

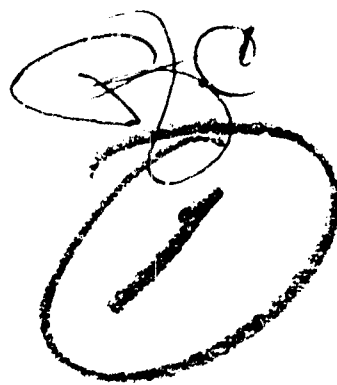
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**AFAPL-TR-70-5
PART II**



**RESEARCH ON HYDROCARBON
FUELS AND RELATED APPLICATIONS**

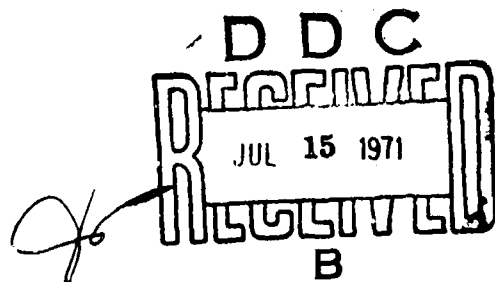
**Robert K. Johnston
Charles M. Monita
Robert E. Linder
et al.**

Southwest Research Institute

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TECHNICAL REPORT AFAPL-TR-70-5, PART II

May 1971



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**Air Force Aero Propulsion Laboratory
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio**

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**AFAPL-TR-70-5
PART II**



RESEARCH ON HYDROCARBON FUELS AND RELATED APPLICATIONS

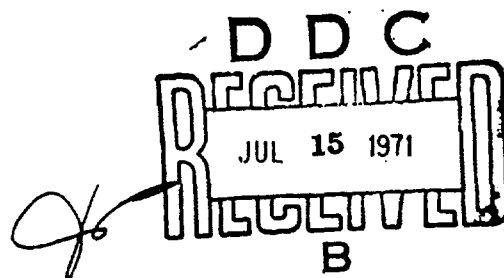
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et al.

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FOREWORD

This report was prepared by Southwest Research Institute, San Antonio, Texas, under Contract F33615-69 C-1231. The contract was initiated under Project Numbers 3048 and 3066. The work was performed by contractor's personnel using Air Force facilities at Area B, Wright-Patterson AFB and the contractor's facilities at San Antonio, Texas. The program was administered by the Fuels Branch of the Fuels and Lubrication Division, Air Force Aero Propulsion Laboratory, Air Force Systems Command, Wright-Patterson AFB, Ohio. The Air Force project engineer during the report period was Mr. Gregory W. Gandee (SFF-1).

In addition to the authors listed, the following members of the contractor's staff were responsible for specific sections of the experimental work and the technical reporting: Larry W. Schenk (fuel stability); Donald V. Reed (fuel corrosion inhibitors, fuel and lubricant analysis and testing).

This is a Technical Report covering the second year of work performed under subject contract, during the period from 1 December 1969 through 30 November 1970. The report was submitted by the authors in December 1970. Contractor's identifying numbers are Project Number 12-2497 and Report Number RS-561.

This technical report has been reviewed and is approved.

Arthur V. Churchill

Arthur V. Churchill, Chief
Fuels Branch
Fuels and Lubrication Division
Air Force Aero Propulsion Laboratory

ABSTRACT

Work has been continued on fuel stability, using gas-drive cokers and other test equipment to investigate the effects of dissolved metals, fuel additives, fuel-system materials, and test conditions on thermal stability ratings. In high-quality fuels, dissolved lead, zinc, iron, and copper in amounts as low as 25-100 parts per billion have been found to degrade the fuel quality seriously. New test devices for measuring fuel stability have been evaluated critically, and new techniques have been developed. The effects of flexible-tank liner materials on JP-7 fuel thermal stability are being studied in long-term simulated storage tests. Apparatus for measuring fuel lubricity has been set up, improved, and operated to show how fuel composition can affect control valve sticking behavior. A new fuel corrosion test has been developed and applied in drafting a recommended specification for fuel corrosion inhibitors. Gas-chromatographic techniques for the analysis of fuels and synthetic lubricants have been developed and improved, and luminescence and phosphorescence spectroscopy have been studied and adapted for this purpose. Short-term studies have been made of coagulant additives, fuel dyes, analytical methods for fuel system icing inhibitor, lead-corroding behavior of fuel corrosion inhibitors, and new methods for measuring fuel demulsification properties. Turbine engine instrumentation and control concepts have been developed to the stage of experimental hardware and are being integrated into a complete control system for a J-85 engine. Operation of an existing information retrieval system has been continued and expanded.

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SECTION I

INTRODUCTION AND SUMMARY

1. GENERAL

This report describes the results obtained in the second year of a three-year program of research and development on aerospace fuels and related areas. This work is being performed by Southwest Research Institute personnel in Air Force facilities at Wright-Patterson AFB, supplemented in certain areas of the program by technical guidance, consultation, and design and experimental work by staff members located at SwRI's main facilities in San Antonio, Texas.

Long-term investigations included in this program are described in detail here and in other Technical Reports. Descriptions of short-term studies are also included when the results are of general interest or are pertinent to other aspects of the program.

The program is divided into five general areas:

- Fuel research and development
- Optical techniques for chemical analysis
- Turbine engine control instrumentation
- Fire and explosion detection
- Information retrieval.

The work on fuel research and development continues as the major activity. Work on fire and explosion detection was terminated during the past year.

2. FUEL RESEARCH AND DEVELOPMENT

a. Fuel Stability

The stability of advanced and current hydrocarbon fuels has been investigated at normal and elevated temperatures. Work on development, improvement, and evaluation of test devices and techniques has been continued, and these devices and techniques have been applied in problem-solving and in long-term investigations.

Modified fuel cokers with gas drive had become well standardized during the first year of this program, and only minor efforts have been made toward further improvements. These cokers have been used in a detailed investigation of the effects of dissolved iron, copper, lead, and zinc on the thermal stability of JP-7 fuel. It was found that the amounts required to degrade thermal stability are lower than expected; the addition of as little as 15 ppb copper, 25 ppb iron, 100 ppb zinc, or 125 ppb of lead can degrade JP-7 fuel significantly. The true threshold concentrations may be even lower, since some of the added metal is usually lost in handling, transferring, sampling, and analyzing the fuel samples. This work on dissolved metals also pointed out the need for better definition of deposit rating color codes when the deposits do not conform to the existing standards.

Two relatively new thermal stability test devices, the Alcor JFTOT and the Erdco Precision Coker, have been studied in connection with cooperative evaluation programs. Data have been reported to the coordinating group for detailed analysis and are presented here as a basis for discussion and comparison of the two devices. Extensive operating experience with the JFTOT has provided a further basis for critical evaluation and minor modifications of the equipment. Detailed data have been accumulated on JFTOT temperature profiles, and preliminary studies have been made of a step-test procedure in which power input requirements give some indication of deposit formation. In the usual JFTOT procedure, major problems still exist in rating and interpreting the deposits formed on JFTOT tubes. Some of these problems may be resolved by the use of nonvisual rating systems.

Fuel cokers of several types have been used in short-term investigations of the thermal stability of intermixed fuels, the effects of fuel additives, and the effects of various fuel-system materials. A long-term investigation of fuel-elastomer compatibility is in progress, aimed primarily at elastomers used as liners for flexible fuel-storage tanks. Large differences have been found among the elastomers used or proposed as liners.

b. Fuel Lubricity

Fuel lubricity has been investigated in relation to fuel control-valve sticking, using a lubricity simulator previously designed and constructed for the Air Force. Much of the effort during the past year has been devoted to identifying and solving the various problems encountered in mechanical operation, component redesign and replacement, instrumentation, and test technique. Work has progressed sufficiently to demonstrate that the simulator can discriminate between a pure hydrocarbon and the same hydrocarbon with 50 mg/liter of oleic acid. It is now probable that a valid fuel rating technique can be developed using a reference fuel and determining relative rather than absolute values.

c. Fuel Corrosion Inhibitors

A modified rusting test has been developed in order to improve the precision of results when testing the presently qualified or proposed fuel corrosion inhibitors. This rusting test, along with changes in other tests, has been incorporated in recommendations for revision of the present specification, MIL-I-25017B. This work has indicated the need for a storage stability test for corrosion inhibitors.

d. Fuel and Lubricant Analysis and Testing

Work has been continued in the development and improvement of gas-chromatographic techniques for identification and analysis of synthetic lubricants and lubricant components. New liquid phases have opened up several possibilities in analysis that were not available before, and these are being explored. Existing techniques have been standardized for accumulation of a "fingerprint" file that has already proven very useful in identifying unknown lubricants and mixtures and in following the progressive changes in lubricant composition during service or engine tests. Gas chromatography has also been applied in the development of an analytical technique for fuel vapor content in fuel-tank atmospheres, and numerous analyses have been run in support of an Air Force program relating to fire hazards.

Methods for determining the content of fuel system icing inhibitors have been compared. No great differences in precision were found. Coagulating agents for the removal of particulate matter from fuel have been studied briefly. Lead corrosion tests on commonly used fuel corrosion inhibitors have indicated that lead reaction products can be formed and could be a factor contributing to certain fuel pump freezeup problems. The need for a general study of nonferrous metal corrosion by "corrosion inhibitors" is clearly indicated. Water separator tests have been studied briefly in relation to sample-can contaminants and cleaning. A new approach to fuel demulsibility testing has been examined briefly, with somewhat encouraging results.

3. OPTICAL TECHNIQUES FOR CHEMICAL ANALYSIS

Studies of fluorescence and phosphorescence spectra of synthetic lubricants and additives have been supplemented by NMR and optical absorption spectrometry. Use of these techniques makes it possible to identify or "fingerprint" lubricant formulations and in many cases to detect batch-to-batch variations of the same lubricant formulation. Work is being continued to explore the range of applicability of these techniques in specific analytical problems and to obtain a better definition of optimum combinations of techniques.

4. TURBINE ENGINE INSTRUMENTATION AND CONTROL

Work during the past year has been directed toward development of a sophisticated, flexible facility for work in the area of engine dynamics. A multichannel data acquisition and external control system is being implemented, so that a J-85-7 test engine can be interfaced with an IBM-1800 process control computer. The engine instrumentation plan has been drawn up in detail, and a number of probes already built.

5. INFORMATION RETRIEVAL

Storage and retrieval of material, primarily technical reports, have been continued. The system has been expanded so as to assimilate the documents relating to fire protection, which had previously been kept separate from those on fuels and lubricants. New acquisition rate has been about 30 documents per month.

SECTION II

FUEL STABILITY

1. GENERAL

The solution of problems in the thermal-oxidative and storage stability of jet fuels has continued to receive major emphasis. Efforts have been directed primarily toward refining the present methods and developing new methods for evaluating fuel stability. Such methods have then been used to determine not only the suitability of fuels, but also the suitability of materials for use in fuel systems.

The first year of contractual effort in this area was aimed mainly at the development, evaluation, and improvement of methods for determining thermal stability.* During the second year, this work has been continued, but emphasis has been shifted to using these test methods on a broader scale to evaluate the effects of materials on fuel thermal stability. Work has progressed in a long-range study of the effects of dissolved metals on thermal stability. Many short-term problems have been investigated, including the effects of proposed fuel antioxidants, fuel dyes, coating materials, and storage system materials on fuel thermal stability. As in the past, the scope of such evaluations has been somewhat restricted by excessive requirements for fuel sample and testing time when using standard test methods. New test equipment and procedures currently in use have relieved this problem to some extent and should improve matters still more in the future. Such new methods may also lead to better test precision and thus alleviate a problem that has hampered all work on fuel thermal stability.

2. GAS-DRIVE FUEL COKER STUDIES

a. Test Equipment and Procedures

All gas-drive coker tests were run on four semiautomatic fuel cokers equipped with modified test sections conforming to current requirements of the Coordinating Research Council (CRC). Two of the cokers were equipped with standard CRC flanged-pipe reservoirs. The other two cokers were equipped with stainless steel oxygen bottles as fuel reservoirs. These were used for the work reported in this section but were later replaced with flanged-pipe reservoirs. In subsequent discussion, the flanged-pipe reservoirs are termed "standard" and the oxygen bottles are termed "nonstandard" reservoirs.

