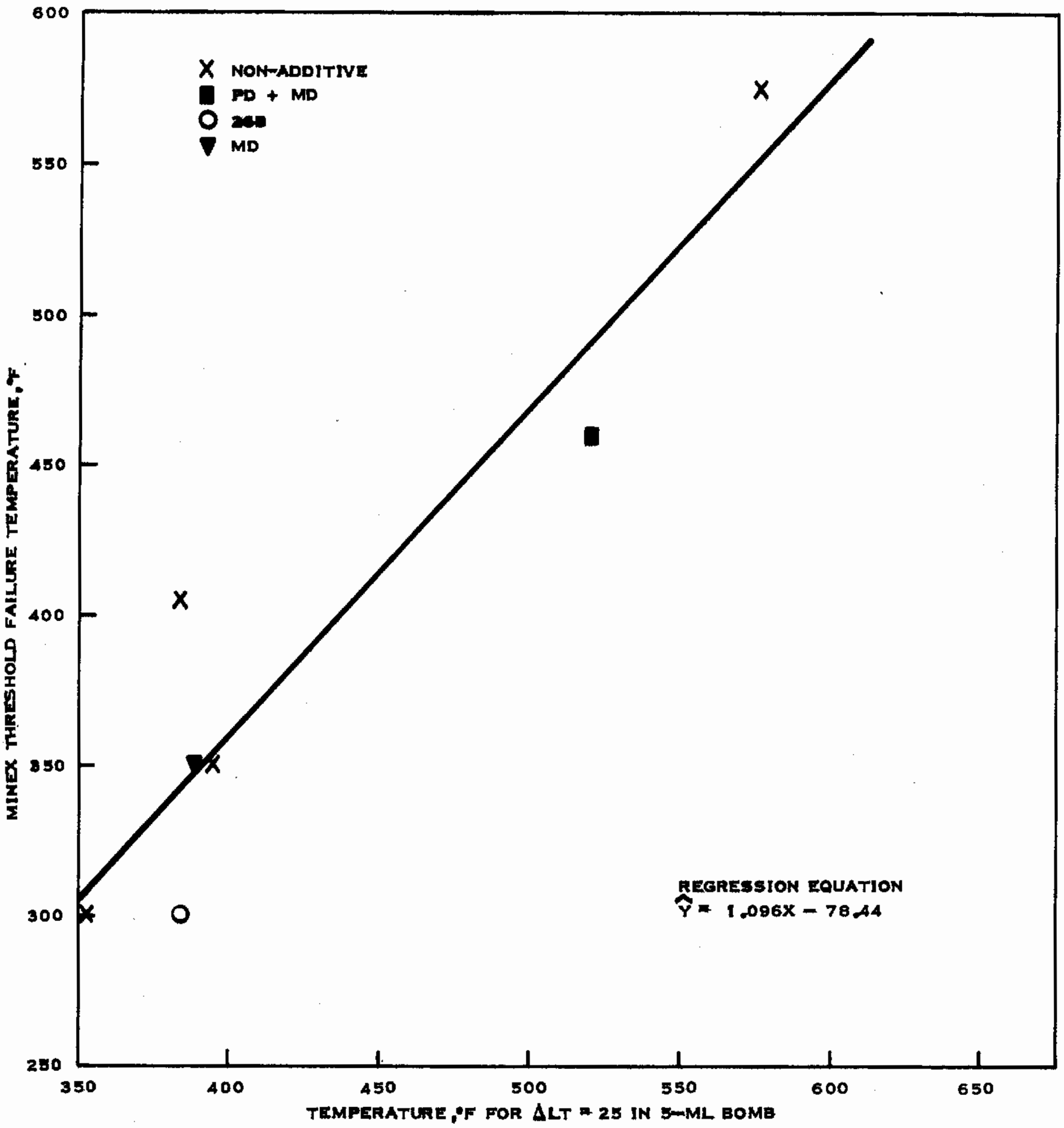


FIGURE 5 RELATIONSHIP BETWEEN 5-ML BOMB AND MINEX RATINGS

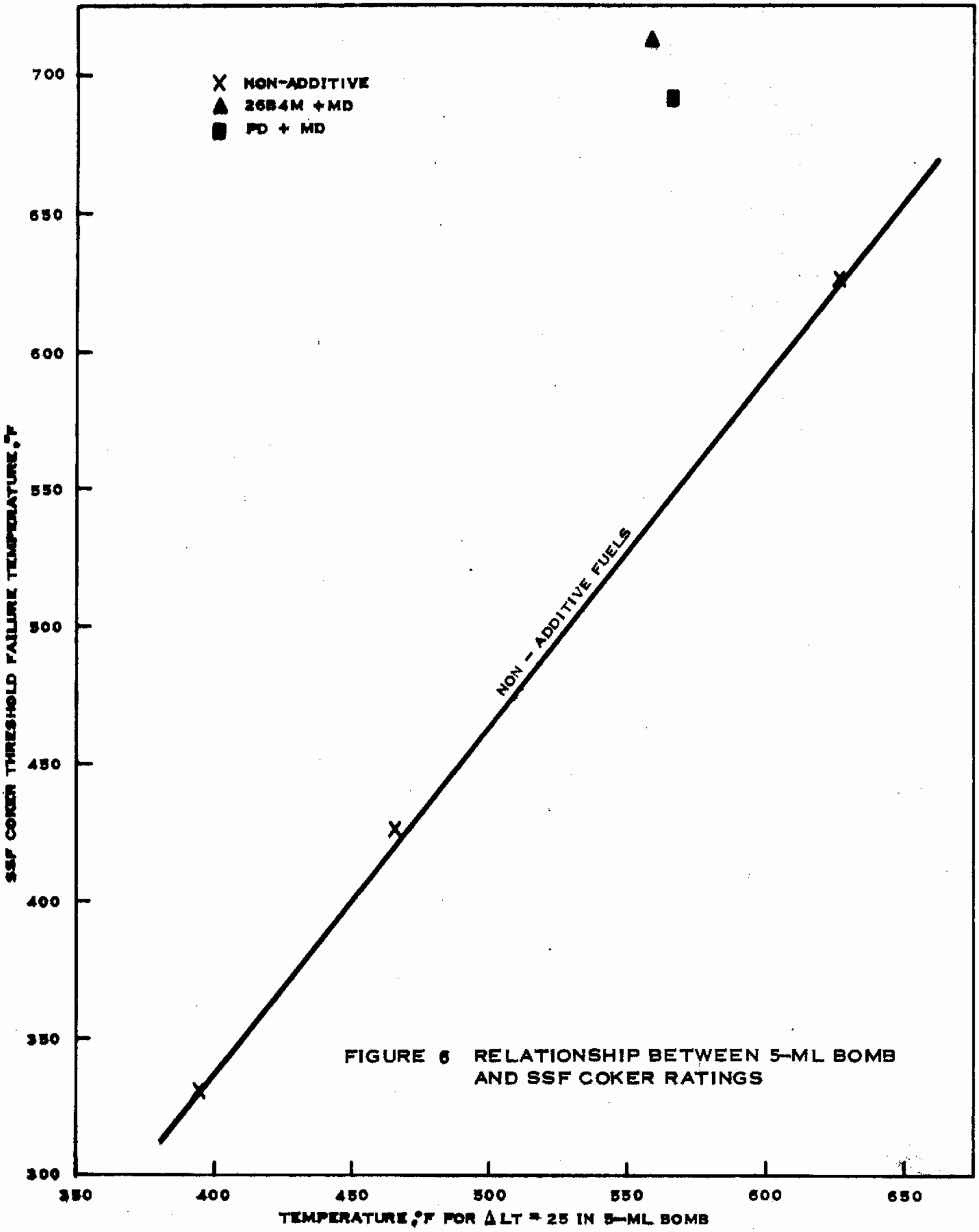


FIGURE 6 RELATIONSHIP BETWEEN 5-ML BOMB AND SSF COKER RATINGS

The literature reports the work of numerous investigators who have attempted to develop such a test procedure based on accelerated gums, peroxides, etc. formation (References 10, 11, 12, 13, 14, 15, 16). None of these studies have resulted in a procedure wholly acceptable by the industry.

The work done by Phillips in recent years under Air Force sponsorship (Refs 2 and 3) has consistently shown that as a result of either ambient or accelerated aging of fuels, significant changes in light transmittancy occur in the range of 300 to 550 millimicron wave lengths. Further exploratory work has been conducted to determine if this loss in light transmittance, induced by short term aging procedures, bears any relationship to the loss in ASTM-CRC Coker thermal stability ratings during long term storage.

A. Effect of 26 Weeks, 110°F Aging on Light Transmittance and Thermal Stability Quality

Before developing an accelerated storage stability test based upon inducing loss in light transmittance, it was considered desirable to determine if change in light transmittance during long term storage correlated with the change in Coker ratings for the same fuels and storage period.

Data were available from the previous Air Force contract(3) to permit such an analysis. Thirty fuels, Table 1, representing four base stocks with and without a variety of additives had been aged in a temperature controlled hot room at 110°F for 26 weeks and (1) duplicate CRC Fuel Coker and (2) light transmittance spectra (340-550 mμ wave lengths) were obtained before and after aging. ASTM-CRC Coker tests were made at 450/550°F/6 pph conditions for an isoparaffinic base fuel containing selected pure aromatic additives and 425/525°F/6 pph conditions for three JP-6 type fuels containing a variety of antioxidants and a metal deactivator. A summary of the Coker data is shown in Table 2. Light transmittance data at 340, 350 and 365 millimicrons wave lengths before and after aging are shown in Table 3.

Also shown in Table 2 are RTD-TSR Coker ratings for these same fuels. The CRC Coker data were converted to the RTD-TSR by a slight modification of a method proposed by the Air Force(7) to combine the effect of preheater deposit code and filter pressure rise into a single value. In brief this proposal suggests that the maximum preheater code be added to a number arbitrarily assigned to different ranges of Δ filter pressure rise according to the following scheme:

TABLE 1

DESCRIPTION OF PSEUDO-FRESH JP-6 FUELS USED IN AGING STUDIES

BJ62-10-	Description
J15	Phillips Isoparaffinic Base Oil No. 1 (1962 Production Batch)
J16	Base Oil + 2.0 wt % Cumene (Phillips Pure Grade)
J17	Base Oil + 2.0 wt % Mesitylene (Eastman Grade)
J19	Base Oil + 2.0 wt % Secondary Butylbenzene (Phillips Pure Grade)
J23	Base Oil + 2.0% Durene (Eastman Practical Grade--Redistilled)
J24	Base Oil + 100 ppm Indene (Eastman Practical Grade--Redistilled)
J25	Base Oil + 100 ppm Indene + 0.02 wt % Sulfur (1)
J27	Base Oil + 100 ppm Indene + 0.2 ppm Copper (2)
J28	Base Oil + 2.0 wt % Cumene + 0.2 ppm Copper (2)
J29	Base Oil + 2.0 wt % 2-Methylnaphthalene (Eastman Grade)
K20	Air Force JP-6 (SF6-6201) + .0029 wt % 2,6-Ditertiarybutylphenol (26B) + 0.0007 wt % Metal Deactivator MD (3)
J32	Air Force JP-6 (SF6-6201) + 0.0044 wt % 2,6-Ditertiarybutyl-4-Methylphenol (26B4M) + 0.0029 wt % 26B + 0.0007 wt % MD
J33	Air Force JP-6 (SF6-6201) + 0.0044 wt % 2,4-Dimethyl-6-tertiarybutylphenol (24M6B) + 0.0029 wt % 26B + 0.0007 wt % MD
J34	Air Force JP-6 (SF6-6201) + 0.0063 wt % (26B) + 0.0007 wt % MD
J35	Air Force JP-6 (SF6-6201) + 0.0063 wt % 26B4M + 0.0044 wt % MD
J36	Air Force JP-6 (SF6-6201) + 0.0063 wt % 26B + 0.0044 wt % MD
K43	Air Force JP-6 (SF6-6207) + 0.0029 wt % 26B + 0.0007 wt % MD
J44	Air Force JP-6 (SF6-6207) + 0.0044 wt % 26B4M + 0.0029 wt % 26B + 0.0007 wt % MD
J45	Air Force JP-6 (SF6-6207) + 0.0044 wt % 24M6B + 0.0029 wt % 26B + 0.0007 wt % MD
J46	Air Force JP-6 (SF6-6207) + 0.0063 wt % 26B + 0.0007 wt % MD
J47	Air Force JP-6 (SF6-6207) + 0.0063 wt % 26B4M + 0.0044 wt % MD
J48	Air Force JP-6 (SF6-6207) + 0.0063 wt % 26B + 0.0044 wt % MD
J62	West Texas JP-6 (50-50 Blend of West Texas Turbine Fuel + Paraffins)
J64	West Texas JP-6 + 0.0073 wt % 26B4M
J67	West Texas JP-6 + 0.0073 wt % 26B
J68	West Texas JP-6 + 0.0037 wt % Experimental Phenol (EP) (4)
J69	West Texas JP-6 + 0.0073 wt % 24M6B
J70	West Texas JP-6 + 0.0073 wt % 26B4M + 0.0044 wt % MD
J71	West Texas JP-6 + 0.0073 wt % 26B + 0.0044 wt % MD
J78	West Texas JP-6 + 0.0037 wt % EP + 0.0044 wt % MD

(1) Ditertiaryhexyldisulfide (Phillips--Redistilled)
(2) NBS Copper Cyclohexanebutyrate
(3) Disalicylal Propylenediamine (DuPont)
(4) Antioxidant AN 105 (Ethyl Corp.)

Note: Fuels SF6-6201 and 7 already contained 8 lb/1000 bbl (0.0029 wt %) of 26B and 2 lbs/1000 bbl (0.0007 wt %) MD when received. These percentages are included in the compositions shown above.

TABLE 2

CONVERSION OF ASTM-CRC COKER DATA TO AIR FORCE RTD-TSR COKER RATINGS

CRC FUEL COKER DATA 425/525°F

	Filter ΔP, "Hg.		Heater Deposit Max. (No. of Seg.)		RTD-TSR COKER RATINGS		
BJ62-10-	Fresh	Aged	Fresh	Aged	Fresh	Aged	Avg. Δ
J15	0.1	1.2	1(4)	0(13)	2	3	+ .5
	0.1	1.0	1(3)	0(13)	2	2	
J16	0.0	0.2	0(13)	0(13)	0	1	+2.0
	0.1	0.7	0(13)	2(4)	1	4	
J17	0.1	0.1	0(13)	1(3)	1	2	+0.2
	0.6	0.2	2(4)	1(4)	4	3	
	0.0		2(1)		2		
J19	0.2	1.4	2(7)	1(4)	3	4	0
	0.8	5.8	3(3)	1(2)	5	4	
J23	0.2	0.2	4(3)	4(3)	5	5	-1.0
	0.7	0.2	4(2)	3(3)	6	4	
J24	2.5	0.0	2(1)	1(5)	5	1	-3.0
	0.1	0.1	2(4)	0(13)	3	1	
J25	0.7	0.1	2(2)	1(6)	4	2	-2.5
	0.6	0.1	3(4)	1(5)	5	2	
J27	0.4	2.0	2(2)	0(13)	3	3	+0.5
	0.0	7.4	3(2)	1(4)	3	4	
J28	0.1	4.4	4(2)	0(13)	5	3	-2.0
	0.1	0.7	3(2)	0(13)	4	2	
J29	0.1	4.7	1(2)	3(3)	2	6	+4.5
	0.1	7.5	1(3)	4(4)	2	7	

CRC FUEL COKER DATA 450/550°F

K20	10.0	25/269 min	3(2)	0(13)	6	5	-0.7
	11.2	25/183	4(1)	0(13)	8	5	
	5.2		0(13)		3		
J32	12.8	25/250	1(2)	0(13)	5	5	0
	21.0	25.0	4(1)	4(2)	8	8	
J33	20.3	9.6	3(3)	0(13)	7	3	-3.0
	25/220 min	15.1	1(3)	0(13)	6	4	
J34	25/282	25/251	4(1)	0(13)	9	5	-4.0
		25/293		0(13)		5	
J35	25/177	8.8	3(2)	0(13)	8	3	-4.0
	25/201	4.3	1(2)	0(13)	6	3	
J36	25/199	25/140	0(13)	4(2)	5	9	+3.0
	25/178		2(1)		7		

(Continued)

TABLE 2 (Continued)

CRC FUEL COKER DATA 450/550°F

BJ62-10-	Filter ΔP, "Hg.		Heater Deposit Max. (No. of Seg.)		RTD-TSR COKER RATINGS		
	Fresh	Aged	Fresh	Aged	Fresh	Aged	Avg. Δ
K43	8.3 4.7 3.9	25/175 25/168	1(3) 3(2) 3(1)	3(2) 3(3)	4 6 6	8 8	+2.7
J44	23.9 25/255	2.2 11.3	1(3) 0(13)	0(13) 1(2)	5 5	3 5	-1.0
J45	13.0 25/283	0.5 1.7	0(13) 0(13)	0(13) 0(13)	4 5	1 3	-2.5
J46	8.0 25/280	15.0 25/270	0(13) 0(13)	0(13) 0(13)	3 5	4 5	+0.5
J47	10.8	25/235 25/169	0(13)	0(13) 4(1)	4	5 9	+3.0
J48	15.0	25/281 10.5	0(13)	1(3) 0(13)	4	6 4	+1.0
J62	0.5 0.2 0.7	0.0 0.6	0(13) 3(2) 2(4)	3(3) 3(2)	1 4 4	3 5	+1.0
J64	0.6 2.2	0.4 0.1	3(1) 3(3)	1(4) 3(5)	5 6	2 4	-2.5
J67	2.3 0.8	0.1 0.1	2(1) 3(1)	2(4) 3(4)	5 5	3 4	-1.5
J68	0.8 25/296 10.2	7.5 3.7	2(2) 4(1) 4(2)	8(5) 8(2)	4 9 8	11 11	+4.0
J69	0.7 1.3	1.8 2.3	2(3) 2(3)	1(3) 0(13)	4 5	4 3	-1.0
J70	1.8	0.6 0.9	1(2)	0(13) 0(13)	4	2 2	-2.0
J71	1.0	0.1 0.2	0(13)	0(13) 1(4)	2	1 2	-0.5
J78	1.3	0.3 0.3	0(13)	0(13) 0(13)	3	1 1	-2.0

TABLE 3

LIGHT TRANSMITTANCE DATA RESULTING FROM 26 WEEKS, 110°F AGING

	PerCent Light Transmittance								
	340 Millimicrons			350 Millimicrons			365 Millimicrons		
Fuels	Fresh	Aged	Δ	Fresh	Aged	Δ	Fresh	Aged	Δ
J15	59	59	0	68	68	0	75	74	1
J16	58	56	2	67	64	3	77	75	2
J17	60	60	0	67	67	0	76	75	1
J19	55	49	6	65	58	7	75	70	5
J23	56	56	0	65	65	0	76	75	1
J24	57	57	0	66	66	0	75	74	1
J25	57	57	0	66	65	1	76	74	2
J27	58	42	16	66	52	14	76	62	14
J28	58	42	16	66	51	15	75	66	9
J29	56	47	9	66	54	12	75	66	9
K20	78	71	7	84	77	7	89	81	8
J32	79	74	5	85	81	4	91	85	6
J33	80	74	6	85	79	6	91	82	9
J34	76	69	7	82	74	8	88	75	13
J35	57	48	9	72	58	14	84	69	15
J36	56	43	13	70	53	17	82	62	20
K43	85	76	9	90	81	9	94	83	11
J44	83	80	3	88	83	5	93	90	3
J45	85	79	6	90	85	5	94	89	5
J46	83	78	5	88	83	5	92	86	6
J47	60	49	11	73	61	12	83	71	12
J48	56	52	4	69	62	7	81	73	8
J62	76	74	2	82	78	4	89	85	4
J64	76	76	0	81	81	0	88	88	0
J67	70	70	0	82	81	1	84	82	2
J68	75	30	45	81	40	41	89	40	49
J69	75	75	0	83	82	1	89	89	0
J70	56	56	0	71	71	0	85	85	0
J71	56	50	6	72	65	7	86	74	12
J78	54	54	0	68	66	2	85	83	2

Note: Data obtained from curves shown in Air Force Report ASD-TR-61-238, Part III

Coker Results	RTD-TSR* System
Maximum Preheater Ratings:	
0 - bright polish	0
1 - dull but no color	1
2 - slight discoloration	2
3 - light tan	3
4 - medium tan	4
5 - light brown	5
6 - dark brown	6
7 - grey	7
8 - black	8

Filter Pressure Rise: Inches Hg		(Minutes)	
0		300	0
0.1 - 0.5		300	1
0.6 - 1.0		300	2
1.1 -10.0		300	3
10.1 -25.0		300	4
25.0	Less Than	300	5

*Air Force Research and Technology Division - Thermal Stability Rating

Differences in the average RTD-TSR Coker ratings before and after aging is the parameter used to measure storage stability quality. Positive value indicates the extent of degradation, negative values the extent of improvement resulting from storage.

Calculations of standard deviation and precision data for the pooled ΔRTD-TSR Coker ratings resulted in the following:

Standard Deviation	±1.25
Least Significant Difference for 99 Percent Confidence	3.33
Least Significant Difference for 95 Percent Confidence	2.50
Least Significant Difference for 90 Percent Confidence	2.09

To determine the relationship of loss in light transmittance at 350 mμ (resulting from aging) with the converted Coker data, a correlation plot of the two procedures for all fuels was made in Figure 7. The resulting scatter of data points was so great that no attempt was made to determine a correlation coefficient. Analyzing the data with respect to the absence of phenolic type inhibitors (Figure 7) also resulted in no correlation. Figures 8 and 9 show the scatter that exists when a comparison is made within a given base fuel (irrespective of additives).

Although these results are discouraging, failure to show an acceptable correlation was not construed to mean that no relationship could be expected to exist. Possible reasons for the poor correlation for all types of fuels might be

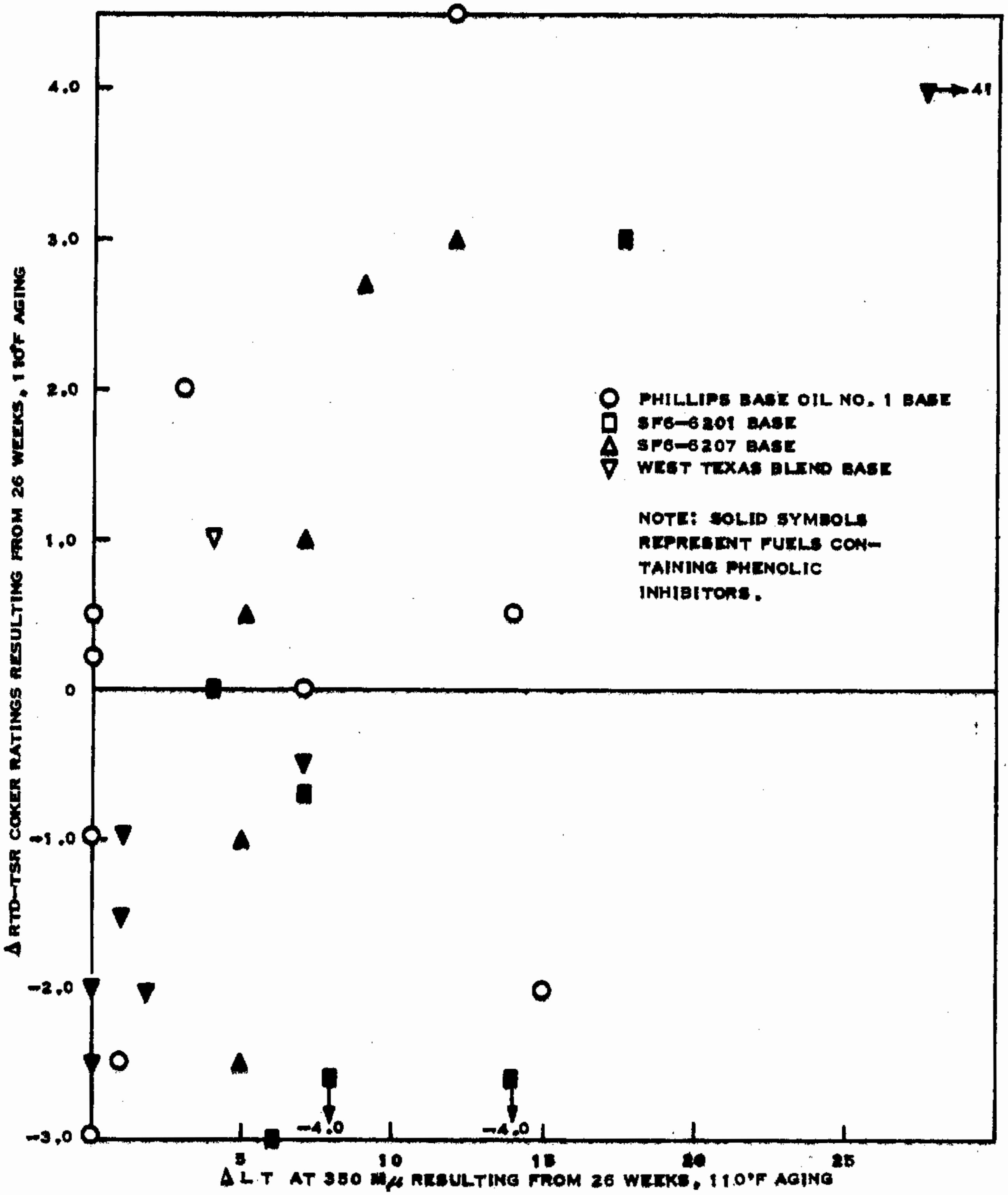


FIGURE 7 RELATIONSHIP OF LIGHT TRANSMITTANCE DETERIORATION WITH CHANGES IN RTD-TSR COKER RATINGS RESULTING FROM 26 WEEKS, 110°F HOT ROOM AGING-FOR ALL FUELS

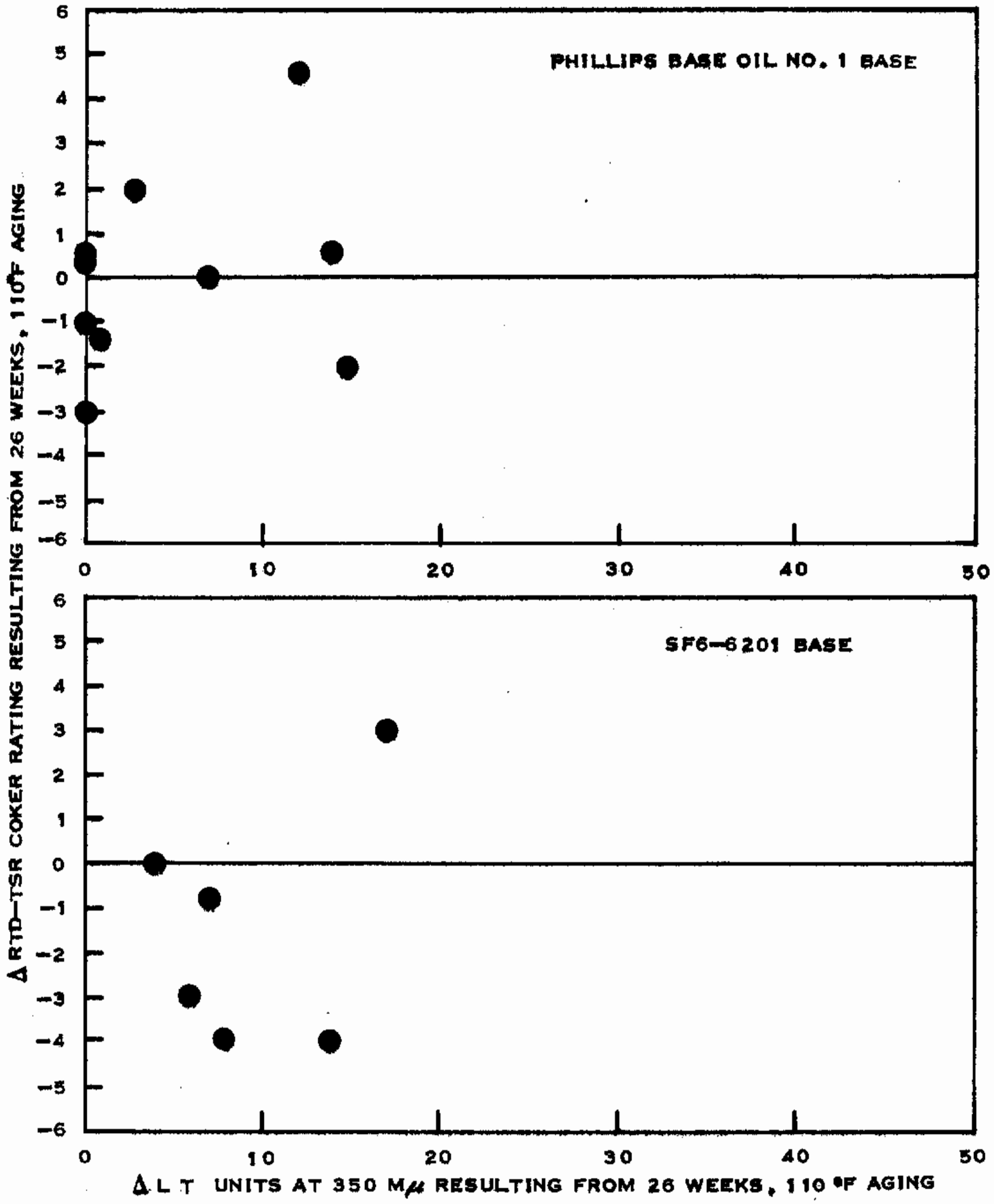


FIGURE 8 Δ LIGHT TRANSMITTANCE VERSUS Δ RTD—TSR COKER RATINGS FOR TWO DIFFERENT BASE FUELS CONTAINING ADDITIVES

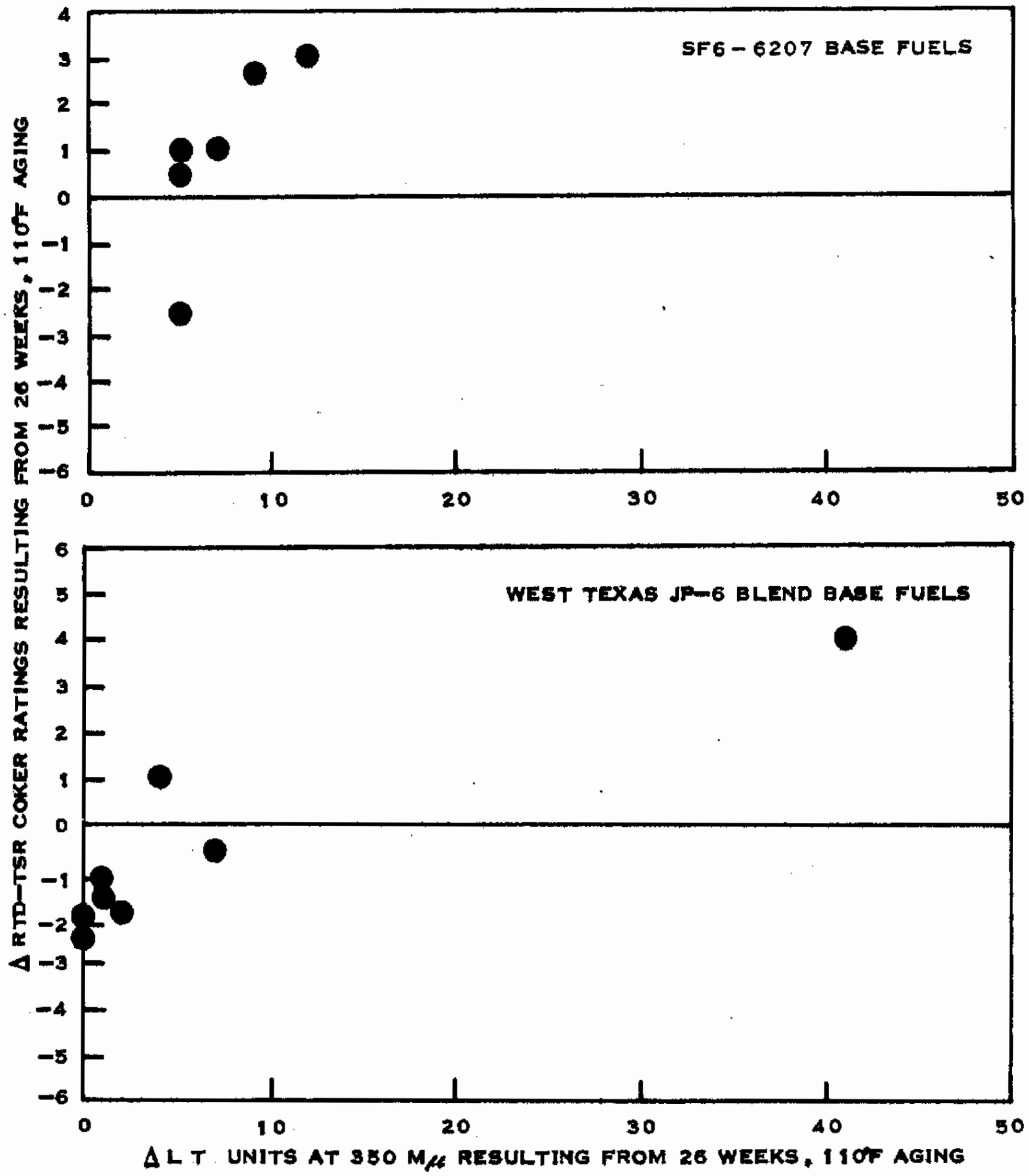


FIGURE 9 Δ LIGHT TRANSMITTANCE VERSUS Δ RTD–TSR COKER RATINGS FOR TWO DIFFERENT BASE FUELS.

(1) the lack of precision of the Coker data as determined statistically (2) the gross differences in substrate compositions whereby the mechanisms leading to deterioration might vary from fuel to fuel and would be unrelated to ultraviolet absorption.

The calculated precision for the pooled ΔRTD-TSR Coker ratings was shown to require a change of 2.5 code numbers (LSD at 95 percent confidence) for a given fuel to have changed significantly during 26 weeks, 110°F storage. From Table 2 and Figure 7 it is seen that eighteen of the thirty fuels did not significantly change in storage, seven improved and five deteriorated. The meaning of improvement of certain fuels in storage as measured by losses in light transmittance is not entirely understood. Since only five significantly deteriorated in storage, these were analyzed to see if a correlation existed. The following tabulation shows the Coker and light transmittance data for these fuels:

Fuel	ΔRTD-TSR Rating	ΔLight Transmittance @ 350 mμ Wave Length
K43	+2.7	9
J47	+3.0	12
J36	+3.0	17
J68	+4.0	41
J29	+4.5	12

As these figures indicate all fuels show significant losses in light transmittance but are not separated in a satisfactory manner in relation to the Coker data. Thus the difficulty of establishing a correlation between fuels, known to have deteriorated in storage is recognized.

In order to minimize interpretation of the data and to determine if a specific correlation might exist for fuels of a given substrate, all fuels in this group which were shown to have significantly deteriorated with respect to ΔRTD-TSR Coker ratings were selected for study. Fortuitously it was observed that each substrate had at least one additive composition that definitely failed during storage. In order to study the possibility of correlating differential Coker with differential light transmittance data within a given substrate fuel, it was necessary to select different additive fuels of the same substrate which did not significantly change during long term storage for comparison. Fortunately, data were also available on fuels in this study which could be used for this comparison. It should be pointed out that it is not the purpose of this report to study the effect of slight differences in additive composition and/or additive concentrations on storage stability quality but it will be obvious that the only reason for storage instability of certain fuels within a given substrate is due to these slight differences. A description of the fuels selected on this basis is shown in Table 4.

For two fuels within a given substrate to be considered significantly different the LSD at 95 percent confidence of 2.5 was arbitrarily multiplied by $\sqrt{2}$ to give 3.5 units. The differences shown between stable and unstable fuels within a substrate are separated by at least this amount and increases the confidence that the Coker data are reliable for this comparison.

TABLE 4

COMPARISON OF DIFFERENTIAL LIGHT TRANSMITTANCE AND DIFFERENTIAL RTD-TSR COKER RATINGS RESULTING FROM 26 WEEKS, 110°F AGING FOR SELECTED FUELS

Fuel	Description	ΔRTD-TSR Rating	Assigned Storage Stability	Δ Light Transmittance Units At following Wave Lengths		
				340	350	365
J23	Base Oil #1 + 2.0 Wt % Durene	-1.0	Stable	0	0	1
J29	Base Oil #1 + 2.0 Wt % Me-Naph.	+4.5	Unstable	9	12	9
K20	SF6-6201 + 0.0029 Wt % 26B + 0.0007 Wt % MD	-0.7	Stable	7	7	8.
J34	SF6-6201 + 0.0063 Wt % 26B + 0.0007 Wt % MD	-4.0	Stable	7	8	13
J36	SF6-6201 + 0.0063 Wt % 26B + 0.0044 Wt % MD	+3.0	Unstable	13	17	20
J44	SF6-6207 + 0.0044 Wt % 26B4M + 0.0029 Wt % 26B + 0.0007 Wt % MD	-1.0	Stable	3	5	3
K43	SF6-6207 + 0.0029 Wt % 26B + 0.0007 Wt % MD	+2.7	Unstable	9	9	11
J47	SF6-6207 + 0.0063 Wt % 26B4M + 0.0044 Wt % MD	+3.0	Unstable	11	12	12
J78	West Texas Blend (JP-6) + 0.0037 Wt % KP + 0.0037 Wt % MD	-2.0	Stable	0	2	2
J68	West Texas Blend (JP-6) + 0.0037 Wt % EP	+4.0	Unstable	45	41	49

The precision of light transmittance measurements at 350 mμ wave length was shown to be ±3.1 units for the differences of single determinations at 95 percent confidence. The Least Significant Difference (LSD) between fuels is obtained by multiplying 3.1 x $\sqrt{2}$ or 4.3 light transmittance units. Data in Table 4 show that all of the Δlight transmittance values within a given substrate differ by amounts greater than necessary to indicate a significant separation in storage stability quality (as measured by significant changes in Coker performance). It is also apparent that all unstable fuels in this group showed light transmittance losses of at least eight units which is indicative of a possible criterion for rating storage stability in terms of differential light transmittance.

As a result of these considerations and as shown in Figure 10 a favorable relationship between differential light transmittance and Coker data is apparent. The correlation, however, is valid only for fuels within a given substrate which are similar as to additive compositions and/or concentrations and are known to be significantly separated by fuel Coker data. Additional fuels of related compositions should be tested to determine a more realistic regression.

B. Accelerated Aging Using Ultraviolet Irradiation

From a practical viewpoint, accelerated aging with ultraviolet light might not be expected to simulate reactions occuring during long term storage since fuels stored in drums would receive very little or no ultraviolet irradiation. However, since it is not possible for an "accelerated" procedure to duplicate the "long-term" storage environment any method is potentially as good as another. The criteria that must be met are that the reaction mechanisms resulting in deterioration, activation energy requirements and reaction rates will be simulated by the accelerated procedure. Since the ability of ultraviolet irradiation to age fuels is well known, this was studied using differential light transmittance as a criterion to ascertain if it would correlate with known changes in storage stability quality resulting from 26 weeks, 110°F storage as measured by Fuel Coker performance changes.

Since the fuels shown in Table 4 represented the most reliable changes in storage stability quality of similarly related fuels, and various substrates and additive compositions are included, these were used for a preliminary survey. It was assumed that if an accelerated procedure simulated the same reactions occuring in 26 weeks, 110°F storage a correlation with losses in light transmittance and changes in RTD-TSR Coker ratings resulting from long-term storage would be observed. A failure to recognize a correlation with these parameters was interpreted to mean that the accelerated procedure was not satisfactory as a potential predictive test method.

Accelerated aging of fuels by UV-irradiation at an elevated temperature was accomplished with the use of a specially designed oven (Figure 11). A Sylvania Sun Lamp (110-v AC, 275 watts) was used as a source of thermal and ultraviolet light energy. Approximately 10-ml samples were filtered through 0.45 micron/ Millipore membrane and the initial per cent light transmittance using isooctane as a reference (100 percent) and a Bausch and Lomb Spectronic 20 spectrophotometer

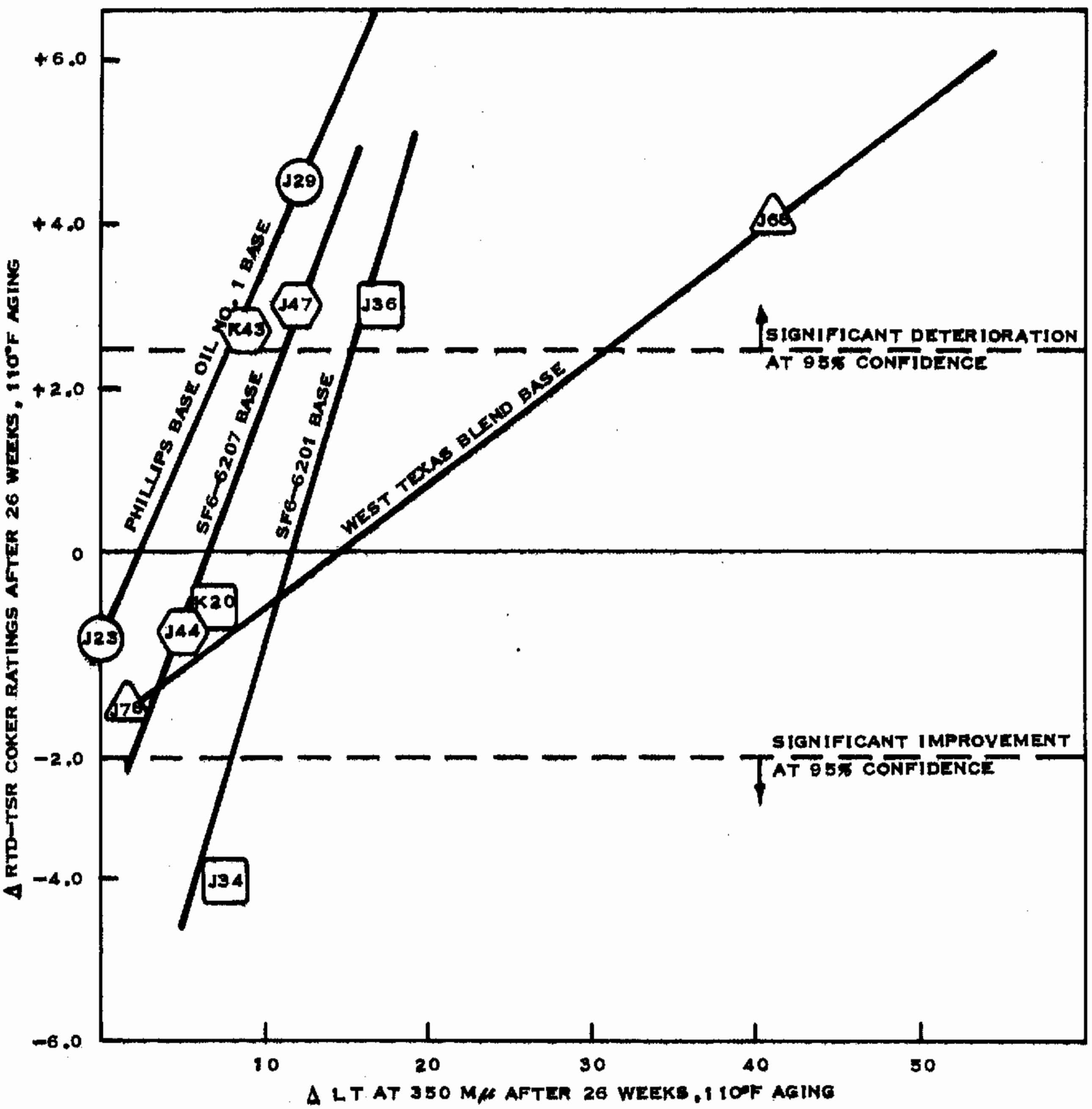
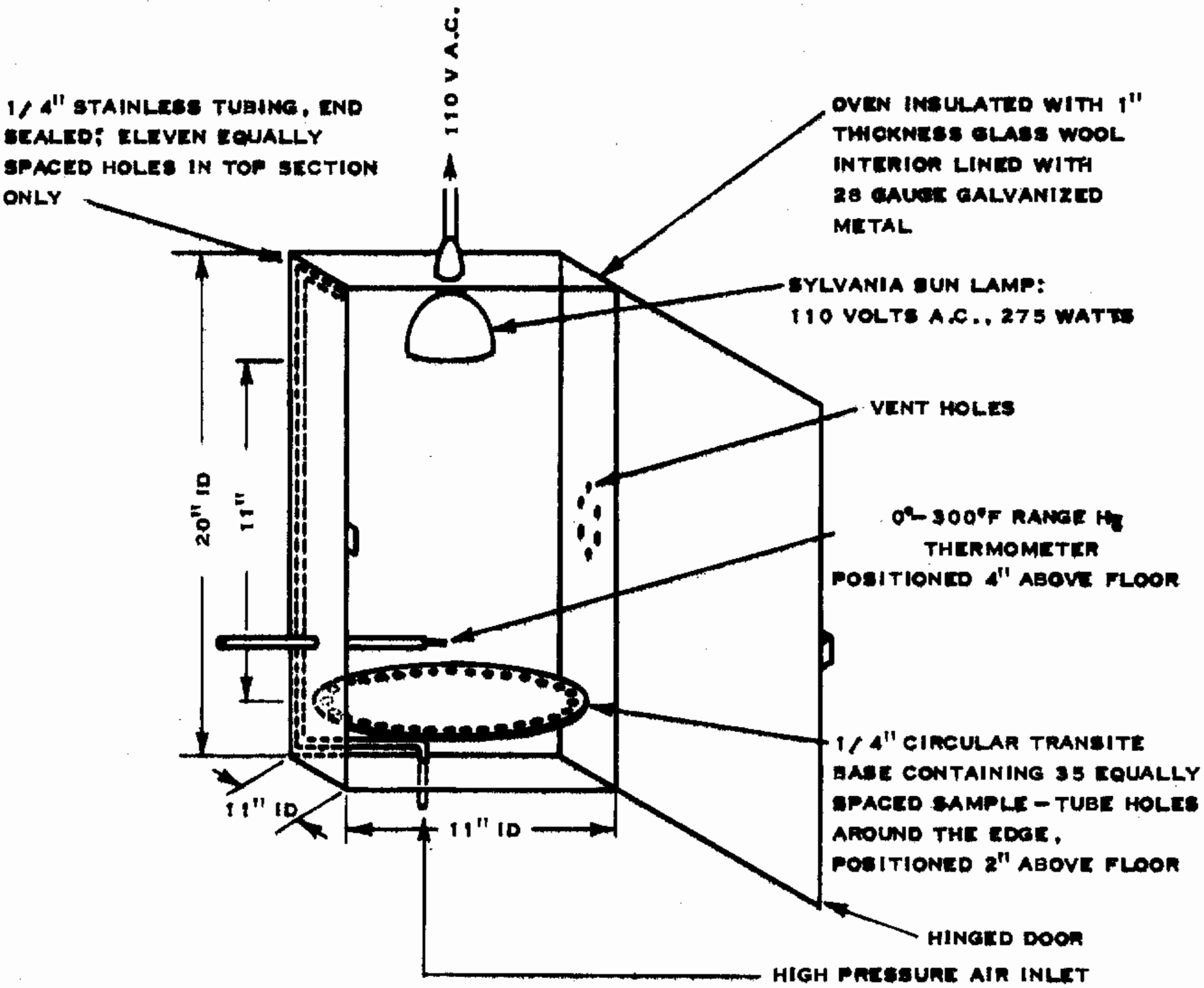


FIGURE 10 RELATIONSHIPS OF Δ LT WITH Δ COKER RATINGS RESULTING FROM 26 WEEKS, 110°F AGING FOR DIFFERENT ADDITIVE TREATMENTS WITHIN A GIVEN BASE FUEL



NOTE: OVEN CAN BE MADE FROM ANY SUITABLE COMMERCIAL PICNIC ICE–CHEST THIS BOX SUPPLIED BY HETTRICK, TOLEDO, OHIO

FIGURE 11 SCHEMATIC DIAGRAM OF ULTRAVIOLET IRRADIATION OVEN were obtained. The samples were placed into 15 ml Pyrex centrifuge tubes and irradiated at 180°F for 0.5, 1.0 and 1.5 hours. Since degradation reactions normally occurring in storage could possibly be catalyzed by the iron surface of the drum walls, matching samples each containing a polished soft iron rod (1 1/2 x 1/8 inch) were irradiated for 1.0 and 1.5 hours. (No attempt was made to study the effect of iron oxide at this time.) After irradiation, light transmittance spectra were obtained without an intervening filtration. The differential light transmittance spectra obtained as a result of ultraviolet irradiation for samples without any iron present are compared with differential RTD-TSR ratings in Table 5.