Flow diagrams of the cokers are presented in Figures 1 and 2. The "CRC flow configuration" with standard reservoir (Figure 1) conforms to the CRC requirements for gas-drive cokers.^{(1)†} The "SwRI flow configuration" with nonstandard reservoir (Figure 2) represents the modified version described previously.⁽²⁾ In the work reported here, two cokers have been operated in each configuration.

The primary difference between the two flow configurations, apart from the difference in fuel reservoir, is the absence of an in-line filter ahead of the test section in the SwRI configuration. This leads to a difference in test procedure: The usual order, fuel prefiltration followed by aeration, is reversed in the SwRI configuration. This is necessary to ensure that filtration is the last step before the actual test is started.

All tests were run for 5 hours with a fuel flow rate of 2.5 ± 0.1 lb/hr. Coker warmup procedures were standardized so that the preheater and filter normally reach their respective test temperatures 15 to 20 minutes after startup. In the tests reported here, almost all warmup times fell between 13 and 23 minutes, with a few as long as 30 minutes.

*Since the high-temperature degradation of jet fuels in service is primarily an oxidative process, the stability of fuels at elevated temperatures is sometimes called "oxidation stability." We have retained the older term, "thermal stability," which is understood to mean the resistance of a fuel to degradation at elevated temperatures, normally with some oxygen present.

†Superscript numbers in parentheses refer to the List of References at the end of this report.

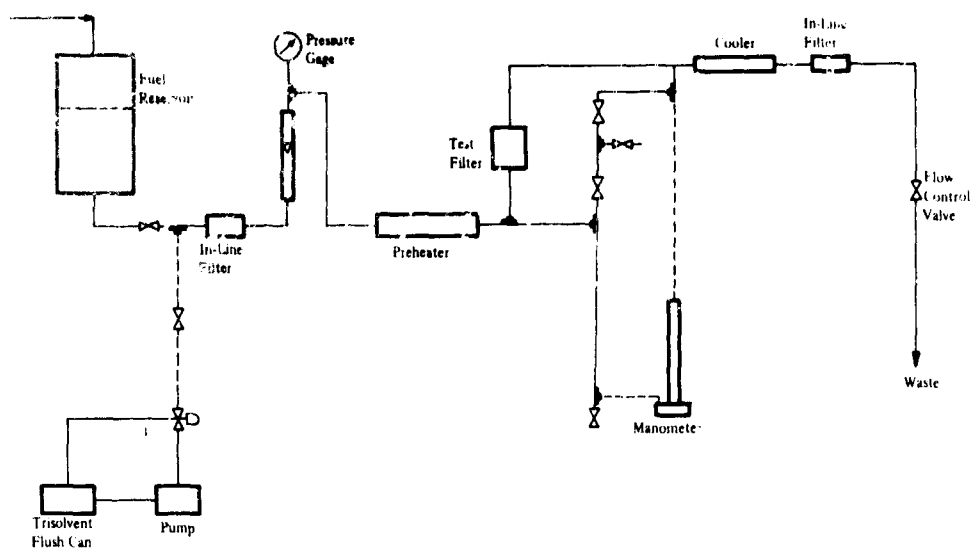


FIGURE 1. GAS-DRIVE COKER, CRC CONFIGURATION

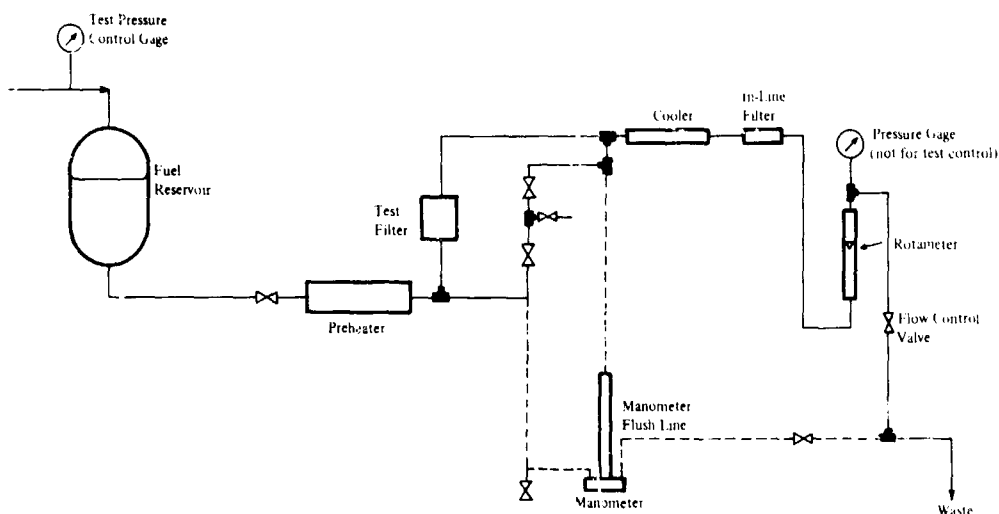


FIGURE 2. GAS-DRIVE COKER, SwRI CONFIGURATION

Preheater tubes were rated after test in both unwiped and wiped conditions, using the standard Tuberator. The breakpoint of a given fuel is defined as the lowest preheater fuel-out temperature giving either an *unwiped* preheater tube rating of Code 3 or darker, or a filter pressure drop of 2.0 in. Hg or more.*

The basic operating procedures, starting with ASTM D 1660, are further defined for the CRC gas-drive cokers in Reference (1). Additional modifications are given in Reference (2) and are understood to apply here unless otherwise indicated.

For all of the work reported here, nitrogen was used as the drive gas. A drive pressure of 250 psi was used in all except a few tests in which fuel boiling necessitated an increase to 280 psi.

* The criterion of 2.0 in. Hg was adopted several years ago for the gas-drive coker in work performed by SwRI. Currently, the *standard* coker criterion usually cited in specifications is 3.0 in. Hg. A scaled-down to the flow rate in the gas-drive coker would give 1.25 in. Hg, but the older criterion of 2.0 in. Hg has been retained.

Preheater tubes were polished with A-1 metal polish in accordance with ASTM D 1660-69 unless indicated otherwise. A few tests were run with a substitute polish, which was under consideration by the ASTM at that time and subsequently was adopted as standard.

b. Test Fuels

Six test fuels were used in comparing various equipment and procedure modifications. These are identified here by type and specification number:

Fuel A	JP-7	MIL-F-38219
Fuel B	JP-8	MIL-T-83133
Fuel C	JP-7	MIL-F-38219
Fuel D	JP-6	MIL-J-25656B
Fuel E	JP-5	MIL-T-5624C
Fuel F	JP-4	MIL-T-5624G

c. Test Results and Discussion

(1) Comparison of Configurations and Procedures

Comparative tests were run using the two equipment configurations (CRC and SwRI, Figures 1 and 2) and the respective procedures associated with these configurations. The results of these tests are summarized in Table 1. The breakpoints obtained by the two methods usually coincided. The comparison was sometimes obscured by the rather poor repeatability of results obtained with both configurations. For Fuels A and B, the breakpoints with the CRC configuration were 25-50°F higher than with the SwRI configuration; this is hardly a significant difference in view of the occasional out-of-line results. Several mechanical and operating problems arose during these series of tests, and interpretations were further complicated because of tube deposits that were abnormal in terms of location, shape, or color.

TABLE 1. FUEL BREAKPOINTS WITH TWO CONFIGURATIONS OF GAS-DRIVE COKER

Fuel	Heater breakpoint, °F, for Code 3	
	CRC	SwRI
JP-7, Fuel A	700	675
JP-8, Fuel B	475	425-450*
JP-7, Fuel C	650-700	650
JP-6, Fuel D	550-575†	550-575†
JP-5, Fuel E	400‡	400‡
JP-4, Fuel F	450	450

*Gold end tube deposits at 425°F.
†Fuel boiling.
‡Filter plugging as well as tube deposits.

There was no significant difference in results obtained with the two configurations; this is evidence that system cleanliness was comparable in the two configurations. In subsequent work, we have considered the two configurations to be equivalent in rating level. Later in the program, the two cokers originally set up in the SwRI configuration were equipped with standard CRC fuel reservoirs but still operated in the SwRI flow configuration. It appears that this combination will provide optimum insurance against contamination of test fuel.

(2) Evaluation of Metal Polish for Preheater Tube

The A-1 metal polish, long used to prepare preheater tubes for both standard and gas-drive coker tests, is now unavailable. A metal polish known as "Silvo" was under consideration by the ASTM as a replacement at the time of the work reported here. This polish has since been adopted, and its use is specified for the standard coker in ASTM D 1660-70.

For the purposes of the present program, the two polishes were compared in gas-drive coker tests. Breakpoints were determined for three test fuels, using preheater tubes polished with A-1 polish. Then the breakpoints were determined for the same three fuels, using Silvo-polished tubes. New tubes were installed in the cokers at the start of the A-1 series and again at the start of the Silvo series. For both polishes, the tube cleaning and polishing

procedure was that specified for the standard coker at the time (ASTM D 1660-69) and for the gas-drive coker.⁽¹⁾ As noted subsequently, this procedure is not suitable for use with the Silvo polish.

TABLE 2. EFFECT OF HEATER TUBE POLISH ON GAS-DRIVE FUEL COKER BREAKPOINTS

The breakpoints listed in Table 2 indicate that the change in metal polishing compound did not change the thermal stability ratings for these fuels. The series was, in fact, an over-rigorous test of the new polish, since the incorrect rinsing procedures undoubtedly left significant amounts of the polishing compound on the tube surface.

Fuel	Heater breakpoint, °F for Code 3	
	A-1 polish	Silvo polish
JP-7, Fuel A	675	675
JP-7, Fuel C	650	650
JP-5, Fuel E	400*	375*
*Filter plugging as well as tube deposits.		

When using the original A-1 polish, the polished tubes are rinsed with pentane or hexane. This same procedure was applied to the Silvo polish in this series of tests, as we were not aware at that time of ASTM work indicating that a rinse in toluene/acetone/isopropanol trisolvant is necessary to remove the residues left by the Silvo polish.

Hence, the Silvo-polished preheater tubes in these tests must be regarded as highly contaminated. It was noted that the Silvo-polished tube surfaces before test were a nearer match to a No. 1 or 2 color code standard than to the No. 0 which supposedly represents a freshly polished tube. Even with the older standard A-1 polish, a real match to the No. 0 standard could hardly ever be achieved, but the Silvo-polished tubes in this series were definitely duller than the A-1 polished tubes.

Subsequently, the use of Silvo polish was studied using the ASTM-recommended procedure⁽³⁾, which includes a rinse with trisolvant after polishing. Silvo-polished surfaces still had a duller finish than A-1 polished surfaces. This difference was most pronounced when polishing new preheater tubes. The Silvo is not as efficient as the A-1 in removing the original oxide discoloration, and it requires more work to arrive at a reasonably bright finish. With some new tubes that are heavily discolored, polishing with Silvo is very laborious and time-consuming.

Although this problem with the polish is not critical, it would be desirable to find a better substitute. Some effort along this line is being continued.

3. EFFECT OF DISSOLVED METALS ON FUEL STABILITY

a. Background

Earlier studies^(2,4) had demonstrated that JP-7 fuel thermal stability is affected very adversely by contact with certain metals and elastomers during storage, and that the fuel quality deterioration is associated with increases in content of dissolved copper, iron, zinc, or lead. The present work has been aimed at a clear definition of the role of these four metals in degrading the thermal stability of JP-7 fuel, with a view toward using metal analyses for control tests during storage and material compatibility studies.