TABLE 5

EFFECTIVENESS OF DIFFERENTIAL LIGHT TRANSMITTANCE RESULTING FROM ULTRAVIOLET IRRADIATION AT 180°F AS AN ACCELERATED AGING PROCEDURE

	Storage Stability Quality(a)		Δ Light Transmittance Units @ 365 mμ Wave Length After Irradiation At 180°F For Following Hours		
Fuel	ARTD-TSR Fuel Coker Ratings	Δ Light Transmittance Units(b)	0.5	1.0	1.5
J23	Stable	1	0	1	6
J29	Unstable	9	1	1	1
K20	Stable	8	4	5	13
J34	Stable	13	8	7	20
J36	Unstable	20	5	5	9
J44	Stable	3	1	2	8
K43	Unstable	11	(c)	(c)	(c)
J47	Unstable	12	3	4	8
J78	Stable	2	7	10	10
J68	Unstable	49	6	3	5

(a) As a result of 26 weeks, 110°F aging

(b) At 365 mμ wave length

(c) Data not available due to fuel depletion

Note: Repeatability for light transmittance values shown is ±3.6 units for differences of single determinations at 95 percent confidence.

These data show that UV-irradiation can affect light transmittancy for some of the fuels especially after 1.0 hours of irradiation, but the trend is not consistent. The differential loss in light transmittance within any given substrate is not in the proper order when compared to the known storage behavior as measured by the Coker.

Table 6 shows similar results using iron metal in the fuel samples to simulate a drum environment. The use of iron metal does not show any ability to rate storage effects in the proper order as measured by the Coker.

TABLE 6

EFFECTIVENESS OF DIFFERENTIAL LIGHT TRANSMITTANCE RESULTING FROM ULTRAVIOLET IRRADIATION AT 180°F IN THE PRESENCE OF FE-METAL AS AN ACCELERATED AGING PROCEDURE

	Storage Stability Quality(a)		Δ Light Transmittance Units @ 365 mμ Wave Length After Heating At 180°F For Following Hours	
Fuels	ARTD-TSR Fuel Coker Ratings	ΔLight Transmittance Units(b)	1.0	1.5
J23	Stable	1	2	4
J29	Unstable	9	0	2
K20	Stable	8	7	16
J34	Stable	13	9	23
J36	Unstable	20	9	21
J44	Stable	3	3	13
K43	Unstable	11	(c)	(c)
J47	Unstable	12	7	10
J78	Stable	2	8	14
J68	Unstable	49	3	5

(a) As a result of 26 weeks 110°F aging
(b) At 365 mμ wave length
(c) Data not available due to fuel depletion

NOTE: Repeatability for light transmittance values shown is ±3.6 units for differences of single determinations at 95 percent confidence.

From these preliminary experiments it is concluded that the conditions chosen were severe enough to degrade the fuels significantly with respect to light transmittance characteristics but these changes are not related to the reactions responsible for storage instability as measured by the Coker or as measured by losses in light transmittance from 26 week storage.

C. Accelerated Aging By Thermal Stressing

Because the degradation reactions taking place during long term storage are believed to be temperature dependent, conditions were chosen to keep the aging temperature to a minimum while maintaining a reasonable test duration. Earlier work(3) indicated that aging the test fuels 16 hours at 212°F was more than sufficient to recognize light transmittance deterioration. Accordingly, the available selected fuels (Table 4) were aged at 180°F for 48 hours in conjunction with differential light transmittance measurements at 365 millimicrons wave length in the following manner.

Approximately 10 ml of each fuel (except fuel K43) shown in Table 4 were filtered through 0.45 micron/Millipore membrane and initial light transmittance spectra over 340-550 mμ wave length range were obtained using a Bausch and Lomb Spectronic 20 spectrophotometer. All samples contained a polished iron rod and were heated in 15 ml Pyrex centrifuge tubes in the absence of light for 48 hours using a Hotpack oven (Model 1354; 115-v ac 20 amps) capable of controlling temperatures in the range of 95-356°F. (The particular oven used in this work was obtained from the Hotpack Corporation, Philadelphia, Pennsylvania.) After heating, the fuels were cooled to room temperature slowly in the absence of light and light transmittance spectra were redetermined with no intervening filtration.

The results of this experiment at 365 millimicrons wave length are shown in Table 7.

TABLE 7

EFFECTIVENESS OF DIFFERENTIAL LIGHT TRANSMITTANCE RESULTING FROM THERMAL STRESSING AT 180°F IN PRESENCE OF FE-METAL AS AN ACCELERATED AGING PROCEDURE

	Storage Stability Quality(a)		
Fuels	ΔRTD-TSR Fuel Coker Ratings	Δ Light Transmittance Units(b)	Δ Light Transmittance Units @ 365 mμ Wave Length After 48 Hrs Heating @ 180°F In Presence of FE-Metal
J23	Stable	1	3
J29	Unstable	9	4
K20	Stable	8	9
J34	Stable	13	3
J36	Unstable	20	4
J44	Stable	3	4
K43	Unstable	11	(c)
J47	Unstable	12	3
J78	Stable	2	4
J68	Unstable	49	19

(a) As a result of 26 weeks, 110°F aging
(b) At 365 mμ wave length
(c) Data not available due to fuel depletion

NOTE: Repeatability for light transmittance values shown is ±3.6 units for differences of single determinations at 95 percent confidence.

The data indicate that thermal stressing under the conditions described above could only separate one set of fuels (J78 and J68) out of four as to the proper storage stability quality as measured by the Coker or light transmittance changes. Since the total loss for all fuels except K20 and J68 was four light transmittance units and less, the conditions may not have been severe enough to obtain a reasonable separation. Further work should be done at lower temperatures to develop a possible relationship since it was shown in earlier work(3) that higher temperature (212°F) aging with ultraviolet light did not correlate with the known storage behavior of these fuels.

D. Accelerated Aging In The Presence Of Iron And Iron Oxide

In order to determine the possible catalytic effects of drum walls on fuel deterioration during long term storage, and to ascertain whether such effects could be used to accelerate aging such as to simulate deterioration normally occuring, eight of the ten fuels (Table 4) were aged in Pyrex containers in the presence of (1) polished iron metal rods, (2) matching iron metal rods previously oxidized to give a coating of black iron oxide, (3) finely divided black Fe_3O_4 powder and (4) finely divided red Fe_2O_3 powder. Matching samples containing no iron were also included for comparison. The iron oxide coatings were prepared by heating soft iron rods (1 1/2 x 1/8 inch) at 280°F for 15 hours in air. A black coating of presumably Fe_3O_4 was visually apparent. The weights of iron oxide deposited were less than 0.2 milligrams. For samples tested in the presence of powdered catalyst one tenth per cent by weight of black Fe_3O_4 (Fischer Scientific Purified Lot No. 713508) and red Fe_2O_3 (Matheson Coleman & Bell, Reagent Grade CB385) were used. Aging was accomplished by heating the samples at 180°F for 48 hours in a Hotpack oven. Separate sets of fuel samples were aged (1) by sealing the containers immediately after the fuel temperature reached the desired control temperature (approximately 5 minutes) and (2) sealing the containers at ambient temperature prior to heating. Initial experiments contained rubber seals in the system. Because of the possibility that contamination from the rubber was responsible for the effects observed, subsequent experiments were conducted which eliminated this possibility. As the data will indicate, such effects are not attributed to contamination from the seals.

The effect of iron and iron oxide on light transmittance and a comparison with the known storage behavior are shown in Table 8. Three experiments without any catalyst present in the fuel show only small changes (deterioration) resulting from aging at 180°F for 48 hours. The repeatability of the method is good and in general the differences from the mean value of the three trials are within the precision of light transmittance measurements (1.8 units at 95 percent confidence for the differences in the averages of triplicate determinations).

For any catalyst to have significantly (95 percent confidence) affected the magnitude of deterioration as a result of accelerated aging a change of 2.5 light transmittance units should be recognized, (calculated by multiplying 1.8 x $\sqrt{2}$). On the basis of this criterion the use of iron metal did not have any effect on fuel deterioration since the mean values in the presence of iron did not exceed this amount when compared with the mean values of fuels containing no catalyst.

TABLE 8

EFFECTIVENESS OF DIFFERENTIAL LIGHT TRANSMITTANCE RESULTING FROM THERMAL STRESSING AT 180°F IN THE PRESENCE OF IRON OXIDE AS AN ACCELERATED AGING PROCEDURE

Storage Stability Qual.(a)			Δ Light Transmittance Units @ 350 mμ Resulting from Heating 48 Hours at 180°F														
	Δ RTD-TSR Fuel Coker	Δ (350 mμ) Transmittance	No Catalyst				Fe-Metal Rod				Oxidized Fe-Metal Rod					Finely Divided Catalysts	
			(b)	(c)	(d)		(b)	(c)	(d)		(b)	(c)	(d)	(e)	(g)	(e)	(e)
Fuels	Ratings	Units	Trial 1	Trial 2	Trial 3	Mean	Trial 1	Trial 2	Trial 3	Mean	Trial 1	Trial 2	Trial 3	Trial 4	Mean	Fe_3O_4	Fe_2O_3
J23	Stable	0	2.0	-0.2	3.7	1.8	2.0	2.0	3.7	2.6	2.5	2.0	3.9	0.8	2.2	0.3	1.3
J29	Unstable	12	1.2	1.0	2.1	1.4	3.2	1.3	3.5	2.7	1.9	0.0	-0.2	0.5	0.1	-1.4	1.1
J34	Stable	8	3.0	3.0	2.8	2.9	4.8	5.2	5.2	5.1	11.1	3.7	3.8	2.5	3.3	5.5	16.9
J36	Unstable	17	3.1	3.7	2.3	3.0	3.8	3.2	3.3	3.4	10.1	3.2	1.2	1.5	2.0	1.8	5.1
J44	Stable	5	2.0	0.9	2.8	1.9	5.0	4.9	4.1	4.7	7.7	3.0	2.6	0.9	2.2	0.9	9.0
J47	Unstable	12	3.4	2.3	3.5	3.1	4.7	5.5	5.0	5.1	12.9	5.3	4.3	0.3	3.3	3.3	10.8
J78	Stable	2	1.2	1.2	2.1	1.5	3.4	3.2	15.6(f)	3.1	12.4	2.5	3.1	1.5	2.4	1.8	4.0
J68	Unstable	41	2.4	1.0	2.0	1.8	1.5	4.5	5.3	3.8	2.4	2.7	3.1	1.0	2.3	1.3	1.8

(a) As a result of 26 weeks, 110°F aging

(b) Containers sealed with rubber stoppers after fuels reached equilibrium with oven temperature

(c) Containers sealed with rubber at ambient temperature prior to oven heating

(d) Containers sealed without rubber at ambient temperature prior to oven heating

(e) Containers sealed without rubber after fuels reached equilibrium with oven temperature

(f) Doubtful data (not included in mean)

(g) For last three trials only

When oxidized iron metal rods were used as a possible catalyzing agent to accelerate fuel aging the first attempt (Trial I) showed a significant change for all fuels containing metal deactivator. Subsequent trials did not confirm these results. The means of the three tests (excluding the first trial) in the presence of oxidized iron rods also showed no significant effect on light transmittance deterioration as a result of accelerated aging. A single determination using finely divided black iron oxide (Fe_3O_4) powder in place of the oxidized iron rods also showed no effect resulting from aging which confirms the results found for three of the four trials with the oxidized rods. A single run using finely divided red iron oxide (Fe_2O_3) however, shows a significant effect for those fuels containing metal deactivator additive. Since this experiment confirms the first trial using the oxidized iron rods, it is possible that the oxidized rods used may have contained iron oxide in the Fe_2O_3 state. Additional tests using Fe_2O_3 powder are presently being made to confirm this finding. It is of particular interest that fuels containing metal deactivator are susceptible to light transmittance deterioration in the presence of a particular type of iron oxide. This clue may indicate a possible mechanism for degradation reactions occuring in ambient storage. Drums used for storage, when new, have a black Fe_3O_4 coating and even though the drums are sealed, the presence of small amounts of water and air in the fuel possibly could further oxidize (over prolonged periods) some of the Fe_3O_4 to Fe_2O_3. Circulation effects due to changes in day to night temperatures would not only help to oxidize the iron walls but would also aid in exposing susceptible molecules to these surfaces.

Although the data indicate that Fe_2O_3 is responsible for light transmittance deterioration, there is no indication that these reactions simulate the degradation reactions measured by the Coker or light transmittance changes. Accelerated aging in the presence of Fe_2O_3 as well as the other iron-type catalyst failed to separate the fuels as to the proper storage stability quality.

It should be remembered that the Coker data in these experiments were obtained on samples stored in a controlled 110°F hot room. Accordingly, circulation effects would be minimized and also possible oxidation and catalytic effects which are dependent on sufficient contact of the fuel with the surface. These observations suggest that accelerated aging in the presence of Fe_2O_3 catalyst (using differential light transmittance as a criterion) might show the proper separation of fuels if Coker data were available on fuels stored in ambient rather than controlled hot room storage.

E. Accelerated Aging in the Presence of Azodiisobutyronitrile (ADN)

The mechanisms of fuel deterioration leading to harmful deposits in heat exchangers, fuel nozzles, etc. are believed by many investigators to proceed via free radical formation. A typical, simplified mechanism based on this theory is represented by the following:

I $RH \longrightarrow R\cdot$ Initiation
II $R\cdot + O_2 \longrightarrow RO_2\cdot$ Free radical peroxidation
III $RO_2\cdot + RH \longrightarrow RO_2H + R\cdot$ Chain propagation
IV $RO_2H + [RH, RH(O)_x + RH(S)_x---] \longrightarrow$ Polymerization

In order to accelerate aging or more specifically to accelerate reactions which lead to deterioration as shown in Reaction IV, an attempt was made to increase the rate of Reaction I by adding a free radical initiator.

Since differential light transmittance was to be used to measure the extent of deterioration by measuring the change in the formation of light absorbers, it was recognized that ADN free radicals could react with any of the fuel components, free radicals, metal deactivators and inhibitors that are present to form possible light absorbing reaction products which would complicate interpretation.

To study the effect of free radical initiation, the same fuels used in the previous accelerated aging experiments (Table 4) were selected with the exception of fuels K20 and K43 which were not included because of depletion. Four ml of each fuel were combined with 0.5 ml of a saturated (ambient temperature) solution of azodiisobutyronitrile (ADN) in isooctane. (ADN was obtained from Matheson, Coleman, Bell, Code AX1825). The fuel containing the ADN solution was filtered through 0.45 micron/Millipore paper and placed in 15 ml Pyrex containers and a polished, soft iron rod (1 1/2 x 1/8 inch) was also placed into each tube. Matching samples containing an iron rod and 0.5 ml of the isooctane solvent only were also prepared for comparison. All samples were heated simultaneously in a Hotpack oven and heated at 110 ±5°F for a total of 109.5 hours. To obtain rate data, the samples were removed periodically from the oven and triplicate light transmittance readings at 350 millimicrons wave length over a two hour period were obtained using a Bausch and Lomb Spectronic 20 spectrophotometer. While the fuels were out of the oven precautions were taken to eliminate possible photochemical reactions resulting from exposure to light.

Data on the rate of light transmittance loss with and without ADN present are shown in Table 9 and the rate curves are shown in Figures 12, 13 and 14. The rate curves in general show that all fuels (with and without ADN present) deteriorate linearly after about 15 hours heating. Some fuels show slightly different rates during the first 15 hours, however no attempt was made to attach any significance to the variation in rates during this period. It is apparent from the curves that the presence of ADN accelerates light transmittance deterioration for all fuels. The slight increases in rates shown, however, for fuels J23 and J47 are not considered significant.

Since these results indicate that the presence of ADN materially affected the rates of light transmittance deterioration for most of the fuels the data were analyzed to determine if the effect was due to a reaction between ADN and the additives that were present. Figure 15 shows the relationship of light transmittance losses for the fuels with and without the presence of ADN. If ADN had no effect in any of the fuels the data points would be expected to follow the "line of equality". The points appear to define a line significantly different from the line of equality. Since these fuels represent a variety of substrates, additives and contaminants, these data indicate that the light transmittance deterioration is not due to selective reactions of ADN with any particular additive or type of additive.

TABLE 9

RATE OF LIGHT TRANSMITTANCE LOSS FOR VARIOUS JP-6 TYPE FUELS WITH AND WITHOUT ADN AT 110°F IN THE PRESENCE OF IRON METAL

	Addition of Isooctane Only						
	Percent L.T. After Following Time, Hrs					Δ L.T. After	Rate x 100
Fuels	0	2.5	16.5	38.5	109.5	109.5 Hrs	Δ L.T./Hr(b)
J23	67.4(a)	67.3	66.8	66.8	63.7	3.7	3.4
J29	69.1(a)	68.5	66.7	65.3	64.5	4.6	2.4
J34	77.8		76.8		70.5	7.3	6.6
J36	56.7		55.5		46.3	10.4	12.9
J44	82.1(a)	81.8	79.3	77.5	73.0(a)	9.1	6.8(c)
J47	62.0		59.7		52.7	9.3	8.9
J78	69.5(a)	69.3	68.7	67.3	64.5	5.0	5.1
J68	50.8		49.0		41.3	9.5	8.6
	Addition of Isooctane + ADN						
J23	66.7(a)	66.5	65.0	65.2	63.0	3.7	3.7
J29	69.1(a)	68.5	66.3	65.5	62.8	6.3	4.2
J34	75.5		74.3		54.7	20.8	21.2
J36	55.8		52.0		30.0	25.8	23.5
J44	82.1(a)	82.2	77.5	74.0	63.2	18.9	10.3
J47	60.0		57.8		48.2	11.8	10.4
J78	69.0	68.7	65.7	63.3	58.2	10.8	7.8
J68	52.8		48.2		28.7	24.1	22.2

(a) Extrapolated

(b) Obtained from linear segment of smooth rate curves between 16.5 and 109.5 hours

(c) Obtained from linear segment of rate curves between 16.5 and 38.5 hours

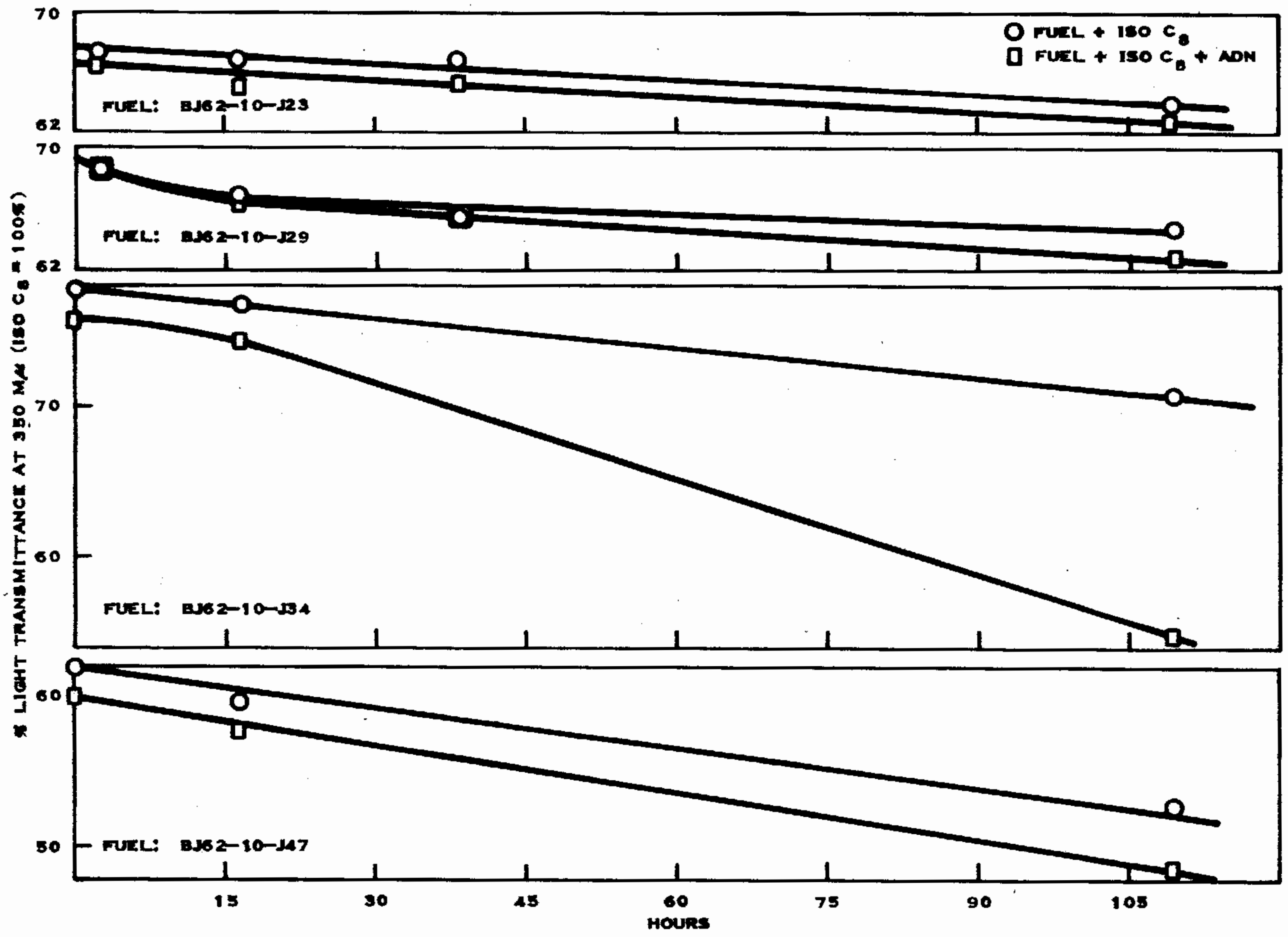


FIGURE 12 EFFECT OF ADN (AZODIISOBUTYRONITRILE) ON RATE OF CHANGE IN LIGHT TRANSMITTANCE AT 110°F

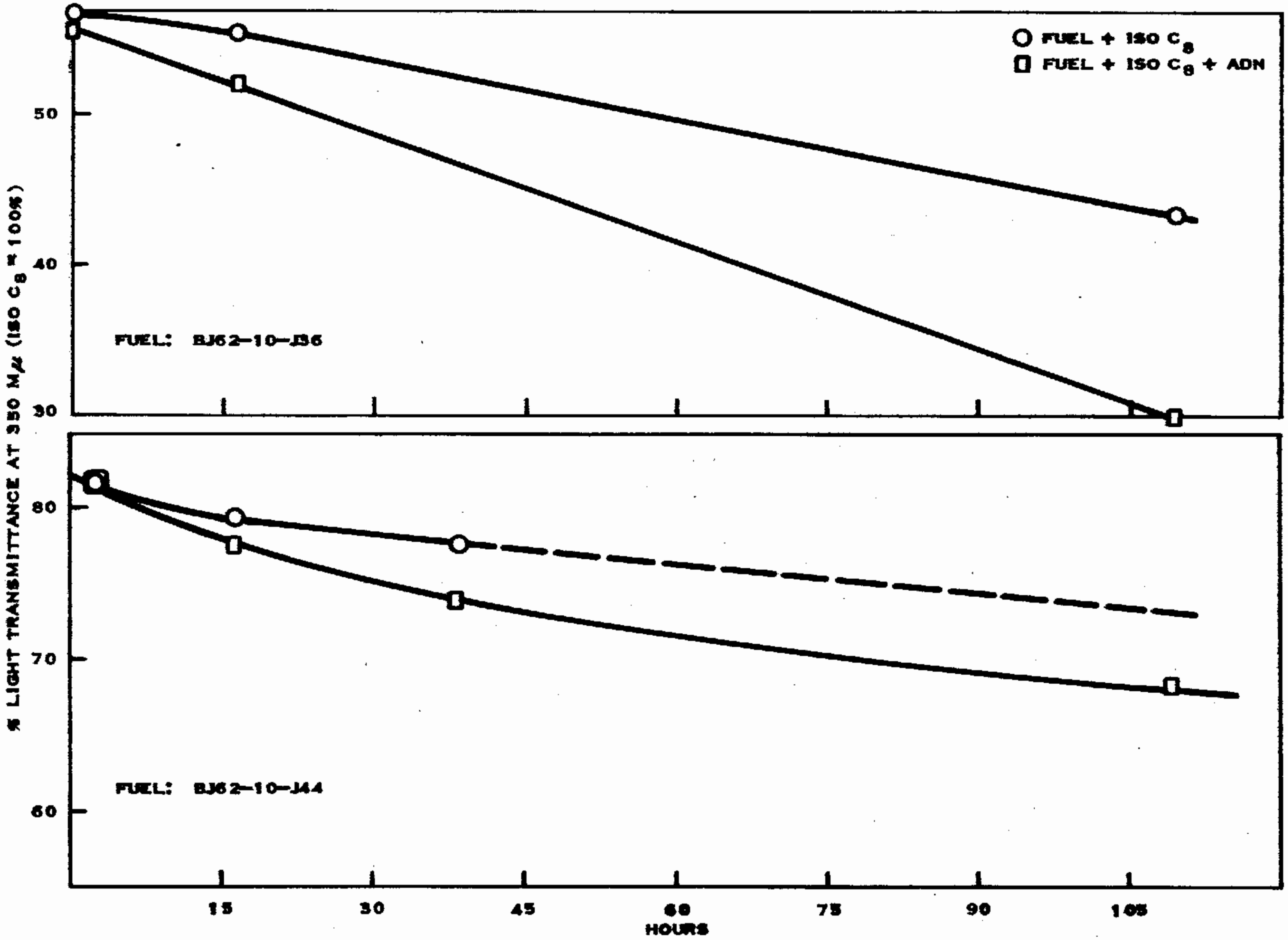


FIGURE 13 EFFECT OF ADN (AZODIISOBUTYRONITRILE) ON RATE OF CHANGE IN LIGHT TRANSMITTANCE AT 110°F

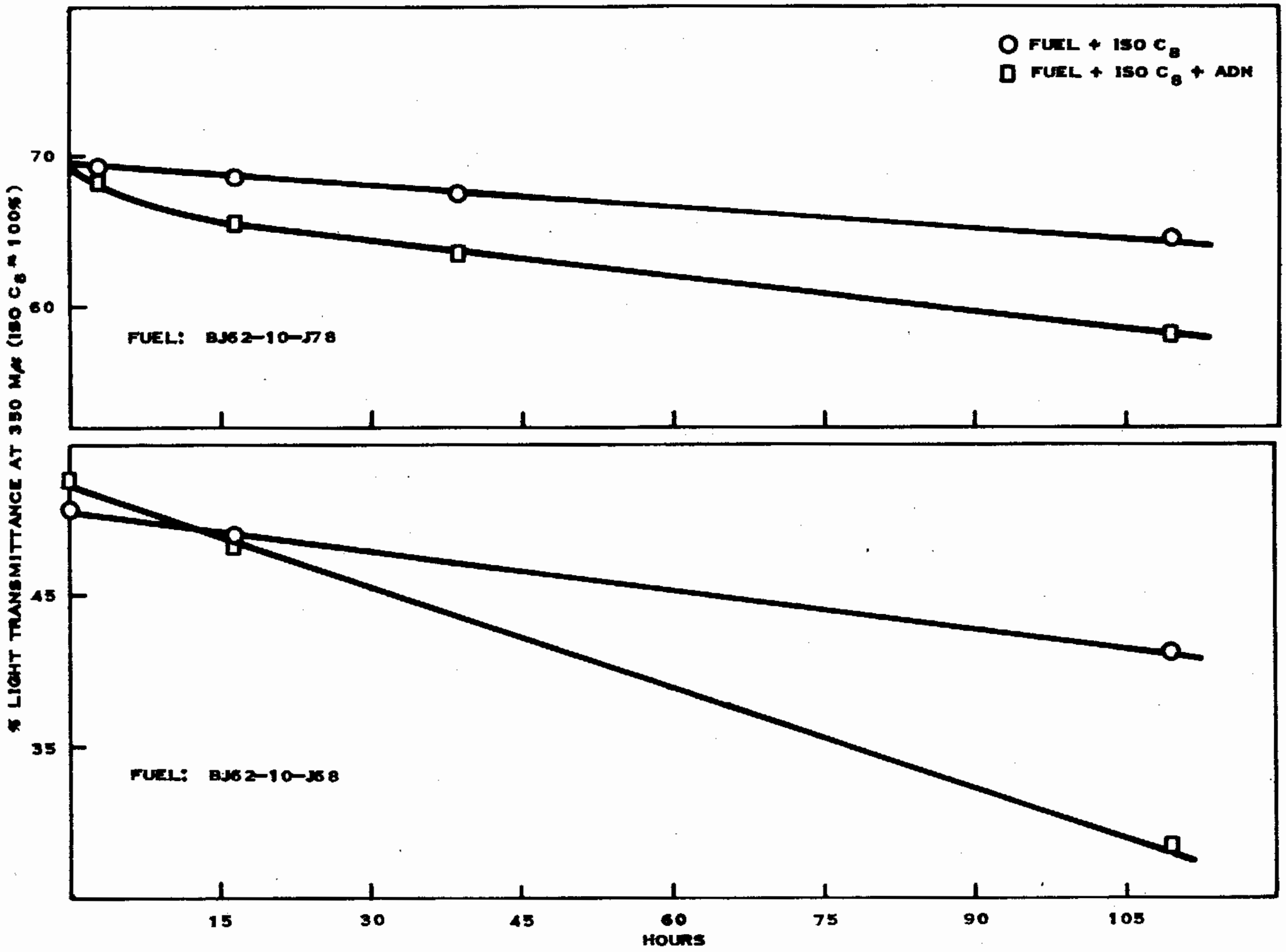


FIGURE 14, EFFECT OF ADN (AZODIISOBUTYRONITRILE) ON RATE OF CHANGE IN LIGHT TRANSMITTANCE AT 110°F

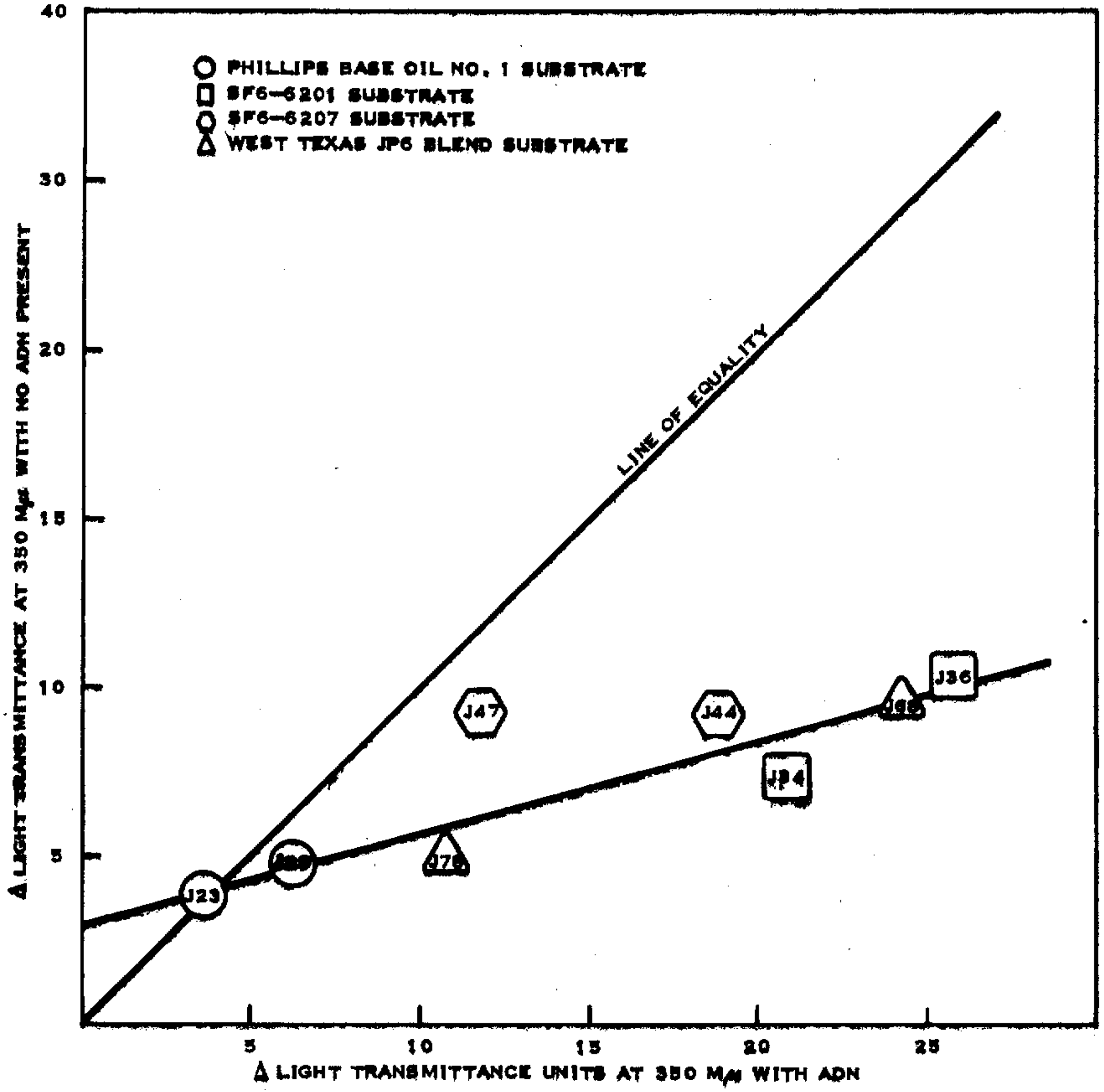


FIGURE 15 EFFECT OF AZODIISOBUTYRONITRILE (ADN) ON DIFFERENTIAL LIGHT TRANSMITTANCE RESULTING FROM THERMAL STRESSING AT 110°F, FOR 109.5 HOURS IN PRESENCE OF IRON METAL

Comparison of fuel deterioration as measured by differential light transmittance using ADN with ARTD-TSR Coker ratings and light transmittance changes are made in Table 10. Data are shown with and without ADN with respect to (1) the overall loss in light transmittance after 109.5 hours heating at 110°F, (2) the rate of loss, (3) the difference between rate losses (rate with ADN - rate without ADN) and (4) the percentage increase in the rate of loss due to the influence of ADN.

The following observations are indicated by the data:

1. None of the accelerated aging methods based on differential light transmittance correlates satisfactorily all types of fuels with the known storage behavior as measured by changes in Coker performance and changes in long term light transmittancy at 350 mμ wave length. For example, some stable substrates show greater overall losses in light transmittance (resulting from accelerated aging) than for other substrates shown to be unstable. This is also shown for rating parameters based on rates of deterioration. This may indicate the impossibility of developing a test method that will predict storage stability quality for all types of fuels containing a variety of additives.

2. The overall loss in light transmittance resulting from heating at 110°F for 109.5 hours without ADN present shows a separation in the right order within each substrate but the magnitude of the differences can only be considered significant for the West Texas Blends (J78, J68).

3. The overall loss in light transmittance in the presence of ADN shows a reversal for one pair of fuels (J44, J47). The magnitude of the separation for two of the remaining three pairs J34, J36, and J78, J68) is significant and the one remaining pair (J23, J29) is not significant.

4. A consistent separation for all pairs of fuels is observed based on the rates of deterioration in the presence of ADN, however, the magnitudes of these differences for all but one pair (J78, J68) is so slight that little significance can be attached to the separation. Possibly a different selection of variables such as greater concentration of ADN, other aging temperatures and/or aging times would show a greater magnitude of separation.

5. No consistent separation in the proper order or magnitude can be observed using the difference between the rate of deterioration in the presence of ADN and the rate of deterioration without ADN.

6. No consistent separation in the proper order or magnitude can be observed using percentage increase in the rate of deterioration resulting from the presence of ADN.

The procedure of heating fuels for 109.5 hours at 110°F without ADN present appears to be as effective as any procedure studied. Such a procedure is particularly interesting because it is not an "accelerated" technique with respect to temperature. (The reference storage stability data were obtained on samples also heated at 110°F but for 26 weeks.) Since Δ light transmittance data at 110°F for

TABLE 10

EFFECTIVENESS OF DIFFERENTIAL LIGHT TRANSMITTANCE RESULTING FROM THERMAL STRESSING AT 180°F WITH AND WITHOUT AZODIISOBUTYRONITRILE (ADN) AS ACCELERATED AGING PROCEDURES

Fuels	Storage Stability Qual.(a) ARTD-TSR Fuel Coker Ratings	Storage Stability Qual.(a) Δ (350 mμ) L.T. Units	Δ Light Transmittance Units @ 350 mμ Resulting From Heating 109.5 Hrs At 110°F		Rate of Light Transmittance Loss, Δ L.T. Units Per Hour x 100(b)		(Rate of Light Transmittance Loss With ADN Minus Rate Without ADN) x 100	% Increase In Rates Resulting From ADN
			No ADN	With ADN	No ADN	With ADN		
J23	Stable	0	3.7	3.7	3.4	3.7	0.3	11
J29	Unstable	12	4.6	6.3	2.4	4.2	1.8	75
J34	Stable	8	7.3	20.8	6.6	21.2	14.6	220
J36	Unstable	17	10.4	25.8	12.9	23.5	10.6	80
J44	Stable	5	9.1(c)	18.9	6.8(d)	10.3	3.5	50
J47	Unstable	12	9.3	11.8	8.9	10.4	1.5	20
J78	Stable	2	5.0	10.8	5.1	7.8	2.7	50
J68	Unstable	41	9.5	24.1	8.6	22.2	13.6	160

(a) Storage stability after 26 weeks, 110°F heating

(b) Rates based on linear segment of rate curves between 16.5 and 109.5 hours

(c) Extrapolated value

(d) Obtained from linear segment of rate curves between 16.5 and 38.5 hours these fuels are available (Table 3) for the 26 week storage period, a comparison with the loss in light transmittance for 109.5 hours is shown in Table 11.

TABLE 11

SHORT TERM VERSUS LONG TERM AGING AT 110°F AS MEASURED BY DIFFERENTIAL LIGHT TRANSMITTANCE

Fuels	ARTD-TSR Fuel Coker Ratings	Δ Light Transmittance @ 350 mμ After 110°F Aging 26 Weeks[(b)]	109.5 Hours[(a)]
J23	Stable	0	3.7
J29	Unstable	12	4.6
J34	Stable	8	7.3
J36	Unstable	17	10.4
J44	Stable	5	9.1
J47	Unstable	12	9.3
J78	Stable	2	5.0
J68	Unstable	41	9.5

(a) Samples contained approximately 12.5 percent isooctane

(b) Data taken from curves shown in Air Force Report ASD-TR-61-238 Part III.

One discrepancy is apparent in this comparison. For example some of the storage stable fuels show <u>greater</u> losses in light transmittance after 109.5 hours than was experienced after 26 weeks. The reason for this discrepancy is not known. However, there are two differences in these tests: (1) The influence of isooctane on light transmittancy might vary from one fuel to another. (2) Since the mechanism of deterioration is not known with respect to time for the samples aged 26 weeks, and since ice-box storage (~40°F) has been found inadequate(6) to preserve the original freshness for certain fuels, the time intervals over which Δ light transmittance is measured may not be comparable with the interval over which the Coker data were obtained. Experiments are presently being conducted to determine if 0°F storage temperatures can maintain fuel freshness to alleviate this problem.

Although these data are preliminary and are difficult to interpret, they indicate the possibility of measuring storage stability quality by simply measuring the loss in light transmittance during the first few days of 110°F storage. As additional fuels and data become available, this possibility will be investigated.

IV. ENVIRONMENTAL FACTORS AFFECTING STORAGE STABILITY QUALITY

It has been recognized by the Air Force(7) that fuels can be supplied by contractors to meet the present thermal stability specifications for JP-6 fuels. However, after only three weeks of storage some fuels fail the Standard Fuel Coker test for thermal stability. Possible causes for storage instability have been attributed to contamination during handling and transfer, poor Coker reproducibility as well as fuel deterioration. Two objectives of the present program are (1) to study the effects of various storage temperatures from ambient to 300°F (2) to determine the effect of dissolved oxygen concentration (<1 ppm to ~ 80 ppm) at the various storage temperatures on storage stability quality. The data obtained from this study will permit an evaluation of the rate of deterioration as a function of temperature. In addition an attempt will be made to (1) establish activation energies from Arrhenius plots necessary for various types of fuels to deteriorate and (2) obtain some insight on the mechanism of deterioration as influenced particularly by temperature and dissolved oxygen. From such a study it is hoped that corrective measures might be found to prevent or combat storage instability.

A. Test Fuels

Test fuels have been selected to provide gross variations in hydrocarbon structure and thermal stability quality. These fuels are:

Storage Fuel No. 1. (BJ63-10-B75). Phillips Base Oil No. 1 is a kerosine boiling range fraction of HF Alkylate, isoparaffinic in structure and low in aromatics. This fuel contains no additives.

Storage Fuel No. 2. (BJ63-10-G74). CRC SST Rig Fuel No. 1 is an "average quality" commercial turbine fuel, ASTM Type A, supplied by Standard Oil Company of California. This fuel contains no additives.

Storage Fuel No. 3. (BJ64-10-G71). Texaco SO_2 extracted naphthenic kerosine. This fuel contains 5 lbs/1000 barrels 26B4M and 2 lbs/1000 barrels MD additives.

Storage Fuel No. 4. (BJ64-10-G107). Texaco SO_2 extracted paraffinic kerosine. This fuel contains 5 lbs/1000 barrels N,N'disecondary butyl paraphenylenediamine and 2 lbs/1000 barrels of MD additives.

Storage Fuel No. 5. (BJ64-10-G166). Hydrotreated West Texas kerosine supplied by Phillips. A portion of the fuel was collected from the refinery unit without exposure to the atmosphere (<1 ppm dissolved oxygen) and is being maintained under a nitrogen blanket. This fuel contains no additives.

These fuels have been secured and have been placed into storage. The procedure for handling fuels for the storage program including the method for removing dissolved oxygen from the fuels is as follows:

1. Each fuel sample supplied in 55-gallon drums was made uniform by connecting the drums in series and circulated by pumping until the fuels were "turned over" at least twice. After mixing, the fuels were transferred to new, clean, soft-rolled-iron 15-gallon drums (painted black). Fourteen gallons were charged to each drum and sealed. Samples not placed into ambient storage were placed in 40°F ice-box storage until needed.

2. Samples that are stored with dissolved oxygen removed were first transferred to 15-gallon stainless steel containers and dissolved oxygen was removed by the following method:

a. Purge sample for 30 minutes with prepurified nitrogen through a tube extending to the bottom of the container. Rate of nitrogen flow should be sufficient to give violent agitation of the sample.

b. Place container on a mechanical shaker and, after pressurizing to 6 psig with prepurified nitrogen, shake for 5 to 10 minutes.

c. With the shaker stopped connect the container to a vacuum source and reduce the pressure to 29^+ inches of mercury vacuum.

d. Shake the sample for 10 minutes and again evacuate to 29^+ inches of mercury vacuum. Repeat shaking and evacuating until there is no pressure buildup on shaking the evacuated sample.

e. Pressure the container to 6 psig with prepurified nitrogen and shake to saturate the fuel. Repeat if necessary.

f. Measure the dissolved oxygen content of the fuel and if it is <1 ppm the fuel is ready to transfer to a 15-gallon drum for storage.

g. For storing fuel with dissolved oxygen removed, the drum is equipped with an induction tube with a valve at the outer end, inserted through a cross and extending to the bottom of the drum. Also connected to the cross are a pressure gage and a valve to release or add nitrogen as a gas cap. In preparation for storing a sample in the drum it is purged with prepurified nitrogen through the induction tube and out the valve in the cross. In filling the drum from the stainless steel container fuel is forced into the drum through the induction tube and nitrogen released through the valve in the cross while holding a 3 psig back pressure on the drum.

h. Measure dissolved oxygen content of the fuel and if it remains <1 ppm the drum is ready for storage.

3. In order to determine the feasibility of producing fuels free of dissolved oxygen (<1 ppm) at the refinery stream, Storage Fuel No. 5 was secured from a West Texas Refinery without exposure to air. New, clean 15-gallon drums and fittings for the dissolved oxygen removed samples were supplied and samples

were collected in a nitrogen atmosphere. At the same time samples for storage with dissolved oxygen present were obtained and after shipment were prepared for storage as described in Section A.1. No problems were encountered in the sampling and delivery of the dissolved oxygen-free samples. All drums contained less than 1 ppm oxygen after delivery.

B. Storage Conditions

Storage conditions planned for this program include the following:

1. Ambient. Three drums of each fuel with dissolved oxygen and three with dissolved oxygen removed will be placed in open field storage. These drums will be stored one foot off the ground and positioned on end. This will provide samples for evaluation after approximately 3, 9 and 18 months storage.

2. Hot Room. Three drums of each fuel with dissolved oxygen and three with dissolved oxygen removed will be placed in constant temperature storage at 130°F in a hot room. Samples will be removed for testing after approximately 2, 6, and 12 weeks.

3. 212°F Storage. One drum of each fuel with and without dissolved oxygen will be aged at 212°F for approximately 1, 3, and 6 days.

4. 300°F Storage. Tentative plans are to age a drum of each fuel with and without dissolved oxygen removed at 300°F for approximately 1, 3 and 6 hours. However, drums of each fuel will be aged for 3 hours first and on the basis of the deterioration in threshold failure temperature the other two aging times may be lengthened or shortened.

Because the quantities of the storage fuels are limited, it is important that the maximum storage period produce a definite failure in threshold failure temperature. Equally important is the possibility of failing to recognize severe deterioration occurring sooner than expected. To aid in establishing when samples should be removed from storage for Coker tests, per cent light transmittance will be obtained periodically during the storage periods to monitor changes in storage stability quality.