As in the previous studies, the effects of metals on fuel stability were measured by thermal stability tests using the gas-drive coker. All metal analyses were performed at Monsanto Research Corporation under the direction of Dr. W.G. Scribner. The methods for trace amounts of copper, iron, and zinc have been summarized by Lander⁽⁵⁾, the method for iron has been discussed in more detail by Scribner, et al.⁽⁶⁾, and the method for lead has been presented in a recent report by Scribner and Borchers.⁽⁷⁾ For the present work, minor changes were made in the test methods listed in these references in order to adapt the procedures to the special problems involved.⁽⁸⁾ In our discussion of the results of lead determinations, we have quoted from private communications with Dr. Scribner, without giving specific acknowledgment in all instances. We wish to acknowledge here the close cooperation and valuable comments of Dr. Scribner in this work.

b. Fuel Blending and Metal Analyses

The metals used in this program were in the form of commercial naphthenates.

Early work in this program, reported previously⁽²⁾, was confined to determining the effects of dissolved lead and zinc on the thermal stability of JP-7 fuel. Test results on two samples, unavailable for reporting at that time, are included here. The JP-7 base fuel for this earlier work was designated 10-12-T.

The base fuel for the more recent part of the program was a JP-7, here designated Fuel A. Analysis for fuel system icing inhibitor (FSII) indicated a trace, less than 0.02% by volume, which may well reflect the peculiarities of the test method rather than any actual FSII in the fuel. Analyses for metal content of the fuel showed less than 5 parts per billion for each of the four metals involved. During the program, the fuel was stored in two aboveground, 1000-gallon, unlined steel tanks, vented to the atmosphere. The fuels are designated A-8 and A-9 to differentiate the two storage tank lots.

In the earlier work, the 10-12-T fuel was blended with lead and zinc naphthenates to prepare concentrates containing 250 and 2000 ppm of metal, respectively; these were kept in cold storage in one-gallon amber glass bottles. No precipitate or other evidence of insolubility was observed. Final test blends, generally 14 gallons, were prepared in stainless steel containers by adding the concentrate to 10-12-T fuel. Samples of concentrates and test blends were submitted to Monsanto for metal content determination.

In the more recent part of the program, similar concentrates of iron, lead, zinc, and copper naphthenate were blended with Fuel A-8, each concentrate containing 1000 ppm of a single metal. These concentrates were blended in one-gallon clear glass bottles and stored in the dark at ambient temperature. Cold storage had been ruled out previously because of a solubility problem with the iron naphthenate. Even at ambient temperature, the iron concentrate was quite unstable, and precipitate could be observed within about two weeks after blending. Frequent preparation of fresh concentrate was required.

Test blends were prepared from these concentrates in the same manner as in the earlier work, here using JP-7 Fuel A-8 or A-9.

As reported previously⁽²⁾, test blends in the 10-12-T series containing lead in the parts-per-billion concentration range lost much of this lead at some stage in the sample handling. It was suggested by Dr. Scribner of Monsanto that the most likely cause of the lead loss was adsorption on the interior surfaces of the glass sample bottles. In order to eliminate the possibility of such loss, a new sampling technique was adopted for lead samples: Each sample bottle is precleaned with hot nitric acid, distilled water, and reagent-grade acetone; a fuel sample of the proper size is poured into the bottle and weighed; this sample is then analyzed, using nitric acid for quantitative transfer of the lead from the sample bottle.

In the current series of tests and in the 10-12-T tests reported here, this sampling technique was applied to all metal naphthenate solutions, both test blends and concentrates, to prevent any recurrence of similar problems with the other metals. The only change in technique that was necessary was the use of hot hydrochloric acid instead of nitric acid to prepare bottles for iron-containing fuel blends.

Metal contents of the original naphthenates and of the concentrates, as determined by Monsanto, are listed in Table 3. The metal contents found for the naphthenates were close to the nominal contents. Also, the metal contents found for the concentrates were close to the nominal (added) values. Agreement was particularly good for the iron and copper concentrates in the new series, based on one analysis of each. The iron concentrates were found to be quite unstable in storage, and additional concentrates have been prepared and samples submitted for analysis to make sure that the calculated amounts of iron are really being transferred via the concentrates to the test blends. No serious metal loss problems have been encountered with any of the concentrates.

In contrast, serious problems still exist with metal loss from the final test blends containing less than 1 ppm metal. The results from analysis of such blends are shown in Table 4. It is apparent that the recovery of lead

TABLE 3. METAL CONTENTS OF NAPHTHENATES
AND FUEL CONCENTRATES

Naphthenate	Nominal concentration, % metal	% metal found			Average % metal
<i>10-12-T series</i>					
Z-1 (zinc)	8.0	8.34*	8.39†	8.43‡	8.39
L-1 (lead)	24.0	24.2**	24.2**		24.2
<i>JP-7, Fuel A series</i>					
Z-2 (zinc)	8.0	7.99**	7.99**	8.02**	8.00
L-2 (lead)	24.0	23.97**	24.09**		24.03
C-2 (copper)	8.0	8.13††	8.11††	8.06††	8.10
I-2 (iron)	6.0	5.84‡‡	5.82‡‡		5.83
Metal-fuel concentrate	Nominal concentration, ppm metal	• ppm metal found			Average ppm metal
<i>10-12-T series</i>					
S-1 (zinc)	2000	2090**	2090**		2090
S-II (lead)	250	247***			247
<i>JP-7, Fuel A series</i>					
S-III (iron)	1000	1005	1000‡‡		1002
S-IV (copper)	1000	1005	1004‡‡		1004
S-V (iron)	1000		†††		-
S-VI (lead)	1000		†††		-
S-VII (zinc)	1000		†††		-
<p>*Organic material was destroyed with sulfuric-nitric acid. Metal was titrated at pH 10 with (ethylenedinitrilo) tetra-acetate (EDTA) using Eriochrome Black T as indicator. †Two-phase titration with EDTA using Zincon as indicator and 1:1:1 isopropyl alcohol-benzene-water. ‡Sample was dissolved in toluene and the metal ion was extracted with aqueous acid and subsequently titrated at pH 10 with EDTA. **Sample was dissolved in toluene and the metal ion was extracted with aqueous acid and subsequently titrated at pH 5 with EDTA using Xylenol Orange indicator. ††Sample was dissolved in toluene and the metal ion was extracted into aqueous acid and subsequently titrated at pH 5.5 with EDTA using PAN indicator. ‡‡Metal ion was titrated with EDTA at pH 3 using salicylic acid indicator after destruction of organic matter with sulfuric-nitric acids. ***Two-phase titration with EDTA in the presence of 1:1:1 isopropyl alcohol-water-fuel; Xylenol Orange indicator at pH 5. †††EDTA titration at pH 4.0 with 1-(2-pyridylazo)-2-naphthol indicator after destruction of organic matter with sulfuric-nitric acid. ‡‡‡Analytical results unavailable in time for inclusion in report.</p>					

is still very poor, despite the improved sampling technique. In the 10-12-T series, for blend M-18, less than 10% of the 50 ppb added lead was found in the analysis. In view of such problems, several test blends were checked for metal content at the start and end of the fuel coker test series on the particular blend. Such pairs of metal-content results are designated A and B in Table 4. The "A" samples are taken within 1 day after preparing the blend; the "B" samples are taken at the end of the coker test series, generally 4-7 days later.

The results for the iron blends indicate fairly good agreement between the amounts added and found, with recoveries mostly in the 80-95% range. For two blends, the A-B results indicate slight losses of iron during the period of running the coker test series, but the results on the whole are considered satisfactory for present purposes. These comments apply to iron contents in the 100-500 ppb range; no data were available for lower concentrations at the time of reporting.

The results for copper blends indicate fairly good agreement between amounts added and found at the higher concentrations, and excellent agreement at the lower concentrations of 100 ppb and less. Also, the A-B values indicate no significant losses of copper from the blends during the period of running the coker test series.

TABLE 4. METAL CONTENTS OF FUEL-NAPHTHENATE TEST BLENDS

Sample no.	Metal added or sought	Amount added, ppb	Amount found, ppb
<i>10-12-T series</i>			
10-12-T	Fe	0	5
10-12-T	Cu, Zn	0	<5
10-12-T	Pb	0	8
M-16A	Pb	375	323
M-16B*	Pb	375	156
M-18	Pb	50	9
M-19	Zn	100	†
<i>JP-7, Fuel A-8 series</i>			
JP-7, Fuel A-8	Fe, Cu, Pb, Zn	0	<5
M-20	Fe	500	453
M-22A	Fe	500	484
M-22B*	Fe	500	424
M-21	Fe	200	174
M-23A	Fe	200	183
M-23B	Fe	200	160
M-24	Fe	100	†
M-25	Fe	100	133
M-41A	Fe	25	†
M-41B*	Fe	25	†
M-42A	Fe	5	†
M-42B*	Fe	5	†
M-32	Cu	500	451
M-26	Cu	200	146
M-27	Cu	200	†
M-29	Cu	100	98
M-28	Cu	50	58‡
M-31	Cu	50	57
M-30	Cu	25	32
M-37A	Cu	25	29
M-37B*	Cu	25	31
M-39	Cu	15	17
M-40A	Cu	15	22
M-40B*	Cu	15	†

TABLE 4. METAL CONTENTS OF FUEL-NAPHTHENATE TEST BLENDS (Cont'd)

Sample no.	Metal added or sought	Amount added, ppb	Amount found, ppb
<i>JP-7, Fuel A-8 series (cont'd)</i>			
M-33	Cu	5	8
M-34	Cu	5	7
M-36	Cu	5	6
M-38A	Cu	5	9
M-38B*	Cu	5	10
M-35	Cu	0	5
M-54	Zn	200	†
M-48A	Zn	100	†
M-48B*	Zn	100	†
M-51A	Zn	100	†
M-51B*	Zn	100	†
M-49A	Zn	75	†
M-49B*	Zn	75	†
M-47	Zn	50	†
M-44	Zn	25	†
M-43	Zn	5	†
M-53A	Pb	500	431
M-53B*	Pb	500	253
M-52A	Pb	200	181
M-52B*	Pb	200	99
M-50A	Pb	100	37
M-50B*	Pb	100	6
M-46	Pb	50	14
M-45	Pb	25	<5
<i>JP-7, Fuel A-9 series</i>			
M-56A	Zn	100	†
M-56B*	Zn	100	†
M-57A	Pb	435	†
M-57B*	Pb	435	†
M-55A	Pb	300	224
M-55B*	Pb	300	†
<p>* Lettered samples A and B represent samples drawn (A) at same time as first coker test and (B) after last coker test respectively. † Results unavailable at time of reporting. ‡ Laboratory analysis results indicate this blend was possibly contaminated.</p>			

At the time of reporting, no zinc-content data were available for the current series of 100 and 200 ppb blends. As reported previously⁽²⁾, zinc blends in the 500-6000 ppb range had shown zinc contents (by analysis) substantially lower than the amounts added, but these data were obtained before improving the sampling technique.

The results for the lead blends (Table 4) are generally similar to those reported previously.⁽²⁾ Initial samples taken within one day after blending show considerable loss of lead, particularly at added concentrations of 100 ppb and less. Further loss occurs while the coker test series is being run, as demonstrated by the drop in metal content in the A-B pairs. During such a testing period, the fuel is stored in the stainless steel blending container. In the earlier 10-12-T series, lead loss in the sample bottles was undoubtedly a major factor. For the more recent work, however, it must be concluded that lead is lost either by adsorption on the stainless steel container walls or by actual precipitation that cannot be detected visually.

In any case, the lead losses were often reflected in the coker results on a given fuel blend. Interpretation of such coker data is difficult, since the fuel thermal stability often appears to improve as the testing progresses and the lead concentration decreases.

The metal contents reported in the following discussion of thermal stability are the nominal amounts added, but the uncertainties in actual metal contents must be kept in mind when interpreting the thermal stability test results, particularly those on lead-containing blends.

c. Gas-Drive Coker Results on Metal Naphthenate Blends

Breakpoints for the test fuel blends are listed in Table 5. The "base fuel" samples shown in this table are samples taken from 55-gallon lined drums used for fuel transfer from the storage tanks to the blending operation. The "control fuel" samples are those taken from the stainless steel blending containers without any added metal. Some of the tests were run with the SwRI coker configuration (Figure 2) with nonstandard fuel reservoirs; the later tests were run in the same configuration but with standard CRC fuel reservoirs. No differentiation is made between tests conducted with standard or nonstandard fuel reservoirs. Breakpoints are in many cases composite results based on several fuel blends. The previous series (10-12-T) is included and brought up to date for comparison.