C. Thermal Stability Evaluations With the Supersonic Fuel (SSF) Coker Configuration

Because several fuels in this program have threshold failure temperatures above the limits of the Standard ASTM-CRC Fuel Coker, it was requested by the Air Force that another Coker configuration be used for this study that would correlate with the CRC Research Fuel Coker. At a meeting of the CRC-Aviation Modified Coker Panel of the Group on High Temperature Stability for High Performance Aircraft in New York City on April 27, 1964 it was agreed that a modified Coker configuration consisting of a 14 second fuel residence time in the preheater, using a Northern Ordnance pump and a once through fuel system would be acceptable for this study and was designated the Supersonic Fuel (SSF) Coker. A schematic diagram of this configuration is shown in Figure 16.

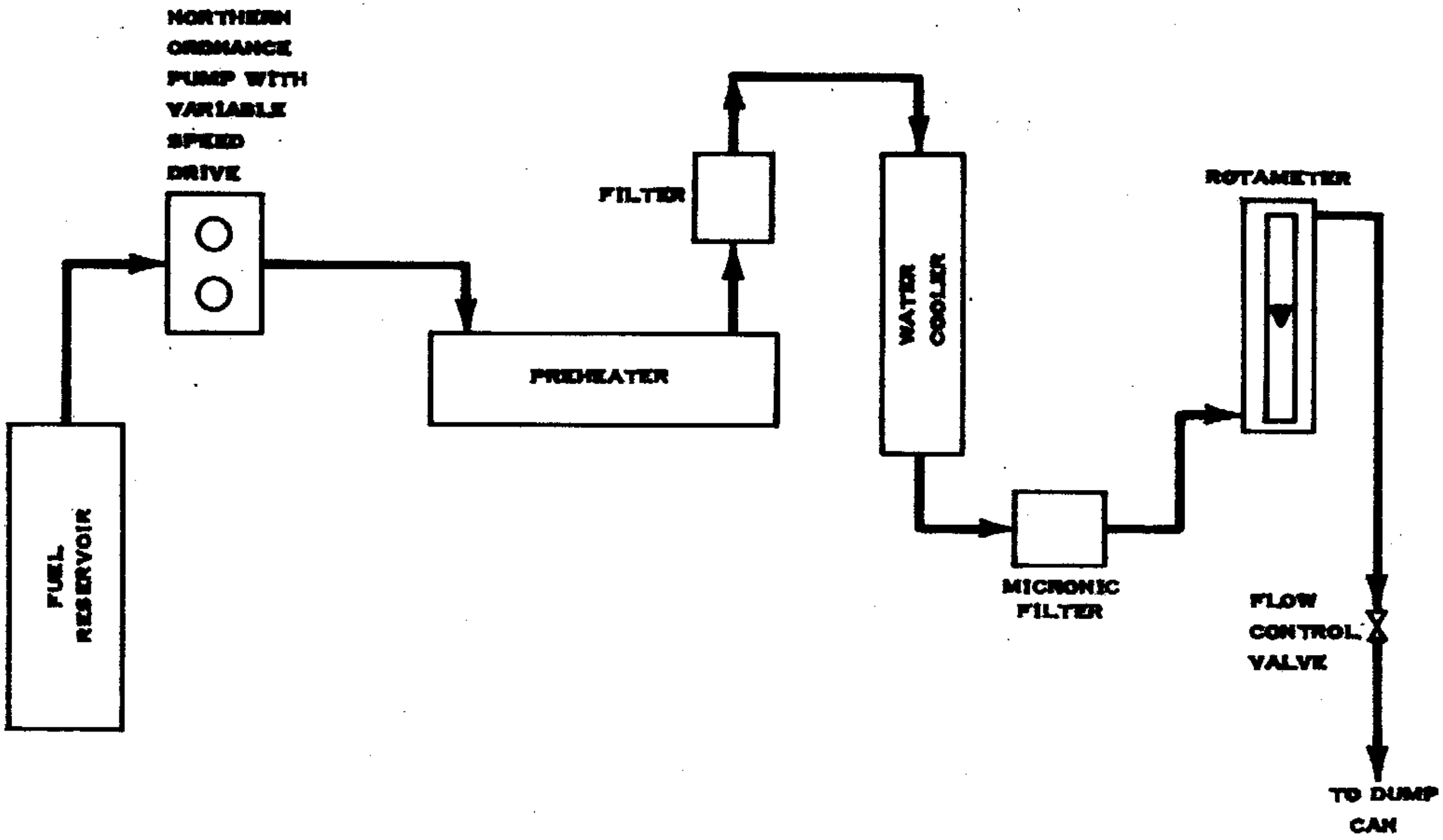


FIGURE 16 SCHEMATIC OF SUPERSONIC FUEL COKER FUEL SYSTEM

Evaluation of storage effects will be based on changes in threshold failure temperatures as shown by the SSF Coker and Phillips Modified 5-ml Bomb.

Fuel samples with dissolved oxygen removed, which have been aged under a nitrogen atmosphere, will be maintained under a nitrogen atmosphere during evaluation. Sufficient fuel will be abailable to provide up to five Coker tests on each sample for establishing threshold failure temperature. Dissolved oxygen determinations will be made toward the end of each coker run (280 min) before and after the fuel goes through the Coker. Percent light transmittance at 350 $m\mu$ wave length will be obtained on all Coker tests at 0, 150, and 280 minutes during the run. Various other physical and chemical properties will be acquired as discussed below.

A minimum of four quarts of each fuel for each storage period and temperature will be retained at deep freeze (0°F) temperatures in brown Pyrex bottles for use in the development of a small scale, accelerated test procedure.

D. Initial Characterization-Physical and Chemical Properties

In addition to the Coker tests, dissolved oxygen consumption and Δ light transmittance through Coker chemical properties have been obtained to characterize the fuels initially. Table 12 lists the test methods and Table 13 shows the data that have been obtained on the fuels as they were placed into storage.

After each storage period certain physical and chemical properties (marked with an asterisk in Table 13) will be obtained to ascertain if any qualitative or quantitative relationship exists between changes in these properties and storage instability.

E. Initial Characterization--SSF Coker Data

Threshold failure temperature data as determined by the SSF Coker for the five storage fuels as placed into ambient storage are tabulated in Table 14 and shown graphically in Figures 17 and 18. Changes in threshold failure temperatures resulting from the storage conditions discussed above will be used as a basis for determining storage stability quality.

F. Oxygen Consumption in the SSF Coker

Table 15 shows the data for dissolved oxygen consumption for all the storage fuels as characterized initially with the SSF Coker. Figure 19 shows the relationship of dissolved oxygen consumption through the coker as a function of the maximum (filter block) recorded temperature. In general, oxygen consumption increases with temperature.

The relationship of the percent of the dissolved oxygen that is consumed at the threshold failure temperature for the storage fuels is shown in Table 16.

TABLE 12

PHYSICAL AND CHEMICAL PROPERTIES--TESTS METHODS

Tests	Test Methods
Distillation, °F	ASTM D-86
Smoke point, Mm	ASTM D1322-59T
API Gravity @ 60°F	ASTM 287-55
Existent gum, Mg/100 ml	ASTM D381-58T
Total potential gum, Mg/100 ml	ASTM D873-57T
Insoluble potential gum, Mg/100 ml	ASTM D873-57T
Lamp sulfur (Wickbold), ppm	ASTM D1266
Mercaptan sulfur, ppm	$Hg(ClO_4)_2$ Titration
Freezing point, °F	ASTM D1477-57T
Net heating value, BTU/lb	Fed Std No. 791-2502
Kinematic viscosity, CS @ -40°F	ASTM D445-53T
Aromatics, Vol % (FIA)	ASTM D1319-58T
Olefins, bromine no., Vol %	Coulometric Method
Corrosion, copper strip	ASTM D130-56
Water reaction	ASTM D1094-57
Aniline point, °F	ASTM D611-55T
Neutralization No., Mg KOH/gram	ASTM D664-58
Flash point, °F	ASTM D93-58T
Total naphthalenes, Wt %	Ultraviolet spectrophotometry
Indenes, ppm	Anal. Chem. 21, 1528 (1949)
Pyrrole nitrogen, ppm	Anal. Chem. 30, 1528 (1958)
Basic nitrogen, ppm	Phillips Method 142-57R
Total nitrogen, ppm	Anal. Chem. 30, 1528 (1958)
Trace copper, ppb	Phillips Method NR-60R
Soluble iron, ppm	Phillips Method OG-61R
Soluble lead, ppb	Phillips Method 100-58R
Water content, ppm	Karl Fisher
Phenols, ppm	Ind. Engr. Chem. Anal. Ed. 18, 103(1946)
Peroxides, ppm	Phillips Method 133-57R
Dissolved oxygen, ppm	Phillips Chromatographic Method RK-63R
Total oxygen, Wt %	Direct Combustion and Adsorption
Hydrogen content, Wt %	Direct Combustion and Adsorption
Saybolt color	ASTM D156-53T
% Light transmittance @ 350 mμ (iso C_8 = 100%)	Bausch & Lomb Spectronic 20 spectrophotometer
Threshold failure temperature, °F	Phillips Modified 5-ml Bomb and SSF Coker

TABLE 13

PHYSICAL AND CHEMICAL PROPERTIES OF JET FUELS FOR STORAGE PROGRAM-INITIAL CHARACTERIZATION

Storage Fuel No.	1	2	3	4	5
*Distillation, °F IBP	362	332	361	381	356
10%	372	361	382	400	386
50%	394	402	420	418	422
90%	480	464	463	456	474
EP	552	508	512	502	511
Residue, Vol %	2.0	1.0	0.5	1.0	1.0
Dist. Loss, Vol %	0.0	0.0	0.0	0.0	0.0
Smoke point, mm	41.0	22.8	21.8	36.4	28.6
API Gravity @ 60°F	52.5	43.9	36.7	46.7	44.0
*Existent gum, mg/100 ml	1.1	0.0	0.0	0.0	0.2
*Total potential gum, mg/100 ml	6.6	0.2	7.0	0.7	3.8
*Insoluble potential gum, mg/100 ml	1.1	0.2	0.3	0.5	0.0
Lamp sulfur, ppm	3	820	47	28	10
Mercaptan sulfur, ppm	<2	4	<2	<2	<2
Freezing point, °F	-78	-58	-100	-72	-46
Net heating value, Btu/lb	18,950	18,550	17,500	18,700	18,550
Kinematic viscosity, CS @ -40°F	21.34	10.14	21.33	14.28	13.21
*Aromatics, vol % (FIA)	3.4	13.5	2.3	1.8	14.5
*Olefins, vol %	1.79	<0.10	0.41	0.12	0.21
Corrosion, copper strip	1A	1A	1A	1A	1A
Water reaction	1	1	0	0	1
*Neutralization No., mg KOH/gram	0.05	0.07	<0.05	0.05	<0.05
Aniline point, °F	189.2	143.3	143.2	165.5	148.3
Flash point, °F	144	130	146	160	146

* These properties will also be determined after each storage period.

TABLE 13 (Continued)

Storage Fuel No.	1	2	3	4	5
*Total naphthalenes, wt %	< 1	2.0	< 1	< 1	2.0
*Indenes, ppm	< 5	< 5	< 5	< 5	< 5
*Pyrole nitrogen, ppm	0.10	0.30	< 0.01	0.02	0.15
*Basic nitrogen, ppm	< 1.0	2.3	1.1	< 1.0	2.0
*Total nitrogen, ppm	< 1	2	5	4	< 1
*Trace copper, ppb	< 10	21	< 10	18	< 10
*Soluble iron, ppm	< 1	< 1	< 1	< 1	< 1
Soluble lead, ppb	7	10	16	19	13
Water content, ppm	20	23	17	10	40
*Phenols, ppm	< 2	18	< 2	< 2	< 2
*Peroxides, ppm	< 2	< 2	< 2	2	< 2
*Dissolved oxygen, ppm	74	59	53	64	62
*Total oxygen, wt %	0.079	0.098	0.120	0.210	
Hydrogen content, wt %	15.1	14.0	13.8	14.2	13.9
*Saybolt color	+27	+18	+28	+29	+30
*% Light transmittance @ 350 mμ (iso C_8 = 100%)	63.4	98.0	93.6	97.3	98.9
*Threshold failure temperature, °F (Phillips Modified 5-ml Bomb)	626	395	558	566	466
*Threshold failure temperature, °F (SS Fuel Coker)	625	332	712	692	425

* These properties will also be determined after each storage period.

TABLE 14

SS FUEL COKER DATA FOR JET FUEL STORAGE PROGRAM-INITIAL CHARACTERIZATION (FUEL FLOW RATE: 2.5 Lbs/Hr)

S. F. Fuel	Date Run	Temperatures, °F Pre-Heater	Temperatures, °F Filter	Filter Δ Pressure "Hg	Filter Δ Pressure Min.	Preheater Deposit Color Ratings Unwiped	Preheater Deposit Color Ratings Wiped
No. 1	4-9-64	525	625	0.00	300	1111111222222	1111111111111
	4-10-64	575	675	0.00	300	1111111222222	1111111111111
	4+13-64	600	700	0.00	300	1111111222222	1111111111111
	4-14-64	650	750	0.00	300	1111111233552	1111111113432
	4-15-64	625	725	0.00	300	1111111222332	1111111111222
No. 2	3-24-64	375	475	25.0	79.0	1111111112232	0000000001130
	3-25-64	350	450	25.0	224	1111111111332	1111111111443
	3-26-64	325	425	0.00	300	1111111111111	1111111111111
	3-31-64	325	425	0.00	300	1111111111111	1111111111111
	4-1-64	350	450	25.0	157	1111111111111	1111111111111
	4-2-64	350	450	25.0	205	1111111111111	1111111111111
No. 3	4-16-64	600	700	0.15	300	1111112222222	0000000000000
	4-17-64	650	750	0.05	300	1111112222222	1111111111111
	4-20-64	700	800	0.00	300	1111122222332	0000001111111
	4-21-64	725	825	0.00	300	1111112222333	0000011111111
	4-22-64	775	875	0.10	300	0000222222226	0000001111226
	4-23-64	750	850	0.05	300	0111222242227	0000111111227
	5-4-64	675	775	0.05	300	1111111112222	1111111112111
	5-5-64	700	800	0.00	300	1111111122222	0011111111111
	5-6-64	725	825	0.00	300	1111111223443	0001111112222
No. 4	4-24-64	650	750	0.00	300	1111111112221	1111111111111
	4-27-64	700	800	0.00	300	0000011123332	0000011111111
	4-28-64	750	850	0.00	300	0000111112224	0111122242224
	4-29-64	725	825	0.00	300	0111122242224	0001111122222
	4-30-64	675	775	0.00	300	0111111112221	0111111111111
	5-1-64	700	800	0.00	300	0001111114314	0001111112111
	5-7-64	675	775	0.00	300	1111111222221	0001111111111
No. 5	5-8-64	375	475	0.60	300	1111111111122	0011111111111
	5-11-64	425	525	0.30	300	1111111122444	1111111111443
	5-12-64	400	500	0.20	300	1111111112222	0000111111111
	5-13-64	400	500	0.20	300	1111111122222	0001111111111
	5-14-64	425	525	0.00	300	1111111111222	0000111111111
	5-15-64	450	550	0.05	300	1122222222333	0000011121111
	5-18-64	425	525	0.15	300	1111111113444	0000111111111
	5-19-64	450	550	0.10	300	1111111234444	0000111111112
	5-20-64	475	575	0.0	300	1222221115111	0000001115111

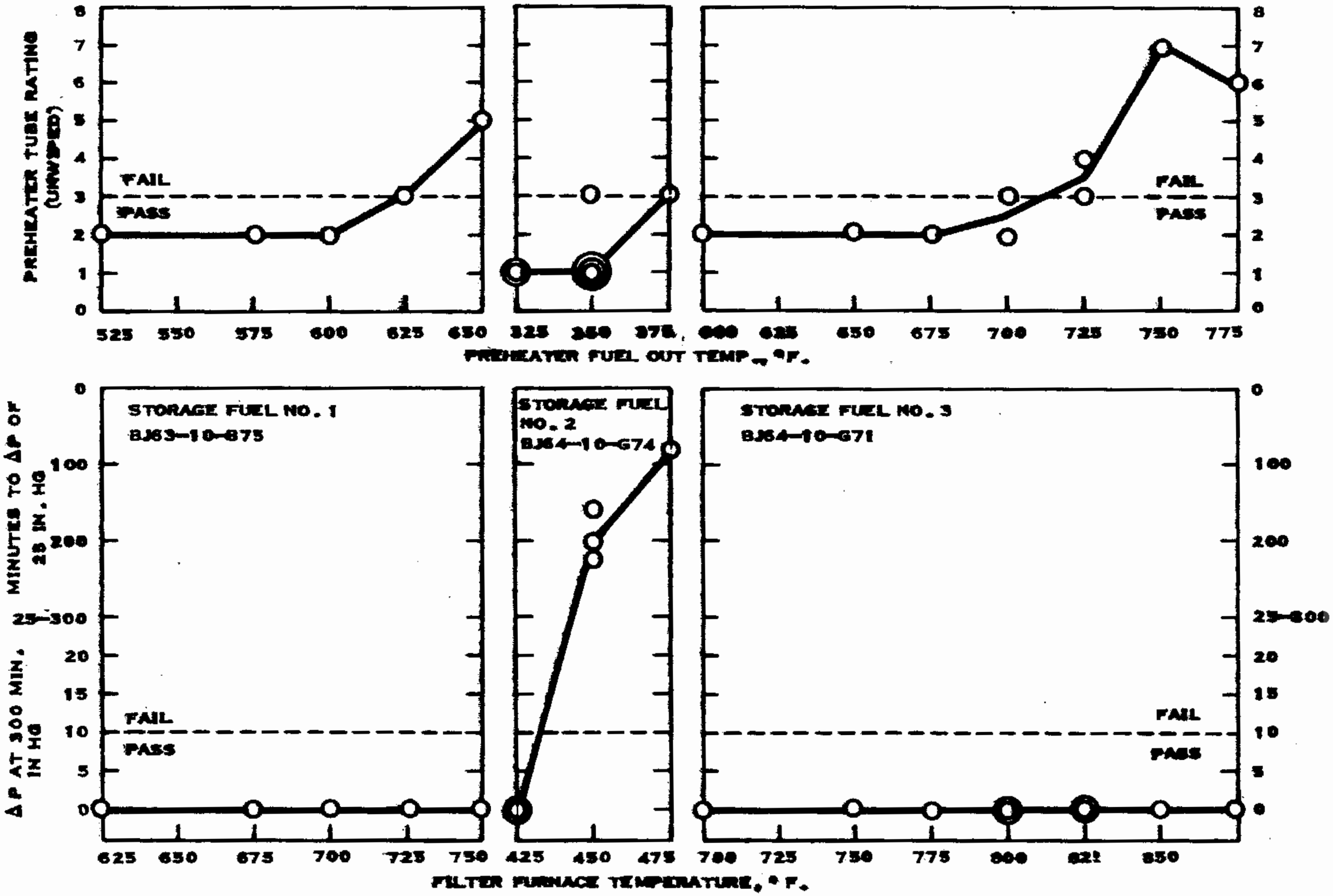


FIGURE 17 THERMAL STABILITY OF STORAGE FUELS 1, 2, 3, AS DETERMINED BY SSF COKER—INITIAL CHARACTERIZATION

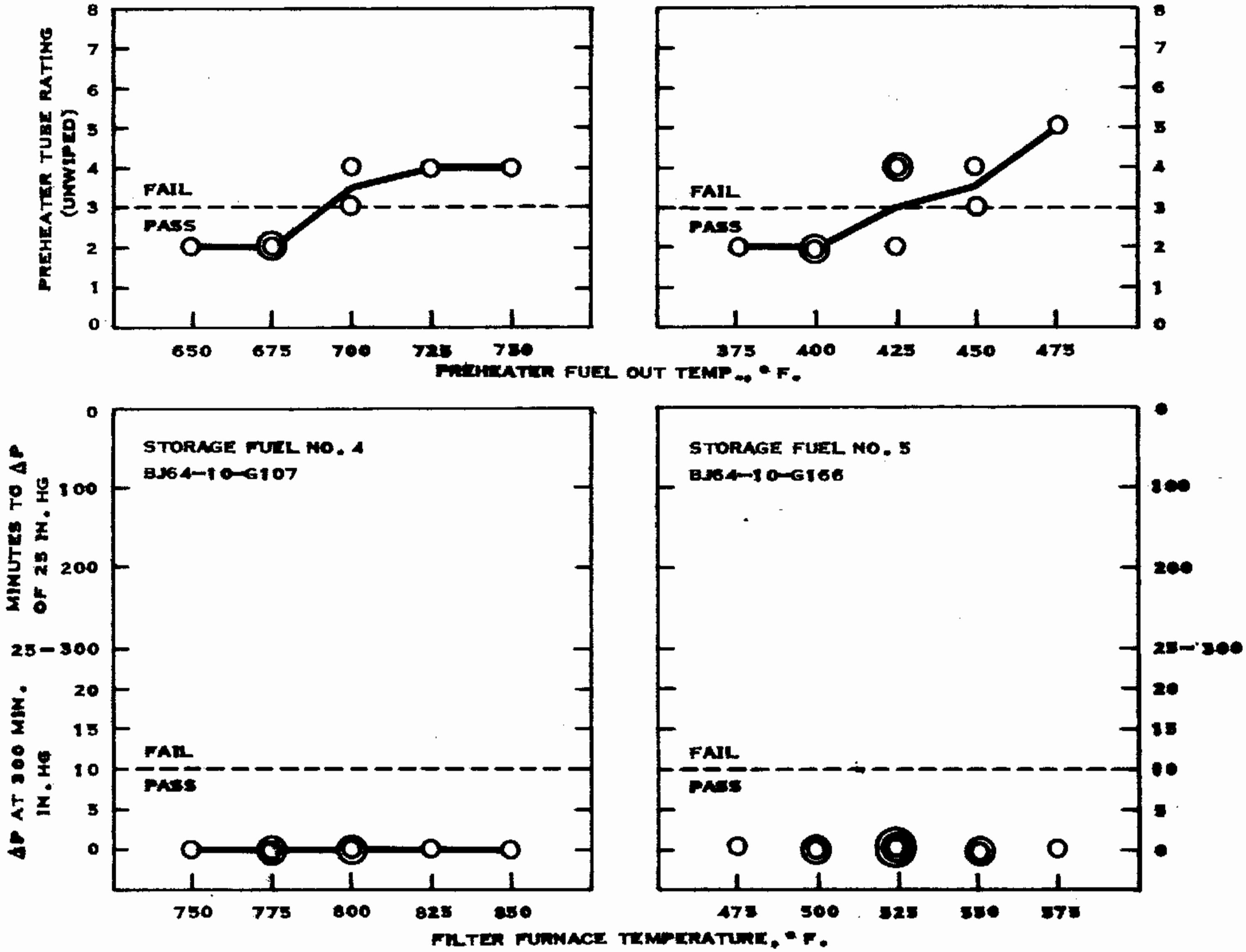


FIGURE 18 THERMAL STABILITY OF STORAGE FUELS 3, 4 AS DETERMINED BY SSF COKER—INITIAL CHARACTERIZATION

TABLE 15

OXYGEN CONSUMPTION AND Δ LIGHT TRANSMITTANCE THROUGH SSF COKER

Storage Fuel	Run Date	Filter Temp., °F	Dissolved O_2 Thru Coker, ppm				% Light Transmittance At 350 mμ					
			Before	After	Δ	Percent Consumed	Into Coker	Coker Out 150 min	Δ	280 min	Δ	% Δ After 280 min
No. 1	4-9-64	625	74.7	9.0	65.7	88.0	65	59	6	60	5	7.7
	4-10-64	675	70.0	3.6	66.4	94.9	63	61	2	61	2	3.2
	4-13-64	700	77.3	4.3	73.0	94.4	63	60	3	61	2	3.2
	4-15-64	725	74.0	7.1	66.9	90.4	64	59	5	59	5	7.8
	4-14-64	750	80.4	4.1	76.3	94.9	62	59	3	60	2	3.2
No. 2	3-26-64	425	64.5	54.5	10.0	15.5	98	93	5	94	4	4.1
	3-31-64	425	55.2	38.3	16.9	30.6	98	92	6	92	6	6.1
	3-25-64	450	62.0	53.1	8.9	14.4	98	86	12	92	6	6.1
	4-1-64	450	57.0	47.2	9.8	17.2	98	85	13	98	0	0
	4-2-64	450	56.7	47.4	9.3	16.4	98	84	14	98	1	1.0
	3-24-64	475	45.0(a)	32.0(a)	13.0(a)	28.9(a)	98	89	9	91	7	7.1
No. 3	4-16-64	700	48.3	5.9	42.4	87.8	94	92	2	90	4	4.3
	4-17-64	750	48.6	3.3	45.3	93.2	90(a)	87	3(a)	90	0(a)	0
	5-4-64	775	56.3	3.7	52.6	93.4	94	92	2	90	4	4.3
	4-20-64	800	50.0	1.8	48.2	96.4	92	89	3	91	1	1.1
	5-5-64	800	57.6	2.4	55.2	95.8	94	91	3	92	2	2.1
	4-21-64	825	58.5	3.2	55.3	94.5	93	90	3	92	1	1.1
	5-6-64	825	54.7	3.0	51.7	94.5	-	-	-	-	-	-
	4-23-64	850	49.4	5.7	44.7	90.5	95	90	5	92	3	3.2
	4-22-64	875	52.1	3.2	48.9	93.8	93	91	2	89	3	3.2

(a) Doubtful data

(Continued)

TABLE 15 (Continued)

Storage Fuel	Run Date	Filter Temp., °F	Dissolved 02 Thru Coker, ppm				% Light Transmittance At 350 mμ					
						Percent	Into	Coker Out				% Δ After
			Before	After	Δ	Consumed	Coker	150 min	Δ	280 min	Δ	280 min
No. 4	4-24-64	750	64.0	4.2	59.8	93.4	97	94	3	94	3	3.1
	4-30-64	775	63.0	2.4	60.6	96.2	97	95	2	96	1	1.0
	5-7-64	775	62.4	2.8	59.6	95.5	97	95	2	94	3	3.1
	4-27-64	800	66.7	3.3	63.4	95.1	96	95	1	96	0	0
	5-1-64	800	67.6	4.1	63.5	93.9	98	96	2	94	4	4.1
	4-29-64	825	64.6	2.7	61.9	95.8	98	95	3	94	4	4.1
	4-28-64	850	58.0	4.1	53.9	92.9	98	94	4	94	4	4.1
No. 5	5-8-64	475	65.7	6.3	59.4	90.4	103	97	6	97	6	5.8
	5-12-64	500	62.6	2.9	59.7	95.4	97	93	4	96	1	1.0
	5-13-64	500	60.2	2.9	57.3	95.2	98	96	2	97	1	1.0
	5-11-64	525	58.5	5.3	53.2	90.9	96	92	4	94	2	2.1
	5-14-64	525	58.6	2.8	55.8	95.2	100	94	6	96	4	4.0
	5-18-64	525	58.5	4.1	54.4	93.0	98	95	3	96	2	2.0
	5-15-64	550	-	-	-	-	99	93	6	95	4	4.0
	5-19-64	550	58.3	3.2	55.1	94.5	99	95	4	96	3	3.0
	5-20-64	575	59.3	2.8	56.5	95.3	100	94	6	95	5	5.0

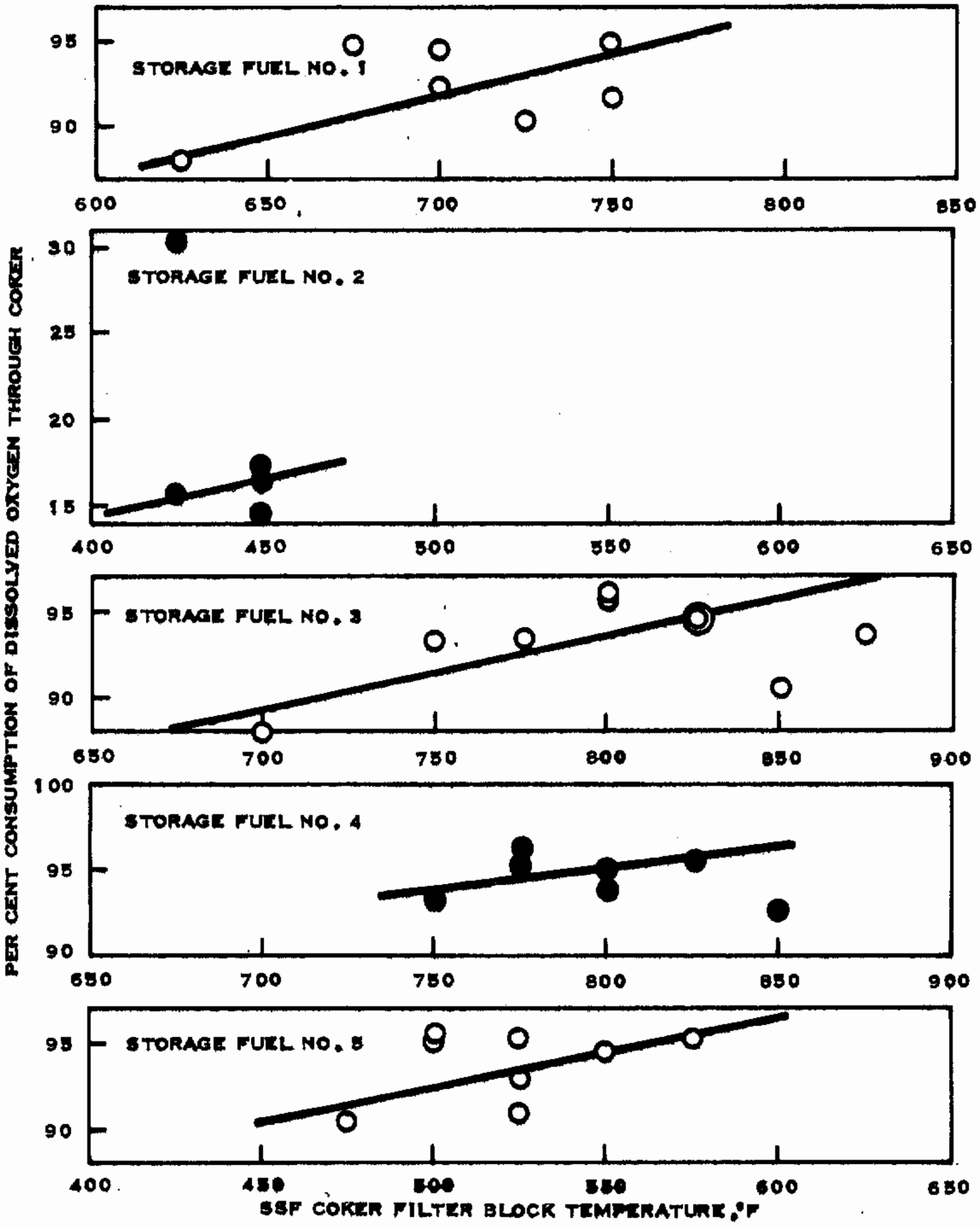


FIGURE 19 RELATIONSHIP OF SSF COKER FILTER BLOCK TEMPERATURE AND DISSOLVED OXYGEN CONSUMPTION

TABLE 16

DISSOLVED OXYGEN CONSUMPTION OF STORAGE FUELS THROUGH SSF COKER AT THEIR THRESHOLD FAILURE TEMPERATURES

Storage Fuel	Threshold Failure Temperature, °F	Percent Oxygen Consumption At Threshold Failure Temperature
2	332(a)	15.8
5	425	93.4
1	625	93.0
4	692	95.0
3	712	94.0

(a) Fails on basis of Δ filter pressure

These data indicate almost all of the dissolved oxygen in the various fuels is consumed at the threshold failure temperature and that the magnitude of oxygen consumption is not able to separate the fuels in the proper order of threshold failure temperatures. It is planned to continue collecting dissolved oxygen consumption to determine if changes in this property can be related to storage stability quality.

G. Light Transmittance Changes of Fuels Through the SSF Coker

Table 15 also shows the changes in light transmittance as fuels go through the Coker. A plot of these data shown in Figure 20 indicates that in general percent loss in light transmittance is constant over the temperature range that determines the threshold failure temperature. The magnitude of the losses is surprisingly small in view of the high temperatures involved and may reflect (1) the necessity of much longer residence time for fuels to show appreciable light transmittance deterioration, (2) a complete conversion of the low concentration unstable components to light absorbing compounds, or (3) a possible "saturation" of the fuels with the light absorbers after which further conversion results in deposits showing up on the preheater and/or filter.

The relationship of threshold failure temperature and percent light transmittance at 350 mμ wave length through the Coker (at the threshold failure temperature) as shown by the curves in Figure 20 is shown in Table 17.

TABLE 17

LIGHT TRANSMITTANCE LOSS THROUGH SSF COKER OF STORAGE FUELS AT THEIR THRESHOLD FAILURE TEMPERATURE

Storage Fuel	Threshold Failure Temperature F	Percent Light Transmittance Loss Through Coker at Threshold Failure Temperature
2	332 (a)	4
5	425	4
1	625	6
4	692	3
3	712	3

(a) Fails on basis of Δ filter pressure

The data indicate that there is no apparent relationship between threshold failure temperature and percent loss in light transmittance at the threshold failure temperature. Collection of this type of data will be continued, however, to determine if changes in light transmittance through the Coker can be related to storage stability quality.

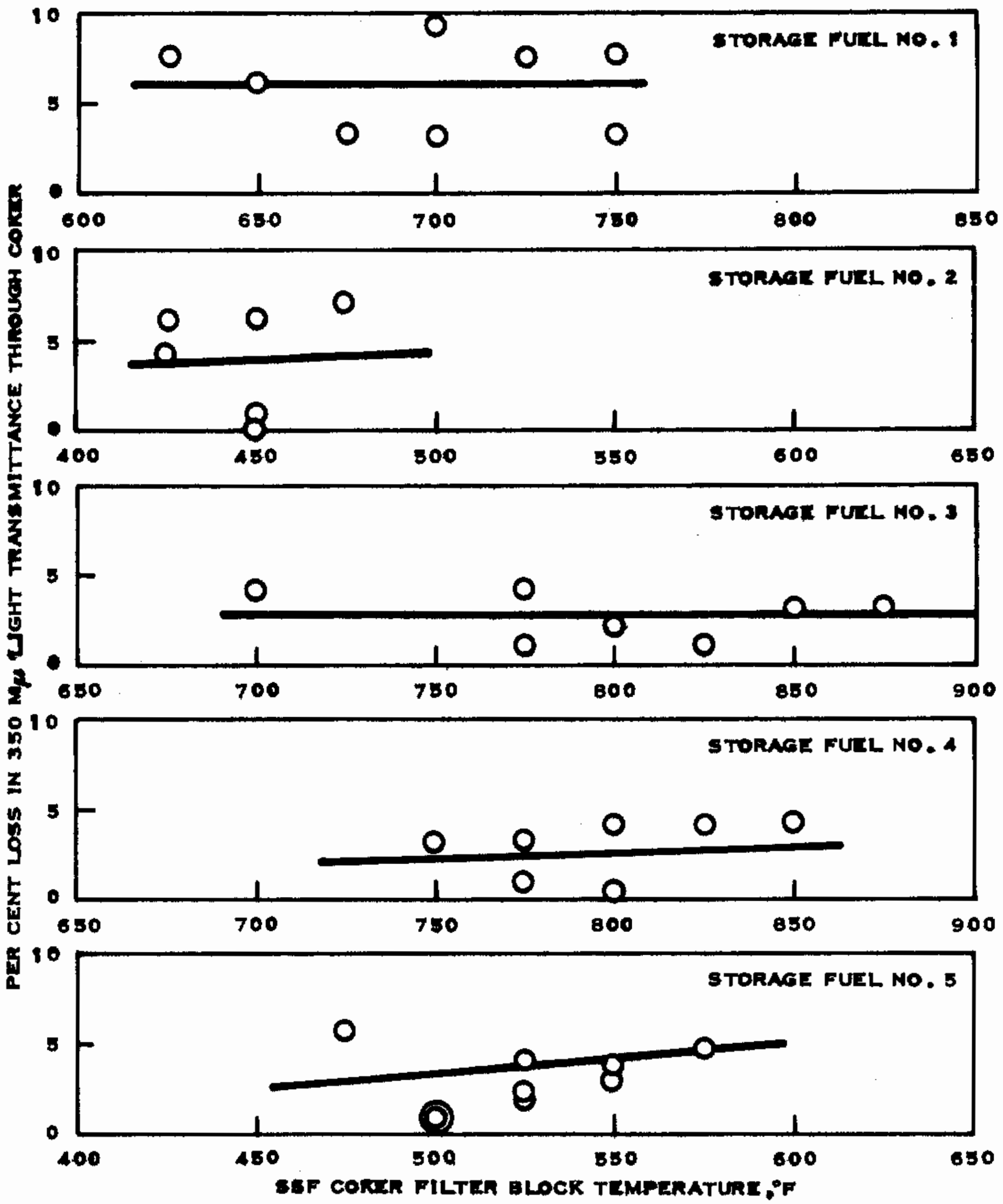


FIGURE 20 RELATIONSHIP OF SSF COKER FILTER BLOCK TEMPERATURE AND PER CENT LOSS IN LIGHT TRANSMITTANCE

V. CONCLUSIONS

A. Studies to Improve Precision of the Standard 5-ml Bomb Test Method

1. Modifications the Standard 5-ml Bomb thermal stability test procedure have resulted in improved precision. Most of the improvement is attributed to change in procedure for cleaning the bomb. A slight improvement may result from the minor modifications of cooling techniques and minimizing voltage input fluctuations to the furnace.

2. Proposed modifications of (1) changing from a variable heating rate to a constant heating rate, (2) using a higher resolving Beckman DB spectrophotometer in place of the Bausch and Lomb Spectronic 20 and (3) changing the wave length for measuring light transmittance from 350 to either 340 to 365 millimicrons showed no improvement in precision.

3. Statistical methods which have been developed for obtaining and interpreting the 5-ml Bomb data are essential to a realistic evaluation of the procedure. These include (1) obtaining nine points over a specified light-transmittance-loss range, (2) use of linear regression techniques for determining temperature for a given light-transmittance-loss and (3) defining the maximum standard deviation from regression (4.0) for a satisfactory set of data.

B. Ability of the Modified 5-ml Bomb Test Procedure to Recognize the Effects of Antioxidants

1. The standard 5-ml Bomb procedure could not detect the effect of antioxidants; however, in a program with one base fuel and three types of antioxidants the modified 5-ml Bomb procedure consistently showed that additives increased the temperature for 0, 15 and 25 light-transmittance-loss.

C. Repeatability of the Modified 5-ml Bomb Procedure

1. In a program with one base fuel and the same fuel with two concentrations of antioxidants repeatability of threshold failure temperature was good; however, when a wide variety of fuels and additives were tested overall repeatability was poorer.

2. Variances for each of the 18 fuels are non-homogeneous. While the repeatability for most of the fuels are good wide differences in repeat tests were found for some fuels.

3. The test severity at the 25 light-transmittance-loss level appears to have changed between the additive study and the repeatability program.

D. Correlation With ASTM-CRC Fuel Coker and Modified 5-ml Bomb

1. Relationships were established between the Modified 5-ml Bomb at 10, 15 and 25 light-transmittance-loss levels and the ASTM-CRC Coker for non-additive fuels. These relationships were significant at the 99+ percent confidence level.

2. A light-transmittance-loss level of 25 provided the best relationship between the Modified 5-ml Bomb and the Coker.

3. With fuels containing antioxidants the threshold failure temperature determined with the Modified 5-ml Bomb were higher than expected from the relationship between the 5-ml Bomb and the Coker on non-additive fuels. This indicates that the antioxidant effect on the 5-ml Bomb results were greater than for the Coker.

4. With fuels containing antioxidants plus metal deactivator threshold failure temperatures determined with the Modified 5-ml Bomb were lower than expected from the relationship between the 5-ml Bomb and the Coker on non-additive fuels. In one case it was found that the addition of a metal deactivator increased the threshold failure temperature as measured by the 5-ml Bomb.

E. Correlation With MINEX and Modified 5-ml Bomb

1. Relationships were established between the MINEX and the Modified 5-ml Bomb procedures at 10, 15 and 25 light-transmittance-loss levels. The relationships at 15 and 25 light-transmittance-loss levels were significant at the 99+ percent confidence level and at the 10 loss level it was significant at the 95 percent confidence level.

2. A light-transmittance-loss level of 25 provided the best relationship between the MINEX and the Modified 5-ml Bomb.

3. The MINEX and the Modified 5-ml Bomb recognized the presence of additives and additive types more nearly the same than did the 5-ml Bomb and the Coker.

F. Relationship Between SSF Coker and 5-ml Bomb

1. A linear relationship was found for three non-additive fuels in SSF Coker and the 5-ml Bomb.

2. Fuels containing metal deactivator in combination with antioxidants were recognized by the SSF Coker in the same manner as with the ASTM-CRC Coker.

3. Comparing the relationships of the ASTM-CRC Fuel Coker and the SSF Coker with 5-ml Bomb ratings the SSF Coker was more severe at low levels of thermal stability quality and milder at the upper limit of rating ability of the ASTM-CRC Coker.

G. With Respect To The Relationship of Changes in Light Transmittance To Changes in Fuel Coker Performance Resulting From 26 Weeks, 110°F Hot Room Storage, The Following Were Concluded:

1. No satisfactory correlation of differential light transmittance with differential Coker performance was found for thirty fuels representing four different bases each containing a variety of additives and variable additive concentrations.

2. No satisfactory correlation of differential light transmittance with differential Coker performance was found for any individual base fuel containing a variety of additives and variable additive concentrations.

3. A favorable relationship between differential light transmittance and differential Coker data within each set of base fuels was found to exist providing fuels were selected such that they differed, only slightly in additive concentrations and/or composition and providing that the differences between storage-stable and storage-unstable fuels as measured by the standard CRC Fuel Coker were significant (at 95 percent confidence).

H. Exploratory Studies to Find a Small Scale Procedure to Predict Storage Stability in Terms of Differential Light Transmittance Resulted in the Following:

1. Irradiation of nine fuels in the presence of no catalyst or iron metal with ultraviolet light at 180°F for 1.5 hours resulted in significant losses in light transmittance but the losses did not satisfactorily separate the fuels in the proper order of storage stability quality.

2. Thermal stressing of nine fuels in the presence of iron metal in the absence of light at 180°F for 48 hours showed significant but only slight light transmittance deterioration for seven fuels and moderately severe deterioration for two fuels. The changes in light transmittance did not satisfactorily separate the fuels in the proper order of storage stability quality.

3. Thermal stressing of eight fuels in the presence of 10 percent iso-octane at 110°F for 109.5 hours resulted in significant losses in light transmittance deterioration and these losses separated the fuels in the proper order of storage stability quality, but the magnitude of these changes could not be considered significant for two of the four pairs of fuels.

4. Thermal stressing of eight fuels in the presence of black Fe_3O_4 catalyst at 180°F for 48 hours showed no to only slightly significant light transmittance deterioration.

5. Thermal stressing of eight fuels in the presence of red Fe_2O_3 shows significant light transmittance deterioration for all fuels (a total of five) containing metal deactivator. The losses in light transmittance did not satisfactorily separate the fuels in the proper order of storage stability quality.

6. Using ADN (azodiisobutyronitrile) to chemically initiate free radical reactions to accelerate deterioration at 110°F for 109.5 hours resulted in marked increases in the overall loss in light transmittance as well as the rate of light transmittance deterioration. Using rates of deterioration as a parameter to measure storage quality resulted in the proper order of separation of all pairs of fuels, however, since the precision of this method is not known, it can not be stated to what extent the separations are significant.

VI. RECOMMENDATIONS

Future work on the 5-ml Bomb procedure should be directed in the following areas:

1. Improvement in Precision of Modified 5-ml Bomb

The temperature for a light-transmittance-loss of 25 in the Modified 5-ml Bomb has been established as giving the best relationship with other thermal stability test methods. Confidence limits of a regression are at a minimum at the mean of the data points. Concentrating the data in the 15 to 35 light-transmittance-loss range rather than the 3 to 35 range should improve the precision at the 25 light loss point. A number of fuels used in the repeatability program should be retested using the 15 to 35 range to determine if precision is improved.

2. Confirmation of Established Relationships

As thermal stability data on other non-additive fuels are obtained they should be used to test the validity of the relationship developed between the Modified 5-ml Bomb and other thermal stability test methods.

3. Correction Factors for Antioxidants and Metal Deactivator

By using a number of the non-additive fuels available from the repeatability program which cover the range of thermal stability quality of interest, a study should be made of the extent of increase or decrease of 5-ml Bomb ratings for various amounts and combinations of antioxidants and metal deactivator. From this study correction factors should be developed for correcting 5-ml Bomb data for additive content. These correction factors could then be verified by data on additive fuels now available.

4. Establishing Reproducibility

A variety of additive and non-additive fuels should be run by different operators and equipment.

Additional work to find a test procedure to predict storage stability quality by accelerating changes in ultraviolet light transmittancy should be continued. To reduce the problem associated with the doubtful precision of duplicate Coker tests at one set of conditions, fuels should be made available that have well defined threshold failure temperatures before and after storage. Samples of such fuels should be retained in cold storage (preferably 0°F) to minimize deterioration effects during the period these fuels are aging.

If fuels, retained at ice box or deep freeze temperatures, and threshold failure temperature data before and after storage can be made available (1) the rate of deterioration at 110°F for the first 200 hours should be determined with and without the presence of isooctane as measured by differential light transmittance, (2) the change in light transmittance characteristics in the presence of free radical initiators such as azodiisobutyronitrile and tertiary butylperoxide should be determined at temperatures between 110 and 180°F and (3) the effect of red Fe_2O_3--all should be studied further.

Contaminants known to degrade fuels such as olefins, diolefins, sulfur compounds and copper compounds should be added to fuels and the effects on light transmittance after storage at various temperatures should be measured.

In addition to differential light transmittance as a correlating parameter with differential Coker data, other methods should be studied such as (1) differential thermal analysis (DTA), (2) differential chromatographic analysis and (3) changes in other small scale thermal stability test methods such as Phillips Modified 5-ml Bomb.

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APPENDIX I

SMALL SCALE (5-ML BOMB) TEST
METHOD FOR THERMAL STABILITY
OF JET FUELS

APPENDIX I

SMALL-SCALE (5-ML BOMB) TEST METHOD FOR THERMAL STABILITY OF JET FUELS

(1) Objective:

To evaluate thermal stability of jet fuels and other petroleum distillates at temperatures from 300 to 650°F with 100-ml samples.

(2) Outline of Method:

A stainless steel bomb is charged with five ml of fuel which has been filtered through 0.45 micron porosity millipore filter paper and air-blown. The bomb is lowered into a 500 watt tubular electric furnace and power is applied at a wattage selected to produce some given temperature after 20 minutes time. The power is then turned off and the bomb removed and air-quenched down to room temperature. Fuel thermal instability is evaluated in terms of the losses in ability to transmit 0.35 micron wavelength ultraviolet light after heating over a range of temperatures. These losses in light transmittance are thought to result both from scattering and absorption by dispersed suspended particles and from moleculer absorption by fuel-soluble oxidation products having absorption bands at 0.35 micron wavelength.

(3) Apparatus:

(a) Stainless steel bomb as shown in Figures 21 and 22 is made from type 304 chrome-nickel alloy steel.