Interpretation of these results is complicated not only by uncertainty as to what metal contents are actually "seen" by the fuel coker, but also problems in interpreting off-color deposits, as discussed in subsequent paragraphs. Here we will attempt only a qualitative comparison of results on the basis of added-metal contents.

The two JP-7 fuels used in this work were comparable in stability as measured by breakpoint, the Fuel A breaking at 650-700°F and Fuel 10-12-T breaking at 625°F. Effects of metals on these two fuels would be expected to be at least of the same order of magnitude. It is believed that loss of metals, particularly lead, is the major source of discrepancies in the data, and that difference between the two fuels plays a relatively minor part.

Qualitatively, we can conclude that as little as 15 ppb copper, 25 ppb iron, or 100 ppb zinc can cause significant degradation of JP-7 thermal stability. The analogous value for lead was 125 ppb of added metal in one series of tests and 500 ppb of added metal in another series, with the true lead contents of the coker feedstocks much lower than these values. The most surprising aspect of the threshold metal-content values is the low level of dissolved iron concentration at which thermal stability effects are observed. Certainly this amount of iron could be picked up very easily by JP-7 fuels that were handled in unlined steel equipment under improper conditions.

The preceding conclusions and comments are based on tests in which the metals were present in the form of salts of organic acids; this form should be fairly typical of the contaminant metals encountered in service. Some thought has been given to determining whether the naphthenate portion of the metal-salt molecule or the mineral spirits used as a diluent in the commercial naphthenates can have any effect on fuel thermal stability. No feasible approach to this question has yet been found.

Any more profound conclusions on the effects of these metals on thermal stability will have to wait for completion and analysis of the data for the entire group of tests. Meanwhile, certain observations of tube deposit phenomena are of independent interest and are discussed here.

TABLE 5. EFFECT OF METAL CONTENT ON FUEL BREAKPOINT IN GAS-DRIVE COKER

Metal added, ppb	Breakpoint, °F	Remarks
<i>JP-7, Fuel A-8</i>		
None (base fuel)	675-700 (tube)	Occasional test passes in the breakpoint range.
None (control fuel)	650->700 (tube)	One set of control tests passed up to 700°F.
5 Fe	>650	
25 Fe	550 (tube)	Occasional tube failures as low as 500°F.
100 Fe	550 (tube)	One test passed at 500°F.
200 Fe	500 (tube)	Occasional filter failures as low as 350°F.
500 Fe	400 (tube)	
5 Cu	650-675 (tube)	Occasional tube failures for one blend as low as 550°F.
15 Cu	575 (tube)	Results erratic at 550°F.
25 Cu	550 (tube)	One failure at 525°F.
50 Cu	525 (tube)	Occasional tube failures below 525°F.
100 Cu	550 (tube)	
200 Cu	525 (tube)	Occasional tube failures and erratic filter plugging below 525°F.
500 Cu	450 (tube)	Tube failures accompanied by heavy filter plugging (ΔP).
25 Pb	>650	
50 Pb	>650	
100 Pb	>650	
200 Pb	>650	
500 Pb	600 (filter)	Filter plugging (ΔP) for all test temperatures; 575°F marginal pass.
5 Zn	>650	
25 Zn	>650	
50 Zn	>650	
75 Zn	650 (tube)	Occasional tube passes at 650°F.
100 Zn	550 (tube)	Tube passes as high as 650°F, 11 tests run.
200 Zn	550 (tube)	
<i>JP-7, Fuel A-9</i>		
None (control fuel)	>700	
300 Pb	>700	Occasional filter plugging (ΔP).
435 Pb	625 (filter)	Filter plugging for all tests.
100 Zn	650 (tube)	One tube failure at 625°F; only 6 tests run.
<i>JP-7, Fuel 10-12-T</i>		
None	625 (tube)	
50 Pb	575 (tube)	
125 Pb	500 (tube)	
250 Pb	500 (tube)	Results erratic at 450°F.
375 Pb	300 (filter)	No tube failures up to 350°F.
530 Pb	300 (filter)	Occasional tube failures above 375°F.
100 Zn	350 (filter)	Occasional tube failures at filter breakpoint.
500 Zn	350 (filter)	Tube failure at 375°F.
1000 Zn	375 (tube)	Filter also plugged at 375°F.
3000 Zn	350 (tube)	Results erratic at 325°F.
6250 Zn	350 (tube)	Only two tests run.

In rating coker tube deposits, iridescent "peacock" colors are sometimes observed. These are commonly considered to be thin deposits, somewhat equivalent to normal-color deposits corresponding to a No. 2 color code. The ASTM D 1660 instructions for rating such peacock deposits state that they are to be recorded as "P" in rating the tube sections but are not to be used in arriving at the final "maximum" rating that is the single number reported for the test. The ASTM instructions do not deal with deposits of any color other than peacock or the normal brownish hue. In practice, odd-colored deposits are sometimes encountered when testing supposedly normal fuels from regular production. As expected, "off-color" deposits were encountered with great frequency in the tests reported here, and in some cases these could be related to type and content of metal.

For the iron blends, heavy peacocking was observed in a number of tests. The color was rather far on the greenish side and was sometimes wipable, sometimes not. It occurred *only* among tests on 500-ppb iron blends that also gave failures on the basis of normal-color deposits. It did not occur for any tests at lower iron concentrations.

For the zinc and lead blends, there were several instances of bluish-white "haze" at the hot end of the tube. Such deposits were observed frequently in tests on a 435-ppb lead blend and on 100-ppb zinc blends. The bluish-white haze ranged from faint to moderately heavy and appeared at the hot end of the coker tube, almost always adjacent to "failing" deposits of normal color (Code 3 or darker). The bluish-white deposits actually did not look like deposits, but rather more like reflected bluish-white light on the tube. The color was quite characteristic and could not be mistaken for peacocking or for the blue deposits so frequently caused by copper. The blue-white haze usually wiped off quite readily. The presence of these particular deposits was very specific to certain fuel blends: i.e., it would not appear over a wide range of concentrations of lead or zinc. Since these deposits were almost invariably associated with darker deposits of normal hue, they did not cause any problems in assigning pass-fail ratings to the test results.

For the copper blends, random peacocking occurred in tests at the 500-ppb level. However, the characteristic feature for the copper blends was the occurrence of blue deposits in nearly all tests on blends with 25 ppb or more copper. The color was typically a light blue, appearing as a band covering as much as three inches of tube length and often extending completely around the tube. The blue bands for the higher concentrations (200 and 500 ppb) were generally darker and larger than for lower concentrations, and the blue bands were often divided into several smaller, separate bands. In these instances, the entire deposit would sometimes appear in a chevron pattern with multiple color bands in a distinct "V" pattern on the tube with the tip of the "V" pointed toward the hot end of the tube. In such cases, the deposit color was sometimes light green toward the tip of the "V," grading into light blue at the other end. This chevron pattern did not appear at copper contents below 200 ppb; areas of continuous light-blue stain were generally present at copper contents of 25-100 ppb.

All of the blue deposits from copper blends occurred upstream of the hottest position of the tube. Evidently these deposits form at tube-surface temperatures lower than those required for "normal" deposits with a given fuel. The positions of the blue deposits were correlated with tube surface temperature, using the relations given in Reference (9); it was found that the blue deposits formed at a tube-surface temperature of about 475-500°F, regardless of the metal concentration of the blend and regardless of the test temperature (fuel-out temperature).

All of the blue deposits were wipable, i.e., they were removed completely by wiping the tube once with a soft lab towel.

The presence or absence of blue deposits had nothing whatever to do with the presence or absence of normal-colored deposits on the rest of the tube. For instance, in 35 tests on blends containing 50-500 ppb of copper, the blue stain appeared in all but one test. This same group included tests above and below the "breakpoint" as defined by the normal-colored deposits; i.e., it included tests with preheater ratings of well below 3 as well as tests with preheater ratings of 3 and above. The nature and amount of blue deposits appeared to vary primarily with the copper content of the test fuel, while the nature and amount of normal-colored deposits was influenced by test temperature and copper content.

In 24 tests on blends containing 5 ppb of added copper, blue deposits were observed only once; and in 12 tests on blends containing 15 ppb of added copper, no blue deposits whatever were observed. These groups of tests also included "failing" and "passing" tests as rated by the normal-colored deposits.

In assigning the breakpoint temperatures to these fuel blends, we have chosen arbitrarily to ignore the blue deposits and to base the ratings solely on the normal-colored deposits. The question of how to rate tubes having odd-colored deposits (other than peacock) is not covered in ASTM D 1660, and, in fact, no firm guidelines exist. We understand that certain British work has demonstrated a drop in heat transfer coefficient caused by blue deposits at temperatures too low to cause any "failing" deposits in coker tests. However, so far as we know, details of this work have not been made generally available in the United States. The problem of rating abnormal-colored deposits does merit further study, since such deposits are encountered in real fuels as well as in laboratory blends of the type tested here. One must doubt whether such deposits can ever be rated adequately by any method of visual rating or by light-reflectance techniques.

The blue deposits obtained in the tests reported here did show a rather startling color change when viewed in ordinary "cool white" fluorescent light in the laboratory, rather than in the Tuberator. The light blue color observed in the Tuberator generally became a distinct brown under fluorescent light, sometimes corresponding quite closely to a No. 3 or 4 color standard. This change is the reverse of what would be expected, since fluorescent lighting will ordinarily accentuate rather than suppress the blue tones. This observed change does introduce even more unknowns into the questions of deposit significance and rating techniques.

A summary of the presence or absence of blue deposits in testing fuels with various contents of added copper is given in Table 6. Also shown are the corresponding breakpoints based on "normal" deposits only. The 5 ppb of added copper had little or no significant effect on breakpoint, and blue stain was observed only once. This small amount of copper could well be "lost" before it reaches the coker hot section, and the presently available analytical techniques are not adequate to follow changes in

copper content in this extremely low range. With 15 ppb of added copper, the breakpoint dropped some 100°F, but no blue deposits appeared. With 25 ppb of added copper, there was very little added effect on breakpoint, but blue deposits did show up in most of the tests. Further increases in added copper content up to 200 ppb gave only moderate decreases in breakpoint, and the blue deposits persisted and generally became greater in amount. Only at 500 ppb of added copper was there a marked drop in breakpoint.

One of the most interesting features of these results is the marked effect of as little as 15 ppb of added copper on the fuel breakpoint, and the lack of any further drastic effects when up to 100 or even 200 ppb of copper is added. Apparently a very small amount of copper is needed to catalyze the reactions that form "normal" deposits in this particular fuel. When more copper is added, the excess simply reacts and forms blue deposits on the tube at temperatures below those needed to form the "normal" deposits. This hypothesis is a reasonable explanation of the observed facts, but it should not be expected to apply at still higher copper concentrations or with other base fuels.

d. Auxiliary Tests

Various samples of the JP-7 Fuel A test blends were checked for WSIM and other specification requirements, to determine whether the added metals had changed any fuel characteristics other than thermal stability. Test results are still incomplete. Thus far, it has been found that no significant changes were caused by 5-100 ppb added copper, 5-25 ppb added iron, 25-50 ppb added lead, or 5-50 ppb added zinc. Blends with 50 and 100 ppb added copper gave essentially the same WSIM values as the base fuel. Blends with 500 and 200 ppb added iron gave WSIM values lower than that of the base fuel by some 30 units on the 100-WSIM scale.

TABLE 6. OCCURRENCE OF BLUE STAINS IN COKER TESTS ON COPPER-CONTAINING BLENDS

Copper added, ppb	Total number of coker tests	Coker tests with blue stain	Test fuel breakpoint, °F
500	6	6	450
200	12	12	500-525
100	5	5	550
50	12	11	525
25	12	8	550
15	12	0	575
5	24	1	650-675

e. Status and Future Plans

When the gaps in the data for the present series have been filled in, this work on the effect of single metals on JP-7 thermal stability will be complete. An attempt will be made to analyze the data so as to relate fuel thermal stability to actual metal contents of the samples at the time of the fuel coker tests. This will be difficult, since the test series had been predicated on obtaining stable metal solutions. However, it should be possible to obtain a better quantitative picture than is now available. Some additional work may be necessary to define metal loss rates, in order to predict actual metal contents of the test blends.

Several other investigations of contaminant-metal effects had been planned, but the difficulties encountered with the present approach suggest that broad changes should be made. Some consideration has been given to dispensing with the metal analyses altogether, simply adding known amounts of metal concentrates to the fuel in the coker reservoir immediately before testing. Consideration has also been given to use of a testing device other than the gas-drive coker. The possible advantages of such changes must be balanced against the loss of continuity of background data already obtained. These questions will be resolved in the near future.

Work is planned in determining whether combinations of two or more contaminant metals have synergistic effects in degrading fuel quality. This question is of considerable practical importance, since fuel handling systems obviously cannot be relied upon to contribute a single metal to the fuel. It is also of some importance to determine whether lead in the form of tetraethyllead will behave in the same manner as lead in the form of the naphthenate. If this proves to be the case, studies of other metals may be warranted. Finally, it would be desirable to extend the studies to other types of fuels and to determine whether commonly used fuel additives will change the threshold levels at which metal contents become important.

4 EFFECTS OF COATINGS ON FUEL THERMAL STABILITY

Four epoxy coatings for fuel-tank interiors were checked for effects on fuel thermal stability and other fuel properties, using procedures generally similar to those specified in MIL-STD-1262. These tests were intended to determine the suitability of the coatings as qualified MIL-C-4556C materials for use with aircraft turbine-engine fuels.

The four coatings, designated here as A, B, C, and D, were supplied for this work in the form of coated panels.

Effects on fuel thermal stability were evaluated by soak tests in JP-7 fuel, using two coated panels with 10 gallons of test fuel to give an area/volume ratio of 50 in²/gal. For each coating tested, two such assemblies with panels and one control assembly without panels were stored for 35 days at 70-90°F, after which the fuels were tested in gas-drive cokers using the standard CRC configuration.

TABLE 7. EFFECT OF EXPOSURE TO COATINGS ON JP-7 FUEL BREAKPOINT IN GAS-DRIVE COKER

Epoxy coating code no.	Heater breakpoint, °F		
	Base fuel	Control fuel	Soak fuel
A	650-700	700	675
B (test series I)	675	675	625
B (test series II)	650-675	700	650-675
C	650-675	700	675-700
D	675	650-675	650-675

The results of these tests are summarized in Table 7. For coatings A, C, and D, fuels exposed to the coated panels gave breakpoints at least as high as those of the base fuel and control fuel. Fuel exposed to coating B gave a breakpoint 50°F lower than that of the base fuel and control fuel. A repeat of this series of tests indicated that coating B had no degrading effect on thermal stability.

In all of these tests, breakpoints were 625°F or higher, and the variations appeared to be caused by the usual difficulties with lack of precision in coker tests at these high temperatures rather than any effects of

previous exposure to the coatings. All breakpoints were based on preheater tube ratings; filter plugging was observed only in one test. It is concluded that these coatings do not have any significant effect on JP-7 thermal stability.

The coatings were also checked in 2-gallon exposure tests with JP-4 and JP-5 fuels. Inspection tests on the fuels indicated that exposure to the coated panels had not produced any significant changes in fuel properties.

5. EFFECT OF PIPE SEALANT ON JP-7 THERMAL STABILITY

Since JP-7 fuel systems are constructed for the most part of materials that will not contribute contaminants to the fuel, the role of "minor" materials in the fuel system may become quite important. One such material is the sealant used in pipe joints. A sealant identified as Code No. 555 was furnished for evaluation of its effects on JP-7 fuel thermal stability.

Two 55-gallon epoxy-lined drums were filled with a JP-7 fuel, designated Fuel G, filtering through a 0.45- μ membrane filter as the fuel was transferred to the drums. Six cubes of the Code 555 sealant were placed in each drum, giving a nominal surface-to-volume ratio of 2.1 in²/gal. The true ratio during the test is indeterminate because of blocking of some sealant surfaces by contact with the bottom of the drum and because of changes in the sealant surfaces during the fuel storage period. Fuel samples were drawn after 1, 2, 4, and 6 months of storage at indoor ambient temperature.

Thermal stability of the base fuel and of the fuel after exposure to the sealant was determined by tests in the research fuel coker with the fuel reservoir held at 300°F. The breakpoints for these tests are summarized in Table 8. The base fuel gave a sharp breakpoint at 550°F with no evidence of filter plugging; the fuels after exposure to the sealant gave breakpoints about 25-75°F lower, with numerous instances of filter plugging. Most of the filter plugging occurred in the 450-475°F range. Since no tests were run on the base fuel in this temperature range, one cannot be certain that the sealant caused the plugging behavior of the test fuels. The intermediate sample taken at 4 months had the lowest breakpoint and showed the most plugging of the four samples; the 6-month sample from the same drum seemed to have recovered and in fact was essentially as good as the original base fuel.

TABLE 8. EFFECT OF SEALANT ON JP-7 FUEL BREAKPOINT IN RESEARCH COKER

Reservoir temperature, 300°F

Months of storage with sealant	Heater breakpoint, °F	Remarks
0	550	Breakpoint very sharp; no ΔP .
1	500-525	Some ΔP in 450-475°F range.
2	500	Some ΔP at 450°F.
4	500	Some ΔP at 450°F, failing ΔP at 475°F.
6	525	No ΔP .

It will be noted that the greatest decrease in breakpoint was only 75°F. This is fairly minor considering the relatively large amounts of sealant present in each drum. It is concluded that this sealant can cause some degradation of fuel thermal stability, but that no drastic effects were found for the one particular fuel that was tested.

Samples of the base fuel and the 6-month soak fuel were found to conform to JP-7 specification, MIL-T-38219. Specification test results on these two fuels showed no significant differences. The existent gum content increased from 0.2 mg/100 ml (base fuel) to 2.0 mg/100 ml (6-mo fuel), but this is hardly a drastic change.

The sealant cubes were removed from each drum after fuel sampling from that particular drum had been completed. The sealant from the first drum (2 months) appeared to be unchanged, except that some dark brown, viscous material cozed from the sealant cubes when they were stored dry for a few days after removing them from the fuel. The sealant from the second drum (6 months) was badly deteriorated and some of it had fallen apart into layers resembling long, narrow crystals. The sealant was much less pliable than it was originally.

Since this degradation of the sealant was not accomplished by any serious degradation of fuel thermal stability, it is concluded that any fuel-soluble substances in the sealant must have at most minor effects on fuel thermal stability.

6. EFFECT OF FUEL ADDITIVES ON THERMAL STABILITY

a. Effect of "RT" Antioxidant on Thermal Stability

This antioxidant has been proposed for use in military and commercial jet fuels. Tests were run to check for any effects on JP-4 and JP-5 fuel thermal stability at standard test conditions, and a more severe evaluation was performed in JP-7 fuel.

Nonadditive JP-4 and JP-5 fuels were prefiltered (Whatman 2V paper) and then blended with RT at 16.8 lb/1000 bbl, which is twice the antioxidant concentration allowed in MIL-T-5624H. A JP-7 fuel (Fuel A-8 as identified in Section II-3) was prefiltered through a 0.45- μ membrane filter and blended with RT at 8.4 lb/1000 bbl. For each of the three types of fuel, a control fuel without antioxidant was prepared in the same manner.

The fuel samples, with and without antioxidant, were stored at 70-90°F for one month. Standard fuel coker tests were run at 300/400°F on the JP-4 and JP-5 fuel samples in accordance with ASTM D 1660. The JP-7 fuel samples were evaluated in gas-drive cokers in CRC configuration, testing at several temperatures to establish breakpoints. The results of these tests are shown in Table 9.

TABLE 9. EFFECT OF "RT" ANTIOXIDANT ON FUEL THERMAL STABILITY

Fuel grade	Thermal stability test	Base fuel*	Control fuel*	Fuel + RT*
JP-4	Standard coker, 300/400°F			
	Tube rating	1	4†	1
	Filter ΔP , in. Hg	0.0	0.0	0.0
JP-5	Standard coker, 300/400°F			
	Tube rating	1	1	1
	Filter ΔP , in. Hg	0.0	0.0	0.0
JP-7	Gas-drive coker, CRC			
	Breakpoint, °F‡	675	625-675	>675

*Base fuel without additives; control fuel same, after 1-month storage; fuel with RT, after 1-month storage.
†Tube rating decreased to 2 after wiping.
‡All breakpoints based on tube ratings; no filter pressure drop.

In the JP-4 fuel, the single tests at standard temperature conditions indicated a possible beneficial effect of the antioxidant, since the control fuel without antioxidant gave a preheater failure after one month of storage. No effect was detected in the JP-5 fuel; all tests gave passing results. The results on the JP-7 base fuel and control fuel (stored one month) were in line with past history of this particular fuel, which typically had a breakpoint of 675°F with random failures as low as 625°F. The fuel with antioxidant gave two good results at 675°F, indicating a probable beneficial effect of the antioxidant.

Specification testing of the fuel showed no detrimental effect from the antioxidant before or after storage. No detailed evaluations were made of the antioxidant performance of the material.

Based on these tests, it is concluded that the RT antioxidant should have no adverse effects on fuel thermal stability and may have beneficial effects.

b. Effect of Fuel Marker on Thermal Stability

A fuel marker, Code XP, was evaluated at a concentration of 0.5 lb/10,000 gallons in JP-4 base fuel by means of standard coker tests (ASTM D 1660) at 300/400°F. The fuel marker had no effect in these tests. There was no filter plugging in the test on the base fuel or in the two tests on the fuel containing the XP marker. The preheater rating was a Code 1 for the base fuel, Code 1 and Code 2 (wiping to a Code 1) in the two tests on the fuel plus marker.

Further evaluations of the XP fuel marker were run in a JP-5 base fuel, using the Alcor JFTOT unit for thermal stability testing. These tests showed that the base fuel breakpoint of 520-525°F was unchanged by the addition of the XP marker at a concentration of 0.5 lb/10,000 gal. A few instances of filter plugging were observed in these tests, but these occurred at random and were not related to the presence or absence of the marker.

The XP fuel marker does not cause any detectable changes in thermal stability as measured by the standard coker (JP-4 fuel) or the Alcor JFTOT unit (JP-5).

7. MISCELLANEOUS THERMAL STABILITY EVALUATIONS

a. Coker Tests on Various Fuels

Extensive thermal stability testing has been conducted in support of Air Force in-house and contractual research programs. Test results on various fuels in standard, research, and gas-drive fuel cokers are listed in Table 31 (Appendix) for the record.

Most of this work was performed to establish breakpoint values for correlation purposes of other programs or to establish conformance of a given fuel batch to specifications. The data in Table 31 do illustrate certain problems in repeatability of results and the uncertainties that are introduced into determinations of breakpoint. These problems do not appear to be confined to any one type of coker. It is well known that the repeatability of standard coker results leaves something to be desired, and this problem has not been solved by the use of modified cokers. One thought that occurs when looking at poorly repeatable results is how much of this lack of precision should be attributed to the test apparatus and procedure and how much to possible variations in the fuel sample from test to test. Our work on the effects of dissolved metals had indicated large changes in thermal stability that were caused by an unstable fuel blend. In a sense, all practical fuels are unstable systems, since pickup or loss of minor amounts of surface-active substances can change their properties very radically. This possible source of nonrepeatability in thermal stability testing has not received as much attention as it deserves.

b. Thermal Stability of TS/JP-4 Mixtures

As reported previously⁽²⁾, "thermally stable" (TS) fuel can be degraded significantly in thermal stability when mixed with as little as 5% JP-4. In this previous work, the TS fuel was extremely stable, with a gas-drive coker breakpoint above 675°F, and the JP-4 fuel was quite good for its class, having a standard-coker breakpoint of 400°F. With these two fuels, 10% JP-4 brought the stability of the TS fuel about halfway down to the JP-4 level, and a 50-50 blend of the two fuels had the same stability as the JP-4 itself.