(b) Electrical muffle furnace and accessories, approximately 500 watt heating capacity with suitable controls for continuously varying power input. The interior of this furnace should be cylindrical in shape and of a size adequate to admit and fully enclose the bomb in an upright position (about one inch diameter and four inches deep). Figure 23 shows a furnace found to be suitable for this purpose.

(c) Spectrophotometer equipped to handle liquid samples. This instrument should be capable of measuring light transmittance at selected wave lengths with a repeatability of ± 1.0 light transmittance units. At a wave length setting of 350 millimicrons, a nominal band width of 20 millimicrons is acceptable. One such instrument is the Bausch and Lomb "Spectronic 20".

(d) Pressure gauge suitable for use with nitrogen and hydrocarbons. This should be of the indicating type, graduated in intervals of five psi per scale division with a maximum reading of 300 psi.

(e) Self-balancing potentiometer suitable for iron-constantan thermocouples and temperature levels between 300 and 700 F. This should be graduated in intervals of one degree per scale division, accurate within 0.1 per cent of the temperature indicated and having response characteristics of about five seconds full scale.

(f) Iron-constantan thermocouple, 22 gauge, closed end, 1/8 inch diameter, 12 inches long with wire, connectors, etc., for attachment to potentiometer.

(g) Millipore(1) laboratory filtration apparatus suitable for filtering approximately 1/2 pint samples of hydrocarbon distillates through 0.45 micron porosity paper elements.

(h) Laboratory stop watch or clock.

(i) Miscellaneous suitable stainless steel fittings, etc., for attaching pressure gauge, thermocouple and source of nitrogen to stem of bomb; ring stand and accessories for mounting bomb; laboratory table etc.

(4) Material:

(a) Oil-free nitrogen and air in cylinders.

(b) Cleaning solvents for bomb assembly, including detergent, scouring powder, hot water, acetone and metal polish.

(c) Millipore filters, 25 mm diameter, 0.45 micron pore size, type HA.

(d) Spectral grade isooctane for standardizing spectrophotometer.

(5) Preparation of Apparatus:

(a) Clean the bomb assembly thoroughly from all contamination left by previous tests by scrubbing with scouring powder and metal polish followed by washing with hot water-detergent. Then rinse with hot water followed by acetone and dry the components in an oven or by air-blast.

(6) Procedure:

(a) Install the thermocouple in the upper cap of the bomb so that the junction is 1/4 inch above the bottom.

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(1) Available from Millipore Filter Corp., Bedford, Mass.

(b) Measure out 100 ml of the fuel to be tested; filter through 0.45 micron pore size Millipore paper and air-saturate by bubbling oil-free bottled air through the sample for several minutes.

(c) Following aeration, add exactly five ml of fuel to a clean bomb assembly and seal, pressurize to 50 psig with oil-free bottled nitrogen and mount it in the furnace.

(d) Apply electrical power to the furnace at a wattage selected to produce a fuel temperature in the desired range after 20 minutes time. Start stop watch at same time power is turned on, record temperature after exactly 20 minutes, turn power off, raise the bomb assembly from the furnace and cool with a stream of compressed air.

(e) Using precalibrated test tubes, standardize the spectrometer at 100 on spectral grade isooctane, then measure light transmittance of samples of the test fuel taken before and after heating. Subtract to determine light transmittance loss due to heating.

(f) Repeat steps (c) through (e) using different furnace power inputs each time to obtain a series of seven to ten light-transmittance-loss values corresponding to different 20 minute temperature levels. Select power inputs to produce light transmittance losses ranging from minimum values of 5 to 10 at the lowest test temperature up to maximum values between 25 and 30 at the highest test temperature.

(g) Plot the data on linear graph paper as temperature versus light transmittance loss and draw a smooth curve through the points plotted.

(7) Report:

Either the light-transmittance-loss for a given temperature level, considering 25 as "failing" or, alternatively, the temperature level at which a light-transmittance-loss of 25 is reached and exceeded. The latter is to be preferred since it provides information as to the actual working temperature limit of the particular fuel.

(8) Precision:

Not known at this time.

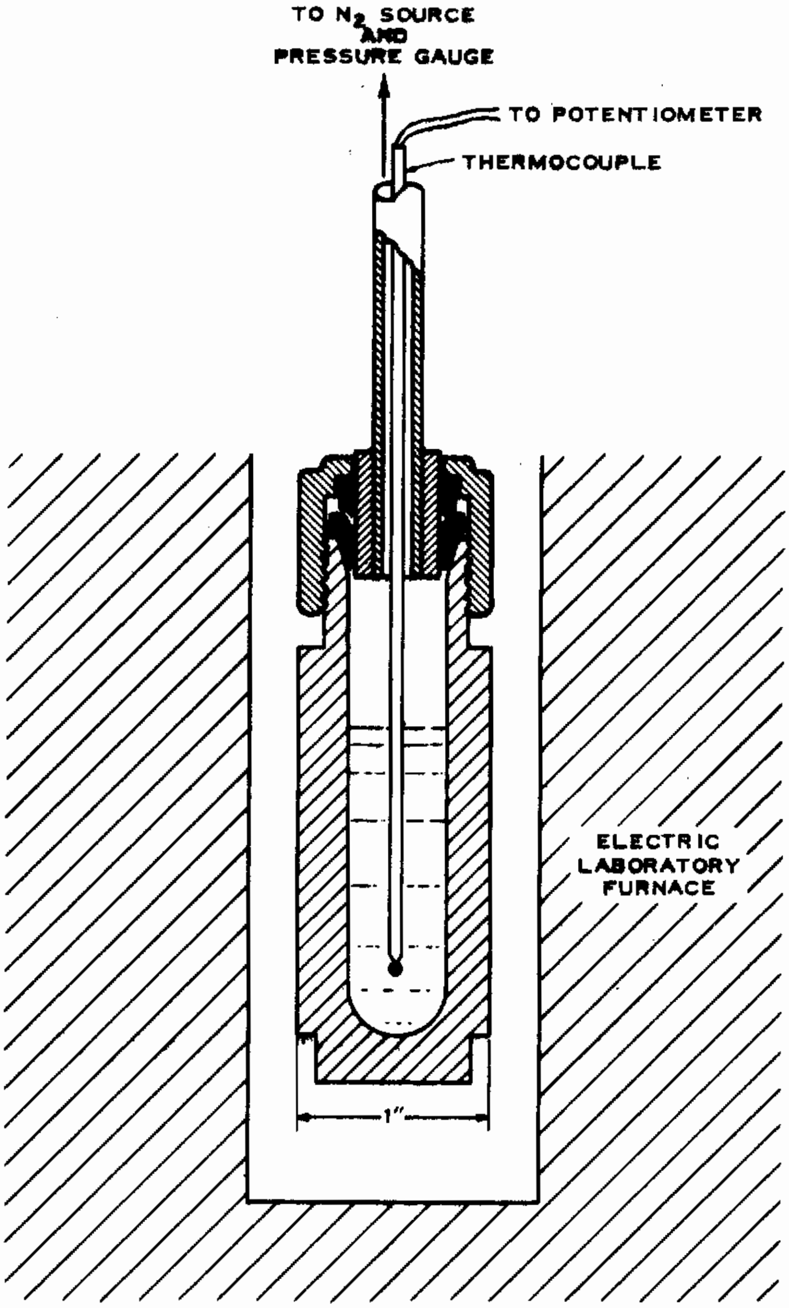
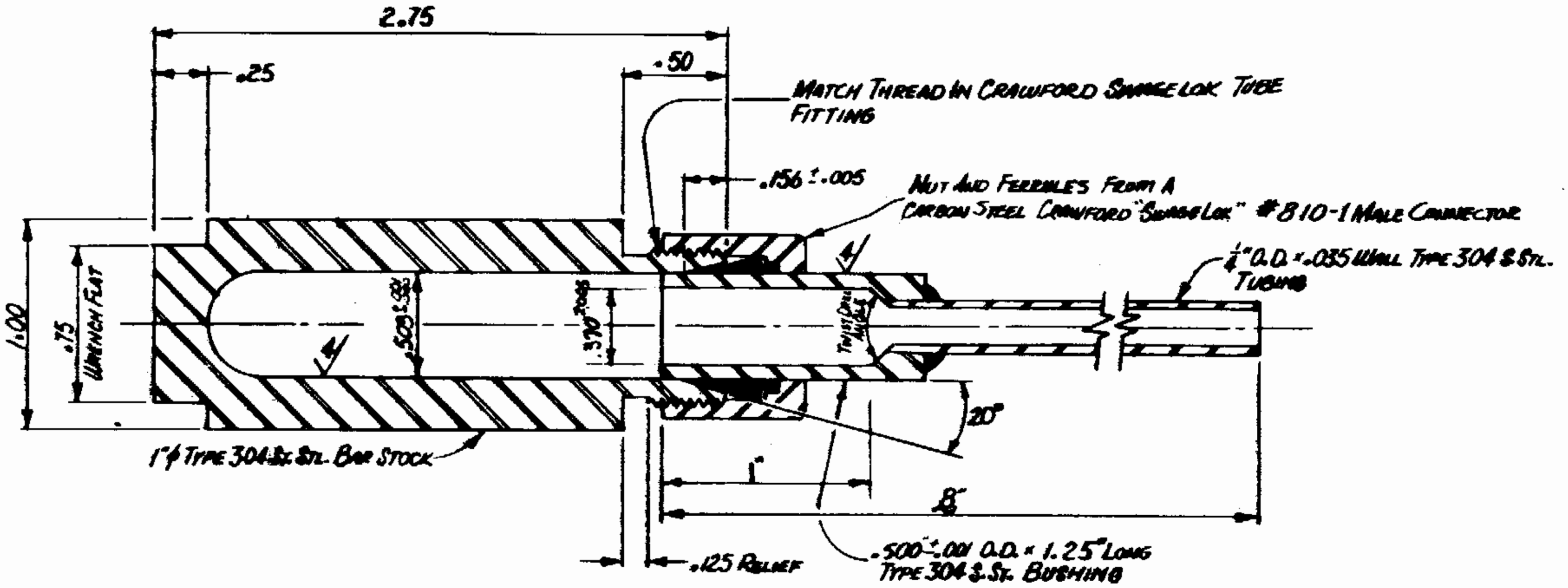


FIGURE 21 EXTERNALLY HEATED JET FUEL THERMAL STABILITY BOMB



MAT'L: AS NOTED

FIGURE 22 REVISED 5-ML THERMAL STABILITY BOMB

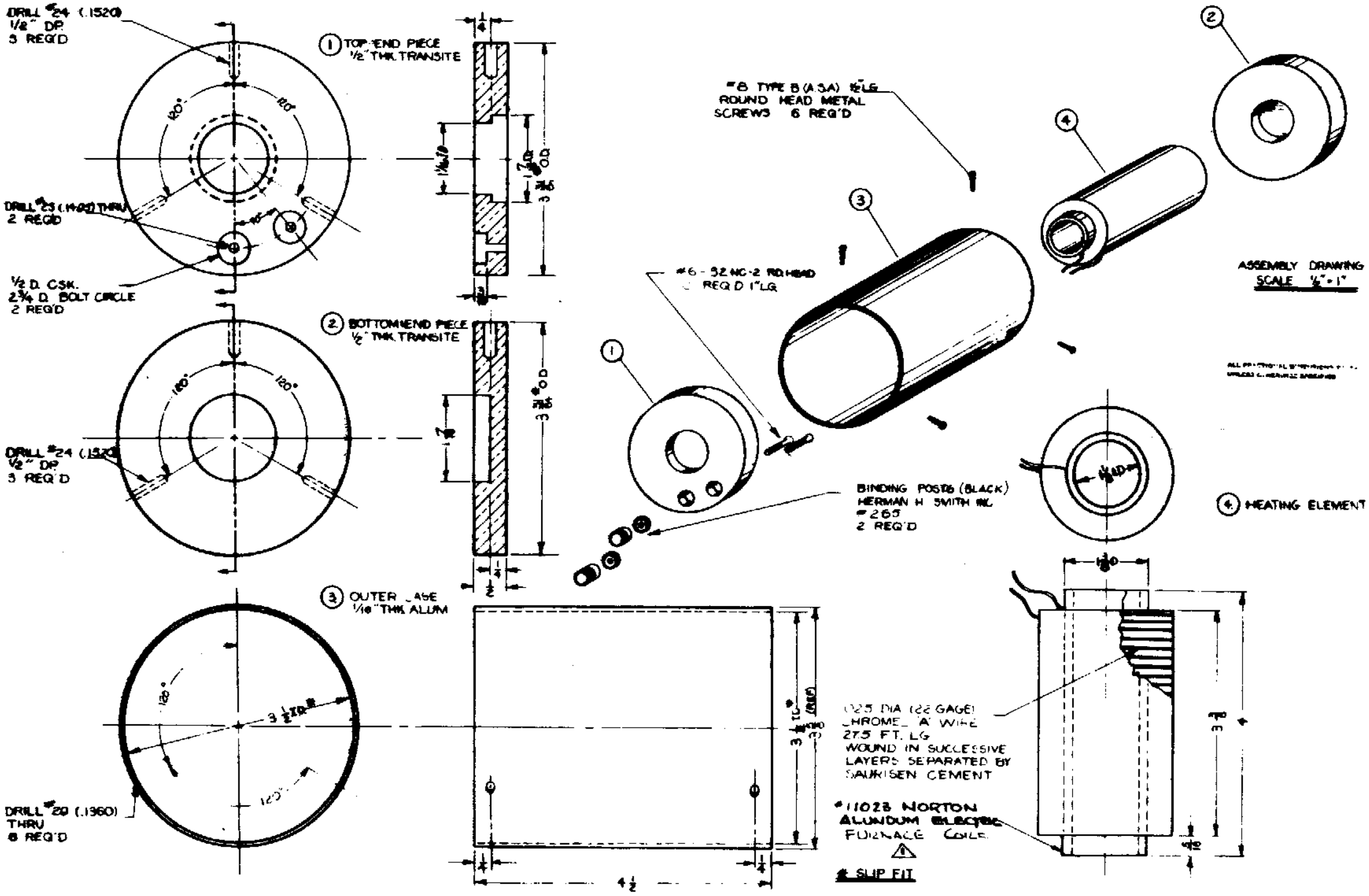


FIGURE 23 ASSEMBLY FOR 500 WATT 5-ML BOMB FURNACE

APPENDIX II

DETAILED TEST DATA AND STATISTICAL ANALYSES TO IMPROVE PRECISION OF ORIGINAL 5-ML BOMB THERMAL STABILITY TEST PROCEDURE

APPENDIX II

STUDIES TO IMPROVE PRECISION OF 5-ML BOMB TEST METHOD

From an examination of 5-ml Bomb data it was concluded that a statistical design for obtaining and handling the data would aid in interpretation and could improve repeatability. The following items were adopted.

(1) Statistical Methods For Obtaining And Interpreting Data

(a) Randomizing Experiments. The order of testing (temperatures) for a given fuel was randomized to eliminate bias and to permit an estimation of error.

(b) Defining the Shape of the Curve, viz, Linear versus Curvilinear. Accumulated 5-ml Bomb data indicated a linear relationship between light transmittance and temperature, therefore linear regression analysis was selected for uniformity of interpretation.

(c) Defining the Limits of Linear Regression. To standardize the procedure the following limits are defined: (a) obtain a minimum of 9 points in the 0 to 35 range of light-transmittance-loss, (b) if values are obtained in the 0 to 3 light-transmittance-loss range use only one value representing the highest temperature, and (c) select the remaining eight points such that two additional points define the lower range, three the upper range and the three remaining between the upper and lower range to confirm that the regression is linear.

(d) Sample Standard Deviation From Regression. The sample standard deviation from regression is a measure of how well the data define a line. As a result of subsequent data a maximum limit of 4 was selected for the sample standard deviation from regression. Any test with a sample standard deviation from regression greater than 4 was rejected.

(e) Multiple Light Transmittance Readings. Since light-transmittance-loss values are differences between measurements on fresh and heated fuels and errors in individual readings are additive or compensative, an increase in precision should result from multiple readings on each sample. A minimum of three measurements at 350 millimicron wave length over a two hour period was selected. Subsequent data confirmed an increase in repeatability of results.

(f) Predicted Temperature. With the regression equation developed from the data on a fuel, the temperature for any given light-transmittance-loss level was calculated for use as a rating criterion. For the subsequent investigation temperature for 0, 15 and 25 light-transmittance-loss levels were calculated.

(2) Repeatability of Bausch and Lomb Spectronic 20 Spectrophotometer

To establish the repeatability of light-transmittance readings obtained with the Spectronic 20 spectrophotometer used with the 5-ml Bomb procedure a series of seventeen fuels were evaluated. Quadruple light transmittance measurements were made over a period of two days by a single operator at 340, 350 and 365 millimicrons wave lengths. These data are shown in Table 26. Using the standard deviations for three programs which are shown in the first line of Table 18 confidence limits were calculated for single and triplicate measurements and differences in single and triplicate measurements. It can be observed that the use of triplicate determinations of light transmittance at 350 millimicrons reduced the confidence limits of a difference from ±3.1 for single determinations to ±1.8 for triplicate determinations. While quadruple determinations would have reduced the confidence interval further (±1.8 to ±1.6) the small additional improvement did not justify the additional testing.

TABLE 18

REPEATABILITY OF BAUSCH AND LOMB SPECTRONIC 20 SPECTROPHOTOMETER

	Millimicrons Wave Length		
	340	350	365
Standard deviation (a)	± 1.3	± 1.1	± 1.3
Confidence limits of a single determination (95%) (a)	± 2.6	± 2.2	± 2.6
Confidence limits of triplicate determinations (95%) (a)	± 1.5	± 1.3	± 1.5
Confidence limits of differences in single determinations (95%) (a)	± 3.7	± 3.1	± 3.7
Confidence limits of differences in triplicate determinations (95%) (a)	± 2.1	± 1.8	± 2.1

(a) Light transmittance units

(3) Comparison of Spectronic 20 and a Beckman DB Spectrophotometer

To determine if a higher resolving instrument would improve the repeatability of the light transmittance readings fresh and heated samples for three fuels were measured with a Beckman DB Spectrophotometer in addition to the Bausch and Lomb Spectronic 20. These data are shown in Table 25. Linear regression analyses were made on each fuel using data from each spectrophotometer(5). While the sample deviations from regression for the Beckman instrument were lower than for the Spectronic 20, the reduction was found to be not statistically significant at the 90 percent confidence level. Thus a change to the higher resolving Beckman instrument was not justified.

(4) Revision of the Standard Procedure of Heating and Cooling 5-ml Bomb Samples

Recognizing that variations in heating could contribute to the lack of precision, a reevaluation of the original heating and cooling procedures was made. The original procedure employs a variable heating rate to obtain end-temperatures after exactly 20 minutes. After heating, the sample is cooled by blowing with high velocity air. The major revision of the procedure consisted of a change from a variable heating rate to a constant heating rate which requires a variable time cycle to obtain the desired end-temperatures. The original cooling technique was changed to an ice-water quench (which has been used in all subsequent modification studies). All runs in this study employed a revised cleaning procedure which will be discussed later.

Other minor revisions which appeared necessary were (1) an elimination of convection currents through the furnace by "sealing" the bottom furnace opening; (2) a minimizing of voltage input fluctuations by installing a Sola constant voltage transformer; and (3) minimizing variations in oxygen concentration by aeration of individual test samples rather than aeration of the total sample. No attempt was made to establish which if any of these "minor" variations were pertinent to precision improvement and they were adopted merely as precautionary measures.

In addition to checking the effect of the heating rate, these revisions were used to evaluate the relative merits of (1) starting the heating cycle with the furnace at about ambient temperature (90°F) versus starting at 1000°F; (2) using a commercial 1000 Watt Hoskins furnace in place of the standard 500 Watt furnace. Data for all of these modifications are shown in Tables 27, 28 and 29. A summary of regression analysis of data from the 5-ml Bomb (variable heating rate) and the constant heating rate procedure are shown in Table 19. It was concluded that (1) no improvement was realized by a change in the heating procedure, based on the standard deviation and (2) the predicted temperature at a 25 light-transmittance-loss level for the modified procedure was poorer than the standard procedure(5).

(5) Revision of the Cleaning Procedure

Before starting the modified heating studies discussed above, attention was given to the cleaning procedures specified for the original 5-ml Bomb. Normally, this is done by "scrubbing" the bomb with a brush and commercial cleanser, rinsing with distilled water, acetone, and finally dried by air-blowing. However, it was apparent that this method was inefficient for removing lacquers, varnish, etc. which were still evident from visual observation. To improve cleaning the following revisions were made: (1) after each run the bomb only was washed in an ultrasonic bath containing Cities Service Solvent S-26 for 10 minutes, (2) the bomb was rinsed with water, (3) the bomb and assembly were rinsed with acetone and (4) the bomb and assembly were dried by air blowing. It was evident from visual examination after a few treatments that most of the brown stains were removed. This procedure was therefore used on the modified heating studies discussed above.

Since no improvement in precision was observed as a result of the modified heating studies using the ultrasonic-chemical cleaning between runs, it was decided to evaluate the merits of the modified cleaning with the original bomb heating procedure. These data are shown in Table 30 and are summarized in Table 19. Although the precision was acceptable a few points were more widely scattered that desired. After further consideration of the cleaning procedure it was decided to eliminate cleaning between runs and use ultrasonic-chemical cleaning only at the start of a series of runs with each new fuel. This would eliminate deposits from the previous fuel and at the same time eliminate possible contamination from cleaning solvents within a series of tests on a fuel.

This "limited" ultrasonic-chemical cleaning along with statistical methods of obtaining and interpreting data were incorporated in a Modified 5-ml Bomb Test Procedure shown in Appendix III.

Using this procedure a number of fuels were evaluated. These data are shown in Table 31 and are summarized in Table 19. An example of data on four repeat tests on one fuel (West Texas hydrotreated kerosine) are shown in Figure 2. In Figure 3 data on this base fuel and the same base with three concentrations of Ionol are shown. The precision of the Modified 5-ml Bomb Procedure was improved over the original 5-ml as indicated by (1) comparison of Figures 1, 2 and 3 (2) a maximum sample standard deviation from regression of 4.1 for the original procedure (3) all regression coefficients were significant above the 99 percent confidence level for the Modified 5-ml Bomb procedure whereas only 5 of 16 were this significant for the original 5-ml Bomb procedure. An example of the data with Standard Deviations From Regression (S.D.F.R.) of from 0.71 to 4.00 are shown in Figure 2.

(6) Repeatability of Predicted Temperatures

Calculated temperatures for given light-transmittance-loss levels are the final results for 5-ml Bomb tests. In Table 19, calculated temperatures at 0, 15 and 25 light-transmittance-loss units are shown for multiple tests on a neat West Texas hydrotreated kerosine (BJ62-16-J1, BJ63-10-G53), a 100 ppm Ionol blend (BJ63-16-J35, BJ63-10-J54), and a 500 ppm Ionol blend (BJ63-16-J6, BJ63-10-J55) for both the original and Modified 5-ml Bomb procedures. Variance analyses of the data from each procedure at each of the three light-transmittance loss levels were made and are shown in Table 20.

TABLE 19

SUMMARY OF REGRESSION ANALYSIS OF 5-ML BOMB DATA

Fuels	Additive	Conc., PPM	Series	No. of Runs Per Series	Mean Temp., F	Mean ΔL.T.	Regression Coef.	Standard Dev. From Regression	Temperature (°F) Calculated For Various Δ L.T. Losses		
									0	15	25
Variable Heating Rate; Time Constant (Standard Bomb Procedure)											
BJ62-16-J1	None	0	1	9	396.1	24.3	0.36**	7.74	330	371	398
			2	6	371.7	18.2	0.27**	2.31	305	360	397
			3	3	358.3	15.7	0.27*	0.72	300	356	393
BJ63-10-G53			4	9	374.4	17.7	0.32**	3.49	319	366	398
			Average						314	363	396
BJ63-16-J35	Ionol	100	1	5	380.0	13.4	0.49(-)	5.50	353	383	404
			2	6	388.3	20.7	0.42*	8.54	339	375	399
			3	3	378.3	20.7	0.78*	1.31	352	371	384
			Average						348	376	396
BJ63-16-J6	Ionol	500	1	8	382.5	18.1	0.44**	7.58	342	375	398
			2	4	375.0	12.3	0.36(-)	5.79	341	383	410
			3	6	397.5	19.8	0.41*	9.83	349	386	410
			Average						344	381	406
BJ62-10-K43 (SF6-6207)	None	0	1	5	381.4	19.4	0.43*	8.10	336	371	394
BJ63-10-J40	Ionol	100	1	5	404.0	15.2	0.23*	3.06	338	403	446
BJ63-10-J41	Ionol	500	1	4	408.8	16.0	0.53*	2.38	378	407	426
BJ62-10-J62	None	0	1	5	401.0	18.4	0.16*	3.07	283	379	443
BJ63-10-J44	Ionol	100	1	6	395.8	18.0	0.43*	10.29	354	389	412
BJ63-10-J45	Ionol	500	1	6	394.2	15.3	0.30**	4.49	344	393	426
Constant Heating Rate; Time Variable; 1000°F Initial Furnace Temperature; Hoskins Furnace(a)											
BJ63-10-G53	None	0	1	13	356.2	15.8	0.39**	4.06	315	354	380
Constant Heating Rate; Time Variable; 1000°F Initial Furnace Temperature; 5-ML Bomb Furnace(a)											
BJ63-10-G53	None	0	1	5	380.0	11.2	0.29(-)	4.69	341	393	427
Constant Heating Rate; Time Variable; 90°F Initial Furnace Temperature; 5-ML Bomb Furnace(a)											
BJ63-10-G53	None	0	1	10	365.2	10.1	0.36*	6.59	337	379	407
			2	5	360.6	9.6	0.34**	1.06	332	377	406
			3	12	406.9	15.5	0.11(-)	5.41	265	402	494
			4	9	377.8	11.6	0.32(-)	6.99	341	388	420
			Average						319	386	432
Variable Heating Rate With Ultrasonic-Chemical Cleaning Between Runs											
BJ63-10-G53	None	0	1	9	368.6	16.9	0.19*	2.46	280	359	411
Variable Heating Rate With Limited Ultrasonic-Chemical Cleaning											
BJ63-10-G53	None	0	1	9	366.6	15.4	0.28**	2.18	312	365	401
			2	9	369.7	16.3	0.32**	3.80	319	366	397
			3	9	373.8	16.3	0.22**	4.00	301	368	412
			4	9	368.8	16.1	0.25**	0.71	305	364	404
			Average						310	366	404
BJ63-10-J72	Ionol	30	1	9	398.8	14.9	0.31**	1.24	351	399	431
BJ63-10-J54	Ionol	100	1	9	399.4	15.9	0.38**	3.47	358	397	423
			2	9	396.6	15.7	0.35**	1.00	351	394	423
			3	9	401.8	17.4	0.46**	0.67	364	396	418
			4	9	399.8	13.3	0.36**	1.07	363	404	432
			Average						359	398	424
BJ63-10-J55	Ionol	500	1	6	395.3	12.9	0.53**	2.00	371	399	418
			2	9	400.2	15.8	0.64**	2.40	376	399	415
			3	9	401.1	12.8	0.53**	4.13	377	405	424
			4	9	398.1	16.0	0.59**	1.64	371	396	413
			Average						374	400	418
BJ63-10-J71	AN701	100	1	9	392.8	16.3	0.44**	0.79	355	390	413
BJ63-10-J67	AN701	500	1	9	397.2	11.6	0.46**	1.13	372	405	427
BJ63-10-J70	DuPont 22	100	1	9	415.6	17.8	1.16**	3.99	400	413	422
BJ63-10-J68	DuPont 22	500	1	16(b)	440.1(c)	14.6(c)	0.35**(c)	1.95(c)	399(d)	441(c)	470(c)

** 99+ per cent confidence

* Between 95-99 per cent confidence

(-) Less than 95 per cent confidence

(a) Ultrasonic-chemical cleaning between runs

(b) More than nine points required to define regression

(c) Values are for regression segment above 400°F

(d) Extrapolated

TABLE 20

ANALYSIS OF VARIANCE FOR TWO PROCEDURES AT THREE LIGHT-TRANSMITTANCE-LOSS LEVELS

Source of Variation	Modified 5-ml Bomb			Standard 5-ml Bomb		
	Calculated Temperature For Light Transmittance Loss = 0					
	d.f. (1)	M.S. (2)	F (3)	d.f. (1)	M.S. (2)	F (3)
Fuels	2	4568.58	126.3(4)	2	1284.50	127.2(4)
Error	9	36.16		7	101.00	
Standard Deviation		6.0			10.0	
	Calculated Temperature For Light Transmittance Loss = 15					
Fuels	2	1456.00	121.1(4)	2	310.15	8.04(5)
Error	9	12.02		7	38.58	
Standard Deviation		3.5			6.21	
	Calculated Temperature For Light Transmittance Loss = 25					
Fuels	2	439.00	13.53(4)	2	102.61	2.18(6)
Error	9	32.44		7	47.09	
Standard Deviation		5.77			6.96	

(1) degrees of freedom
(2) mean square
(3) variance ratio
(4) significant at the 99 percent level
(5) significant at the 95 percent level
(6) not significant at the 90 percent level

An examination of the error mean squares shows that the error for the Modified procedure is less than for the original procedure at each of the three levels of light-transmittance-loss. Examining these data more closely by using an "F" test of the ratios of the error mean squares to determine homogeneity of variance it can be concluded at the 90 per cent confidence level that the error for the Modified procedure is less than for the original procedure for 0 and 15 units light-transmittance-loss while at the 25 units level there is no significant difference. From Table 20 one can conclude with 99 percent confidence that there are significant differences among the three fuels at all three light-transmittance levels, based on the Modified 5-ml Bomb data, however, based on the original 5-ml Bomb data this conclusion could be made only at the 0 light-transmittance-loss level. At the 15 loss level one could conclude that there are differences among the three fuels with 95 percent confidence and at the 25 loss level it should be concluded that there is no difference.

While the above analyses show there are significant differences among the fuels at various light-transmittance-loss levels for the two procedures, a further analysis is necessary to determine which of the fuels show significant differences at the various light-transmittance-loss levels. Table 21 shows a calculated Least Significant Difference (L.S.D.) at 95 percent confidence that the means must exceed for the differences to be significant.

TABLE 21

COMPARISON OF CALCULATED TEMPERATURES FOR VARIOUS FUELS

Light Transmittance Loss Level	Mean Calculated Temp. For Various Additive Concentrations			Mean Differences in Calculated Temperature For Various Fuel Combinations			
	0 ppm	100 ppm	500 ppm	Δ(100-0)	Δ(500-0)	Δ(500-100)	L.S.D.(a)
			Original 5-Ml Bomb Procedures				
0	314	348	344	34*	30*	-4	19.4
15	363	376	381	13*	18*	5	12.0
25	396	396	406	0	10	10	13.2
			Modified 5-Ml Bomb Procedures				
0	309	359	374	50*	65	15*	9.6
15	366	398	400	32*	34	2	5.5
25	404	424	418	20*	14*	-6	9.1

(a) Least significant difference at 95 percent confidence

* Values which represent significant difference, since they are larger in magnitude than the corresponding L.S.D.

The above data show that the modified procedure extends the versatility of the 5-ml Bomb procedure, since it is able to recognize significant differences between neat and additive fuels even at the presently used 25-loss level. In addition, at the 0 loss level, the modified procedure is able to recognize differences between 100 and 500 ppm additive concentrations.

(7) Ability of The Modified Procedure to Detect Differences In Thermal Stability Quality

At the time of this investigation it was not known at what light-transmittance-loss level fuels should be rated for best correlation with other test methods nor the minimum differences necessary to recognize changes in thermal stability quality as determined by other test methods. However, using the error mean squares for the Modified 5-ml Bomb shown in Table 20 it is possible to calculate the Least Significant Difference (L.S.D.) to use in drawing conclusions with 95 percent confidence for means of various numbers of determinations. A number of LSD values are shown in Table 22. The values shown represent the minimum difference in the average temperatures for two fuels at a given light-transmittance-loss level that would justify a conclusion as to a difference in fuel thermal stability quality with 95 percent confidence.

TABLE 22

LEAST SIGNIFICANT TEMPERATURE DIFFERENCE

(95 percent confidence)

Light Transmittance Loss Level	L.S.D. For Comparison of Means of Various Size				
	$1^{(1)}$ vs $1^{(1)}$	$2^{(1)}$ vs $2^{(1)}$	$3^{(1)}$ vs $3^{(1)}$	$4^{(1)}$ vs $4^{(1)}$	$4^{(1)}$ vs $1^{(1)}$
0	19.2	13.6	11.1	9.6	15.2
15	11.1	7.8	6.4	5.5	8.8
25	18.2	12.9	10.5	9.1	14.4

(1) Number of tests on the fuels being compared

(8) Additive Effects by the Revised 5-ml Bomb Procedure

One purpose of this investigation was to determine if the 5-ml Bomb procedure could be modified to the point where it could recognize the small improvements that some additives impart to the CRC Coker thermal stability of some fuels. In Table 21 it was shown that the Modified 5-ml Bomb could recognize the addition of 100 or 500 ppm of Ionol to a neat fuel at three different levels of light-transmittance-loss and could also recognize a difference between 100 and 500 ppm Ionol in a fuel. Single sets of determinations in the Modified 5-ml Bomb were also made on blends of West Texas hydrotreated kerosine with 30 ppm Ionol, 100 and 500 ppm Ethyl AN701 and 100 and 500 ppm du Pont 22. All of the possible comparisons with each additive are shown in Table 23.

TABLE 23

COMPARISONS OF DIFFERENCE IN MEAN TEMPERATURES FOR VARIOUS ADDITIVE CONCENTRATIONS

Light Transmittance Loss Level	Difference in Mean Temperature F, For Difference in Additive Concentration, PPM							
	Ionol							
	(500-0)	(100-0)	(500-100)	LSD	(30-0)	(100-30)	(500-30)	LSD
0	65*	50*	15*	9.6	42*	8	23*	15.2
15	34*	32*	2	5.5	33*	-1	1	8.8
25	14*	20*	-6	9.1	27*	-7	-13	14.4
	AN701							
	(500-0)	(100-0)	LSD	(500-100)	LSD			
0	63*	46*	15.2	17	19.2			
15	39*	24*	8.8	15*	11.1			
25	23*	9	14.4	14	18.2			
	Du Pont 22							
	(500-0)	(100-0)	LSD	(500-100)	LSD			
0	90*	91*	15.2	-1	19.2			
15	75*	47*	8.8	28*	11.1			
25	66*	18*	14.4	48*	18.2			

* Significant difference at 95 percent confidence level

From Table 23 it can be observed that the Modified 5-ml Bomb recognized the effect of 30, 100 or 500 ppm of Ionol at 0, 15 or 25 light-transmittance-loss levels. The 5-ml Bomb was also able to detect differences between 500 and 100 or 30 ppm Ionol at the 0 loss level. An effect of 500 ppm AN701 was shown at all three loss levels, however, 100 ppm AN701 only showed an effect at 0 and 15 loss levels. An effect of 500 over 100 ppm AN701 was shown only at the 15 loss level. With du Pont 22, effects were shown for 100 and 500 ppm at all three loss levels and an effect of 500 over 100 ppm was also shown for 15 and 25 loss levels. From these data it is concluded that the Modified 5-ml Bomb procedure recognized at least directionally, improvements in thermal stability imparted by antioxidants.

TABLE 24

DESCRIPTION OF JP-6-TYPE TEST FUELS

Fuel Number	Description
BJ62-16-J1	West Texas Hydrotreated Kerosine (1962 Production Batch)
BJ63-10-G53	Same as BJ62-16-J1 (Number Reassigned)
BJ63-16-J35	West Texas Hydrotreated Kerosine + 100 ppm Shell IONOL, (2,6-Ditertiarybutyl-4-methylphenol)
BJ63-16-J6	West Texas Hydrotreated Kerosine + 500 ppm Shell IONOL
BJ62-10-K43	Air Force JP-6 (SF6-6207) See Note 1
BJ63-10-J40	SF6-6207 + 100 ppm Shell IONOL
BJ63-10-J41	SF6-6207 + 500 ppm Shell IONOL
BJ62-10-J62	West Texas JP-6 (50-50 Blend of West Texas Turbine Fuel + Paraffins)
BJ63-10-J44	West Texas JP-6 Blend + 100 ppm Shell IONOL
BJ63-10-J45	West Texas JP-6 Blend + 500 ppm Shell IONOL
BJ63-10-J54	Same as BJ63-16-J35 (Second Blend)
BJ63-10-J55	Same as BJ63-16-J6 (Second Blend)
BJ63-10-J67	West Texas Hydrotreated Kerosine + 500 ppm Ethyl AN701 (2,6-Ditertiarybutylphenol)
BJ63-10-J68	West Texas Hydrotreated Kerosine + 500 ppm du Pont 22 (N,N'-Disecondary butylparaphenylenediamine)
BJ63-10-J70	West Texas Hydrotreated Kerosine + 100 ppm du Pont 22
BJ63-10-J71	West Texas Hydrotreated Kerosine + 100 ppm AN701
BJ63-10-J72	West Texas Hydrotreated Kerosine + 30 ppm IONOL

Note 1: SF6-6207 already contained 8 lbs/1,000 bbls (30 ppm) of AN701 and 2 lbs/1,000 bbls (~8 ppm) Metal Deactivator when received.

TABLE 25

PHILLIPS STATIC 5-ML BOMB JET FUEL THERMAL STABILITY TEST METHOD DATA

Fuels Tested + Comments	Date	Run No.	Furnace Condition			Temp. F	Percent Light Transmittance (iC_8 = 100)						
							Bausch-Lomb Spectronic 20						Bec. *
							0.35 µ			0.34 µ			0.35 µ
			Volts	Amps.	Watts		Before	After	Loss	Before	After	Loss	Loss
BJ62-16-J1	7-29-63	L75-12	44	1.60	70.4	330	98	95	3				5.5
(Series 1)		-13	45	1.65	74.2	355	98	90	8				7.0
		-14	46	1.66	76.3	360	98	88	10				
		-15	48	1.76	84.5	395	98	81	17				
		-16	50	1.83	90.5	400	98	60	38				26.0
		-17	52	1.92	99.8	420	98	60	38				
		-18	54	1.98	106.9	460	98	44	54				
		-19	51	1.85	94.3	410	98	76	22				
		-20	53	1.93	102.2	435	98	69	29				
BJ62-16-J1	8-2-63	L77-4	44	1.60	70.4	330	100	95	5				
(Series 2)		-5	46	1.66	76.3	360	100	81	19				
		-6	51	1.85	94.3	415	100	71	29				
		-7	45	1.65	74.2	345	100	90	10				
		-8	47	1.73	81.3	380	100	80	20				
		-9	50	1.83	90.5	400	100	74	26				
BJ63-16-J1	8-21-63	L82-9	46	1.66	76.3	350	102	88	14	96	81	15	5.2
(Series 3)		-10	48	1.76	84.5	390	102	78	24	96	69	27	11.4
		-11	44	1.60	70.4	335	102	93	9	96	85	11	18.6
BJ63-16-J6	7-29-63	L76-1	44	1.60	70.4	330	100	97	3				
(Series 1)		-2	46	1.66	76.3	360	100	97	3				
		-3	48	1.76	84.5	395	100	78	22				
	7-30-63	-4	50	1.83	90.5	405	100	80	20				
		-5	52	1.92	99.8	420	100	58	42				
		-6	47	1.69	79.4	380	100	87	13				
		-7	46	1.66	76.4	370	100	93	7				
		-8	50	1.83	90.5	400	100	65	35				
BJ63-16-J6	8-5-63	L78-1	46	1.66	76.4	345	100	96	4				1.5
(Series 2)		-2	48	1.76	84.5	375	100	94	6				3.3
Doubtful		-3	49	1.69	79.4	385	100	85	15				12.3
Watts		-4	50	1.83	90.5	395	100	76	24				17.7

(*) Beckman Model DB Recording Spectrophotometer

TABLE 25 (Continued)

Fuels Tested + Comments	Date	Run No.	Furnace Condition			Temp.	Percent Light Transmittance (iC_8 = 100)						
							Bausch-Lomb Spectronic 20						Bec. *
							0.35 µ			0.34 µ			0.35 µ
			Volts	Amps.	Watts	F	Before	After	Loss	Before	After	Loss	Loss
BJ62-10-J62	8-16-63	L81-6	48	1.76	84.5	375	80	64	16	72	54	18	
		-7	50	1.83	90.5	405	80	59	21	72	50	22	
		-8	52	1.92	99.8	425	80	62	18	72	52	20	
		-9	54	1.98	106.9	445	80	53	27	72	44	28	
		-10	46	1.66	76.3	355	80	70	10	72	65	7	
BJ63-10-J44	8-16-63	-11	49	1.78	87.2	390	81	64	17	74	56	18	
		-12	46	1.66	76.3	345	81	79	2	74	70	4	
		-13	48	1.76	84.5	375	81	75	6	74	68	6	
		-14	50	1.83	90.5	410	81	68	13	74	59	15	
		-15	52	1.92	99.8	435	81	32	49	74	27	47	
	8-19-63	-16	51	1.85	94.3	420	81	60	21	74	51	23	
BJ63-10-J45	8-19-63	-17	46	1.66	76.3	340	80	78	2	74	70	4	
		-18	48	1.76	84.5	375	80	78	2	74	70	4	
		-19	50	1.83	90.5	400	80	60	20	74	53	21	
		-20	52	1.92	99.8	445	80	50	30	74	43	31	
		-21	51	1.85	94.3	415	80	56	24	74	48	26	
		-22	49	1.78	87.2	390	80	66	14	74	59	15	
BJ63-10-G53	10-21-63	L96-1	46	1.66	76.3	361	102(a)	95(a)	7				
		-2	47	1.72	80.8	370	102	87	15				
		-3	46	1.66	76.3	364	102	87	15				
		-4	49	1.78	87.2	395	102	80	22				
		-5	47	1.72	80.8	377	102	79	23				
		-6	46	1.66	76.3	357	102	87	15				
		-7	49	1.78	87.2	392	102	77	25				
		-8	47	1.72	80.8	367	102	85	17				
		-9	49	1.78	87.2	387	102	82	20				

(a) Light transmittance for remaining runs based on average of three readings

(*) Beckman Model DB Recording Spectrophotometer

TABLE 25 (Continued)

							Percent Light Transmittance (iC8 = 100)						
							Bausch-Lomb Spectronic 20						Bec. *
Fuels Tested	Date	Run	Furnace Condition			Temp.	0.35 μ			0.34 μ			0.35 μ
+ Comments		No.	Volts	Amps.	Watts	F	Before	After	Loss	Before	After	Loss	Loss
BJ63-16-J6	8-22-63	L83-1	46	1.66	76.3	345	100	96	4	94	90	4	2.7
(Series 3)		-2	48	1.76	84.5	375	100	92	8	94	86	8	7.3
		-3	50	1.83	90.5	400	100	85	15	94	80	14	
		-4	52	1.92	99.8	430	100	53	47	94	45	49	
		-5	51	1.85	94.3	415	100	84	16	94	76	18	
		-6	52	1.92	99.8	420	100	71	29	94	61	33	26.4
BJ63-16-J35	7-30-63	L76-9	46	1.66	76.3	360	100	92	8				
(Series 1)		-10	48	1.76	84.5	385	100	82	18				
		-11	50	1.83	90.5	400	100	75	25				
		-12	47	1.69	79.4	380	100	87	13				
		-13	44	1.60	70.4	375	100	97	3				
BJ63-16-J35	8-2-63	L77-11	44	1.60	70.4	340	99	96	3				2.0
(Series 2)		-12	47	1.69	79.4	380	99	85	14				13.0
		-13	50	1.83	90.5	400	99	83	16				
		-14	52	1.92	99.8	425	99	67	32				27.5
		-15	51	1.85	94.3	415	99	54	45				
		-16	46	1.66	76.3	370	99	87	12				11.3
BJ63-16-J35	8-21-63	L82-13	46	1.66	76.3	360	100	93	7	94	87	7	3.2
(Series 3)		-14	48	1.76	84.5	375	100	83	17	94	73	21	13.7
		-15	50	1.83	91.5	400	100	62	38	94	53	41	30.6
BJ62-10-K43	8-13-63	L80-1	44	1.60	70.4	337	85	79	6	80	73	7	
		-2	46	1.66	76.3	360	85	79	6	80	73	7	
		-3	48	1.76	84.5	385	85	67	18	80	63	17	
		-4	50	1.83	90.5	405	85	63	22	80	57	23	
		-5	52	1.92	99.8	420	85	40	45	80	34	46	
BJ63-10-J40	8-13-63	-6	46	1.66	76.3	360	85	77	8	80	72	8	
		-7	48	1.76	84.5	380	85	77	8	80	72	8	
		-8	50	1.83	90.5	405	85	73	12	80	67	13	
		-9	52	1.92	99.8	425	85	65	20	80	59	21	
		-10	54	1.98	106.9	450	85	57	28	80	51	29	
BJ63-10-J41	8-14-63	-11	48	1.76	84.5	385	85	80	5	80	75	5	
		-12	50	1.83	90.5	405	85	73	12	80	67	13	
		-13	52	1.92	99.8	430	85	56	29	80	50	30	
		-14	51	1.85	94.3	415	85	67	18	80	62	18	

(*) Beckman Model DB Recording Spectrophotometer

TABLE 26

DETERMINATION OF PRECISION OF BAUSCH AND LOMB SPECTRONIC 20 SPECTROPHOTOMETER

AT THREE DIFFERENT WAVE LENGTHS

Wave-	Percent Light Transmittance, (Isooctane Reference = 100)																	
Length, mμ	340						350						365					
Series(a)	1	2	3	4	Total	Mean	1	2	3	4	Total	Mean	1	2	3	4	Total	Mean
Fuels																		
SF6-6201	76	75	76	76	303	75.75	82	80	81	81	324	81.00	86	86	86	87	345	86.25
SF6-6303	79	80	78	76	313	78.25	86	86	87	85	344	86.00	96	95	98	95	384	96.00
SF6-6202	98	97	99	98	392	98.00	100	99	100	100	399	99.75	102	100	103	102	407	101.75
SF6-6304	78	80	79	78	315	78.75	86	87	86	87	346	86.50	95	96	94	97	382	95.50
SF6-6203	100	99	98	98	395	98.75	100	100	99	100	399	99.75	102	102	103	105	412	103.00
SF6-6306	103	103	103	102	411	102.75	104	104	104	102	414	103.50	104	104	104	104	416	104.00
SF6-6207	76	76	78	75	305	76.25	81	81	80	80	322	80.50	85	85	86	85	341	85.25
TSF-6307	99	102	102	100	403	100.75	102	103	102	102	409	102.25	103	105	104	104	416	104.00
SF6-6208	89	93	89	88	359	89.75	93	95	93	92	373	93.25	98	102	100	98	398	99.50
TSF-6204	70	73	71	70	284	71.00	79	82	81	79	321	80.25	90	96	91	91	368	92.00
SF6-6213	74	74	74	76	298	74.50	82	81	82	83	328	82.00	89	88	89	89	355	88.75
TSF-6305	63	61	62	63	249	62.25	75	73	75	78	301	75.25	91	90	92	95	368	92.00
TSF-6206	24	25	26	24	99	24.75	30	31	31	30	122	30.50	40	40	41	40	161	40.25
TSF-6312	71	68	72	67	278	69.50	77	74	76	72	299	74.75	83	80	83	81	327	81.75
SF6-6214	75	76	74	73	298	74.50	81	82	80	81	324	81.00	89	89	88	88	354	88.50
SF6-6209	68	69	68	69	274	68.50	75	76	73	75	299	74.75	81	81	80	83	325	81.25
TSF-6306	94	93	94	92	373	93.25	99	98	98	97	392	98.00	102	102	102	102	408	102.00
Standard Deviation						1.3						1.1						1.3

(a) Series 1 ran on 9-26-63 at 8:15 a.m.; Series 2 on 9-26-63 at 11:00 a.m.; Series 3 on 9-27-63 at 8:45 a.m.; Series 4 on 9-27-63 at 3:00 p.m.