It appeared desirable to obtain similar data on other batches of fuel, since the high-stability TS fuel used in the previous work was rather unusual. For this additional work, an "in-flight" JP-4 fuel was obtained, containing all additives, and was checked for conformance to all requirements of MIL-T-5624G before it was used in the stability work. Selection of a suitable TS fuel proved to be very difficult. Initially, we were looking simply for a TS fuel that would pass a standard-coker test at 450/550°F, in conformance with the current MIL-T-25524B. None of the TS fuels that were available would meet that requirement. It was assumed that these had been purchased to an earlier requirement of 400/500°F. Actually, testing at 450/550°F in the standard coker is rather difficult, and strange results must be expected at times. In any case, for this work, a TS fuel passing a 400/500°F standard-coker test was accepted for use.

Blends containing 2 to 50% JP-4 were prepared in 15-gallon lots and tested in the gas-drive coker in the CRC flow configuration. The results obtained on the blends and on the two individual fuels are summarized in Table 10, along with earlier data on TS/JP-4 mixtures.

It will be noted that the JP-4 fuel used in the current program had little margin in thermal stability in relation to the specification requirement of 300°F. For the TS fuel, the preliminary standard-coker results showing a failure at 450°F with filter plugging were *not* confirmed by the gas-drive coker results, in which no failures could be obtained even at 600°F. This discrepancy between the standard and gas-drive coker results probably reflects the

TABLE 10. THERMAL STABILITY OF TS/JP-4 MIXTURES

Vol % JP-4 in TS fuel	Breakpoint, °F*	
	Standard coker	Gas-drive coker
0 (straight TS)	450†	>600‡
2		575‡
5		>575‡
10		>575‡
25		525
50		500
100 (straight JP-4)	325	350†
<i>Earlier data</i>		
0 (straight TS)	400	>675
5		625-650
10		550
50		450
100 (straight JP-4)		450
*Based on tube rating unless indicated otherwise.		
†Based on filter plugging.		
‡Problems with fuel boiling.		

poor operability of the standard coker at 450/550°F, but it could possibly reflect differences in the handling of the preliminary blend and the test blend.

The breakpoints of the blends show only moderate decreases as more JP-4 is added to the blend. Earlier results obtained with two different base fuels were quite different. In the earlier work, it had been concluded that the TS fuel was quite sensitive to small amounts of contamination by JP-4 fuel and that a 50-50 blend was equivalent to the JP-4 itself in thermal stability. In the current work, contamination by JP-4 up to 10% did not have any drastic effects, and even the 50-50 blend was far better than the JP-4 itself. These differences in "response" of TS fuel to JP-4 contamination make it clear that not even approximate guidelines could be established without testing a great many pairs of fuels.

In the current test series, boiling was detected in a number of the coker tests on TS and TS/JP-4 blends, as evidenced by unsteady flow or fluctuations in the manometer reading. In many of the tests at higher temperatures, the test pressure was increased to 275-280 psi to suppress boiling, not always successfully. The observed cases of boiling are fairly well in line with the predicted vapor pressures of the fuels. Most of the cases of boiling occurred when the vapor pressure at the preheater fuel-out temperature was only about 150 psi. Therefore, it is unlikely that any boiling occurred within the

preheater, but rather in the filter, which was 100°F hotter. Such boiling could not promote the formation of preheater deposits via the hot-spot route (boiling in preheater) but might upset the test conditions enough to cause local, temporary overheating in the preheater. In these tests, the boiling problems did appear to be associated with the occasional, random appearance of tube deposits at temperatures well below the nominal breakpoint of the fuel.

The oddest feature of these data is the reasonably good stability of the 50-50 blend. The usual expectation for such mixtures is that the lower-stability component will predominate, and in fact this was observed in the earlier series of tests. Also very surprising is the fact that the JP-4 used in the current series gave severe filter plugging and preheater deposits at fuel-out temperatures from 350 to 450°F, but the 50-50 blend gave no filter plugging at all with temperatures from 475 to 525°F. Decreases in plugging as the temperature is increased is a very common phenomenon in fuel coker testing, but generally tube deposits will show up as the filter plugging disappears. Here, at 475°F fuel-out temperature, the 50-50 blend gave no serious deposits of either kind. It would have been of interest to test the 50-50 blend at lower temperatures to see if we have here a rare case in which failures of both filter and preheater at moderate temperatures will both disappear as the temperature is increased. Such cases have been suspected but never really identified.

There are several possible explanations of why the 50-50 blend was so much better than the JP-4, but all are merely speculative. For instance, it is possible that diluting the JP-4 with TS fuel precipitates or coagulates some "impurity" that is subsequently removed in prefiltration before the coker test.

In this work, as in any study of blend compatibility, each pair of materials is unique. Here, it does not appear possible to work out any general guidelines. All that can be concluded is that minor amounts of JP-4 in TS fuel may cause serious degradation of thermal stability, and such admixtures should be avoided.

8. CRC COOPERATIVE PROGRAMS

During the reporting period, SwRI personnel took part in several cooperative programs in thermal stability that were carried out by CRC and ASTM groups. Some of this work has already been reported through these

channels and will not be repeated here. Work concerned with the Alcor JFTOT coker is discussed in Section II-10. Here we will report certain standard-coker data obtained to provide up-to-date ratings on five fuels being used in JFTOT evaluation programs. These standard-coker data have already been reported to the appropriate CRC group, but are considered to be of enough independent interest to present here also.

Five fuels were tested in accordance with ASTM D 1660-67. Warmup time was controlled very closely and was 20 minutes unless otherwise indicated. The test conditions for each fuel were established during the course of the program by consultation with the other laboratories involved.

The preheater tube from each test was rated by four different persons, taking precautions to ensure that the readings were unbiased. Each tube was rated first within one hour of exposure after the test, and again at least 24 hours later. Each tube was stored in a sealed plastic tube during the period between ratings.

Individual test results and ratings are listed in Table 32 (Appendix). Filter plugging was quite heavy for three of the fuels tested, light but detectable for one fuel (AFFB-4-64), and absent for one fuel (RAF-174). This fuel, RAF-174, gave peacock-colored deposits in all tests; none of the other fuels gave such deposits. The same trend had been found in Alcor JFTOT tests on the same group of fuels. It is probably too much to read any deep significance into this set of facts, but the coincidence of heavy peacocking of the tubes and the absence of filter plugging is quite striking in this group of fuels.

Heater deposit ratings in Table 32 are listed in terms of temperature for inception of deposits corresponding to each color code. Breakpoints are based on the tube temperature corresponding to the inception of a Code 3 deposit or higher (darker). The raters' data giving deposit color vs location were converted to a color/temperature basis by using CRC data⁽⁹⁾ correlating tube temperature with location and fuel-out temperature, taking interpolated values when the actual fuel-out temperature in the tests did not match the temperatures listed in the CRC correlation.

For this work, an inception point for a particular color code is defined as the location on the preheater tube at which the deposit on the tube *first* matches a standard color code; i.e., there are inception points for codes 1, 2, 3, and 4 if each color is present on the tube. Ideally, the tube deposits will define a series of increasing code numbers (darker deposits) as the fuel flow progresses from the cold-end inlet to the hot-end outlet. In actual practice, reversals often occur; i.e., a darker deposit will appear before the lighter deposit. For example, the inception points, progressing in the direction of fuel flow, might be 1-3-2-4 instead of the normal 1-2-3-4. In such instances, the inception points have been reported as found, interpreting the definition of inception point strictly; instances of these reversals are footnoted in Table 32.

Another irregularity in deposit sequence is what we have termed "intermittent" deposits, in which darker deposits alternate with lighter deposits, giving the tube a spotty appearance. This may give "secondary inception points" that can cause large differences in the values assigned by different raters. For example, one rater may see a deposit pattern of 1-0-1-2-3, with the first Code 1 rating at 250°F and the second Code 1 rating at 400°F; he would report an inception point of 250°F for Code 1 deposit. Another rater might not see the first Code 1 deposit and so would report an inception point of 400°F for Code 1 deposit. This problem affects mainly the Code 1 ratings and to some extent the Code 2 ratings; no instances of secondary inception points for Code 3 or 4 deposits have been found. Whenever intermittent deposits are observed, the first appearance of that particular code is regarded as the true inception point, and the secondary inception point is merely indicated by a footnote. These footnoted entries in Table 32 provide a clear description of how the tubes are seen by different raters.

The ratings listed in Table 32 are arranged chronologically; that is, each tube was rated by the four raters in a fixed order. There is no evidence of any short-term trends in color rating; the deposit colors remain stable long enough for all four raters to see the same object.

For a better look at long-term trends in deposit color, deposit inception temperatures are shown in Table 33 (Appendix) as the mean of values from four raters and the corresponding standard deviation. Several of the mean temperatures are based on fewer than four values, indicating that this particular color code was "absent" in the judgment of one or more raters. The "composite breakpoints" are derived by averaging the temperatures (observed

by different raters) at which Code 3 or darker deposit was first observed. Whenever Code 4 deposit was present and the raters disagreed on whether or not Code 3 deposit was present, the composite breakpoint was obtained by averaging some Code 3 inception points along with some Code 4 inception points, so the composite breakpoint does not correspond in all cases to the "average temperature for Code 3 rating."

From Table 33, it can be seen that the deposit inception temperatures did change significantly when the tubes were rerated after 24 hours, particularly for the Code 1 and 2 deposits. Such timewise color changes have also been noted regularly on heater tubes from the Alcor JFTOT coker. For the standard coker data shown here, there was generally little change in the Code 3 and 4 inception temperatures, hence little effect on breakpoints in most cases. In one test on the AFFB-3-64 fuel (test 5750) and in several tests on the RAF-174-63 fuel, the observed breakpoint did change substantially after 24 hours. For the AFFB-3-64 fuel, the 24-hour rating indicated a failure (code 3 deposit), but the 1-hour rating did not. This is a doubtful case, since only two out of the four raters reported the presence of Code 3 deposit in the 24-hour ratings. Also, these two raters were in very wide disagreement on the temperature at which the Code 3 deposit appeared, as evidenced by the large deviation. The two instances of large changes in breakpoint rating between the 1-hour and 24-hour ratings with the RAF-174-63 fuel amounted to +35 and -28°F. The significance of these changes is doubtful, in view of the difficulties in rating these heavily peacocked tubes.

Other than three instances that have been discussed, there were no important changes in breakpoint between the 1-hour and the 24-hour ratings. It is concluded that timewise changes in deposit color did not cause any serious problems in establishing breakpoint ratings for these five fuels.

According to the standard deviation data included for each temperature in Table 33, the raters had much greater difficulty in identifying and agreeing on inception points for Code 1 and 2 deposits than they did for the Code 3 and 4 (breakpoint) inception points. For example, considering both 1-hour and 24-hour ratings, 46 out of the 76 ratings of Code 1 or 2 had standard deviations greater than 25°F, but only 3 out of the 28 composite breakpoint ratings had standard deviations greater than 25°F. The use of Code 1 ratings for analyzing coker data and establishing breakpoints has been suggested.⁽¹⁰⁾ From the comparisons that we have made, using standard coker tubes with the ASTM Tuberator and color standards, it appears that adopting a lighter deposit for rating purposes would merely compound the present rating errors, at least for the standard coker.

The data have been worked up further in illustration of sources of variation in coker ratings. The original visual ratings for each group having identical fuel, test conditions, and rating code have been averaged to give a "multiple rating," and the corresponding "multiple standard deviation" was calculated for each of these sets. Further, the rater-to-rater standard deviation for each group having identical fuel, test conditions, and rating code has been averaged to obtain an "average standard deviation." The multiple standard deviation will be related to the total error (coker test error and rating error), while the average standard deviation is related to the rating error alone. The ratio of these two standard deviations (average/multiple) should give some sort of measure of the relative magnitudes of the two types of error. A more precise definition is not possible, since these quantities were calculated without any great statistical rigor.

The multiple test ratings and the corresponding multiple and average standard deviations are shown in Table 34, along with the average/multiple deviation ratios. It will be noted that the ratio is greater than 50% for 34 of the 48 cases listed, indicating that, for this type of rating of deposit inception temperature, the rating errors are probably at least as important as the remaining errors in the coker test procedure.

It is not planned to attempt a more rigorous analysis of these data, in view of the small number of tests. Presumably, a more rigorous analysis will be made by the CRC, based on these data and those of other laboratories. The comparisons given here will serve to illustrate the relatively large errors in color-code rating and to emphasize the need for improved rating techniques.

9. ERDCO PRECISION FUEL COKER

a. Background

Among the newer approaches to fuel thermal stability testing are two devices developed by equipment manufacturers and submitted to a CRC group for evaluation. These devices utilize the same operating principle as

the standard fuel coker, passing fuel over a heated metal surface and then through a test filter, with ratings based on the color of the hot-surface deposits and the degree of plugging of the test filter. Even though these new devices use the same principle as the standard coker, they are rather beyond the "modified coker" concept, since new approaches in design have been incorporated to eliminate at least some of the major shortcomings of the standard coker and its various modifications.

The two devices are the Erdco Precision Fuel Coker and the Alcor JFTOT (Jet Fuel Thermal Oxidation Tester).

In the current program, an Erdco instrument was made available for a brief evaluation and use in obtaining data for a CRC cooperative program. These evaluations and data are reported here. Similar but much more extensive work on the Alcor JFTOT is reported in Section II-10.

b. Equipment and Procedures

The apparatus, operation, and maintenance procedures for the Erdco Precision Fuel Coker have been described in the manufacturer's operating manual, and only a brief summary will be given here.

The test fuel (3000 ml) is contained in a steel reservoir at ambient temperature, pressurized to 150 psig with nitrogen to drive the fuel through a heated test section and cooler, then out to a waste receptacle. The test section consists of a horizontally mounted tube-in-tube heater with a test filter (not independently heated) mounted directly on the fuel outlet from the heater.

The outer tube of the heater is machined from a solid hexagonal bar of Type 316 stainless steel with a circular bore that is reamed and lapped to a tolerance of 0.001 inch. The inner tube consists of a 800-watt heater, inner tube liner, inner tube, and connectors, resembling a shortened version of the CRC gas-drive coker preheater tube. The inner tube is 6061-T6 aluminum tubing with a wall thickness of 0.060 inch, into which the heater and tube liner are press-fitted as a unit. The outer surface of the inner tube is repolished by the user for each test. In the heater assembly, the fuel flows through an annular space with 0.012 inch radial clearance between the inner and outer tubes. The surface temperature of the inner tube is measured by two thermocouples mounted in the inner tube liner, diametrically opposed, with the active portion (0.040 inch) diameter of their tips pressed against the inside of the inner tube. One of these thermocouples is used for control and the other for reference.

The test filter medium is sintered stainless steel; the pressure drop across the test filter is measured by a standard fuel coker manometer. The flow system includes a 0.45- μ in-line filter ahead of the test section. The fuel leaving the test section passes through a cooler, a micronic in-line filter, a rotameter and flow control valve, and out to waste. Flow rate is controlled manually to 363 ± 2 g/hr as established by gravimetric calibration at the start of each run.

Power input to the heater is controlled automatically to maintain a constant tube surface temperature as indicated by the control thermocouple. The test temperature is monitored by the reference thermocouple and a potentiometer, which are also used to calibrate and correct the temperature controller system before each test. An optional sealed bismuth-freezing-point standard is available for absolute calibration of the controller system.

The temperature profile along the tube is predetermined by the manufacturer and tabulated for each control temperature. These values are used to relate deposit inception points to tube temperature.

Before starting a test, the test section components and fuel inlet lines are disassembled, cleaned, and assembled with a freshly polished inner tube and a new test filter. The reservoir top flange is removed and the reservoir interior is cleaned. The remainder of the flow system (beyond the test section) is cleaned and flushed with a pump-driven system similar to that of the standard coker.

The test fuel sample (3000 ml) is filtered through Whatman No. 2 paper into the fuel reservoir and then aerated for 5 minutes with a small air pump. The reservoir is sealed and pressurized to 150 psig with nitrogen, and

fuel is passed through a flow system to purge the air and set the flow rate. Then power is applied and the test section is brought to the desired temperature. The test period is 5 hours, checking the flow rate every 30 minutes and adjusting if necessary. Filter pressure drop readings are taken every 30 minutes; the test filter may be bypassed if necessary.

After shutdown and disassembly, the inner tube is dipped in pentane and color-rated in a standard ASTM Tubercator equipped with a special 6-inch tube holder. The first rating is made within 30 minutes after removing the tube from the test section; the tube is stored in the original shipping tube and rerated 24 hours later. Inception points for Code 1, 2, 3, and 4 deposits are recorded to the nearest 0.1 inch and translated to metal surface temperatures by means of the temperature profile tables provided by the manufacturer.

For purposes of data analysis and reporting, we have considered the breakpoint to be the deposit inception temperature for Code 3 or darker deposits. This definition is the same as that commonly used with the Alcor JFTOT; both relate to a nominal metal surface temperature rather than to fuel temperature. No criteria have been established for filter plugging breakpoints.

c. Operating Experience with Precision Coker

Test experience for SwRI with this coker has consisted of 20 tests conducted as a part of a CRC program. Based on this rather limited experience, certain observations will be offered on the equipment, procedure, and data obtained.

The temperature controller gave excellent results. The warmup was accomplished automatically with very little overshoot, and stable control was maintained throughout the tests. Very little operator attention was required, no malfunctions were observed, and no maintenance other than calibration was required.

Severe problems in maintaining a constant flow rate were encountered in all tests. The gravimetric method of determining flow rates is very time-consuming and only fairly accurate. The flow rate fluctuated constantly, so that the rig required constant attention throughout the 5-hour test period. Resetting the flow rate on the basis of rotameter settings was at best a guess. Design changes to provide a steady flow rate with minimum attention would be required for any efficient use of the coker in regular testing.

The procedure for assembly, disassembly, and cleaning this unit is rather involved and does not appear to offer any advantage over the standard or gas-drive cokers. Turnaround time for this unit is about the same as for the standard and gas-drive cokers. Some problems were encountered, such as reservoir leaks and difficulty in cleaning the aerator.

The Precision Coker has some advantage in fuel sample requirement (about one gallon) in comparison to the standard coker (five to six gallons) and the gas-drive coker (three gallons). This is not a tremendous reduction in sample size. If a breakpoint could be obtained in a single test, of course, the total sample requirement would be far smaller than in multiple tests on the standard or gas-drive coker. The data that we have obtained with the Precision Coker are insufficient to indicate whether single-test breakpoints are feasible. We suspect that "floating breakpoint" phenomena will be encountered with this unit just as they were with the Alcor JFTOT, since the principle of matching deposit color to tube temperature via a temperature profile is the same for both units.

The Precision Coker includes a large number of desirable modifications but retains a number of the disadvantages of standard and gas-drive cokers. The problems with polishing the tube to a suitable surface, mentioned earlier in this report, remain the same. Color rating problems remain the same, at least until nonvisual rating systems come into use. Mechanical problems appear to be about the same. Flow rate problems, which are minor with the gas-drive coker, have been accentuated by the scaledown in size.

d. Test Results and Discussion

Five fuels were tested in a 20-test series as a part of a CRC cooperative program. These results have been reported to the CRC group involved but will be presented and discussed here as a matter of general interest.

The test procedure was as described in the manufacturer's brochure; the test period was 5 hours, and the flow rate was 363 ± 2 g/hr. Test temperatures were those recommended by the CRC for these particular tests.

The heater tube from each test was rated by four different raters within 30 minutes of tube exposure and again at least 24 hours later. Between the two rating operations, the tube was stored in its original shipping container. Care was taken to assure unbiased ratings. Deposit inception points corresponding to the first appearance of Code 1, 2, 3, and 4 deposits were converted to inception temperatures by means of the tables supplied by the manufacturer. The data were then treated in the same general manner as those obtained in the standard coker for these same fuels (see Section II-8).

The results of individual ratings for tubes from the Precision Coker are shown in Table 35 (Appendix). Also included are the filter plugging data. Three of the fuels gave very little plugging, less than 1 in. Hg; the other two gave plugging on the order of 10 in. Hg in some of the tests. The plugging behavior was very repeatable for the RAF-174 fuel. For the AFFB-9 fuel, plugging was reasonably repeatable at 520°F (9.6, 4.5), but nonrepeatable at 500°F (0.0, 10.0).

The deposit inception temperatures obtained by different raters have been averaged, and the mean values and standard deviations are listed in Table 36 (Appendix). Composite breakpoints are also listed; these were determined as described in Section II-8.

Of the 20 tests that were run, only 11 resulted in breakpoints, and among the 11 the existence of "failing" deposits of Code 3 or higher was not unanimously agreed upon by the four raters. The composite breakpoints that were determined showed standard deviations of less than 25°F. There were no substantial differences between the 30-minute and 24-hour ratings. For the deposit inception temperatures, some 46% of the standard deviations were greater than 10°F, and some 25% of the standard deviations were greater than 30°F. Most of the large deviations were associated with Code 1 and 2 ratings, the same as in the analogous data obtained with the standard coker (see Section II-8).

The deviations just discussed have to do with rating error rather than with overall error. It has been increasingly evident that rating errors must be reduced before any valid comparisons of the precision of different cokers can be made.

The Precision Coker data on AFFB-3 and AFFB-4 (Table 36) included a breakpoint in only one run each, and these were "nonunanimous" among the raters. For AFFB-8, two tests showed breakpoints and two did not; both of the breakpoints were very close to the test temperature, so no conclusion can be drawn on how well the test is repeating. For AFFB-9, breakpoints were obtained in all four tests. With a 20°F spread in test temperatures, the breakpoints ranged from 470 to 502°F. For RAF-174, on which breakpoints were obtained in three tests, the range of breakpoints was 471 to 487°F. These ranges are considerably less than those obtained with the standard coker (Section II-8), but it must be remembered that the standard coker was being rated in a manner for which it was not designed, translating deposit position to deposit inception temperature by means of a correlation hardly translatable from rig to rig.

All that can be said of the precision of the Precision Coker from the few data presented here is that at least a moderate scatter of heater-deposit breakpoints does exist and that one instance of nonrepeatable filter plugging was encountered.

e. Conclusions

From our limited experience with the Precision Coker, we do not feel that its design advantages over the standard and gas-drive cokers are sufficient to warrant its substitution. If it can be demonstrated to be more precise than the Alcor JFTOT as well as the standard and gas-drive cokers, the picture would change considerably. One critical point is whether or not a reasonably valid breakpoint can be obtained in a single test. These questions cannot be resolved fully until the color-rating problems are cleared up or eliminated.

10. ALCOR JET FUEL THERMAL OXIDATION TESTER (JFTOT)

a. General

Of the two thermal stability test devices evaluated by the CRC, we have had more experience with the Alcor JFTOT. Two of these instruments are now being used full-time in the program, and one other was used for several months. The work with these units during the past year has consisted of continuing to evaluate equipment and procedures, investigating possible changes or improvements, participating in CRC cooperative programs, and using the units to study specific thermal stability problems.

b. Equipment and Procedures

The JFTOT units used in this program include one old Model B unit, a Model C, and a new Model B. The old Model B is a prototype, the Model C is a conversion job starting with a "micro coker" from the same manufacturer, and the new Model B is a production unit. The only really significant difference among these units is in the temperature controller. All of the controllers now appear to be satisfactory; the controller on the new Model B is particularly good. The models also differ in minor details such as the device used for thermocouple positioning, location of the aerator pump, and other even less important details.