TABLE 27

MODIFICATION STUDIES OF THE 5-ML BOMB JET FUEL THERMAL STABILITY TEST METHOD

Modifications Employed

1. Constant Heating (Wattage Input) Rate Allowing Time to Vary to Obtain Desired End-Temperatures
2. Substitution of a 1000 Watt Hoskins Furnace
3. Initial Furnace Temperature Increased to 1000°F
4. Ice-Water Quench of Heated Samples
5. Ultrasonic-Chemical Cleaning Between Runs
6. No Opening in Bottom End of Furnace

Fuels Tested + Comments	Run No.	Date	Variac[a]	Peak Fuel Temp., °F	Exit Time, Min	Light Transmittance 0.35 Microns Before	After[b]	Loss	Comments
BJ63-10-G53 (Series 1)	L92-1	10-9-63	46	377	-	100.3	86.0	14.3	Bad data, due to faulty thermocouple
	-2		46	389	7.10		64.3	36.0	
	-3		40	353	8.40		89.3	11.0	
	-4		52	379	6.85		64.7	35.6	
	-5		51	389	6.71		74.5	25.8	
	-6		49	353	6.21		84.7	15.6	
	-7		46	365	6.41		83.7	16.6	
	-8		48	347	5.67		97.0	3.3	
	-9		48	334	5.28		94.3	6.0	
	-10	10-10-63	47	383	6.50	102.6	54.5	48.1	
	-11		49	338	5.44		89.5	13.1	
	-12		48	359	5.49		87.0	15.6	
	-13		45	348	5.40		89.5	13.1	
	-14		46	367	6.20		84.3	18.3	
	-15		48	344	5.95		89.3	13.3	
	-16		47	374	6.49		76.2	26.4	→ Cloudiness in heated sample
	-17		47	344	6.22		91.2	11.4	
	-18		49	368	6.89		75.7	26.9	

(a) Variac setting required to maintain a 1000°F initial furnace temperature
(b) Values represent the averages of 3 readings over a 2 hour period

TABLE 28

MODIFICATION STUDIES OF THE 5-ML BOMB JET FUEL THERMAL STABILITY TEST METHOD

Modifications Employed

1. Constant Heating (Wattage Input) Rate for all Runs Allowing Time to Vary to Obtain Desired End-Temperatures
2. Initial Furnace Temperature Increased to 1000°F
3. Ultrasonic-Chemical Cleaning Between Runs
4. Ice-Water Quench of Heated Samples
5. No Opening in Bottom End of Furnace

			Furnace Conditions			Peak	Exit	Time	Light Transmittance 0.35 Microns		
Fuels Tested + Comments	Run No.	Date	Volts	Amps	Initial Temp., °F	Fuel Temp., °F	Time, Min	to 300°F, Min	(a) Before	(a) After	Loss
BJ63-10-G53	L93-1	10-11-63	64	2.31	1005	387	2.20	1.55	100.8	86.0	14.8
	-2		64	2.31	1000	359	2.00	1.58		99.2	1.6
	-3		64	2.31	1000	351	1.90	1.50		94.7	6.1
	-4		64	2.31	990	406	2.52	1.70		78.2	22.6
	-5		64	2.31	998	397	2.39	1.61		89.7	11.1

(a) Values represent the average of 3 readings over a 2 hour period

TABLE 29

MODIFICATION STUDIES OF THE 5-ML BOMB JET FUEL THERMAL STABILITY TEST METHOD

Modifications Employed

1. Constant Heating (Wattage Input) Rate for all Runs Allowing Time to Vary to Obtain Desired End-Temperatures
2. Ultrasonic-Chemical Cleaning Between Runs
3. Ice-Water Quench of Heated Samples
4. No Opening in Bottom End of Furnace

			Furnace Conditions						Light Transmittance 0.35 Microns		
Fuels Tested + Comments	Run No.	Date	Volts	Amps	Initial Temp., °F	Peak Fuel Temp., °F	Exit Time, Min	Time to 300°F Min	(a) Before	(a) After	Loss
BJ63-10-G53 (Series 1)	L93-6	10-11-63	80	2.89	90	367	8.23	6.67	100.8	89.7	11.1
	-7		80	2.89	90	342	7.71	6.78		96.5	4.3
	-8		80	2.89	90	377	8.29	6.65		84.7	16.1
	-9		80	2.89	90	353	7.66	6.58		94.7	6.1
	-10		80	2.89	90	364	7.94	6.58		90.5	10.3
BJ63-10-G53 (Series 2)	-11	10-14-63	80	2.89	90	389	8.73	6.80	100.7	72.5	28.2
	-12		80	2.89	90	378	8.18	6.51		81.8	18.9
	-13		80	2.89	90	343	7.32	6.45		90.2	10.5
Cloudiness in heated sample	-14*		80	2.89	90	367	7.99	6.55		86.3	14.4
	-15		80	2.93	90	353	7.49	6.40		97.8	2.9
	-16		79	2.90	90	363	7.99	6.55		94.5	6.2
	-17		79.5	2.90	90	357	7.99	6.75		96.2	4.5
	-18		79.5	2.90	90	372	7.98	6.40		94.2	6.5
	-19		79.0	2.90	90	383	8.17	6.30		93.7	7.0
	-20		79.5	2.90	90	347	7.51	6.50		99.2	1.5
BJ63-10-G53 (Series 3)	L94-1	10-16-63	80.0	2.95	90	404	8.34	6.31	101.0	88.7	12.3
	-2		80.0	2.95	90	374	8.18	6.64		93.0	8.0
	-3		80.0	2.95	90	422	9.26	6.54		73.8	27.2
	-4		80.0	2.95	90	388	8.50	6.69		87.0	14.0
	-5		80.0	2.95	90	413	9.10	6.68		83.3	17.7
	-6		80.0	2.95	90	432	9.58	6.63		87.7	13.3
	-7		80.0	2.95	90	432	9.65	6.71		88.2	12.8
	-8		80.0	2.95	90	407	8.95	6.62		88.3	12.7
	-12	10-17-63	80.0	2.95	90	403	8.98	6.71		78.2	23.8
	-13		80.0	2.95	90	403	8.97	6.73		83.2	18.8
	-14		80.0	2.95	90	402	9.03	6.74		90.3	11.7
	-15		80.0	2.95	90	403	8.93	6.69		88.5	13.5

(a) Values represent averages of three readings

* Upper bomb assembly prior to this run was washed with water + acetone; for all other runs assembly was washed with acetone only

TABLE 29 (Continued)

Fuels Tested + Comments	Run No.	Date	Furnace Conditions Volts	Amps	Initial Temp., °F	Peak Fuel Temp., °F	Exit Time, Min	Time to 300°F Min	Light Transmittance 0.35 Microns (a) Before	(a) After	Loss
BJ63-10-G53	L95-1*	10-18-63	80.0	2.95	90	378	8.27	6.64	100.8	81.7	19.1
	-2		80.0	2.95	90	353	7.61	6.54		100.3	0.5
	-3		80.0	2.95	90	377	8.26	6.64		75.5	25.3
	-4		80.0	2.95	90	402	8.89	6.66		83.3	17.5
	-5		80.0	2.95	90	402	8.55	6.43		84.2	16.6
	-6		80.0	2.95	90	378	8.08	6.44		94.2	6.6
Data doubtful due to improper position of bomb in furnace	-7		80.0	2.95	90	354	7.78	6.70		96.3	4.5
	-8		80.0	2.95	90	363	7.25	5.95		95.7	5.1
	-9		80.0	2.95	90	353	7.94	6.83		102.8	-2.0
	-10		80.0	2.95	90	403	8.86	6.66		84.7	16.1
BJ63-10-G53(b)	L94-9	10-17-63	0	0	90	90	9.00	--	101.0	100.7	0.3
	-10		0	0	90	90	9.00	--		101.0	0.0
	-11		0	0	90	90	9.00	--		100.3	0.7

* Began using a constant voltage transformer

(a) Values represent the average of 3 readings over a 2 hour period

(b) Runs L94-9 through 11 are "blank" runs to check the effect of possible contamination resulting from handling

TABLE 30

MODIFICATIONS OF THE 5-ML BOMB JET FUEL THERMAL STABILITY TEST METHOD

Modifications Employed

1. Ultrasonic-Chemical Cleaning Between Runs
2. Individual Aeration of Samples for Exactly One Minute
3. Installation of a Constant Voltage Transformer
4. Ice-Water Quench of Heated Samples

			Furnace Conditions						Light Transmittance 0.35 Microns (a)		
Fuels Tested + Comments	Run No.	Date	Volts	Amps	Initial Temp., °F	Peak Fuel Temp., °F	Exit Time, Min	Time to 300°F Min	Before	After	Loss
BJ63-10-G53	196-10	10-22-63	47	1.72	90	364	20	14.81	102.0	87.3	14.7
	-11		49	1.78	90	384	20	13.78		79.3	22.7
	-12		49	1.78	90	390	20	13.35		81.3	20.7
This run inadvertently air-quenched	-13		47	1.72	90	379	20	13.64		83.5	18.5
	-14		46	1.66	90	346	20	15.81		85.0	17.0
	-15		49	1.78	90	383	20	13.72		82.7	19.3
	-16		46	1.66	90	358	20	14.75		90.2	11.8
	-17		47	1.72	90	361	20	14.75		87.0	15.0
	-18		46	1.66	90	352	20	15.08		90.0	12.0

(a) Values represent averages of three readings

TABLE 31

MODIFICATIONS OF THE 5-ML BOMB JET FUEL THERMAL STABILITY TEST METHOD

Modifications Employed

1. Limited Ultrasonic-Chemical Cleaning (*)
2. Aeration of Total Volume of Sample for a Series of Runs Rather Than Individual Sample Aeration
3. Installation of a Constant Voltage Transformer
4. Ice-Water Quench of Heated Samples

			Furnace Conditions					Light Transmittance 0.35 Microns		
Fuels Tested + Comments	Run No.	Date	Volts	Amps	Initial Temp., °F	Peak Fuel Temp., °F	Time to 300°F Min	(a) Before	(a) After	Loss
BJ63-10-G53	L93-1	10-25-63	48	1.76	90	372	14:30	103.6	83.0	20.6
(Series 1)	-2		44	1.60		332	17:04		97.5	6.1
	-3		50	1.83		392	16:35		84.7	18.9
	-4		45	1.65		342	16:20		95.3	8.3
	-5		47	1.72		366	14:47		89.0	14.6
	-6		46	1.66		354	15:30		91.0	12.6
	-7	10-28-63	44	1.60		341	16:20		97.0	6.6
	-8		50	1.83		408	12:51		75.0	28.6
	-9		49	1.78		392	13:25		81.7	21.9
BJ63-10-G53	-10		47	1.72		371	14:30		88.5	15.1
(Series 2)	-11		48	1.76		382	14:00		86.5	17.1
	-12		44	1.60		336	16:42		97.5	6.1
	-13		44	1.60		339	16:27		98.0	5.6
	-14		50	1.83		395	13:31		81.7	21.9
	-15		50	1.83		404	12:53		78.5	25.1
	-16		45	1.65		348	15:48		94.7	8.9
	-17	10-29-63	49	1.78		391	13:45		72.0	31.6
	-18		46	1.66		361	14:45		88.0	15.6
BJ63-10-G53	-19		45	1.65		350	15:46		91.7	11.9
(Series 3)	-20		48	1.76		383	13:58		85.3	18.3
	-21	10-30-63	46	1.66		366	14:44		86.2	17.4
	-22		47	1.72		370	14:37		86.8	16.8
	-23		44	1.60		347	15:59		93.3	10.3

(*) Bomb ultrasonically cleaned prior to testing a given fuel with Cities Service S-26 solvent for 10 minutes, washed with water, acetone and air dried. Bomb thermocouple cleaned with crocus cloth, water, acetone and air dried. No further washing between runs except rinsing with the fuel being tested.

(a) Values represent average of three readings

TABLE 31 (Continued)

			Furnace Conditions					Light Transmittance 0.35 Microns		
Fuels Tested + Comments	Run No.	Date	Volts	Amps	Initial Temp., °F	Peak Fuel Temp., °F	Time to 300°F, Min	(a) Before	(a) After	Loss
BJ63-10-G53	L99-1	10-30-63	50	1.83	90	407	12:57	103.6	74.3	29.3
(Series 3)	-2		49	1.78		394	13:28		91.0	12.6
	-3		50	1.83		405	12:58		80.8	22.8
	-4		44	1.60		342	16:15		96.0	7.6
BJ63-10-G53	-5		49	1.78		390	13:40		83.0	20.6
(Series 4)	-6	10-31-63	50	1.83		405	13:00		77.3	26.3
	-7		48	1.76		380	14:02		84.8	18.8
	-8		45	1.66		345	16:08		93.0	10.6
	-9		47	1.72		370	14:42		86.5	17.1
	-10		44	1.60		340	16:12		95.3	8.3
	-11		44	1.60		340	16:25		95.0	8.6
	-12		46	1.66		352	15:40		91.3	12.3
	-13		50	1.83		397	13:21		81.0	22.6
BJ53-10-J55	L100-1	11-1-63	45	1.66		341	16:27	99.0	98.0	1.0
(Series 1)	-2		48	1.76		375	-		97.8	1.2
	-3		49	1.78		395	13:22		88.0	11.0
Did not use in	-4		51	1.85		420	12:25		75.0	24.0
regression	-5		50	1.83		400	13:14		82.0	17.0
analysis	-6		47	1.72		372	14:27		98.0	1.0
	-7		45	1.66		352	15:35		98.0	1.0
	-8		46	1.69		357	15:20		98.0	1.0
	-9		51	1.85		410	12:40		76.0	23.0
BJ63-10-J55	-10	11-4-63	48	1.76		375	14:27	99.0	98.0	1.0
(Series 2)	-11		48	1.76		380	14:30	99.0	98.0	1.0
	-12		51	1.85		414	12:34	100.0	76.0	24.0
	-13		50	1.83		400	13:25		80.0	20.0
	-14		51	1.85		415	13:38		76.0	24.0
	-15		48	1.76		383	14:00		98.0	2.0
	-16		50	1.83		402	13:10		80.8	19.2
	-17		51	1.85		420	12:30		72.8	27.8
	-18		50	1.83		413	12:30		77.0	23.0
BJ63-10-J54	L101-1	11-5-63	48	1.76		382	14:00	100.0	91.7	8.3
(Series 1)	-2		51	1.85		414	12:50		85.2	14.8
	-3		48	1.76		380	13:50		91.0	9.0
	-4		51	1.85		410	12:55		82.7	17.3
	-5		50	1.83		400	13:12		83.0	17.0
	-6		48	1.76		379	14:17		92.0	8.0
	-7	11-6-63	50	1.83		400	13:15		82.0	18.0
	-8		51	1.85		420	12:10		71.3	28.7
	-9		50	1.83		410	12:55		78.3	21.7

(a) Values represent average of three readings

TABLE 31 (Continued)

Fuels Tested + Comments	Run No.	Date	Furnace Conditions Volts	Amps	Initial Temp., °F	Peak Fuel Temp., °F	Time to 300°F, Min	Light Transmittance 0.35 Microns (a) Before	(a) After	Loss
BJ63-10-J54 (Series 2)	L101-10	11-6-63	50	1.83	90	398	13:26	100.0	83.2	16.8
	-11		48	1.76		378	14:47		91.0	9.0
	-12	11-7-63	51	1.85		412	12:35		79.7	20.3
	-13		48	1.76		381	14:02		89.7	10.3
	-14		51	1.85		414	12:36		78.7	21.3
	-15		50	1.83		397	13:36		82.0	18.0
	-16		50	1.83		399	13:20		83.0	17.0
	-17		51	1.85		413	12:44		79.0	21.0
	-18		48	1.76		377	14:42		92.0	8.0
BJ63-10-J54 (Series 3)	L102-1	11-8-63	50	1.83		404	13:04	100.0	81.0	19.0
	-2		48	1.76		384	13:57		90.8	9.2
	-3		51	1.85		416	12:39		75.7	24.3
	-4		48	1.76		379	14:20		93.0	7.0
	-5		48	1.76		384	14:00		90.2	9.8
	-6		51	1.85		417	12:25		75.7	24.3
	-7		50	1.83		406	13:00		80.7	19.3
	-8		50	1.83		407	12:54		81.7	18.3
	-9		51	1.85		419	12:28		74.3	25.7
BJ63-10-J55 (Series 3)	L102-10	11-11-63	51	1.85		413	12:40	99.6	77.5	22.1
	-11		50	1.83		403	13:05		79.7	19.9
	-12		48	1.76		385	13:48		93.8	5.8
	-13		48	1.76		384	13:58		96.2	3.4
	-14		51	1.85		417	12:35		77.0	22.6
	-15		50	1.83		404	13:03		90.0	9.6
	-16		50	1.83		404	13:10		92.0	7.6
	-17		51	1.85		415	12:35		80.3	19.3
	-18		48	1.76		385	13:45		94.7	4.9
BJ63-10-J55 (Series 4)	L103-1	11-12-63	50	1.83		401	13:15	99.0	77.8	21.2
	-2		48	1.76		379	14:15		95.0	4.0
	-3		51	1.85		414	12:30		75.8	23.2
	-4		50	1.83		403	13:15		80.5	18.5
	-5		51	1.85		417	12:37		72.0	27.0
	-6		48	1.76		379	14:09		94.2	4.8
	-7	11-13-63	48	1.76		378	14:18		95.7	3.3
	-8		51	1.85		416	12:32		72.5	26.5
	-9		50	1.83		396	13:26		83.5	15.5

(a) Values represent average of three readings

TABLE 31 (Continued)

Fuels Tested + Comments	Run No.	Date	Furnace Conditions			Peak Fuel Temp., °F	Time to 300°F, Min	Light Transmittance 0.35 Microns		
			Volts	Amps	Initial Temp., °F			(a) Before	(a) After	Loss
BJ63-10-J54	L103-1	11-13-63	50	1.83	90	401	13:12	99.4	86.0	13.4
(Series 4)	-2		50	1.83		396	13:27		88.2	11.2
	-3		51	1.85		414	12:40		81.3	18.1
	-4		51	1.85		412	12:50		80.0	19.4
	-5		48	1.76		382	14:02		91.0	8.4
See Note 1	-6	11-14-63	48	1.76		386	13:40		92.0	7.4
	-7		48	1.76		386	13:40		91.8	7.6
	-8		51	1.85		416	12:34		81.0	18.4
	-9		50	1.83		405	12:57		83.8	15.6
BJ63-10-J67	L104-1	11-14-63	48	1.76		383	13:58	100.0	96.0	4.0
	-2		51	1.85		405	13:02		83.3	16.7
	-3		51	1.85		415	12:41		81.0	19.0
	-4		48	1.76		378	14:16		96.5	3.5
	-5		58	1.83		400	13:10		89.0	11.0
	-6	11-15-63	48	1.76		380	14:15		95.7	4.3
	-7		50	1.83		400	13:17		87.0	13.0
	-8		51	1.85		412	12:50		81.0	19.0
	-9		50	1.83		402	13:10		86.0	14.0
BJ63-10-J68	L104-10	11-15-63	50	1.83		399	13:15	27.3	22.0	5.3
	-11		51	1.85		415	12:35		21.5	5.8
	-12		48	1.76		383	13:52		23.5	3.8
	-13		52	1.92		422	12:23		20.5	6.8
	-14		54	1.98		446	11:35		9.7	17.6
	-15	11-18-63	53	1.95		434	11:56		14.5	12.8
	-16		52	1.92		421	12:24		20.0	7.3
	-17		54	1.98		448	11:29		6.0	21.3
	-18		55	2.00		463	11:04		7.0	20.3
	-19		53	1.95		437	11:49		12.0	15.3
	-20		54	1.98		443	11:35		10.0	17.3
	-21		55	2.00		463	-		6.0	21.3
BJ63-10-J68	L105-1	11-19-63	46	1.66		353	15:41	27.0	25.0	2.0
	-2		47	1.72		367	14:55		24.5	2.5
	-3	11-20-63	54.5	1.99		457	11:12		7.0	20.0
	-4		52.5	1.92		432	12:02	27.5	18.0	9.5

(a) Values represent average of three readings

Note 1: Starting with this run a slight change in cleaning was adopted. After ultrasonic cleaning the bomb was ultrasonically rinsed with deionized water.

TABLE 31 (Continued)

Fuels Tested + Comments	Run No.	Date	Furnace Conditions			Peak Fuel Temp., °F	Time to 300°F, Min	Light Transmittance 0.35 Microns		
			Volts	Amps	Initial Temp., °F			(a) Before	(a) After	Loss
BJ63-10-J72	L106-1	11-22-63	50	1.83	90	400	13:00	98.0	84.5	13.2
	-2		53	1.95		436	12:00		70.2	27.8
	-3		46	1.66		361	15:06		94.7	3.3
	-4		53	1.95		431	12:05		73.0	25.0
	-5		49	1.78		392	13:25		87.0	11.0
	-6	11-25-63	46	1.66		355	15:33		95.7	2.3
	-7		53	1.95		437	11:51		70.7	27.3
	-8		46	1.66		363	14:57		94.0	4.0
	-9		51	1.85		414	-		78.0	20.0
BJ63-10-J71	L107-11	12-2-63	50	1.83		407	12:55	99.0	77.2	21.8
	-12		46	1.66		360	15:07		97.0	2.0
	-13		52	1.92		430	12:06		66.5	32.5
	-14		48	1.76		385	12:55		86.5	12.5
	-15	12-3-63	51	1.85		421	12:37		69.0	30.0
	-16		46	1.66		359	15:17		97.0	2.0
	-17		51	1.85		415	12:47		73.0	26.0
	-18		46	1.66		361	15:13		96.0	3.0
	-19		49	1.78		397	13:25		82.0	17.0
BJ63-10-J70	L108-1	12-3-63	50	1.83		401	13:10	58.0	52.5	5.5
	-2		54	1.98		453	11:17		19.0	39.0
	-3		52	1.92		424	12:15		28.0	30.0
Did not use in	-4	12-4-63	51	1.85		419	12:23		44.0	14.0
regression	-5		50	1.83		403	13:03		53.8	4.2
analysis	-6		52	1.92		429	12:08		23.0	35.0
	-7		51.5	1.90		420	12:32		33.7	24.3
	-8		52	1.92		427	12:23		25.0	33.0
	-9		50	1.83		407	13:01		52.0	6.0
	-10		50.5	1.85		410	12:46		50.0	8.0

(a) Values represent average of three readings

APPENDIX III

MODIFIED 5-ML BOMB TEST PROCEDURE FOR IMPROVED PRECISION

APPENDIX III

MODIFIED 5-ML BOMB TEST PROCEDURE FOR IMPROVED PRECISION

(1) Objective:

To evaluate thermal stability quality of jet fuels and other petroleum distillates at temperatures from 300 to 850°F using 200 ml samples with a precision that will permit recognizing small changes as may result from storage instability and/or antioxidant effects.

(2) Outline of Method:

A stainless steel bomb is charged with five ml of fuel which has been filtered through 0.45 micron porosity filter paper and air-saturated. The bomb is lowered into a 500 watt tubular electric furnace and power is applied at a wattage selected to produce some given temperature after 20 minutes time. The bomb is then removed and quenched, in ice water, to 90°F. Power is turned off and the furnace is cooled to 90°F. Fuel thermal instability is evaluated in terms of the losses in ability to transmit 0.35 micron wavelength ultraviolet light after heating over a range of temperatures.

(3) Apparatus:

(a) Stainless steel bomb (as shown in Figure 21 and 22 in Appendix I) made from type 304 chrome-nickel alloy steel.

(b) Electric muffle furnace and accessories, approximately 500 watt heating capacity with suitable controls for continuously varying power input. The interior of this furnace should be cylindrical in shape and of a size adequate to admit and fully enclose the bomb in an upright position (about one inch diameter and four inches deep). Figure 24 in Appendix I shows a furnace found to be suitable for this purpose. A satisfactory method of furnace control consists of using a constant voltage transformer as a source for a variable voltage transformer in the furnace circuit. Experience with a furnace made in accordance with the reference drawing has shown that a change of one volt in the setting of the variable transformer will change the fuel temperature at the end of the 20 minute heating period by about 10°F. A voltmeter graduated to 0.1 volts will aid in attaining preselected temperatures. This is particularly important with fuels which show relatively large changes in light transmittance loss with temperature.

(c) Spectrophotometer equipped to handle liquid samples. A Bausch and Lomb "Spectronic 20" instrument has been satisfactory. At a wavelength setting of 0.35 micron and a nominal band width of 20 millimicrons, a repeatability study using multiple determinations on a series of fuels showed a standard deviation of 1.1 light transmittance units.

(d) Pressure gauge suitable for use with nitrogen and hydrocarbons. This should be of the indicating type, graduated in intervals of five psi per scale division with a maximum reading of 300 psi.

(e) Self-balancing potentiometer equipped with an iron-constantan thermocouple and capable of measuring temperature between 0 and 1000°F. This instrument should be graduated in intervals of one degree per scale division and accurate within 0.1 per cent of the temperature indicated.

(f) Iron-constantan thermocouple, 22 gauge, closed end, 1/8 inch diameter, 12 inches long with wire, connectors, etc., for attachment to potentiometer.

(g) Millipore(1) laboratory filtration apparatus suitable for filtering approximately 1/2 pint samples of hydrocarbon distillates through 0.45 micron porosity paper elements.

(h) Laboratory stop watch or clock.

(i) An ultrasonic cleaning system with a generator output of 80 KC -80 watt average and a tank of 0.5 gallon capacity for cleaning bomb assembly. A satisfactory system is supplied by Ultrasonic Industries, Ames Court, Engineers Hill, Plain View, L.I., New York.

(j) Miscellaneous suitable stainless steel fittings, etc. for attaching pressure gauge, thermocouple and source of nitrogen to bomb assembly; ring stand and accessories for mounting bomb.

(4) Materials:

(a) Oil-free nitrogen.

(b) Cleaning solvents for bomb assembly, including Cities Service Solvent S-26(2), hot water, deionized water and acetone.

(c) Millipore(1) filters, 25 mm diameter, 0.45 micron pore size, type HA.

(d) Spectral grade isooctane(3) for standardizing spectrophotometer.

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(1) Available from Millipore Filter Corp., Bedford, Massachusetts
(2) Available from Cities Service, 60 Wall Street, New York, New York
(3) Available from Phillips Petroleum Company, Bartlesville, Oklahoma

(5) Preparation of Apparatus:

(a) Prior to a series of tests on a fuel, clean the bomb assembly (except the thermocouple) thoroughly of all contamination left from previous tests by washing in an ultrasonic bath containing Cities Service Solvent S-26 for 10 minutes followed by rinsing in a stream of hot water to remove solvent, then wash in deionized water in an ultrasonic bath for 10 minutes. Then rinse with acetone and dry by air-blast. The thermocouple is polished with crocus cloth, rinsed with acetone and air dried.

(b) After the above cleaning and prior to each test in a series on a fuel add approximately 6 ml of filtered fresh fuel to be tested to the bomb, assemble, invert rapidly several times to rinse the assembly. Dismantle the assembly and drain the fuel.

(6) Procedure:

(a) Install the thermocouple in the upper cap of the bomb so that the junction will be 1/4 inch above the bottom of the bomb when assembled.

(b) Measure 200 ml of the fuel to be tested; filter through 0.45 micron pore size Millipore paper and pour into a 4 ounce brown bottle. Air-saturate by vigorous shaking, removing the bottle cap to replenish air removed by solvent and recapping ten times.

(c) Following aeration, add approximately six ml of fuel to the bomb assembly and seal, invert rapidly several times to rinse and then dismantle and drain. Add exactly five milliliters of fuel to the bomb, seal, and pressurize to 50 psig with oil-free bottled nitrogen and mount in furnace.

(d) Apply electrical power to the furnace at a wattage selected to produce a fuel temperature in the desired range after 20 minutes time. Start stop watch at same time power is turned on. After exactly 20 minutes, raise bomb assembly from furnace and quench in ice water without agitation to 90°F. Record temperature of the fuel which was reached at the time the bomb was removed from the furnace. Turn off power and cool furnace with high velocity air to 90°F.

(e) Using precalibrated test tubes(1), standardize the spectrometer to 100 percent with spectral grade isooctane, then measure light transmittance of test fuel after heating. Three light transmittance readings, spaced at least 20 - 30 minutes apart should be made on each heated sample and the results averaged. At least three determinations should be made on the fresh fuel during a series of runs and the results averaged. Subtract averages to determine light transmittance loss due to heating.

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(1) Available from Bausch and Lomb

(f) Repeat steps (c) through (e) using different power inputs each time to obtain a series of nine light transmittance loss values corresponding to different 20 minute temperature levels to define a line. Select power inputs to obtain three points in the 22 to 30 light transmittance loss range, three in the 2 to 10 range and three in the intermediate range. Any point with a light transmittance loss above 35 should not be used. For most fuels at temperatures below some threshold value, light transmittance loss will fall between 0 and 3 units. Only the point for the highest temperature for a 0 to 3 unit loss should be used. Any point not used will be replaced by a point in the proper range. In most cases the nine points will define a straight line. In exceptional cases the data between 3 and 30 or 35 light-transmittance-loss values will be defined by two rather than one straight line, in this case additional data will be required to determine the point at which the slope changes.

Caution: In the event the absolute light transmittance for a fresh (unheated) fuel is below 40 units, power input should be controlled such that after heating, a fuel will continue to transmit 5 units of the light. A failure to observe this may result in a departure from linearity at the 30 to 35 light-transmittance-loss level.

(g) Plot data on linear graph paper as temperature versus light-transmittance-loss. Draw a straight line through the points based on a regression analysis. (See following sample calculations). In case the data are so scattered as to give a standard deviation from regression of greater than 4.0 units of light-transmittance-loss it is recommended that the data be discarded and the test repeated.

Sample Calculations of Regression Line (Reference 8)

Six quantities provide the information necessary for completing the computation of regression. The quantites are:

n = number of determinations
$\bar{x}$ = mean or average of coded(1) temperature
$\bar{y}$ = mean or average of light transmittance loss
Σx^2 = sum of squares of deviation from mean temperature
Σy^2 = sum of squares of deviation from mean light transmittance loss
Σxy = sum of products of deviations x and y
Σ = summation or sum of

These quantities can be calculated as follows:

$$\bar{x} = (X_1 + X_2 + \ . \ . \ . \ X_n)/n = (\Sigma X)/n$$

$$\bar{y} = (Y_1 + Y_2 + \ . \ . \ . \ Y_n)/n = (\Sigma Y)/n$$

$$\Sigma x^2 = (X_1^2 + X_2^2 + \ . \ . \ . \ X_n^2) - (\Sigma X)^2/n = \Sigma X^2 - (\Sigma X)^2/n$$

$$\Sigma y^2 = (Y_1^2 + Y_2^2 + \ . \ . \ . \ Y_n^2) - (\Sigma Y)^2/n = \Sigma Y^2 - (\Sigma Y)^2/n$$

$$\Sigma xy = (X_1Y_1 + X_2Y_2 \ . \ . \ . \ X_nY_n) - (\Sigma X)(\Sigma Y)/n = \Sigma XY - (\Sigma X)(\Sigma Y)/n$$

b = sample regression coefficient or slope is $b = \Sigma xy / \Sigma x^2$

The sample regression equation is

$\hat{Y} = \bar{y} + b\left[X - (\bar{x} + k)\right]$ if temperatures have been coded or
$\hat{Y} = \bar{y} + b(X - \bar{x})$ if coding was not used

The deviation from regression, $dy.x = Y - \hat{Y}$, measures the failure of the line to fit the data.

$$\Sigma dy.x^2 = \Sigma y^2 - (\Sigma xy)^2/\Sigma x^2$$

$sy.x^2 = \Sigma dy.x^2/(n-2)$ is the mean square deviation from regression

$sy.x = \sqrt{sy.x^2}$ is the sample standard deviation from regression and corresponds to the standard deviation in a single-variable problem.

$s_b = sy.x/\sqrt{\Sigma x^2}$ is sample standard deviation of regression coefficient

$t = b/s_b$, d.f. $= n - 2$ is a test of significance of sample regression coefficient

(Continued)

Point No.	Final Fuel Temp., F.	X Coded (1) Temp., F.	Y Mean Light Transmittance Loss
1	400	100	13.2
2	436	136	27.8
3	361	61	3.3
4	431	131	25.0
5	392	92	11.0
6	355	55	2.3
7	437	137	27.3
8	363	63	4.0
9	414	114	20.0

$\Sigma X = 889.0$ $\qquad \Sigma Y = 133.9$

$n = 9 \qquad \bar{x} = \Sigma X/n = 98.78 \qquad \bar{y} = \Sigma Y/n = 14.88$

$\Sigma X^2 = 96{,}601.00 \qquad \Sigma XY = 15{,}987.70 \qquad \Sigma Y^2 = 2{,}870.55$

$(\Sigma X)^2/n = \underline{87{,}813.44} \qquad (\Sigma X)(\Sigma Y)/n = \underline{13{,}226.34} \qquad (\Sigma Y)^2/n = \underline{1{,}992.13}$

$\Sigma x^2 = 8{,}787.56 \qquad \Sigma xy = 2{,}761.36 \qquad \Sigma y^2 = 878.42$

$b = \Sigma xy/\Sigma x^2 = 2{,}761.36/8{,}787.56 = 0.31$ (2)

$\hat{Y} = \bar{y} + b\,[X - (\bar{x} + 300)] = 14.88 + 0.31(X - 398.78) = 0.31X - 108.74$ (3)

$\Sigma dy.x^2 = \Sigma y^2 - (\Sigma xy)^2/\Sigma x^2 = 878.42 - 2{,}761.36^2/8{,}787.56 = 10.70$

$sy.x^2 = \Sigma dy.x^2/(n-2) = 10.70/7 = 1.53$

$sy.x = \sqrt{sy.x^2} = \sqrt{1.53} = 1.24$ (4)

$s_b = sy.x/\sqrt{\Sigma x^2} = 1.24/\sqrt{8{,}787.56} = 0.013$ (5)

$t - b/s_b = 0.31/0.013 = 23.846^{**}$ d.f. = 7 (6)

(1) If numbers are large in the original data, calculations can be simplified by subtracting a constant from each number before proceeding with the calculations. In the example 300 was subtracted from each temperature measurement.

(2) Sample regression coefficient

(3) Sample regression equation

(4) Sample standard deviation from regression

(5) Sample standard deviation of regression coefficient

(6) Test of significance of sample regression coefficient[** value significant above 99 percent level].

APPENDIX IV

DETAILED MODIFIED 5-ML BOMB DATA AND STATISTICAL ANALYSES FOR REPEATABILITY AND CORRELATION STUDIES

APPENDIX IV

REPEATABILITY AND CORRELATION OF MODIFIED 5-ML BOMB DATA

With the demonstration that the Modified 5-ml Bomb procedure could recognize the small effects of antioxidants on thermal stability quality, the second phase of the program with the 5-ml Bomb was initiated. This phase consisted of determining the repeatability of the 5-ml Bomb as modified in this program with a wide range of fuels over a considerable period of time and determining the extent of correlation with other thermal stability test methods. The Air Force furnished Phillips Petroleum Company a number of fuels on which threshold failure temperatures had been or would be established by the ASTM-CRC Coker, Research Coker with ambient reservoir or the MINEX Rig. Other fuels which Phillips had evaluated in the ASTM-CRC Coker or were part of the storage stability program were also included. A list of the fuels with the additive contents are shown in Table 39. Graphs of Coker thermal stability tests for each fuel for determining threshold failure temperatures are shown in Figures 25 to 51. At this time Coker data have not been received on some of the fuels which have been evaluated in the Modified 5-ml Bomb.

1. Repeatability Program

Initial plans were to determine the repeatability of the Modified 5-ml Bomb Procedure which is shown in Appendix III by testing the entire group of fuels once in a random order and then repeating the evaluation for a second time, again selecting the order of fuels at random. The entire program was to be conducted by a single operator with a single set of equipment. Because of unforeseen delays in obtaining a number of fuels it was necessary to abandon the plan of testing all fuels once before making the second test on each fuel. Second tests were started in a random order upon the completion of the first test of all the fuels on hand. As new fuels were received they were added to the group to be tested. A stipulation was made that two tests were not to be made consecutively on any fuel. A total of 60 Modified 5-ml Bomb Procedure evaluations have been made on 38 fuels. Four fuels were evaluated in triplicate, 14 in duplicate and the remainder only once to date. Any evaluation in which the sample deviation from regression exceeded 4 was discarded. Six tests were discarded because of excessive sample standard deviation from regression. Detailed data on the 60 tests used in this program are shown in Table 40. Regression analysis of each 5-ml Bomb test was made using the method shown in Appendix III. Calculated temperatures for 0, 10, 15 and 25 light transmittance-loss-levels are shown in Table 32. Also shown are average temperatures for fuels on which multiple determinations were made. In addition the standard deviation from regression, regression coefficient and light transmittance at 350 millimicrons for the fresh fuel are also shown. All Coker threshold failure temperature data which have been received to date are also shown. Using the data for 18 fuels on which multiple tests are available an analysis of variance was made of the temperatures at 0, 10, 15 and 25 light transmittance-loss-levels. A summary of these analyses is shown in Table 33.

From these data it can be concluded with 99 percent confidence that there are differences among the fuels at each of the four loss levels; however, the error mean squares for the various light transmittance-loss-levels are much higher than in the antioxidant studies shown previously in Table 20. The previous program was confined to one base fuel and the same base plus two concentrations of an antioxidant while the second program represented a wide variety of fuels and additives. To investigate the possible cause of the greater variability of results in this program the temperatures for a light transmittance-loss of 25 for each fuel were examined. These data are shown in Table 34. It will be noted that there are very wide differences in threshold failure temperature for some fuels which result in very wide differences in the error mean square and standard deviation values for these fuels. Using Barttlet's test of Homogeneity of Variance(8) it can be concluded with 95 percent confidence that this is more than a chance variation. The standard deviations and mean temperatures for a light-transmittance-loss of 25 are shown graphically in Figure 24 since standard deviation frequently varies with the size of means. Additives in the fuels are also shown. This figure shows no relationship between standard deviation and temperature or additives. An examination of the data with respect to order of tests shows no trend in test severity and it is not believed that this is contributing to poor repeatability. The light transmittance values for the fresh fuel did not indicate improper selection of test fuels. An examination was also made of the data with respect to the presence or absence and type of additives in the fuel tested immediately preceding each test. No trend could be established although this may be a contributing factor and should be investigated further.

A sample of the same base fuel as used in the previous additive studies (BJ62-16-J1 redesignated BJ63-10-G53) was also included in this program. The error mean square for the pair of tests on this fuel at the 25 light transmittance-loss-level compares favorably with that obtained in the previous program (32.00 vs 32.44); however, the calculated temperatures vary at the three light transmittance-loss-levels (314 vs 309 at 0 loss-level, 420 vs 366 at the 15 loss-level and 490 vs 404 at the 25 loss-level). In fact the temperature at the 25 loss-level for this base fuel is higher than for any of the antioxidant blends in the previous program and suggests a possible shift in test severity. The increasing spread of temperatures with an increase in light-transmittance-loss is a result of a difference in the light-transmittance-loss temperature relationship. The average regression coefficient in the current program is 0.14 and in the previous program was 0.27.

In summary it was shown that good repeatability of threshold failure temperature was obtained on one base fuel and the same fuel with two concentrations of an antioxidant; however, when a wide variety of fuels and additives were tested occasional wide variations in threshold failure temperature for a given fuel were obtained. An examination of dates of testing, light transmittance of the fresh fuel samples, and additives in the prior test fuel failed to explain these variations. Further study is needed to identify the factor or factors which cause these variations.

2. Relationship between the Modified 5-ml Bomb and ASTM-CRC Coker

Coker threshold failure temperatures and 5-ml Bomb temperatures for four light transmittance-loss-levels are shown in Table 32. Two fuels (BJ63-10-B75 and BJ64-10-G163) have Coker data from the Research Coker with an ambient reservoir. Fuels BJ64-10-G71 and BJ64-10-G107 are estimated to have ASTM-CRC Coker threshold failure temperatures of 450+ on the basis of 712 and 692 threshold failure temperatures in the SSF Coker. Coker threshold failure temperatures were plotted against temperatures for a light-transmittance-loss of 0, 10, 15, or 25 and data for a 25 unit loss are shown in Figure 4. The 5-ml Bomb data are either single determinations or the average of the points if multiple determinations are available. After an initial plot of the data at each light-transmittance-loss-level it was decided to code the individual points by additive type to determine if this would resolve the wide scatter of the data. In Figure 4 the non-additive fuels are designated by an "x", antioxidants N,N'-disecondary butylparaphenylenediamine (PD), 2,6 ditertiarybutylphenol (26B) and 2,6 ditertiarybutyl-4-methyl phenol (26B4M) are shown by open points and these antioxidants in combination with metal deactivators are shown by solid points. While it is apparent that no relationship exists for all of the data there appears to be a relationship between the 5-ml Bomb data and Coker threshold failure temperatures for non-additive fuels. Linear regression equations were calculated at each of the four light transmittance-loss-levels to permit a calculation of Coker threshold failure temperature from the 5-ml Bomb temperature for the appropriate light-transmittance-loss. The line shown in Figure 4 represents this regression for the non-additive fuels at the 25 loss-level. In Table 35 the Coker threshold failure temperatures that are calculated from the regression equations for the 5-ml Bomb at the various light-transmittance-loss-levels are shown. Also shown are the differences between measured and calculated threshold failure temperatures, sample standard deviations from regression and the regression coefficients. The relationship at the 0 loss level is not significant at a 90 percent confidence level. While the relationships for 10, 15 and 25 loss levels are all significant at the 99+ percent confidence level, the 25 light-loss level gave the smallest sample deviation from regression. It should be pointed out that the sample standard deviation from regression of 26.9°F approaches the generally accepted repeatability of 25°F for the Coker. These data also indicated that the Modified 5-ml Bomb is more sensitive to changes in thermal stability quality than the Coker. A change of 10°F in the 5-ml Bomb is equivalent to a change of only 6°F in Coker temperature. Also on this basis the standard deviation of the 5-ml Bomb Procedure which was shown in Table 33 to be 43.5°F would be equivalent to 25.9°F for the Coker (43.5 x 0.595). These data demonstrate that the Modified 5-ml Bomb Procedure should be a useful tool for screening the thermal stability quality of non-additive fuels.

With respect to the fuels containing additives an examination of Figure 4 will show that the four fuels containing antioxidants all are to the right of the line for non-additive fuels and all fuels (with one exception) which contain a metal deactivator in addition to an antioxidant are to the left of the line for the non-additive fuels. Previously it has been shown that antioxidants increase the threshold failure temperature of a fuel as measured by the Modified 5-ml Bomb. Apparently the 5-ml Bomb recognizes this effect to a greater extent than does the Coker. Likewise the Coker and the 5-ml Bomb appear to recognize the effect of

TABLE 32

MODIFIED 5-ML BOMB FUEL STUDIES

BJ No.	Threshold Failure Temp. F(1)	Fresh Light Trans-mittance 0.35 Microns	Standard Deviation From Regression	Regression Coefficient	Calculated Temperature (F) for Various ΔLT Losses 0	10	15	25
63-10-K23	415	80.0	1.80	0.60	366	382	391	407
63-10-K24	375	99.0	1.59	0.13	309	386	425	502
63-10-K25	380	98.0	1.55	0.10	327	432	485	590
63-10-K25		100.0	1.57	0.13	342	418	455	531
Avg.		99.0			334	425	470	560
63-10-K26	425	80.0	3.04	0.18	359	415	442	498
63-10-K27	355	31.0	1.97	0.04	122	408	551	836
63-10-K28	400	79.0	2.35	0.58	358	375	384	401
63-10-K29	375	93.0	1.87	0.13	340	420	459	539
63-10-K30	380	73.0	1.78	0.74	364	378	384	398
63-10-K31	415	79.3	3.43	0.47	355	377	387	409
63-10-K31		77.0	1.77	0.19	391	443	470	522
Avg.		78.2			373	410	428	466
63-10-K32	435	78.0	1.84	0.27	334	370	389	426
63-10-K33	440	85.0	3.00	0.36	358	385	399	427
63-10-K34	450	87.0	2.29	0.83	366	378	384	396
63-10-K35	450+	73.4	1.55	0.11	354	447	494	587
63-10-K36	450+	96.0	1.86	0.12	310	397	441	528
63-10-K37	440	104.0	1.92	0.13	313	389	427	502
63-10-K38	410	103.0	2.86	0.14	381	454	491	565
63-10-K38		100.0	2.09	0.14	430	502	539	611
Avg.		101.5			406	478	515	588
63-10-K39	365	93.0	2.48	0.22	336	381	404	450
63-10-K39		95.0	2.06	0.15	314	380	413	479
Avg.		94.0			325	380	408	464
64-10-K143	475	103.0	1.27	0.17	305	365	395	454
64-10-K143		102.0	0.93	0.21	430	478	502	549
64-10-K143		102.0	2.19	0.11	349	443	490	583
Avg.		102.3			361	429	462	529

(continued)

(1) ASTM-CRC Fuel Coker

TABLE 32 (Continued)

BJ No.	Threshold Failure Temp. F(1)	Fresh Light Trans-mittance 0.35 Microns	Standard Deviation From Regression	Regression Coefficient	Calculated Temperature (F) for Various ΔLT Losses 0	10	15	25
64-10-K145	425	104.0	1.77	0.10	315	419	471	576
64-10-K145		100.0	1.55	0.16	413	474	505	567
64-10-K145		103.0	0.96	0.11	338	427	471	560
Avg.		102.3			355	440	482	568
64-10-K147	450	102.0	1.47	0.14	391	461	496	566
64-10-K147		101.0	1.83	0.11	350	438	482	570
Avg.		101.5			370	450	489	568
64-10-K148	(4)	90.0	1.30	0.08	305	425	485	606
64-10-K148		93.0	1.24	0.10	291	393	444	546
Avg.		91.5			298	409	464	576
64-10-K164	(4)	63.0	2.83	0.08	458	586	650	778
63-10-B75	500(3)	66.0	1.60	0.10	272	376	428	532
63-10-B75		66.0	2.37	0.05	224	409	502	687
63-10-B75		65.0	3.18	0.07	292	439	512	660
Avg.		65.7			263	408	481	626
63-10-G74	360	99.0	2.15	0.83	365	377	383	395
63-10-G74		100.0	3.26	0.83	358	370	376	388
63-10-G74		100.0	2.49	0.65	364	379	387	402
Avg.		99.7			362	375	382	395
64-10-G71	450+(2)	97.0	1.83	0.09	328	437	492	602
64-10-G71		97.0	1.19	0.12	314	394	434	514
Avg.		97.0			321	416	463	558
64-10-G107	450+(2)	100.0	1.16	0.10	299	404	457	562
64-10-G107		97.0	1.23	0.10	311	414	466	570
Avg.		98.5			305	409	462	566
62-16-J1	400	99.0	1.32	0.14	311	384	421	494
62-16-J1		98.0	0.80	0.15	316	384	418	486
Avg.		98.5			314	384	420	490
63-17-G3	350	85.6	1.52	0.17	274	333	363	423
63-17-G3		84.0	1.12	0.18	284	339	367	422
Avg.		84.8			279	336	365	422

(1) ASTM-CRC Fuel Coker
(2) Estimated from SSF Coker
(3) Research Coker - Ambient Reservoir
(4) Coker data not available.

(Continued)

TABLE 32 (Continued)

BJ No.	Threshold Failure Temp. F(1)	Fresh Light Trans-mittance 0.35 Microns	Standard Deviation From Regression	Regression Coefficient	Calculated Temperature (F) for Various ΔLT Losses 0	10	15	25
64-10-G144	300	62.0	1.98	0.33	320	350	365	395
64-10-G144		62.0	2.39	0.40	312	336	349	374
Avg.		62.0			316	343	357	384
64-10-G162	360	103.0	3.24	0.46	319	341	352	373
64-10-G162		102.0	3.21	0.67	358	373	381	396
Avg.		102.5			338	357	366	384
64-10-G163	435(2)	81.0	1.63	0.12	354	436	477	559
64-10-G163		83.0	2.51	0.22	368	414	437	483
Avg.		82.0			361	425	457	521
64-10-L152	(4)	86.0	2.09	0.12	335	415	455	535
64-10-L154	(4)	104.0	1.82	0.08	337	460	522	646
64-10-L157	(4)	102.0	2.59	0.09	340	454	511	624
64-10-L161	(4)	99.0	1.35	0.10	278	381	433	536
64-10-L-165	(4)	104.0	2.08	0.09	298	406	461	569
64-10-L200	(4)	93.0	1.60	0.13	327	406	445	523
64-10-G166	(4)	105.1	1.85	0.16	315	378	410	473
64-10-G166	(4)	103.0	1.34	0.18	317	374	402	458
Avg.		104.1			316	376	406	466

(1) ASTM-CRC Fuel Coker
(2) Research Coker - Ambient Reservoir
(3) Estimated from SSF Coker
(4) **Coker data not available.**

TABLE 33

ANALYSIS OF VARIANCE OF CALCULATED TEMPERATURES FOR VARIOUS FUELS WITH THE 5-ML BOMB PROCEDURE

Source of Variation	Degrees of Freedom	Mean Square	"F" Ratio
	For Light-Transmittance-Loss = 0		
Fuels	17	3,038.74	3.34(2)
Error	22	910.50	
S.D.(1)		30.2	
	For Light-Transmittance-Loss = 10		
Fuels	17	2,946.94	3.92(2)
Error	22	752.12	
S.D.(1)		27.4	
	For Light-Transmittance-Loss = 15		
Fuels	17	4,984.74	5.21(2)
Error	22	956.13	
S.D.(1)		30.9	
	For Light-Transmittance-Loss = 25		
Fuels	17	13,311.43	7.02(2)
Error	22	1,896.06	
S.D.(1)		43.5	

(1) Standard Deviation

(2) Significant at the 99 percent Confidence Level

TABLE 34

COMPARISON OF VARIATION IN TEMPERATURE FOR A LIGHT TRANSMITTANCE LOSS OF 25 FOR MULTIPLE TESTS ON VARIOUS FUELS

Fuel BJ NO.	Additives	Temperature, °F for ΔLT = 25			d.f.(1)	E.M.S.(2)	S.D.(3)
		Test: 1	2	3			
63-10-K28	PD	590	531		1	1740.50	41.6
63-10-K31	2,6B + MD	409	522		1	6384.50	79.9
63-10-K38	2,6B4M	565	611		1	1058.00	32.5
63-10-K39	None	450	479		1	420.50	20.5
64-10-K143	None	454	549	583	2	4470.33	66.9
64-10-K145	None	576	567	560	2	64.33	8.0
64-10-K147	None	566	570		1	8.00	2.8
64-10-K148	None	606	546		1	1800.00	42.4
63-10-B75	None	532	687	660	2	6856.33	82.8
63-10-G74	None	395	388	402	2	49.00	7.0
64-10-G71	2,6B4M + MD	602	514		1	3872.00	62.2
64-10-G107	PD + MD	562	570		1	32.00	5.7
62-16-J1	None	494	486		1	32.00	5.7
63-17-G3	None	423	422		1	0.50	0.7
64-10-G144	2,6B	395	374		1	220.50	14.8
64-10-G162	None	373	396		1	264.50	16.2
64-10-G163	PD + MD	559	483		1	2888.00	53.7
64-10-G166	None	473	458		1	112.50	10.6

(1) Degrees of Freedom
(2) Error Mean Square
(3) Standard Deviation

metal deactivators in combination with antioxidants in different ways. A detailed study of the effect of metal deactivator in combination with antioxidants has not been made in the 5-ml Bomb; however, one comparison is available. In Table 32, fuel BJ64-10-L200 is fuel BJ64-10-L154 with the addition of 8.0 pounds per 1000 barrels of metal deactivator. In this case the addition of metal deactivator to the fuel reduced the temperature for a light transmittance-loss of 25 by 123°F (646°F to 523°F).

TABLE 35

COMPARISON OF COKER THRESHOLD FAILURE TEMPERATURE PREDICTED BY THE 5-ML BOMB FOR NON-ADDITIVE FUELS

Measured Coker Threshold Failure Temperature, °F	Calculated Coker Threshold Failure Temperature At Light-Transmittance-Loss Levels							
	0		10		15		25	
	Temp.	Δ(1)	Temp.	Δ(1)	Temp.	Δ(1)	Temp.	Δ(1)
375	409	-34	400	-25	406	-31	413	-38
440	410	30	404	36	408	32	413	27
365	409	-44	393	-28	390	-25	390	-25
475	408	67	448	27	442	33	429	46
425	408	17	461	-36	462	-37	452	-27
450	408	42	472	-22	469	-19	452	
500	411	89	425	75	461	39	487	13
360	408	-48	388	-28	364	-4	349	11
400	410	-10	398	2	401	-1	406	-6
350	411	-61	344	6	348	2	365	-15
360	409	-49	368	-8	349	11	343	17
Sample Standard Deviations From Regression	55.1		36.2		28.0		26.9	
Regression Coefficient	-0.033(3)		1.102(2)		0.974(2)		0.595(2)	

(1) Measured Temperature By Coker Minus Calculated From Regression Equation
(2) Significant at 99+ percent Confidence Level
(3) Not Significant at 90 percent Confidence Level

In summary it has been shown that a relationship exists between the threshold failure temperature determined by the Modified 5-ml Bomb and the ASTM-CRC Coker for non-additive fuels. The increase in threshold failure temperature with the addition of antioxidants, which has previously been demonstrated, with the Modified 5-ml Bomb was greater than would have been predicted from the relationship between the 5-ml Bomb and the Coker for non-additive fuels. Data on one fuel showed that the addition of a metal deactivator to a fuel containing an antioxidant reduced threshold failure temperature as measured by the Modified 5-ml Bomb. The fuels tested in this program containing metal deactivator in combination with an antioxidant had lower threshold failure temperatures as measured by the 5-ml Bomb than would have been predicted from the relationship between the 5-ml Bomb and the Coker for non-additive fuels. Further investigations will be needed to define the additive effects and to determine if corrections can be developed for additives to extend the relationship between the Modified 5-ml Bomb and the ASTM-CRC Coker to all fuels.

3. Relationship Between the Modified 5-ml Bomb and MINEX

Another small-scale method for measuring fuel thermal stability is the MINEX test rig(9). This test method uses heat transfer loss in a single tube heat exchanger as a measure of fuel thermal stability quality. Seven fuels have been tested in the 5-ml Bomb on which MINEX threshold failure temperature data are available. In this case the threshold failure temperature is defined as the highest temperature for no loss of "h" (heat transfer coefficient). Data for these fuels are shown in Table 36.

In Figure 5 the MINEX data are plotted versus the temperature for a light-transmittance-loss of 25 in the 5-ml Bomb. Linear regression equations were developed for calculating MINEX ratings from 5-ml Bomb data at 0, 10, 15 and 25 loss-levels. In Table 37 the temperatures from the regression equations and the differences from the MINEX threshold failure temperatures are shown for each of the four light-transmittance-loss-levels. Also shown are sample standard deviations from regression and the regression coefficients.

The standard deviation from regression with a light-transmittance-loss of 25 in the 5-ml Bomb is less than at other loss-levels and provides the best relationship between the 5-ml Bomb and the MINEX. It should be noted that the MINEX and the 5-ml Bomb appear to recognize the presence of additives and additive types more nearly the same than do the 5-ml Bomb and the Coker.

4. Relationship Between the Modified 5-ml Bomb and the SSF Coker

The SSF Coker is being used in a program to evaluate changes in storage stability quality of five JP-6 type fuels as part of this investigation. The SSF Coker will be described in detail in the discussion of the storage program. The five fuels for the storage program have been evaluated by both the Modified 5-ml Bomb and the SSF Coker and a comparison of other ratings are shown in Table 38. Figure 6 shows the relationship of the SSF Coker and the 5-ml Bomb with respect to non-additive and inhibited fuels with metal deactivator. It is apparent that there is a linear relationship among the three non-additive fuels. The two fuels containing antioxidants plus metal deactivator fall to the left of the line as with the ASTM-CRC Coker, indicating that the 5-ml Bomb does not recognize these fuels in the same manner as the Coker. It is of interest to observe the difference in the relationship between the two Cokers and the 5-ml Bomb. With the ASTM-CRC Coker a 375°F temperature for a 25 unit light-loss in the 5-ml Bomb was equivalent to threshold failure temperature of 327°F while with the SSF Coker it was equivalent to 304°F. At 575°F temperature for a 25 unit light-transmittance-loss the equivalent temperature for the ASTM-CRC Coker is 452°F and for the SSF Coker is 560°F indicating that at the low level of thermal stability quality the SSF Coker is more severe than the ASTM-CRC Coker, but for fuels at the upper level of the rating ability of the ASTM-CRC Coker, the SSF Coker is much milder.

TABLE 36

FUELS FOR STUDY OF RELATIONSHIP BETWEEN MODIFIED 5-ML BOMB AND MINEX TEST RIG

Fuel	Temperature For Light-Transmittance-Loss				Highest Temperature For No Loss of "h" in MINEX
	ΔLT = 0	10	15	25	
Kerosene(1)	273(2)	319(2)	342(2)	388(2)	350(1)
JP-6(1)	296(2)	311(2)	325(2)	353(2)	300(1)
BJ64-10-G162	338	357	366	384	405
BJ63-10-G74	362	375	382	395	350
BJ64-10-G163	361	425	457	521	460
BJ64-10-G144	316	343	357	384	300
BJ64-10-K148	298	409	464	576	575

(1) Data from Reference (9)

(2) Previous data using the Standard 5-ml Bomb Procedure

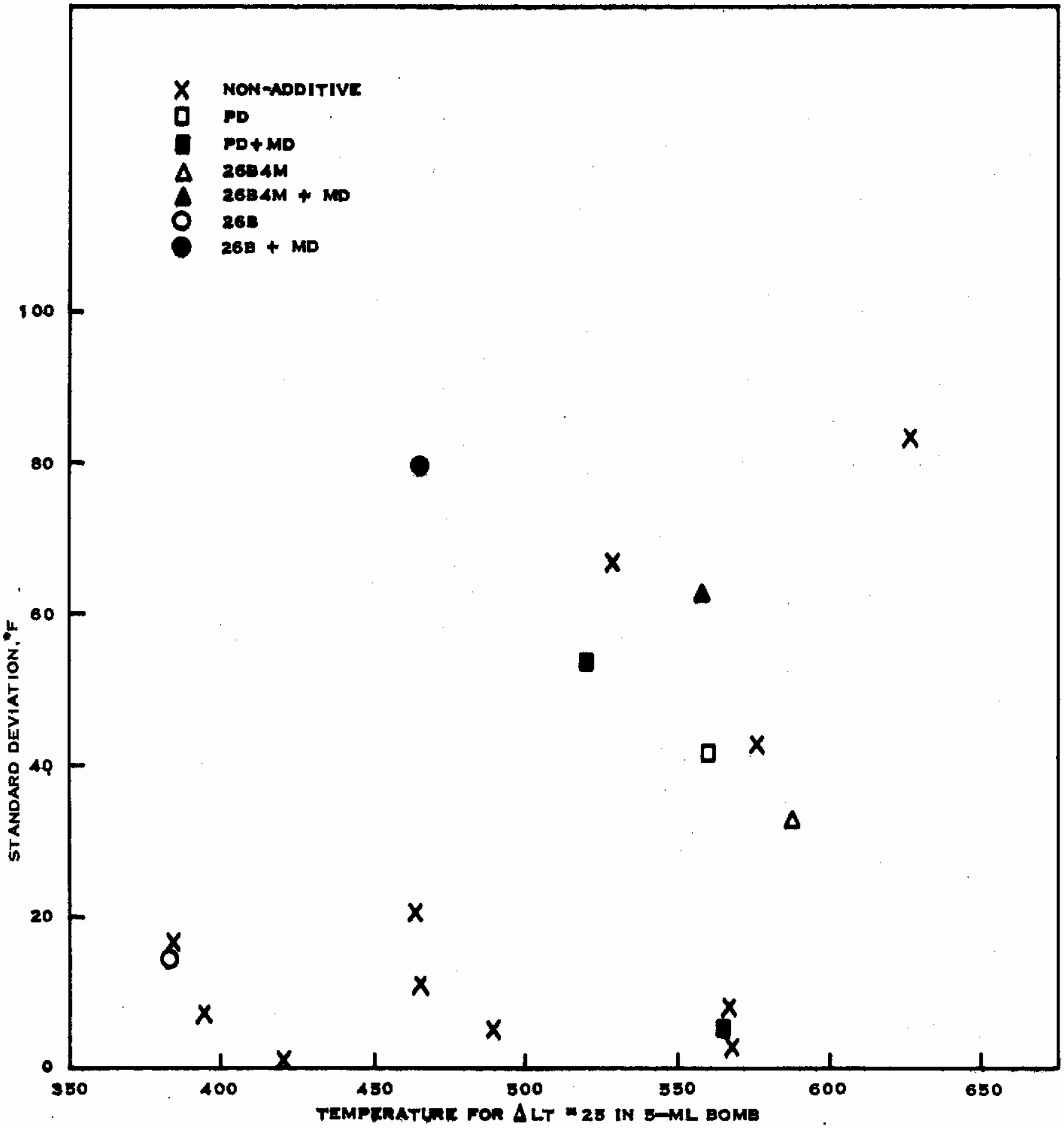

FIGURE 24 VARIATION OF 5-ML BOMB STANDARD DEVIATION WITH TEMPERATURE FOR VARIOUS FUELS

TABLE 37

COMPARISON OF MINEX THRESHOLD FAILURE TEMPERATURE WITH TEMPERATURES CALCULATED FROM THE 5-ML BOMB DATA

MINEX Threshold Failure Temperature, °F	Calculated MINEX Threshold Failure Temperature, °F At Light-Transmittance-Loss Levels							
	0		10		15		25	
	Temp.	Δ(1)	Temp.	Δ(1)	Temp.	Δ(1)	Temp.	Δ(1)
350	379	-29	311	39	322	28	347	3
300	385	-85	296	4	295	5	308	-8
405	396	9	381	24	361	44	342	63
350	402	-52	414	-64	387	-37	354	-4
460	402	58	506	-46	508	-48	492	-32
300	390	-90	355	-55	347	-47	342	-42
575	386	189	476	99	520	55	553	22
Sample Standard Deviation From Regression	108.0		64.9		48.3		38.4	
Regression Coefficient	0.253(4)		1.836(3)		1.616(2)		1.096(2)	

(1) Measured Temperature by MINEX Minus Calculated from Regression Equation

(2) Significant at 99+ percent Confidence Level

(3) Significant at 95 percent Confidence Level

(4) Not Significant at 90 percent Confidence Level

TABLE 38

FUELS FOR STUDY OF RELATIONSHIP BETWEEN MODIFIED 5-ML BOMB AND SSF COKER

Fuel	Temperature °F For Light-Transmittance-Loss of 25 in 5-ml Bomb	Threshold Failure Temperature, °F For SSF Coker
BJ63-10-B75	626	625
BJ63-10-G74	395	332
BJ64-10-G71	558	712
BJ64-10-G107	566	692
BJ64-10-G166	466	425

DETAILED 5-ML BOMB DATA USED IN CORRELATION STUDIES

TABLE 39

DESCRIPTION OF FUELS USED IN 5-ML BOMB REPEATABILITY AND CORRELATION STUDIES

BJ No.		Additives
63-10-K23	SF6-6201	8.0 lb/1000 bbl 26B(1) + 2.0 lb/1000 bbl MD(2)
63-10-K24	SF6-6202	None
63-10-K25	SF6-6203	8.0 lb/1000 bbl PD(3)
63-10-K26	TSF-6204	8.0 lb/1000 bbl PD + 2.0 lb/1000 bbl MD
63-10-K27	TSF-6206	8.0 lb/1000 bbl PD + 2.0 lb/1000 bbl MD
63-10-K28	SF6-6207	8.0 lb/1000 bbl 26B + 2.0 lb/1000 bbl MD
63-10-K29	SF6-6208	3.0 lb/1000 bbl PD
63-10-K30	SF6-6209	8.0 lb/1000 bbl 26B + 2.0 lb/1000 bbl MD
63-10-K31	SF6-6213	8.0 lb/1000 bbl 26B + 2.0 lb/1000 bbl MD
63-10-K32	SF6-6214	8.0 lb/1000 bbl 26B + 2.0 lb/1000 bbl MD
63-10-K33	SF6-6303	8.0 lb/1000 bbl PD + 2.0 lb/1000 bbl MD
63-10-K34	SF6-6304	8.0 lb/1000 bbl PD + 2.0 lb/1000 bbl MD
63-10-K35	TSF-6305	8.0 lb/1000 bbl PD + 2.0 lb/1000 bbl MD
63-10-K36	TSF-6306	15 ppm 26B4M(4) + 5 ppm MD
63-10-K37	SF6-6306	None
63-10-K38	TSF-6307	3.0 lb/1000 bbl 26B4M
63-10-K39	TSF-6312	None
64-10-K143	SF6-6311(5)	None
64-10-K145	SF6-6311(6)	None
64-10-K147	SF6-6311(7)	None
64-10-K148	F-63-18	None
64-10-K164	A	Unknown
63-10-B75	Storage Fuel No. 1	None
63-10-G74	Storage Fuel No. 2	None
64-10-G71	Storage Fuel No. 3	5.0 lb/1000 bbl 26B4M + 2.0 lb/1000 bbl MD
64-10-G107	Storage Fuel No. 4	5.0 lb/1000 bbl PD + 2.0 lb/1000 bbl MD
62-16-J1	B	None
63-17-G3	C	None
64-10-G144	D	3.0 lb/1000 bbl 26B
64-10-G162	E	None
64-10-G163	F	8.0 lb/1000 bbl PD + 2.0 lb/1000 bbl MD
64-10-L152	G	20 ppm 26B4M
64-10-L154	H	20 ppm 26B4M
64-10-L157	J	20 ppm 26B4M
64-10-L161	K	Unknown
64-10-L165	L	20 ppm 26B4M
64-10-L200	M	20 ppm 26B4M + 8.0 lb/1000 bbl MD
64-10-G166	Storage Fuel No. 5	None

(1) 2,6-Ditertiarybutylphenol
(2) N,N'-disalicylidene-1,2-propanediamene
(3) N,N'-disecondary butylparaphenylenediamine
(4) 2,6-Ditertiarybutyl-4-methylphenol

(5) Pseudo Fresh
(6) 6 Months Ambient Storage
(7) 20 Weeks Ambient Storage

TABLE 40

MODIFIED PHILLIPS STATIC 5-Ml BOMB JET FUEL THERMAL STABILITY TEST

METHOD DATA USED FOR CORRELATION STUDIES

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns Before	After	Loss
BJ63-10-K23	L109	400	80.0	56.0	24.0
		414		49.7	30.3
		380		72.0	8.0
		364		80.0	0.0
		372		76.7	3.3
		416		52.0	28.0
		380		73.0	7.0
		420		48.0	32.0
		372		76.0	4.0
BJ63-10-K24	L117	411	99.0	88.0	11.0
		380		89.0	10.0
		332		96.0	3.0
		517		73.7	25.3
		566		66.7	32.3
		331		96.0	3.0
		550		65.0	34.0
		327		96.0	3.0
		557		66.3	32.7
BJ63-10-K25	L120	407	98.0	90.5	7.5
		450		86.3	11.7
		519		79.0	19.0
		575		75.0	23.0
		365		94.3	3.7
		575		72.0	26.0
		367		94.0	4.0
		572		77.0	21.0
		372		94.3	3.7
BJ63-10-K25	L138	540	100.0	76.3	23.7
		581		69.3	30.7
		440		86.7	13.3
		380		96.0	4.0
		493		81.0	19.0
		384		94.3	5.7
		600		65.0	35.0
		388		92.0	8.0
		592		65.0	35.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ63-10-K26	L111	402	80.0	69.7	10.3
		449		59.0	21.0
		472		59.8	20.2
		379		77.0	3.0
		455		64.3	15.7
		489		54.0	26.0
		390		76.0	4.0
		490		61.0	19.0
		384		77.0	3.0
BJ63-10-K27	L125	402	31.0	20.8	10.2
		300		26.0	5.0
		553		13.3	17.7
		657		10.7	20.3
		785		8.7	22.3
		300		27.0	4.0
		463		17.0	14.0
		307		24.3	6.7
		792		10.0	21.0
BJ63-10-K28	L117	404	79.0	51.7	27.3
		386		60.0	19.0
		362		74.5	4.5
		398		53.8	25.2
		372		73.0	6.0
		405		52.2	26.8
		368		73.3	5.7
		405		54.0	25.0
		379		70.7	8.3
BJ63-10-K29	L124	548	93.0	68.2	24.8
		569		62.5	30.5
		373		88.8	4.2
		445		79.0	14.0
		554		67.0	26.0
		502		76.0	17.0
		373		87.7	5.3
		564		62.5	30.5
		375		88.7	4.3

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ63-10-K30	L110	404	73.0	40.7	32.3
		385		55.7	17.3
		396		48.5	24.5
		373		66.7	6.3
		400		46.7	26.3
		370		68.2	4.8
		405		45.0	28.0
		377		65.0	8.0
		405		44.3	28.7
BJ63-10-K31	L120	411	79.3	54.8	24.5
		369		75.3	4.0
		381		70.3	9.0
		390		55.7	23.6
		417		51.3	28.0
		362		75.0	4.3
		412		55.3	24.0
		364		76.3	3.0
		421		46.3	33.0
BJ63-10-K31	L137	436	77.0	70.7	7.0
		466		64.7	12.3
		525		49.7	27.3
		483		63.7	13.3
		553		48.0	29.0
		582		42.0	35.0
		434		70.0	7.0
		577		45.0	32.0
		436		70.0	7.0
BJ63-10-K32	L126	448	78.0	47.8	30.2
		406		60.7	17.3
		361		71.5	6.5
		381		61.5	16.5
		423		53.5	24.5
		437		50.3	27.7
		362		71.0	7.0
		438		48.0	30.0
		360		71.0	7.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns Before	After	Loss
BJ63-10-K33	L123	436	85.0	56.2	28.8
		401		65.3	19.7
		370		81.0	4.0
		391		69.0	16.0
		448		50.2	34.8
		372		81.8	3.2
		450		55.0	30.0
		375		81.7	3.3
		452		52.0	33.0
BJ63-10-K34	L110	403	87.0	57.0	30.0
		381		75.0	12.0
		395		61.7	25.3
		368		82.0	5.0
		390		66.2	20.8
		374		83.5	3.5
		404		57.0	30.0
		375		82.0	5.0
		396		60.0	27.0
BJ63-10-K35	L124	396	73.4	68.0	5.4
		470		59.0	14.4
		552		53.7	19.7
		597		45.3	28.1
		400		69.0	4.4
		595		47.0	26.4
		525		57.0	16.4
		392		69.7	3.7
		582		50.0	23.4
BJ63-10-K36	L113	573	96.0	67.7	28.3
		410		83.0	13.0
		363		92.0	4.0
		478		74.3	21.7
		363		88.7	7.3
		573		64.0	32.0
		367		90.7	5.3
		510		74.3	21.7
		566		67.2	28.8

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ63-10-K37	L121	519	104.0	75.0	29.0
		472		86.7	17.3
		365		98.0	6.0
		413		91.7	12.3
		545		71.5	32.5
		341		99.0	5.0
		537		75.3	28.7
		349		98.0	6.0
		546		73.3	30.7
BJ63-10-K38	L127	445	103.0	97.0	6.0
		527		85.3	17.7
		588		71.0	32.0
		568		77.0	26.0
		498		91.7	11.3
		580		74.3	28.7
		436		95.7	7.3
		580		75.7	27.3
		447		96.0	7.0
		404		99.0	4.0
		314		101.0	2.0
		274		103.0	0.0
		359		101.0	2.0
		427		97.0	6.0
BJ63-10-K38	L138	495	100.0	92.3	7.7
		597		79.7	20.3
		671		65.0	35.0
		620		75.0	25.0
		547		86.0	14.0
		657		69.0	31.0
		491		89.0	11.0
		672		65.0	35.0
		497		89.0	11.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ63-10-K39	L112	401	93.0	75.7	17.3
		418		72.3	20.7
		384		81.0	12.0
		341		92.7	0.3
		362		88.0	5.0
		430		71.3	21.7
		438		75.0	18.0
		453		68.0	25.0
		357		90.0	3.0
		450		58.0	35.0
BJ63-10-K39	L137	466	95.0	73.0	22.0
		508		67.3	27.7
		340		92.3	2.7
		362		88.0	7.0
		422		74.0	21.0
		537		62.0	33.0
		335		92.0	3.0
		516		63.0	32.0
		336		92.0	3.0
BJ64-10-K143	L126	441	103.0	80.0	23.0
		483		73.7	29.3
		396		88.0	15.0
		364		95.7	7.3
		330		99.0	4.0
		476		74.5	28.5
		341		96.3	6.7
		478		73.0	30.0
		337		96.0	7.0
BJ64-10-K143	L135	490	102.0	88.0	14.0
		582		75.7	26.3
		438		95.0	7.0
		545		78.7	23.3
		530		85.0	17.0
		582		74.0	28.0
		443		93.3	8.7
		585		73.0	29.0
		445		95.0	7.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

		Temp.	Light Transmittance,* 0.35 Microns		
Fuels	Log No.	°F	Before	After	Loss
BJ64-10-K143	L142	575	102.0	81.0	21.0
		368		99.7	2.3
		641		73.3	28.7
		500		86.7	15.3
		443		91.3	10.7
		382		99.0	3.0
		643		67.0	35.0
		398		96.0	6.0
		640		69.3	32.7
BJ64-10-K145	L131	489	104.0	90.7	13.3
		559		82.3	21.7
		619		74.3	29.7
		434		93.0	11.0
		388		97.3	6.7
		345		100.0	4.0
		620		73.3	30.7
		353		99.0	5.0
		603		75.0	29.0
BJ64-10-K145	L136	465	100.0	91.3	8.7
		515		84.3	15.7
		575		74.7	25.3
		583		73.0	27.0
		431		96.0	4.0
		591		71.0	29.0
		440		95.0	5.0
		587		70.0	30.0
		445		96.0	4.0
BJ64-10-K145	L141	598	103.0	73.0	30.0
		451		90.0	13.0
		387		97.7	5.3
		494		86.7	16.3
		539		79.0	24.0
		590		74.3	28.7
		371		99.7	3.3
		589		76.0	27.0
		385		97.0	6.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ64-10-K147	L141	564	101.0	78.7	22.3
		593		76.0	25.0
		445		89.3	11.7
		419		93.0	8.0
		490		86.0	15.0
		408		94.3	6.7
		598		70.0	31.0
		413		94.0	7.0
		590		72.0	29.0
BJ64-10-K148	L144	500	90.0	73.0	17.0
		578		67.7	22.3
		348		87.0	3.0
		432		77.0	13.0
		630		62.0	28.0
		351		87.0	3.0
		626		65.0	25.0
		347		87.0	3.0
		632		63.0	28.0
BJ64-10-K148	L145	364	93.0	85.0	8.0
		512		73.0	20.0
		532		67.0	26.0
		425		80.0	13.0
		580		64.7	28.3
		580		65.0	28.0
		360		86.0	7.0
		574		66.0	27.0
		359		87.3	5.7
BJ64-10-K164	L143	578	63.0	54.0	9.0
		622		50.3	12.7
		700		47.3	15.7
		788		34.0	29.0
		741		45.7	17.3
		778		38.0	25.0
		508		58.0	5.0
		802		33.0	30.0
		507		58.0	5.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ63-10-B75	L111	514	66.0	43.3	22.7
		392		54.0	12.0
		488		45.0	21.0
		430		50.7	15.3
		535		38.3	27.7
		385		54.0	12.0
		360		57.0	9.0
		540		43.0	23.0
		324		62.5	3.5
BJ63-10-B75	L116	519	66.0	47.7	18.3
		411		57.0	9.0
		319		60.5	5.5
		605		46.0	20.0
		718		36.7	29.3
		669		45.3	20.7
		670		39.0	27.0
		701		42.3	23.7
		541		50.0	16.0
BJ63-10-B75	L118	632	65.0	46.3	18.7
		325		62.0	3.0
		743		30.0	35.0
		407		56.8	8.2
		512		52.0	13.0
		706		38.8	26.2
		730		31.0	34.0
		328		61.5	3.5
		738		37.0	28.0
BJ63-10-G74	L129	387	99.0	82.0	17.0
		368		93.7	5.3
		396		74.0	25.0
		402		67.7	31.3
		373		94.0	5.0
		380		86.7	12.3
		399		72.0	27.0
		375		91.0	8.0
		396		69.0	30.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ64-10-G107	L129	386	100.0	92.7	7.3
		421		86.7	13.3
		470		83.3	16.7
		540		78.7	21.3
		590		72.0	28.0
		354		95.0	5.0
		591		71.0	29.0
		348		95.3	4.7
		594		73.0	27.0
BJ64-10-G107	L133	370	97.0	90.7	6.3
		464		80.0	17.0
		570		71.0	26.0
		356		93.0	4.0
		587		72.0	25.0
		395		90.0	7.0
		602		69.0	28.0
		357		93.0	4.0
		600		69.0	28.0
BJ62-16-J1	L127	422	99.0	85.7	13.3
		488		74.7	24.3
		530		69.7	29.3
		361		93.3	5.7
		318		97.0	2.0
		465		76.0	23.0
		536		69.0	30.0
		355		93.0	6.0
		536		67.0	32.0
BJ62-16-J1	L134	390	98.0	88.0	10.0
		462		77.0	21.0
		507		71.0	27.0
		345		94.0	4.0
		523		67.0	31.0
		342		94.0	4.0
		534		65.0	33.0
		343		93.0	5.0
		527		67.0	31.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ63-17-G3	L122	402	85.6	67.3	18.3
		450		55.2	30.4
		368		70.8	14.8
		301		80.0	5.6
		306		80.0	5.6
		331		76.3	9.3
		448		54.8	30.8
		308		80.0	5.6
		448		56.0	29.6
BJ63-17-G3	L132	473	84.0	50.0	34.0
		365		69.0	15.0
		305		80.0	4.0
		405		63.0	21.0
		453		55.0	29.0
		300		80.0	4.0
		470		50.0	34.0
		307		81.0	3.0
		463		50.0	34.0
BJ64-10-G144	L130	413	62.0	34.5	27.5
		376		40.0	22.0
		331		58.3	3.7
		345		53.7	8.3
		361		49.5	12.5
		409		30.8	31.2
		333		58.3	3.7
		409		32.0	30.0
		330		58.7	3.3
BJ64-10-G144	L133	376	62.0	36.0	26.0
		388		31.7	30.3
		355		49.0	13.0
		331		57.0	5.0
		320		58.0	4.0
		382		32.0	30.0
		316		58.0	4.0
		382		32.0	30.0
		319		58.0	4.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ64-10-G162	L139	357	103.0	87.7	15.3
		367		77.7	25.3
		330		97.0	6.0
		368		84.0	19.0
		350		89.0	14.0
		382		69.3	33.7
		325		98.0	5.0
		367		85.0	18.0
		332		98.0	5.0
BJ64-10-G162	L147	408	102.0	67.0	35.0
		376		92.0	10.0
		362		96.0	6.0
		385		90.7	11.3
		389		83.0	19.0
		393		78.0	24.0
		403		69.7	32.3
		364		97.0	5.0
		397		76.0	26.0
BJ64-10-G163	L144	459	81.0	65.7	15.3
		528		58.3	22.7
		584		53.0	28.0
		416		74.0	7.0
		420		72.0	9.0
		578		55.0	26.0
		392		79.0	2.0
		588		53.0	28.0
		417		73.0	8.0
BJ64-10-G163	L146	435	83.0	68.0	15.0
		391		77.3	5.7
		503		53.0	30.0
		482		56.0	27.0
		402		58.7	24.3
		392		80.0	3.0
		508		54.0	29.0
		400		77.0	6.0
		501		58.0	25.0

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ64-10-L152	L140	394	86.0	80.0	6.0
		453		71.7	14.3
		518		65.7	20.3
		593		54.7	31.3
		547		57.0	29.0
		388		78.3	7.7
		592		51.0	35.0
		392		78.0	8.0
		592		56.0	30.0
BJ64-10-L154	L142	540	104.0	89.3	14.7
		637		80.3	23.7
		415		96.7	7.3
		360		103.0	1.0
		664		74.0	30.0
		485		93.0	11.0
		660		79.0	25.0
		415		96.0	8.0
		680		77.0	27.0
BJ64-10-L157	L139	348	102.0	100.0	2.0
		666		75.0	27.0
		462		95.0	7.0
		563		86.0	16.0
		633		80.7	21.3
		709		68.7	33.3
		466		95.7	6.3
		709		70.0	32.0
		462		90.7	11.3
BJ64-10-L161	L143	500	99.0	78.3	20.7
		550		74.0	25.0
		327		95.7	3.3
		367		91.3	7.7
		424		82.7	16.3
		542		72.0	27.0
		322		94.0	5.0
		548		73.0	26.0
		327		94.0	5.0

* Bausch-Lamb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns Before	 After	 Loss
BJ63-10-G74	L132	392	100.0	69.0	31.0
		382		81.0	19.0
		373		92.0	8.0
		357		97.0	3.0
		387		79.0	21.0
		360		97.0	3.0
		385		76.0	24.0
		367		94.0	6.0
		394		67.0	33.0
BJ63-10-G74	L136	380	100.0	90.7	9.3
		407		70.0	30.0
		374		93.7	6.3
		394		80.0	20.0
		400		82.0	18.0
		405		72.0	28.0
		374		94.0	6.0
		408		70.0	30.0
		370		94.0	6.0
BJ64-10-G71	L131	371	97.0	90.0	7.0
		440		86.7	10.3
		509		83.7	13.3
		572		75.7	21.3
		688		63.0	34.0
		367		93.7	3.3
		669		65.0	32.0
		709		62.3	34.7
		368		94.0	3.0
BJ64-10-G71	L134	378	97.1	89.7	7.4
		500		74.0	23.1
		590		62.0	35.1
		438		80.0	17.1
		570		67.0	30.1
		380		90.0	7.1
		570		65.0	32.1
		380		88.0	9.1
		579		63.0	34.1

* Bausch-Lomb Spectronic 20,
Isooctane = 100

(Continued)

TABLE 40 (Cont'd)

Fuels	Log No.	Temp. °F	Light Transmittance,* 0.35 Microns		
			Before	After	Loss
BJ64-10-L165	L140	521	104.0	83.0	21.0
		372		96.0	8.0
		475		91.3	12.7
		595		74.0	30.0
		445		92.7	11.3
		563		80.0	24.0
		419		93.0	11.0
		575		78.0	26.0
		311		101.0	3.0
BJ64-10-L200	L146	490	93.0	71.3	21.7
		552		64.0	29.0
		455		74.0	19.0
		415		80.7	12.3
		372		89.0	4.0
		552		64.3	28.7
		374		87.0	6.0
		548		67.0	26.0
		374		88.0	5.0
BJ64-10-G166	L148	459	105.1	83.7	21.4
		511		76.7	28.4
		372		97.0	8.1
		349		101.0	4.1
		407		88.0	17.1
		500		74.0	31.1
		358		99.0	6.1
		506		74.0	31.1
		358		97.0	8.1
BJ64-10-G166	L149	358	103.0	95.7	7.3
		458		78.3	24.7
		503		72.3	30.7
		485		74.3	28.7
		425		84.3	18.7
		358		95.3	7.7
		502		68.0	35.0
		358		96.0	7.0
		498		70.0	33.0
BJ64-10-K147	L135	490	102.0	88.0	14.0
		582		75.7	26.3
		438		95.0	7.0
		545		78.7	23.3
		530		85.0	17.0
		582		74.0	28.0
		443		93.3	8.7
		585		73.0	29.0
		445		95.0	7.0

*Bausch & Lomb Spectronic 20
Isooctane = 100

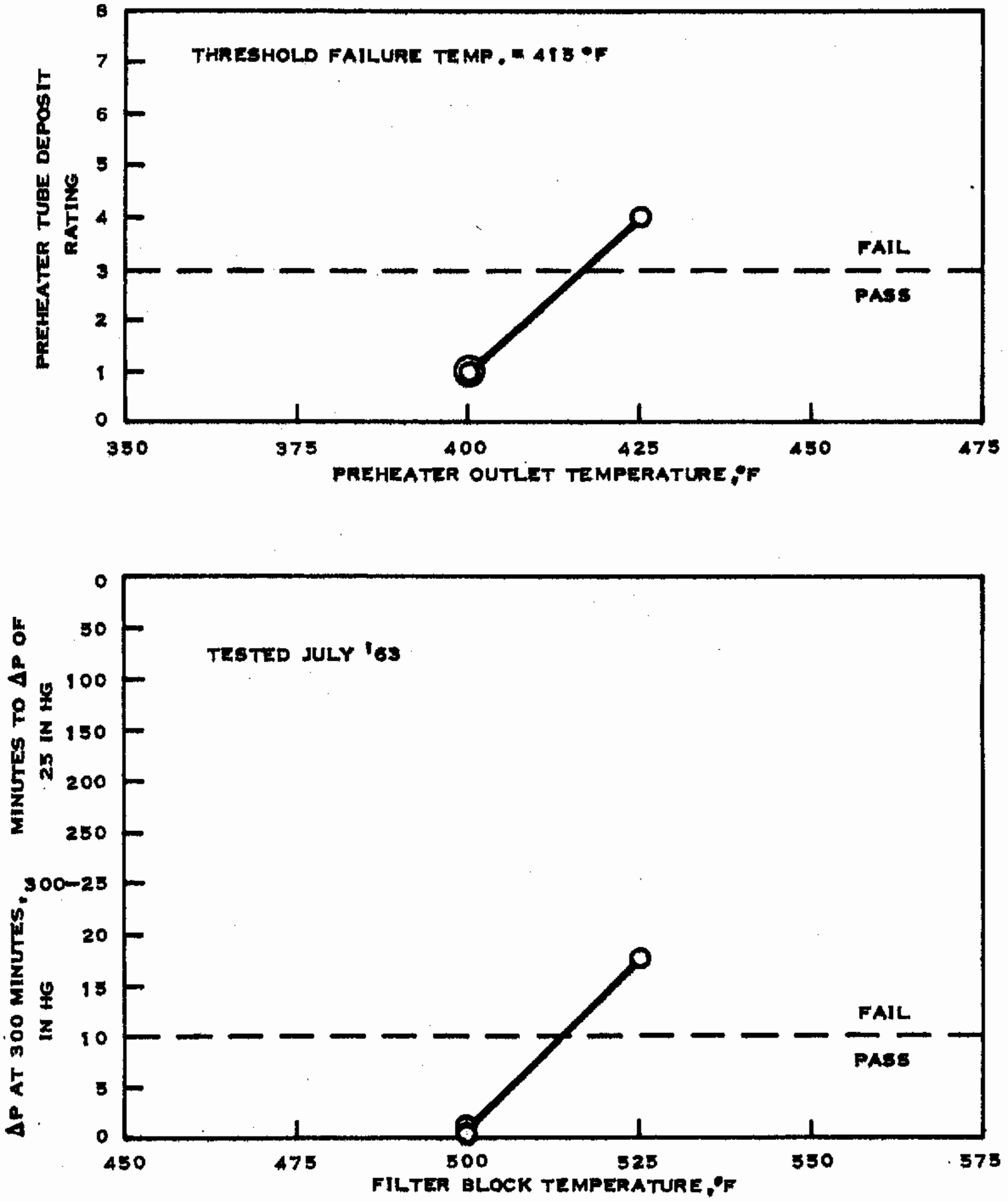

FIGURE 25 THERMAL STABILITY OF BJ63-10-K23 AS DETERMINED BY ASTM-CRC FUEL COKER

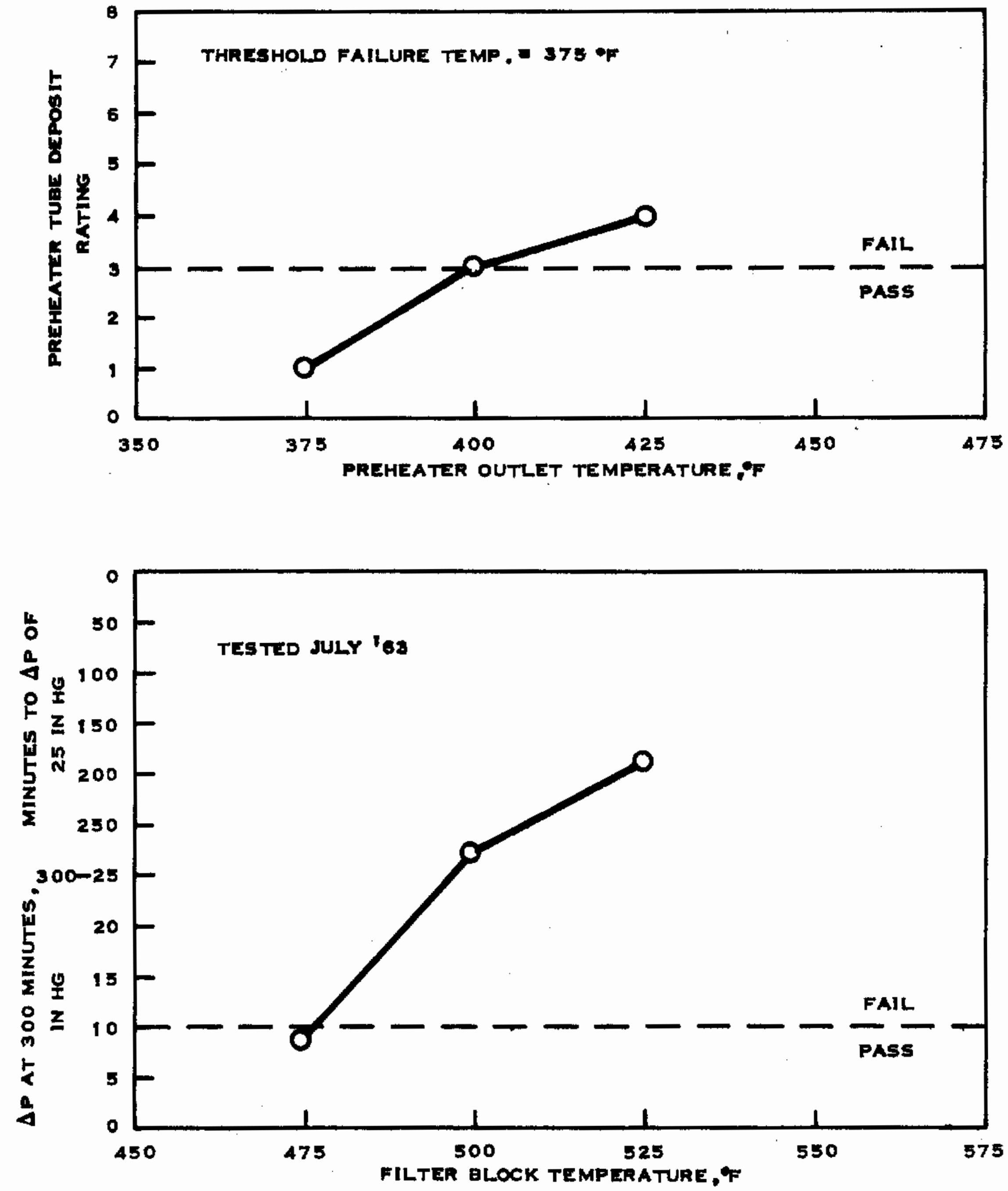

FIGURE 26 THERMAL STABILITY OF BJ63-10-K24 AS DETERMINED BY ASTM-CRC FUEL COKER

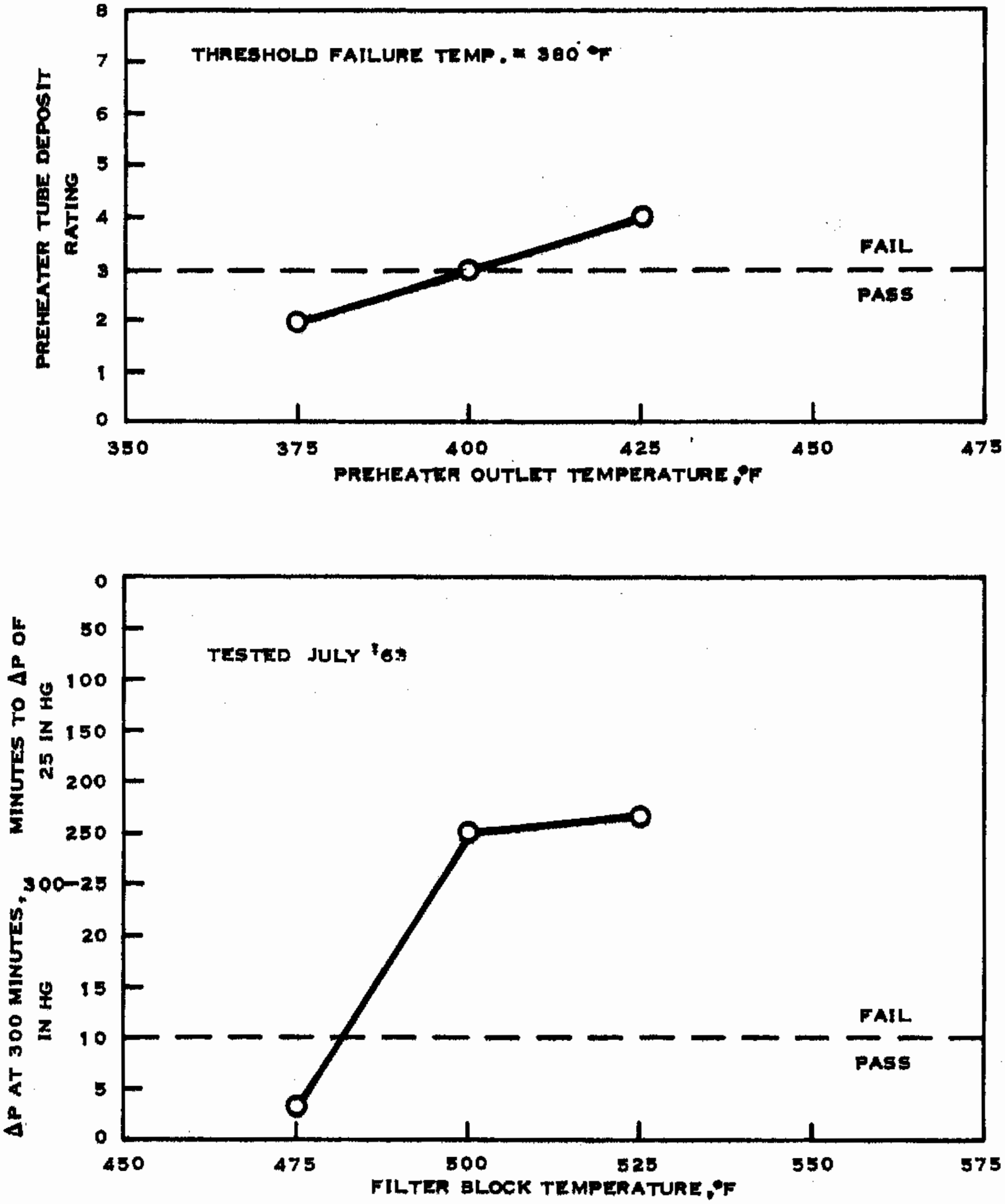

FIGURE 27 THERMAL STABILITY OF BJ63-10-K25 AS DETERMINED BY ASTM-CRC FUEL COKER.

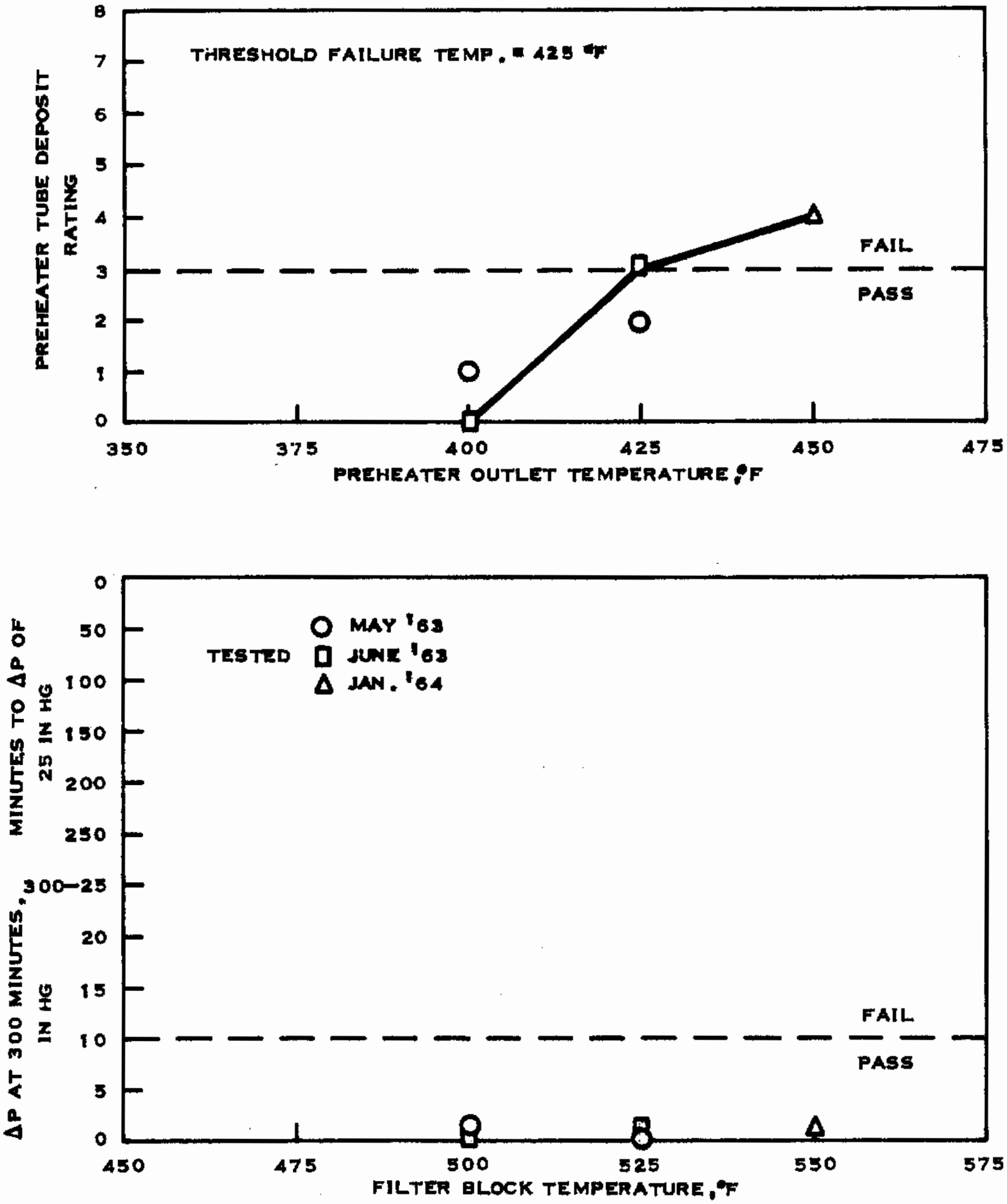

FIGURE 28 THERMAL STABILITY OF BJ63-10-K26 AS DETERMINED BY ASTM-CRC FUEL COKER

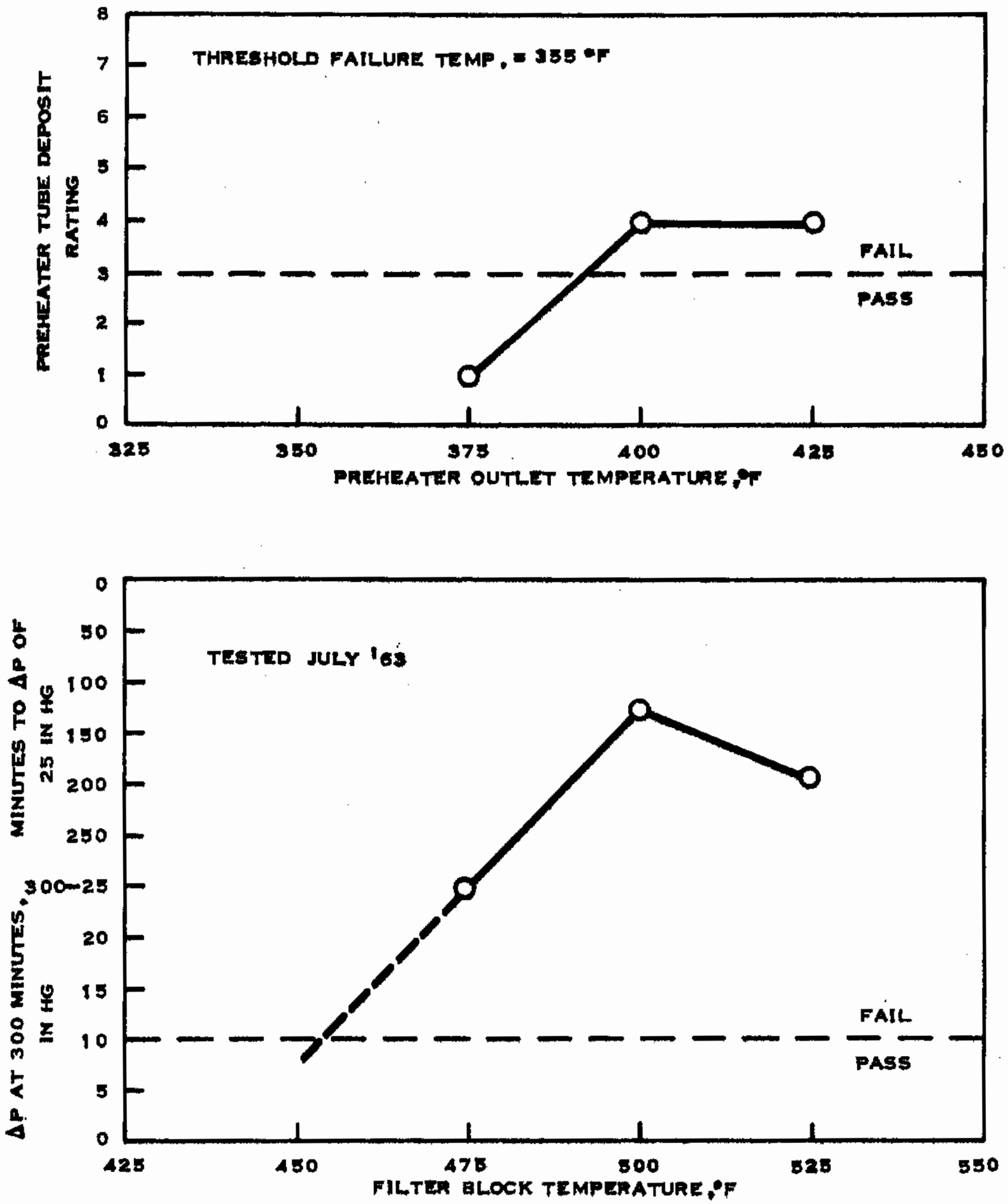

FIGURE 29 THERMAL STABILITY OF BJ63-10-K27 AS DETERMINED BY ASTM-CRC FUEL COKER

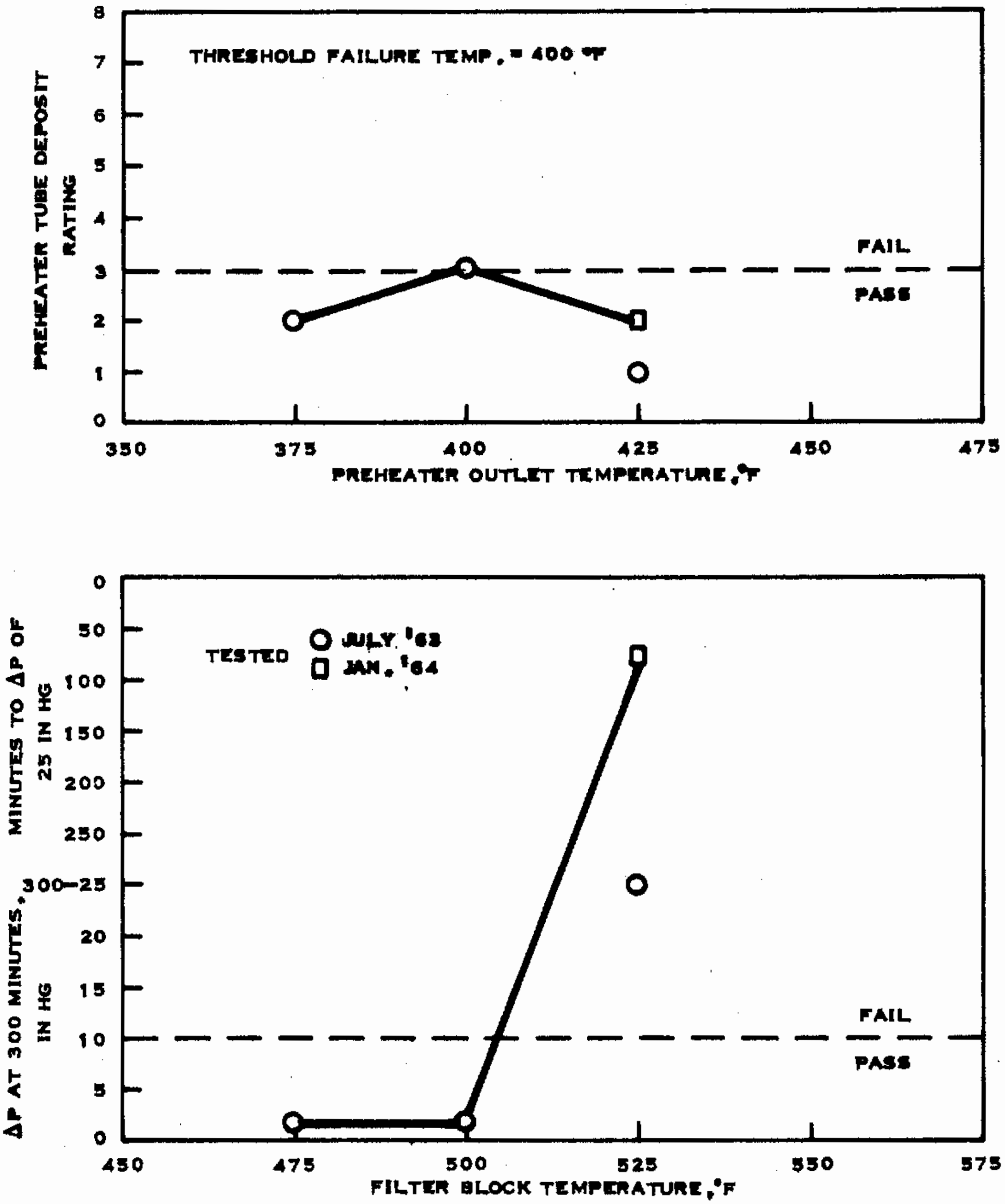

FIGURE 30. THERMAL STABILITY OF BJ63-10-K28 AS DETERMINED BY ASTM-CRC FUEL COKER

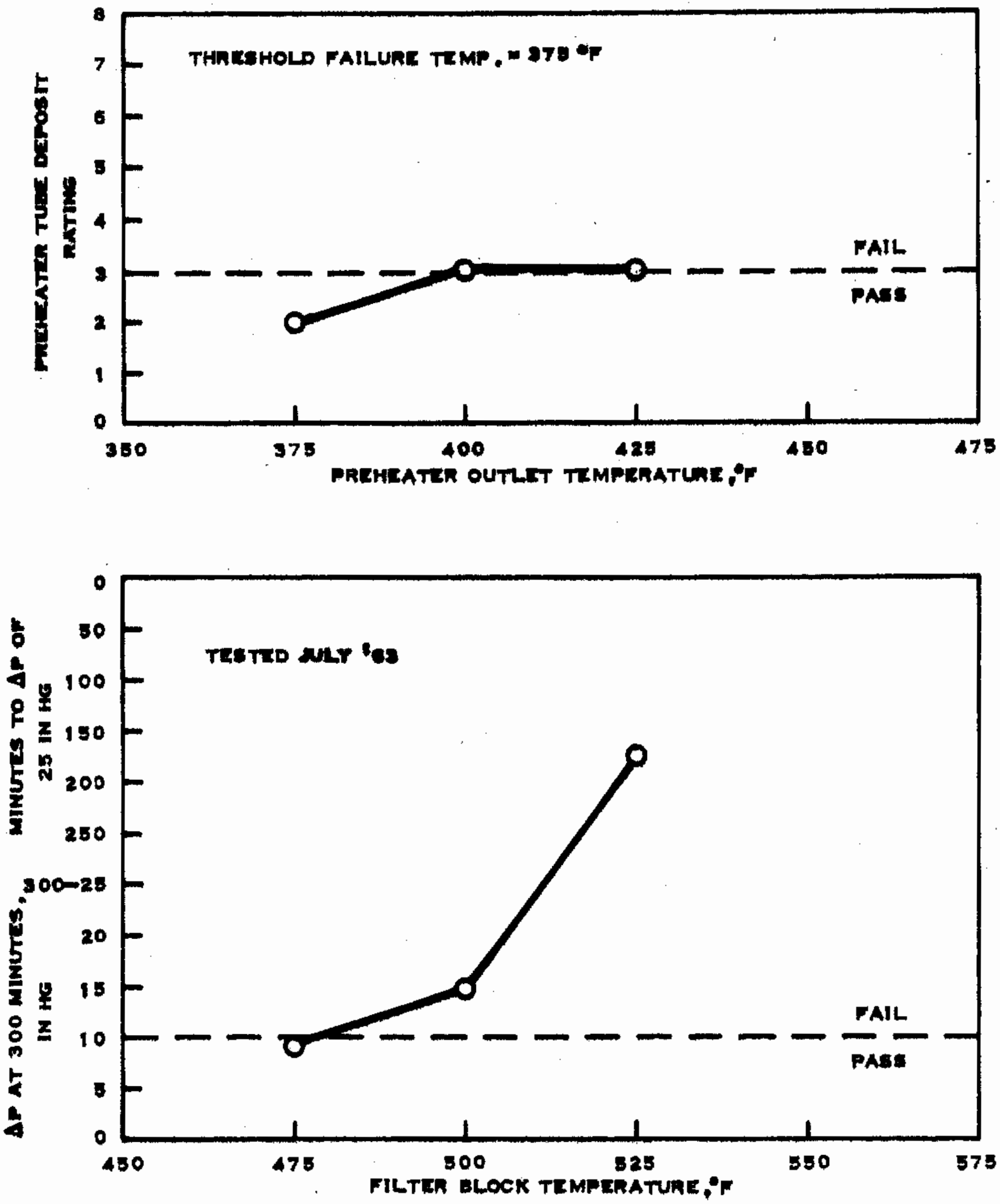

FIGURE 31 THERMAL STABILITY OF BJ63-10-K29 AS DETERMINED BY ASTM-CRC FUEL COKER.

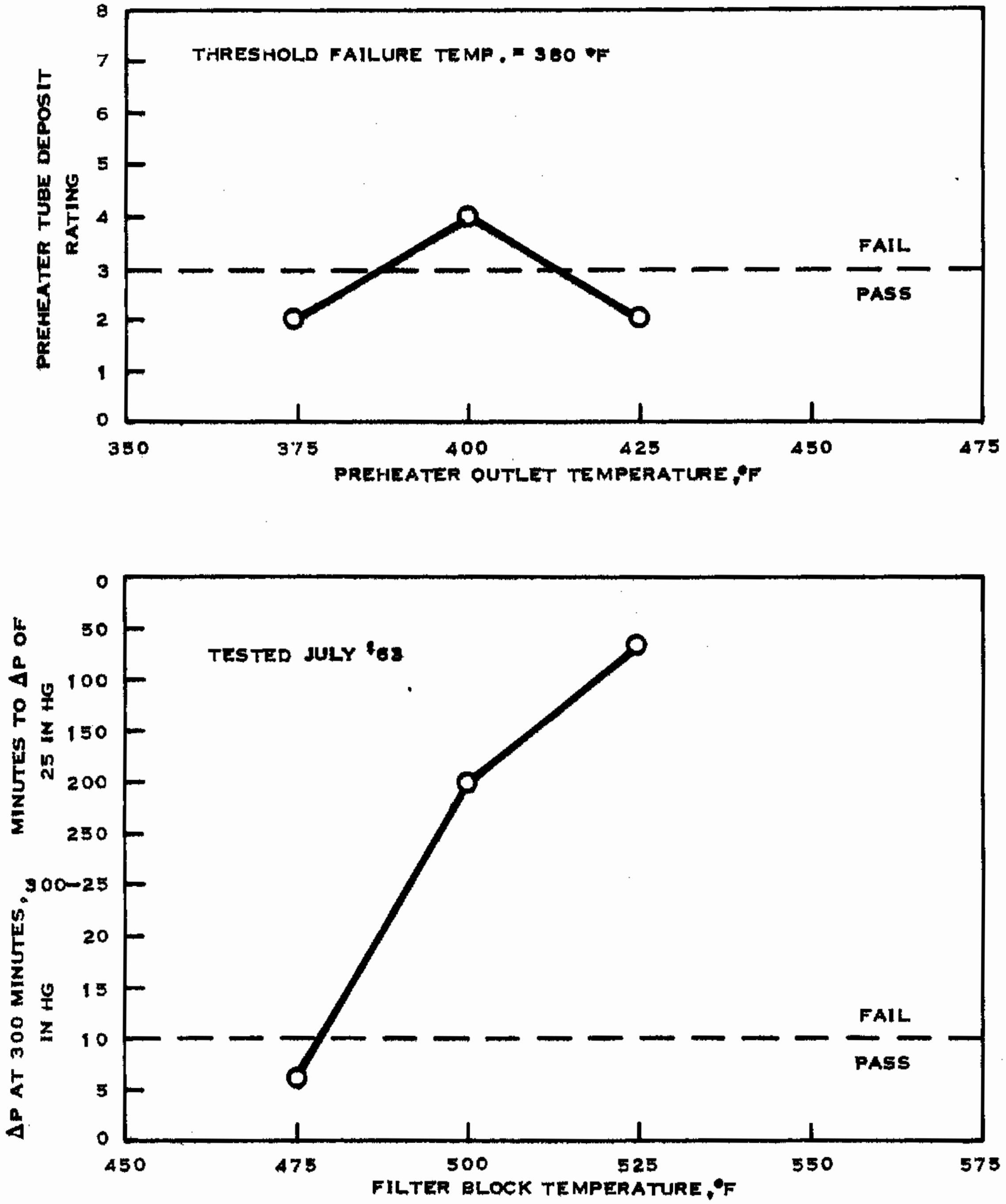

FIGURE 32 THERMAL STABILITY OF BJ63-10-K30 AS DETERMINED BY ASTM-CRC FUEL COKER

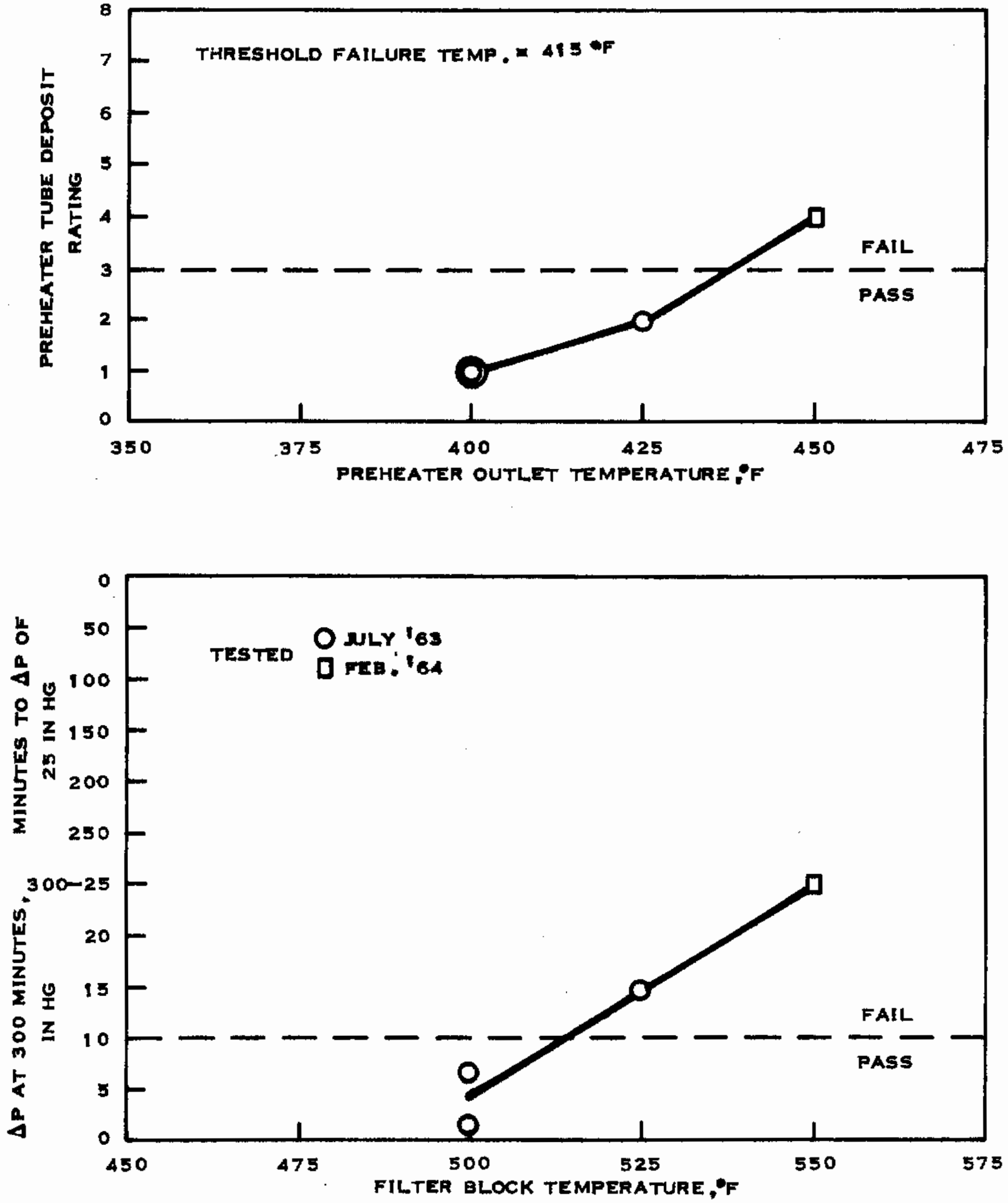

FIGURE 33 THERMAL STABILITY OF BJ63-10-K31 AS DETERMINED BY ASTM-CRC FUEL COKER

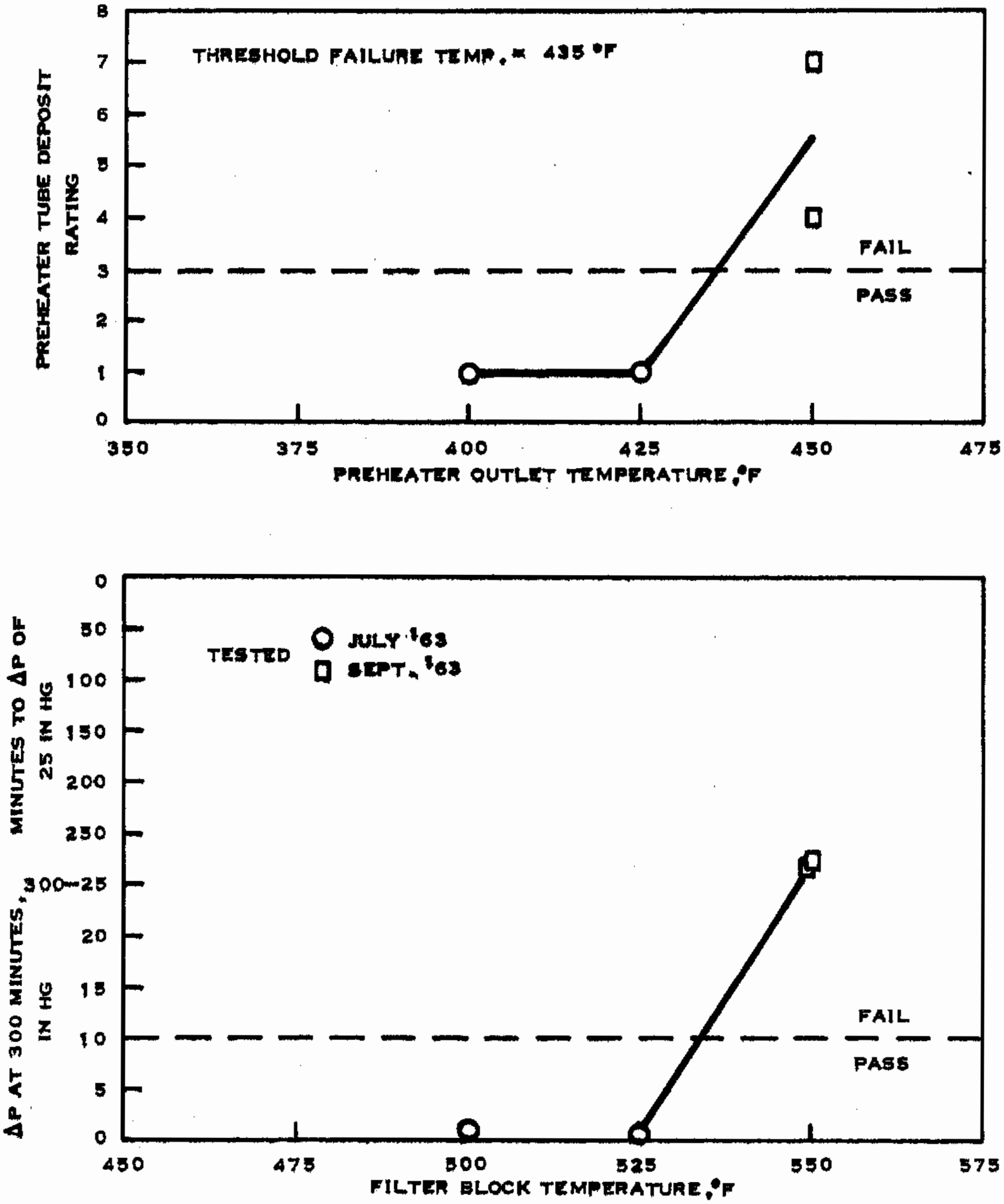

FIGURE 34 THERMAL STABILITY OF BJ63-10-K32 AS DETERMINED BY ASTM-CRC FUEL COKER.

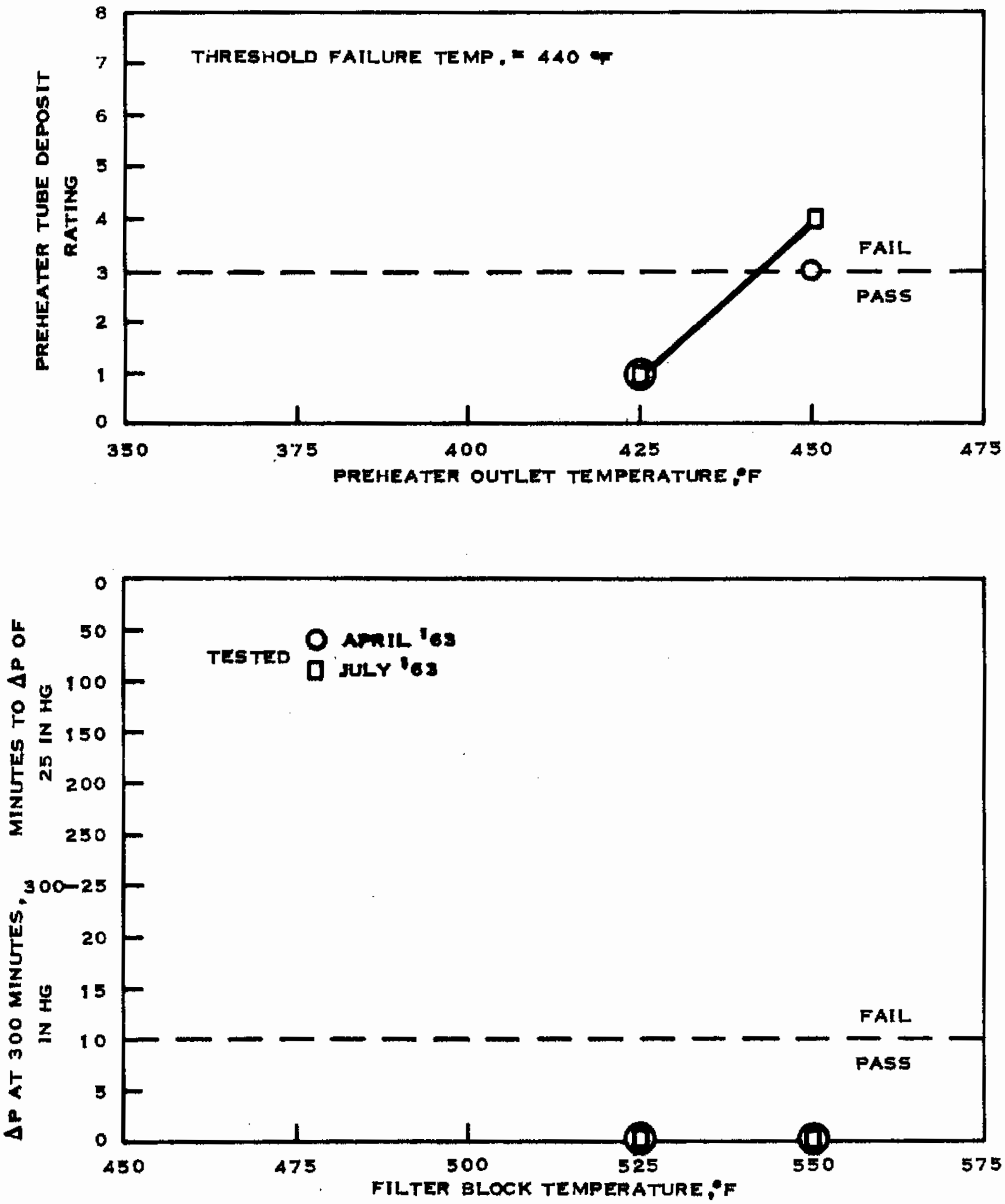

FIGURE 35 THERMAL STABILITY OF BJ63-10-K33 AS DETERMINED BY ASTM-CRC FUEL COKER

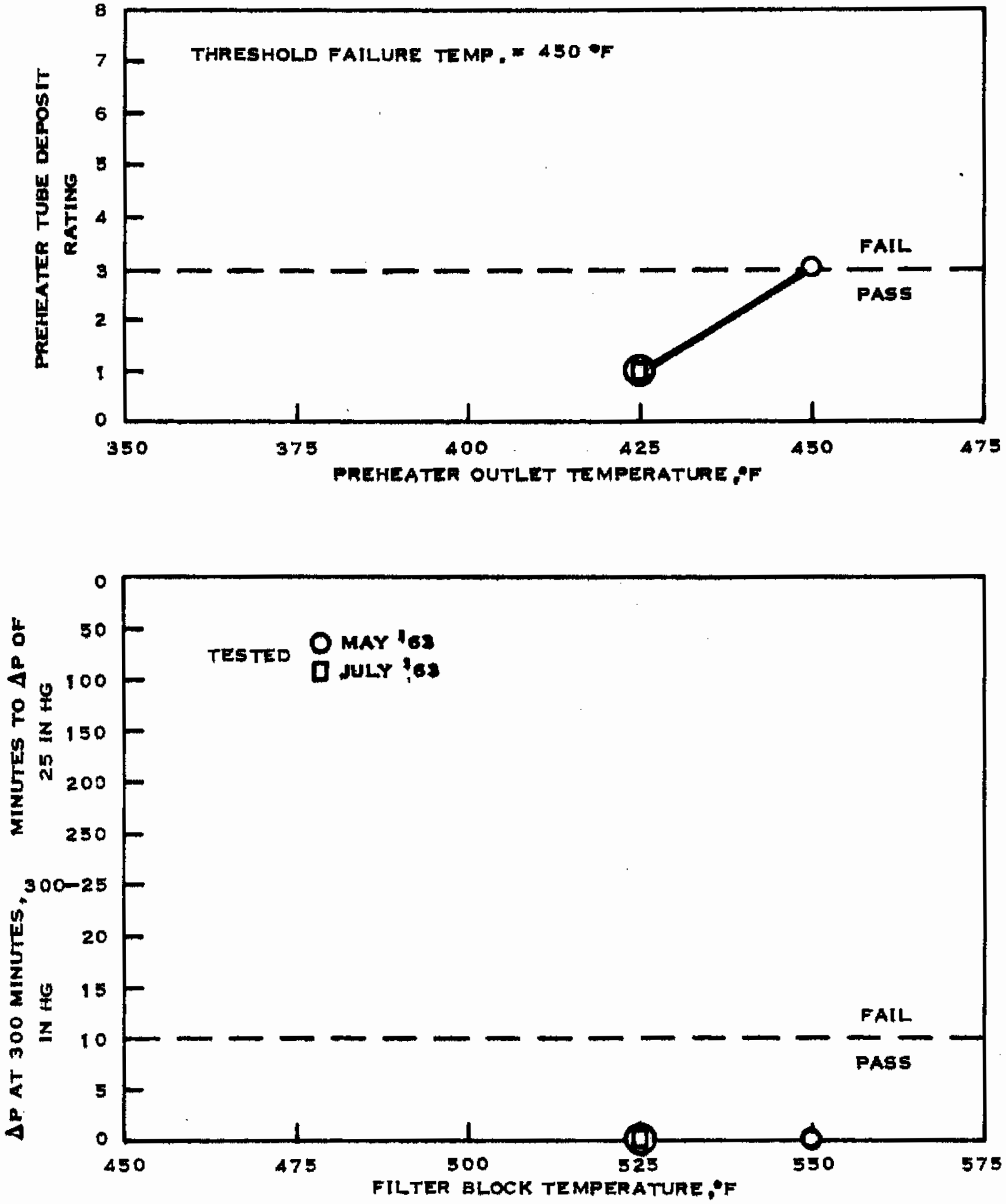

FIGURE 36 THERMAL STABILITY OF BJ63-10-K34 AS DETERMINED BY ASTM-CRC FUEL COKER

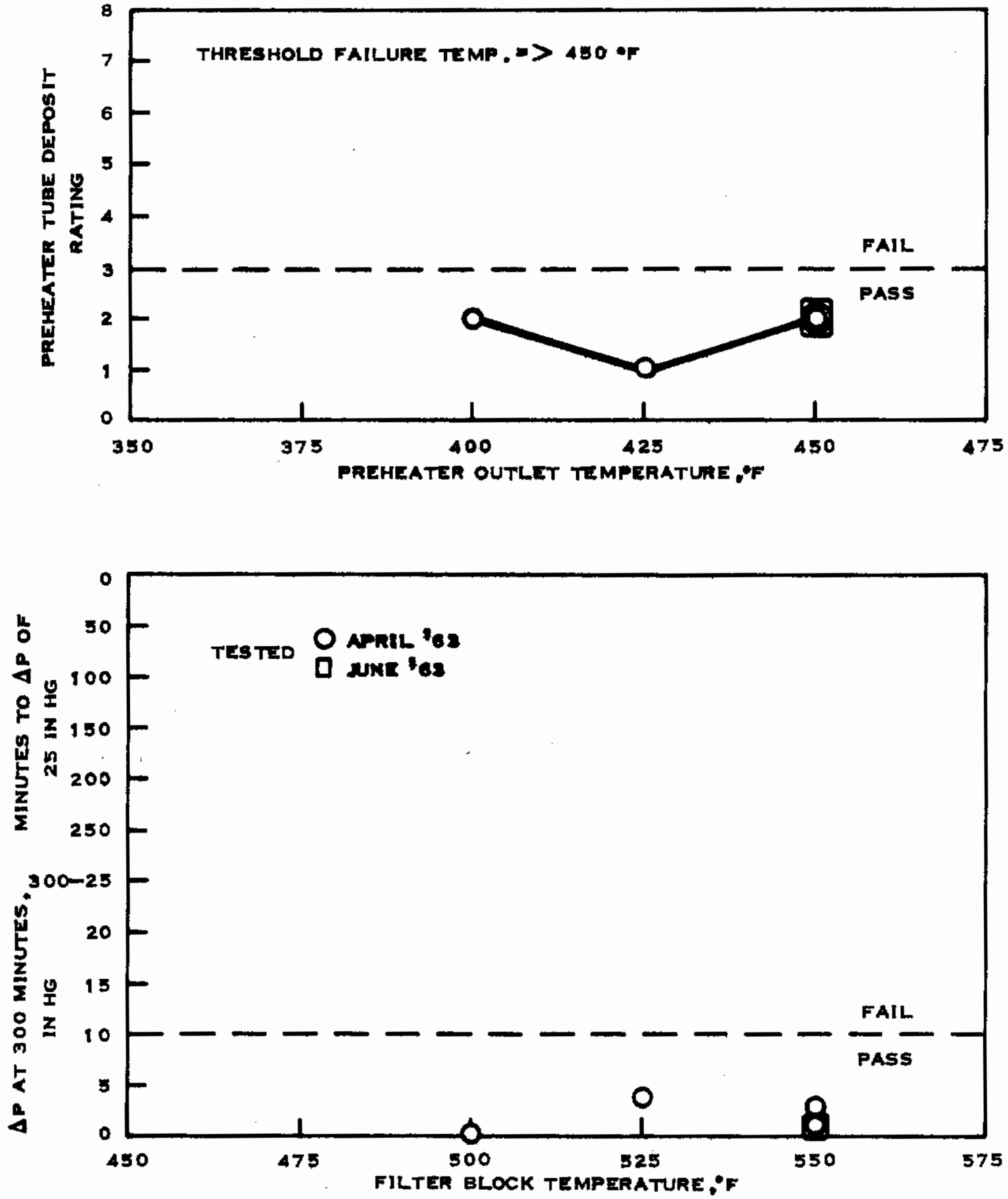

FIGURE 37 THERMAL STABILITY OF BJ63-10-K35 AS DETERMINED BY ASTM-CRC FUEL COKER

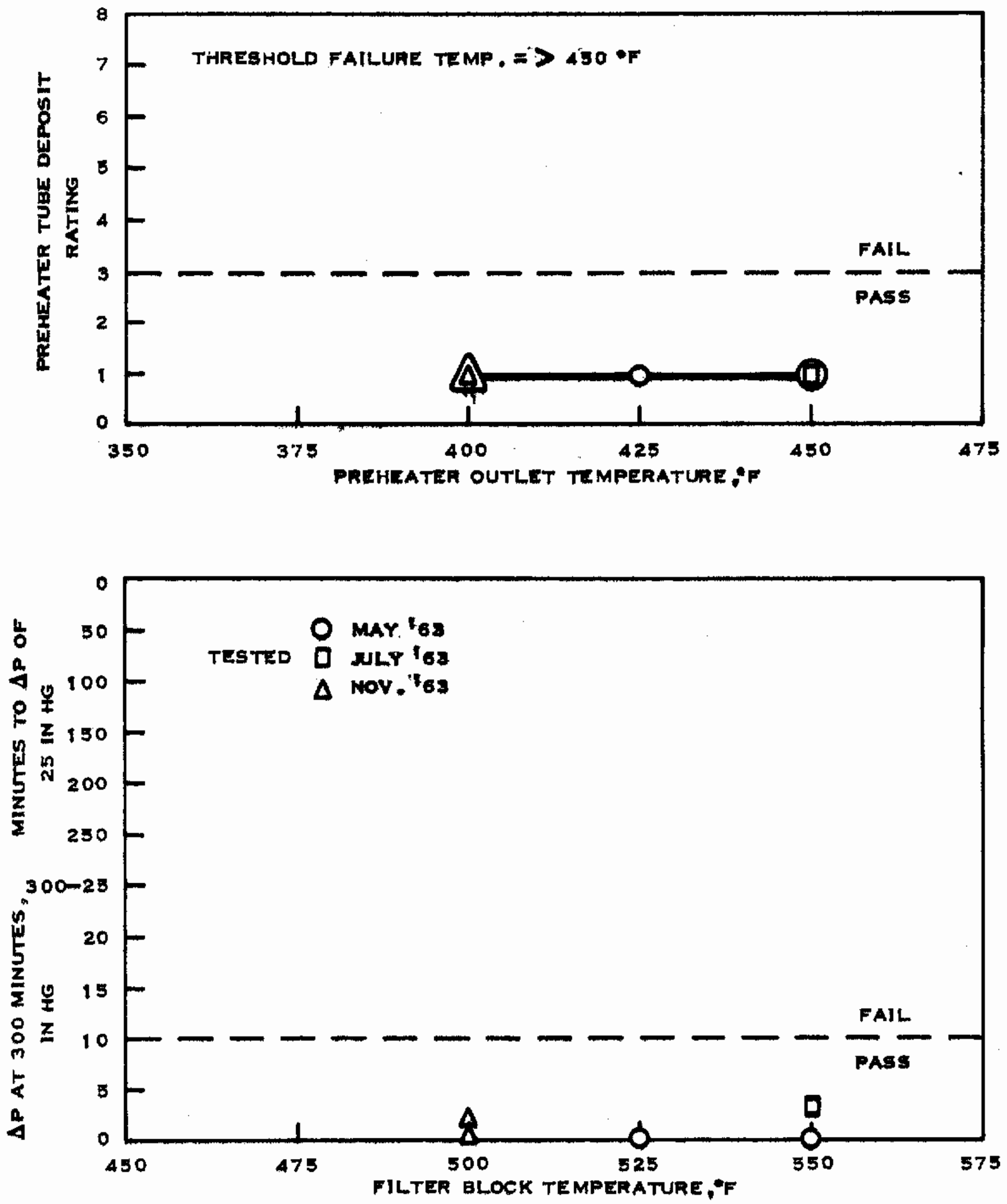

FIGURE 38 THERMAL STABILITY OF BJ63-10-K36 AS DETERMINED BY ASTM-CRC FUEL COKER

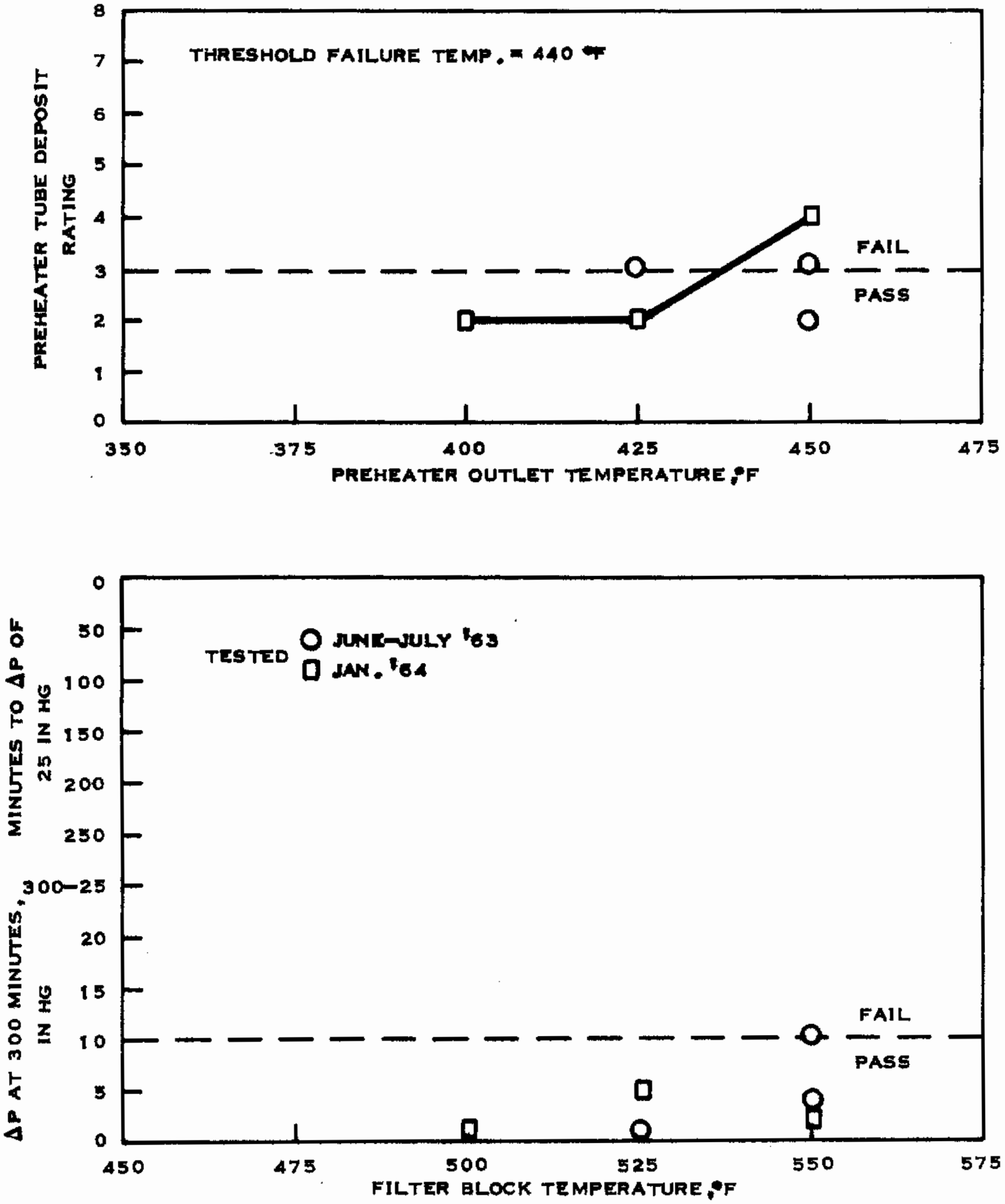

FIGURE 39 THERMAL STABILITY OF BJ63–10–K37 AS DETERMINED BY ASTM–CRC FUEL COKER

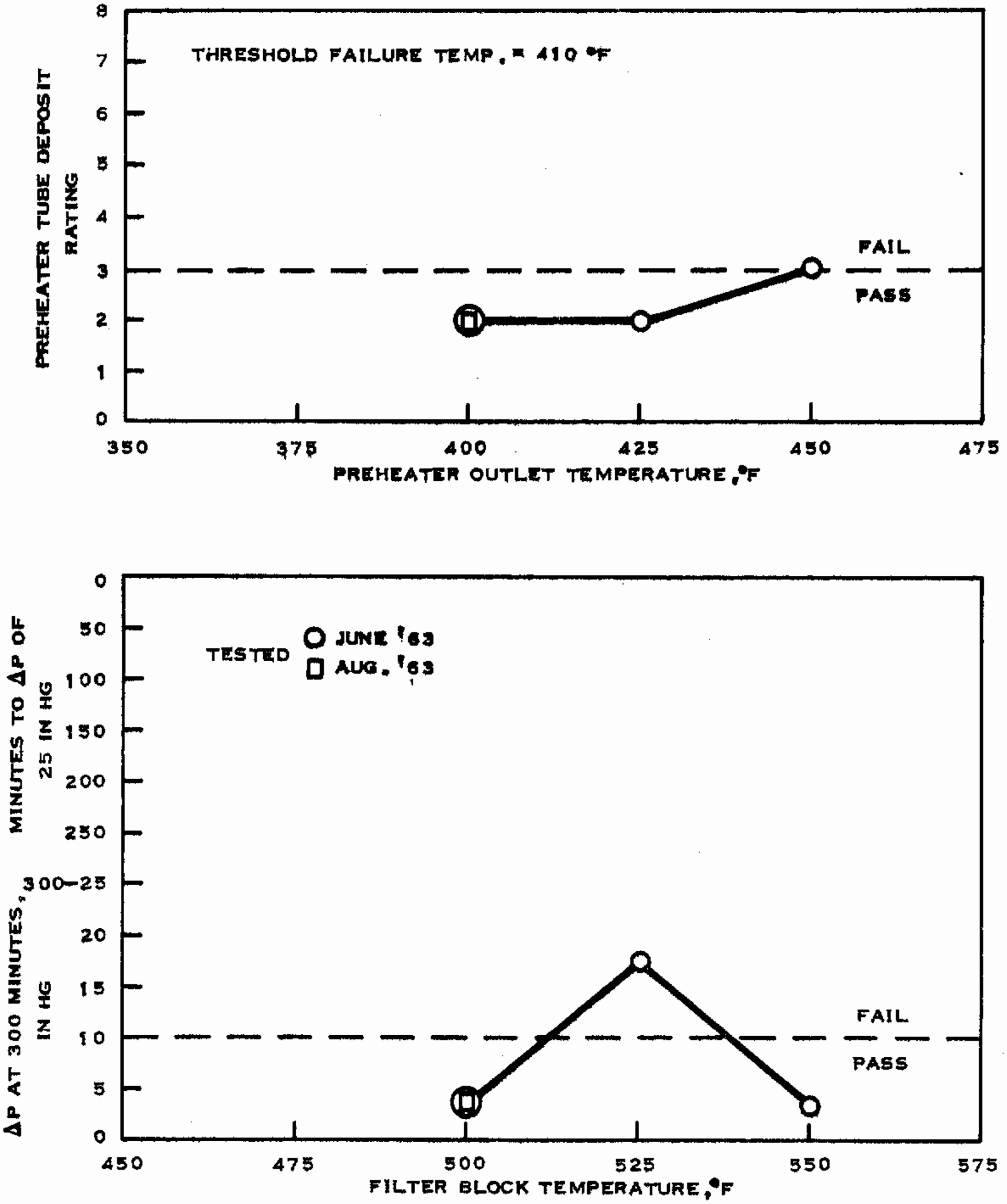

FIGURE 40 THERMAL STABILITY OF BJ63-10-K38 AS DETERMINED BY ASTM-CRC FUEL COKER

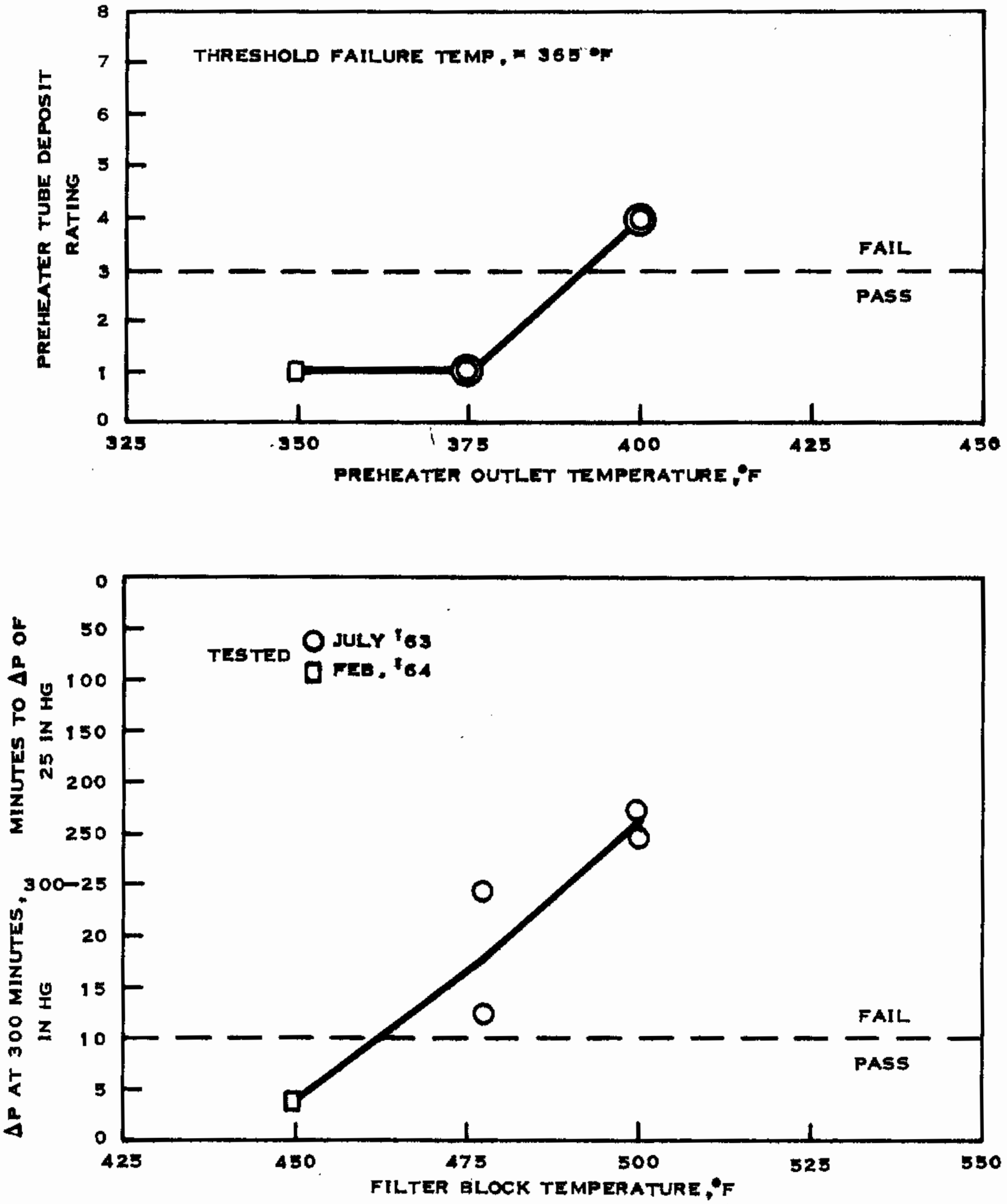

FIGURE 41 THERMAL STABILITY OF BJ63-10-K39 AS DETERMINED BY ASTM-CRC FUEL COKER

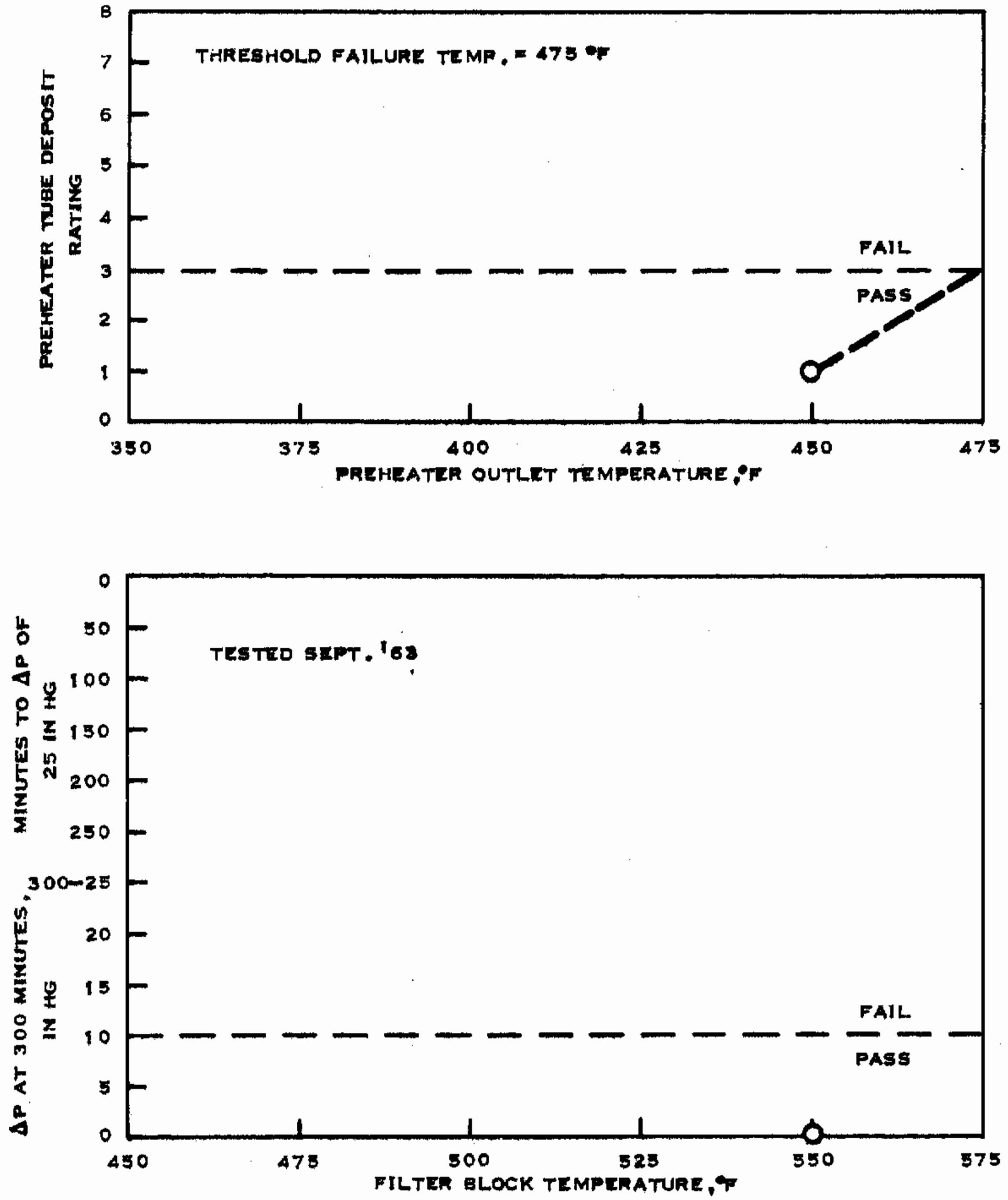

FIGURE 42 THERMAL STABILITY OF BJ63-10-K143 AS DETERMINED BY ASTM-CRC FUEL COKER

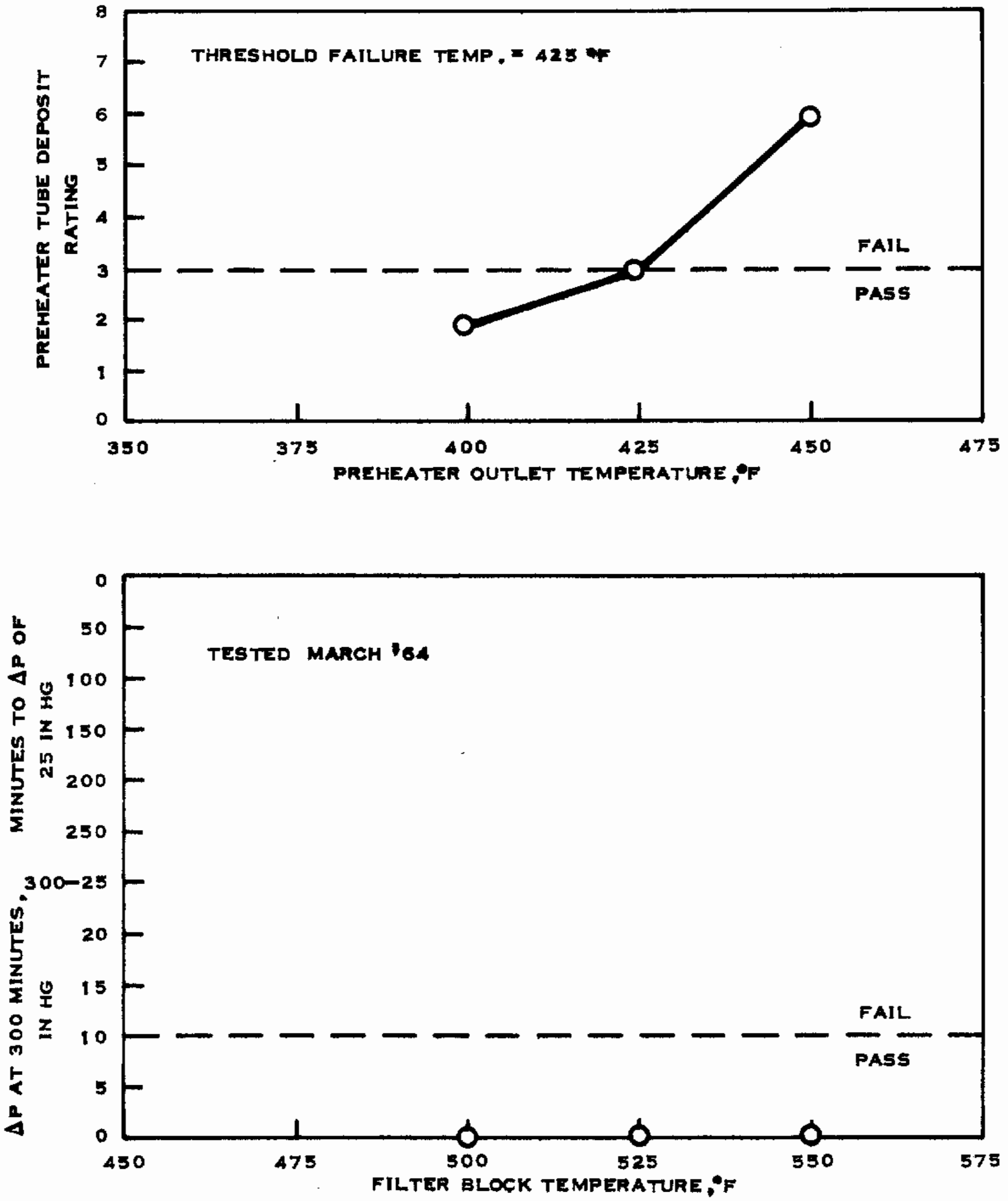

FIGURE 43 THERMAL STABILITY OF BJ64–10–K145 AS DETERMINED BY ASTM–CRC FUEL COKER

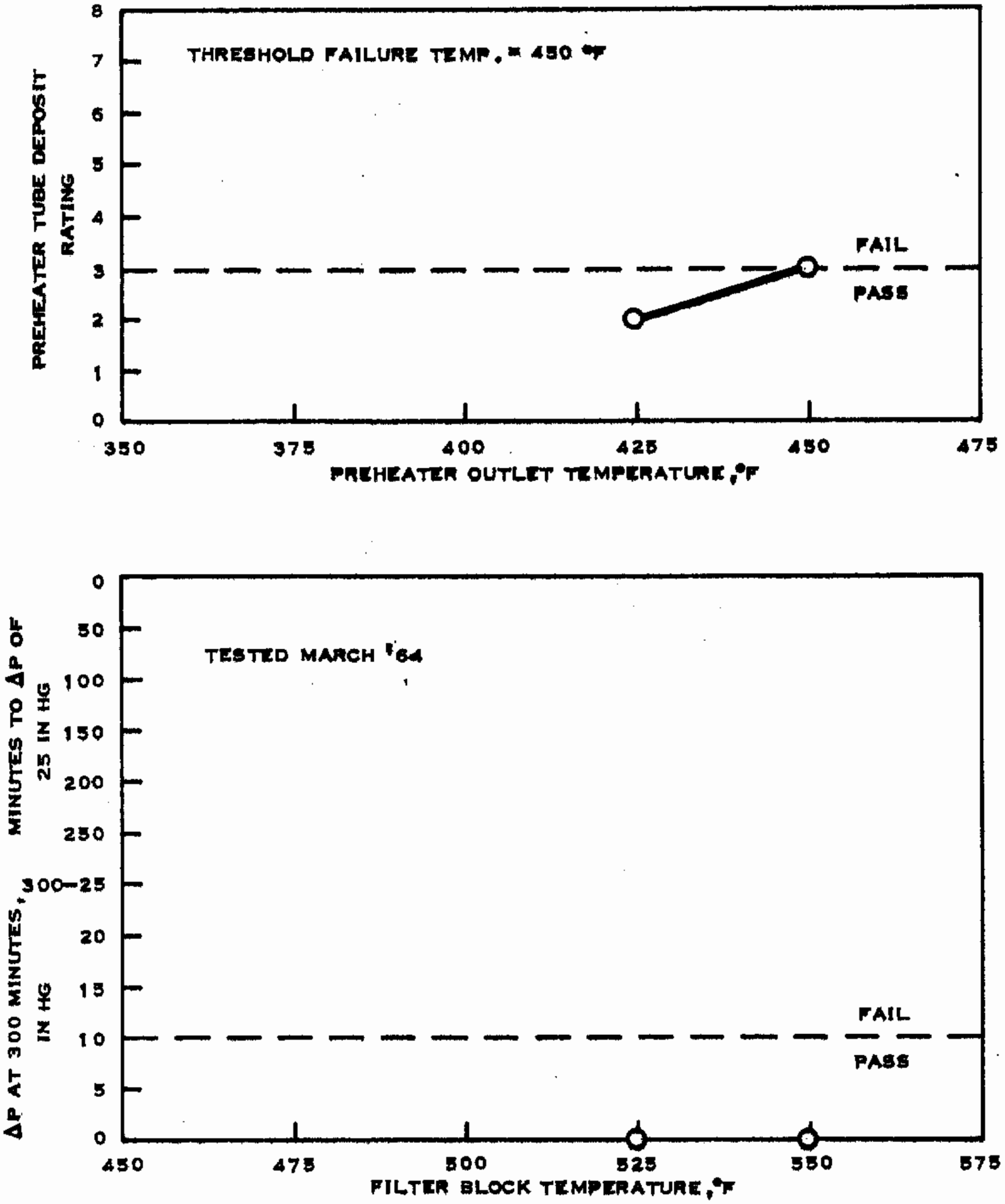

FIGURE 44 THERMAL STABILITY OF BJ64-10-K147 AS DETERMINED BY ASTM-CRC FUEL COKER

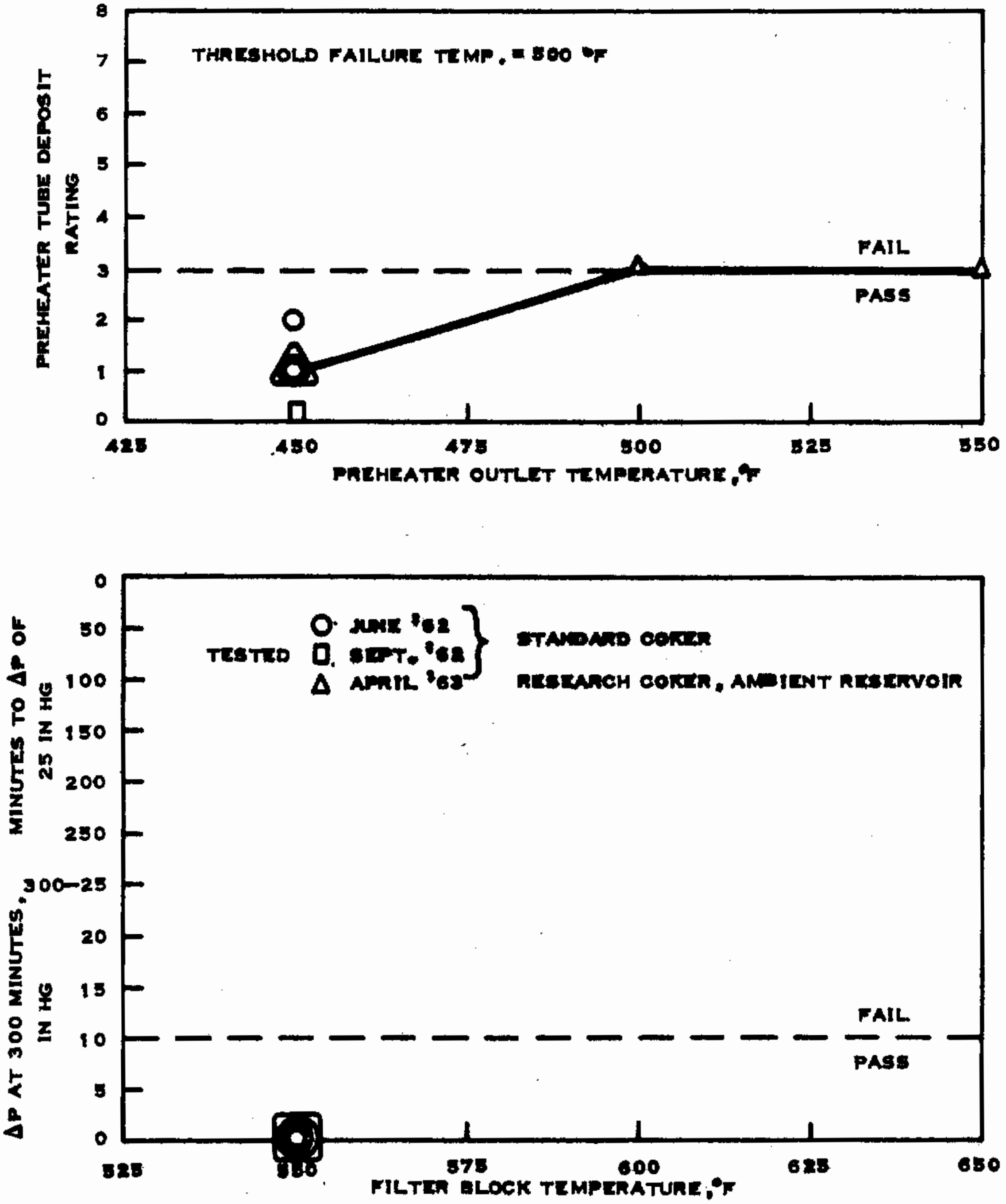

FIGURE 45 THERMAL STABILITY OF BJ64-10-B75 AS DETERMINED BY ASTM-CRC FUEL COKER AND RESEARCH COKER WITH AMBIENT RESERVOIR

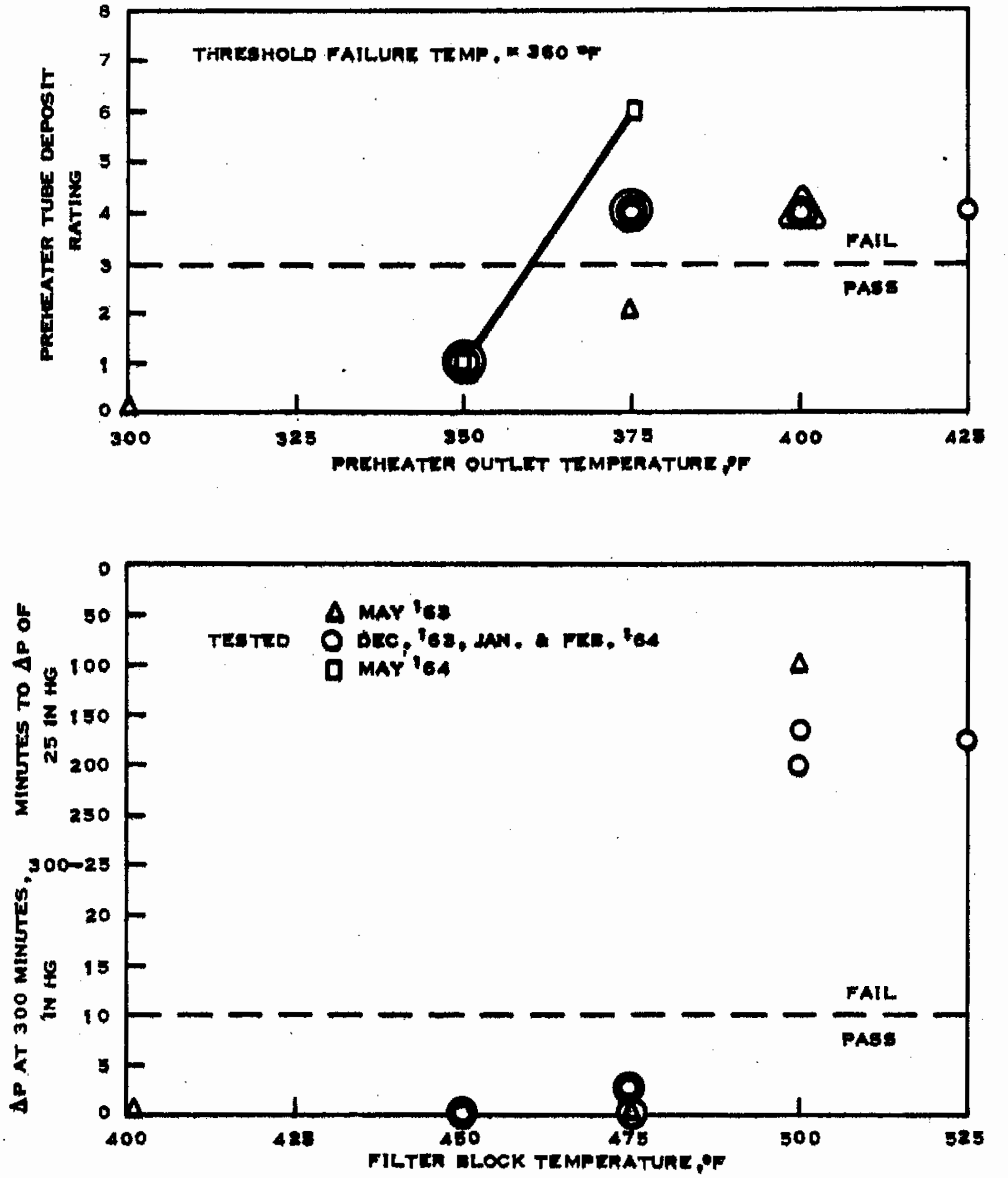

FIGURE 46 THERMAL STABILITY OF BJ63-10-G74 AS DETERMINED BY ASTM-CRC FUEL COKER

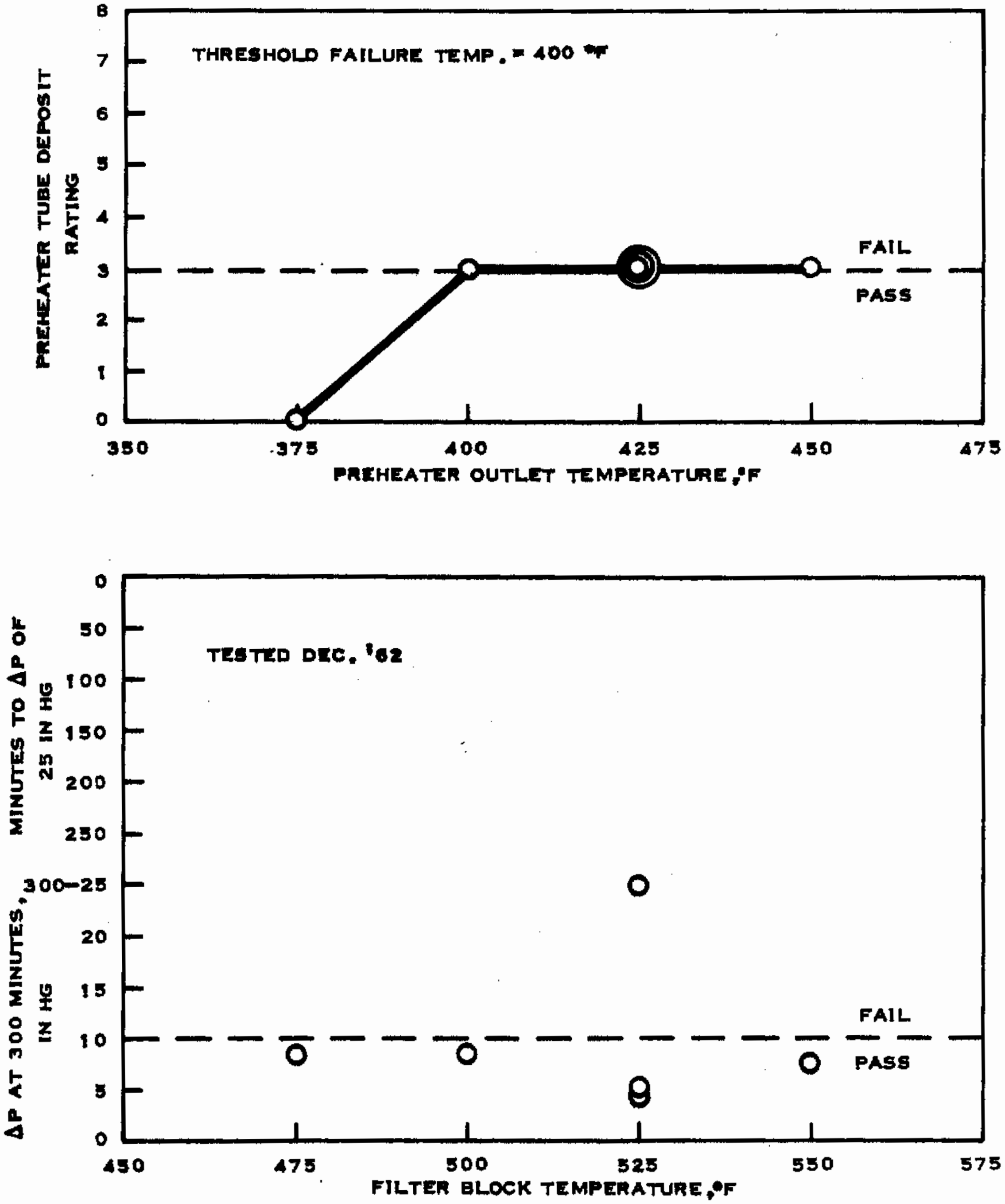

FIGURE 47 THERMAL STABILITY OF BJ62-16-J1 AS DETERMINED BY ASTM-CRC FUEL COKER

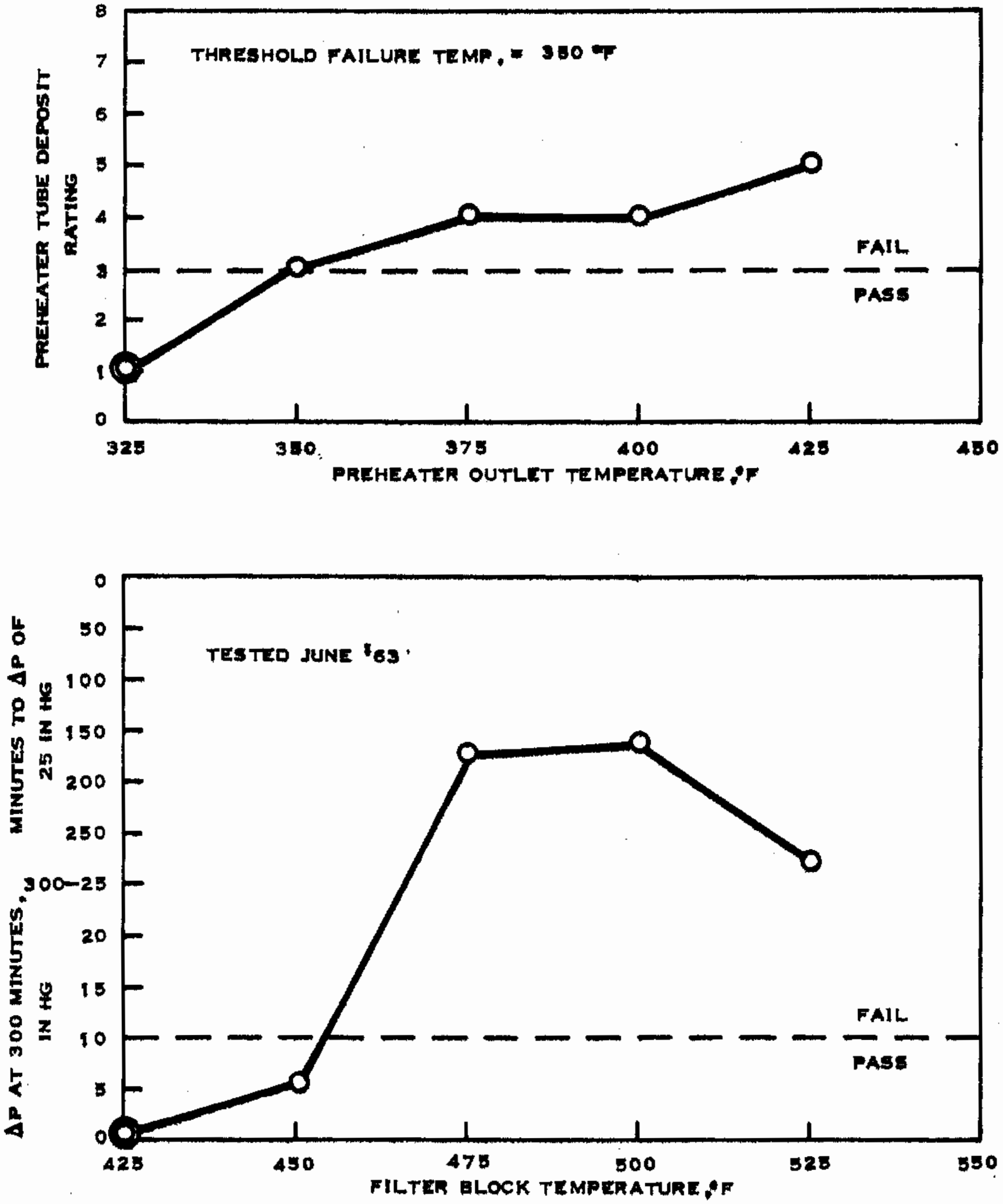

FIGURE 48 THERMAL STABILITY OF BJ63–17–G3 AS DETERMINED BY ASTM–CRC FUEL COKER

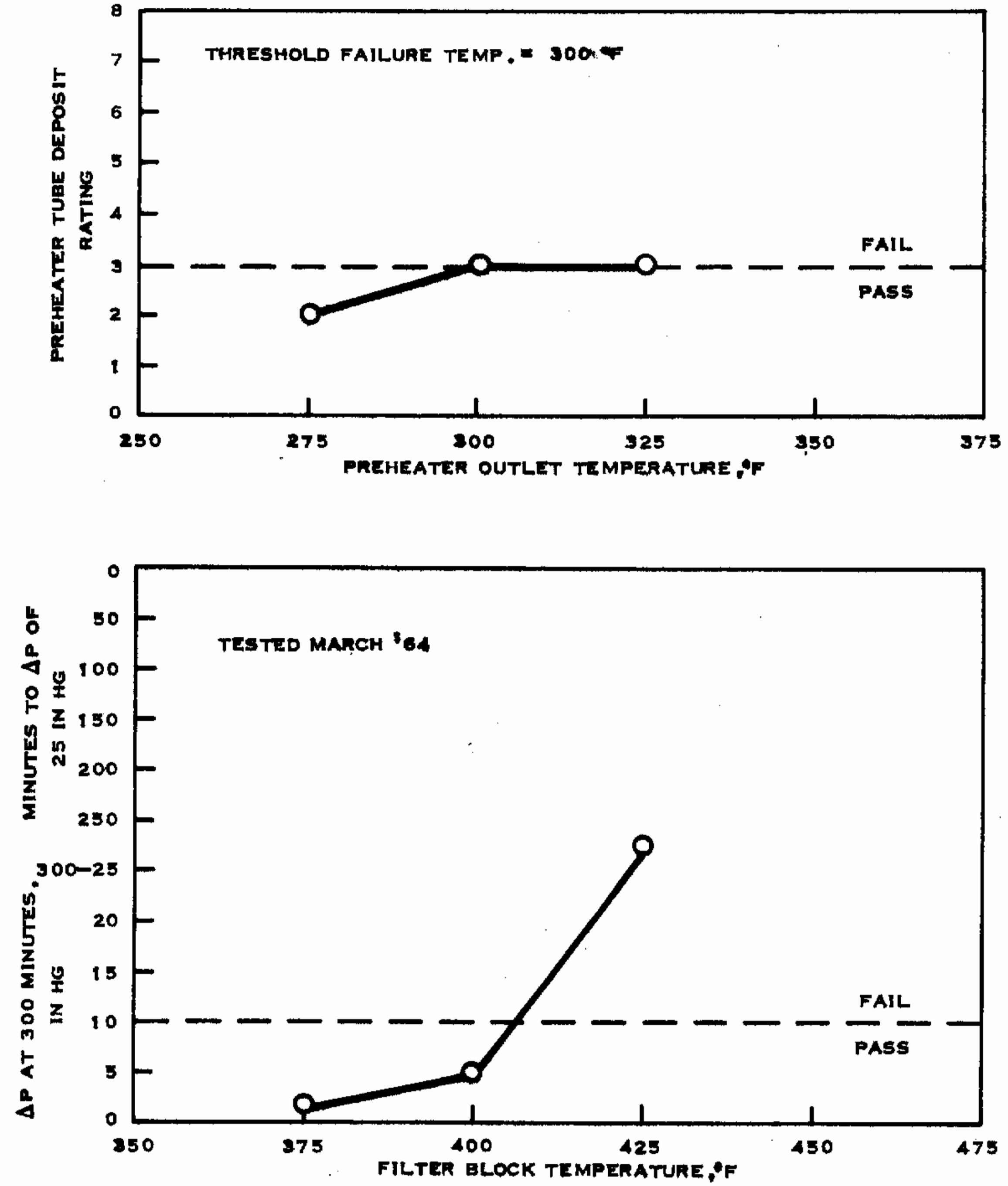

FIGURE 49 THERMAL STABILITY OF BJ64-10-G144 AS DETERMINED BY ASTM-CRC FUEL COKER

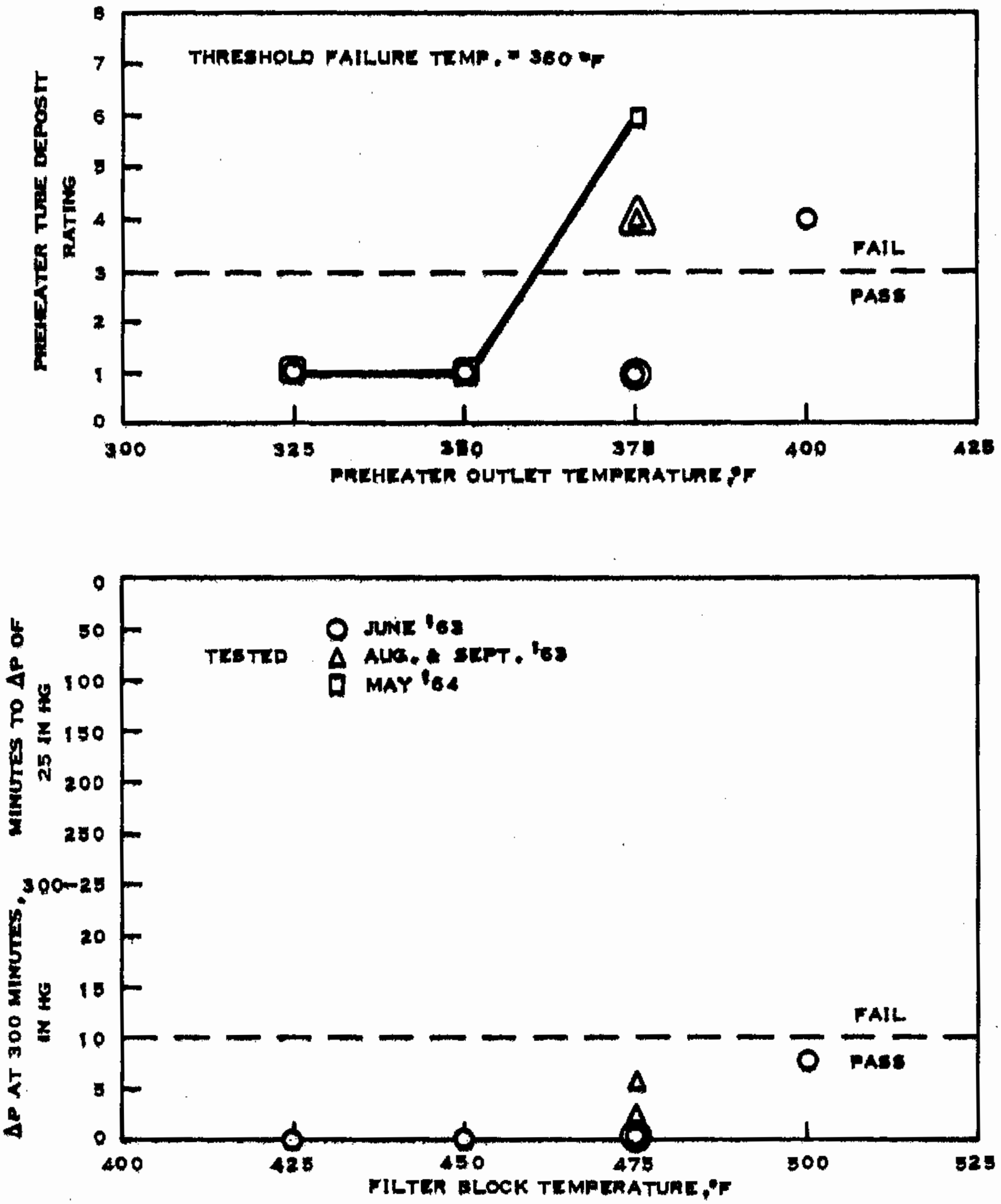

FIGURE 50 THERMAL STABILITY OF BJ64-10-G162 AS DETERMINED BY ASTM-CRC FUEL COKER

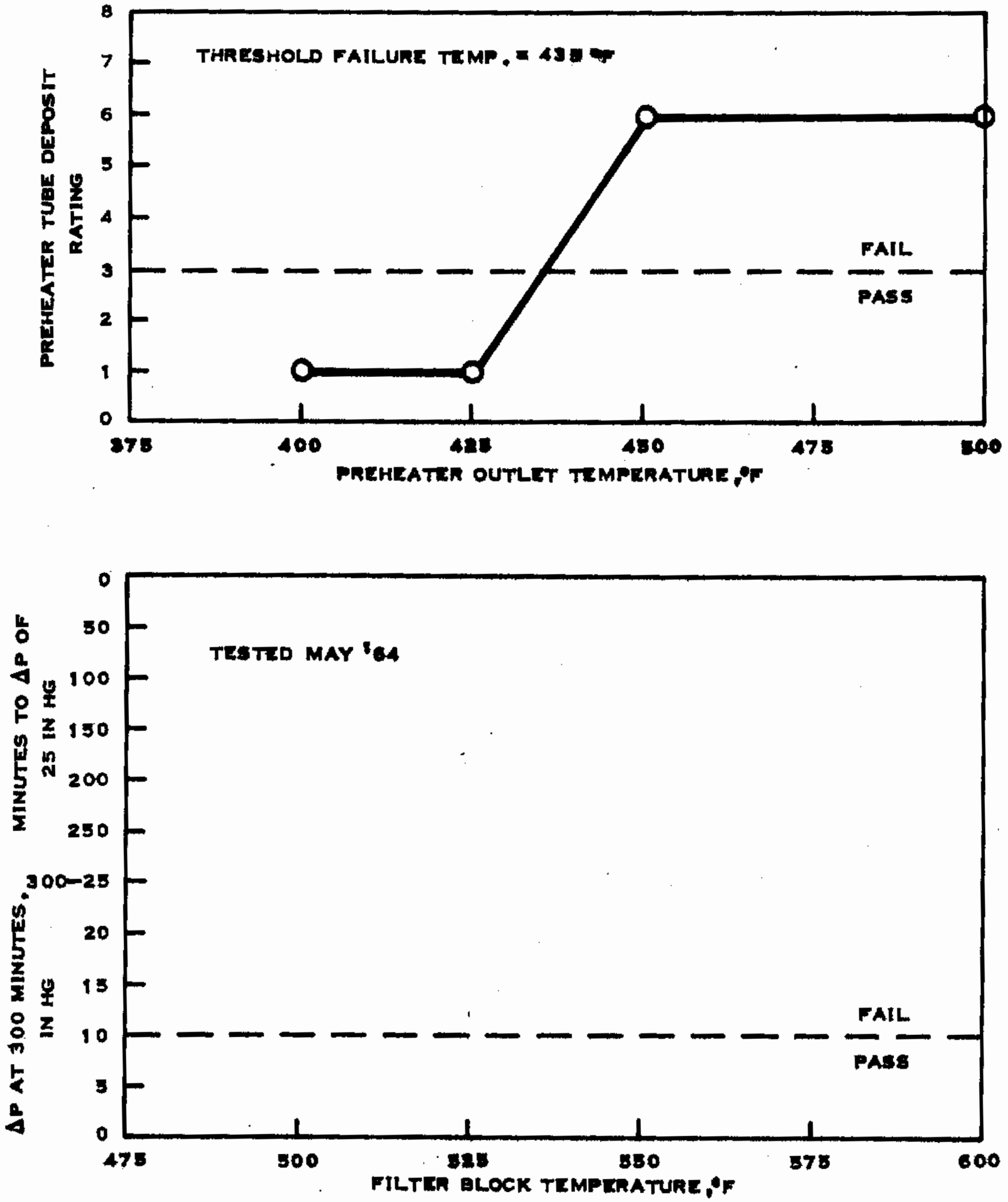

FIGURE 51 THERMAL STABILITY OF BJ64-10-G163 AS DETERMINED BY ASTM-CRC FUEL COKER