This equipment and the procedures for thermal stability testing have been described previously.⁽²⁾ The unit consists of an unheated steel reservoir containing a 1-liter fuel sample, which is pumped in a closed loop through a test section and cooler and back to the reservoir. The entire system is nitrogen-pressurized, with the pump serving only to meter the fuel through the test section at 185 ± 4 ml/hr. The test section consists of a vertical tube-in-tube heater with a test filter mounted directly on the heater exit. The fuel flows upward through the annular space, and the inner tube is resistance-heated by low-voltage current, measuring the tube temperature by a movable thermocouple inside the tube. The thermocouple and temperature indicator are calibrated before each test by immersing the thermocouple in pure, molten tin and observing the freezing point, 449°F .

The test filter medium is stainless steel Dutch-weave screen with a porosity of 17μ and an effective diameter of 0.072 inch. Originally, the in-line filter ahead of the test section used the same $17\text{-}\mu$ filter medium. A $0.45\text{-}\mu$ filter was added later in an effort to improve the repeatability of test-filter plugging behavior.

Another change made during the past year was an increase in nitrogen pressure from 300 psig to 350 psig to reduce the possibility of fuel boiling at very high test temperatures.

For the work reported here, the test period was 5 hours. A tube temperature profile was taken during each run, and deposit colors after test were related to this temperature profile to give deposit inception temperatures for Code 1, 2, 3, and 4 deposits. In line with the usual definition, the inception temperature for Code 3 deposit was considered to be the breakpoint of the fuel. No criterion has been established for filter plugging, and probably none will. Past experience with poor repeatability of plugging ratings, along with some recently reported data⁽¹¹⁾, indicate that filter-plugging breakpoints with this unit are probably impractical.

During the past year, we have made two minor changes in design, one referring to the thermocouple calibration equipment and the other to the thermocouple positioning device.

The Auto Cal calibrator supplied with the unit consists of a special heater tube device with a small well containing pure tin. The device fits into the buss connectors normally occupied by the regular heater tube. Power is turned on until the tin melts; then the thermocouple junction is immersed in the molten tin and the heat is turned off. As the tin cools, the temperature is followed on the indicator of the control instrument to determine the apparent freezing point of the tin. The deviation from the true freezing point of 449°F represents thermocouple error, or, more precisely, combined error of the thermocouple and indicator. Since the error is expected to vary with temperature, it was desired to establish a second reference point at temperatures near the 700°F often used in testing JP-7 fuels. Attempts to use the Auto Cal with zinc (m.p. 787°F) resulted in distortion of the connecting rods holding the small well. A modified calibrator was built with a stainless steel well and heavy copper connecting rods.

This has been operated without difficulty at the higher temperatures and has furnished a second reference point for thermocouple calibration.

We have also modified the thermocouple positioner on the new Model B JFTOT. The positioner is used in determining tube temperature profiles. The old Model B and the Model C units had a positioner outside the cabinet, but this feature had been eliminated in the newer Model B. With the new model, the operator was required to reach into the cabinet to position the thermocouple, running some risk of touching hot-section parts. We extended the positioner rod through a slit in the side of the cabinet for safer operation. An arrangement similar to this was adopted for use on future JFTOT Model B units.

c. Operating Experience with JFTOT

As reported previously⁽²⁾, the most serious problem encountered in early operation was poor temperature control. All JFTOT units in the current program were equipped with new or modified controllers that eliminate most of these problems. Proper temperature control still depends on proper adjustment of all components of the control system and good technique in switching from automatic control to manual and back again when the temperature profile is determined. The new system is not as "touchy" as the old one; that is, small variations in power input do not cause instant and large variations in tube temperature when the unit is on automatic control. The temperature control system appears quite satisfactory on the whole. Now that the more severe problems have been resolved, it should be possible to standardize the controller adjustments, warmup procedures, and automatic/manual/automatic switching procedures so that all laboratories are running the units in the same manner.

As discussed previously⁽²⁾, the JFTOT has very significant advantages over conventional fuel cokers in the ease of operation and maintenance and in the sample size required. It was pointed out previously that these advantages would be multiplied if a valid breakpoint could be established in a single test. More recent data confirm the general impracticality of a single-test approach, particularly when an unknown fuel is being tested. Two to four tests are generally required, more if the breakpoint is missed by a wide margin in the early tests.

The unit productivity (tests per unit per day) is little better than that of the standard coker, so long as a 5-hour test period is used. A 2.5-hour test period is gaining acceptance, since it has been reported recently⁽¹¹⁾ that this trade-off of temperature for time may be feasible. Cutting the test time in half would increase the productivity to two tests per 8-hour day, or six to eight tests per 24-hour day.

An increase in productivity of the unit would be highly desirable and may be feasible when testing fuels that are not extremely stable. With high-quality fuels having breakpoints near 700°F in a 5-hour test, it is not feasible to run up the test temperature enough to give an adequate test in 2.5 hours. Test temperatures much above 700°F are not practical with the aluminum heater tubes now being used. In fact, problems are encountered with bowing of the tubes in tests at 700°F or slightly lower. This sometimes causes a streak of dark deposit on the convex side, presumably because of restricted flow at that point.

For JP-7 or other highly stable fuels, any substantial reduction in test time would require redesign to permit operation at temperatures above 700°F. One obvious possibility is the use of a stainless steel tube, and we understand that this approach has been studied by Alcor and by others. It is also under study in the current program. Caution is indicated by the ill-starred history of the attempt to use a stainless steel preheater in the research coker. Whether the abnormal discolorations at fairly low temperatures in the original research coker were caused by catalytic effect of the stainless steel, poor heat distribution, or some sort of "bluing" phenomenon, the design was obviously unsatisfactory. No serious attempt has been made since that time to use stainless steel in a location where fuel deposits must be rated by color.

In the current program, all JFTOT tube deposits have been rated visually without magnification. Earlier concern over the difficulty of rating these small tubes appears to be unfounded; they are probably no more difficult to rate than are standard-coker tubes. Problems do exist with all visual rating systems, and improvement of the present system is mandatory if any great improvement in precision is to be achieved in any type of coker. Future work with the JFTOT will involve the use of a nonvisual rating device.

The "post-peak" deposits discussed in the previously referenced report have continued to occur, and the method of handling them has been the same as before: Any deposit occurring more than 0.2 inch beyond the maximum temperature point in the direction of fuel flow is ignored in establishing breakpoint ratings. Our experience still indicates that these deposits occur at random rather than with specific fuels. No progress has been made in identifying any cause for these deposits or pattern in their appearance. Any explanation of this phenomenon must be based either on specific fuel behavior or on random variations in tube temperature profile that are not taken into account in the present procedure. Certainly, fuels can differ in temperature/deposition sensitivity and in the mode of deposition. For instance, a fuel sensitive to metal temperature would lay down deposits in the same pattern as the temperature profile; another fuel that is more sensitive to bulk-fuel temperature might deposit in a different pattern. Or, using a more elaborate argument, the "post-peak" deposits might be explained by early formation of soluble gum that is carried along with the fuel while it reacts further to form condensation products; these products may be marginally soluble in the fuel at the maximum test temperature T_{max} but precipitate as the fuel passes that point and reaches a cooler zone. Neither of these hypotheses explains the random nature of the post-peak deposit occurrence.

Another problem encountered frequently in rating JFTOT tubes is the presence of deposits on the cold inlet end, beyond the range of the standard temperature profile, i.e., more than 2.0 inches from the fuel outlet. This problem has been handled by extending the range of the temperature profile. However, many of the values reported in the following section are extrapolated values from tests that were run before the extended profile measurement was instituted. Such extrapolated values are footnoted in reporting the data.

Extending the temperature profile did not really solve the problem, since deposits are often observed at the actual fuel inlet point, i.e., at the beginning of the tube test surface. Such a deposit does not really indicate an inception point, but merely where the heated surface begins; presumably the fuel could have formed deposits at even lower temperatures. In reporting the data, the occurrence of deposits at the fuel inlet is indicated by footnote.

Mention should also be made of deposit color changes that occur on standing. In line with previous practice, all tubes have been rated 1 hour after completion of test and again after 24 hours. The changes occurring during the period between ratings were generally not as extensive or severe in the work reported here as had been noted previously.

d. Test Results and Discussion

(1) JFTOT Tests in CRC Cooperative Program

Five fuels were tested as a part of a CRC evaluation program. All tests were run with an early Model B JFTOT, designated Coker 7 in our laboratory. All tests were conducted in accordance with the procedure then regarded as more or less standard, i.e., that given in the manufacturer's brochure. Nitrogen pressure was 300 psig rather than the 350 psig used in later work, and the original 17- μ in-line filter was used. Test period was 5 hours, and flow rate was 185 ml/hr.

After each test, the heater tube was rated by four different raters within one hour of tube exposure following the test and again at least 24 hours afterward. During this 24-hour period, the tube was stored in its original glass container. Care was taken to assure unbiased ratings.

The results of these tests and the individual ratings are listed in Table 37 (Appendix). The filter pressure drop data indicate severe plugging for all fuels except the RAF-174. This fuel produced heavy peacock deposits in all tests; the other fuels produced either a small amount of peacock deposits or none at all. As mentioned previously, this same peculiarity was noted for RAF-174 in standard-coker tests, where it also produced the sole case of zero filter plugging *and* heavy peacocking on the heater tube. The coincidence is striking, but it may be just that.

In these JFTOT tests, the test temperatures selected by the CRC group were all higher than the respective fuel breakpoints, for some fuels far higher. Hence, deposits rated Code 3 or darker were present in all tests, and Code 4 deposits were present in all but one test.

Many of the problems encountered previously in evaluating JFTOT data were either nonexistent or minor in this series of tests. For example, only one test (No. 92, AFFB-8 fuel) gave a "post-peak" deposit profile in which the fuel has laid down the maximum deposits after passing the maximum temperature point. In this test, the point of maximum deposit was within 0.2 in. of the point of maximum temperature and hence met the criterion we had established for using "post-peak" deposits in defining breakpoint. Another early problem that was conspicuously absent in this series was the change in tube deposit color between the 1-hour and 24-hour ratings.

The absence of earlier problems is gratifying but somewhat puzzling. All of the fuels in this group gave fairly sharp breakpoints at temperatures well within the range of the JFTOT, most of them below 600°F. It is quite possible that these fuels are simply easier to rate than some of those we had worked with previously, and that this accounts for the absence of any real rating problems in the current series.

The deposit inception temperatures obtained by different raters have been averaged, and the mean values and corresponding standard deviations are listed in Table 38 (Appendix). Several of the mean temperatures are based on less than four values when the given color code was "absent" from the tube in the judgment of one or more raters. In some tests, one rater would see both Code 3 and Code 4 deposits, but another rater would see only Code 4. The breakpoint based on the first rater's observations would refer to Code 3 deposit, but the breakpoint based on the second rater's observations would refer to Code 4. In order to arrive at a logical "composite breakpoint," the individual raters' breakpoints were averaged. For the reasons just discussed, this composite breakpoint may not be the same as the "average deposit inception temperature" for Code 3 deposits.

The composite breakpoints (Table 38) show rater-to-rater standard deviations not greater than 21°F, mostly below 10°F. There are no significant deviations between the breakpoints based on 1-hour ratings and those based on 24-hour ratings. Likewise, there were no short-term trends in the color ratings; these would have shown up as consistent bias in the chronological order of rating by the four raters.

The phenomenon described previously⁽²⁾ and termed the "floating breakpoint" was observed to a limited degree in the current series, as can be illustrated by the following list of test temperatures (T_{max}) and corresponding composite breakpoints based on 1-hour ratings.

	AFFB-3		AFFB-4		AFFB-8		AFFB-9		RAF-174		
T_{max}	660	640	520	500	570	550	535	515	565	545	500
Bkpt	614	608	495	494	568	536	501	500	516	509	498
Float	6		1		32		1		7 + 11		

The amount of "float" or decrease in the breakpoint as the test temperature is reduced was quite significant for the AFFB-8 and RAF-174 fuels. This float would be expected in the tests on RAF-174, since the first test temperature that was tried (565°F) was some 50°F higher than the indicated breakpoint, and successive reductions gave a float of 18°F. The float was unexpected for the AFFB-8, since the initial test temperature of 570°F was very close to the indicated breakpoint, yet a 20°F drop in test temperature gave a 32°F float. These results illustrate the practical impossibility of defining a valid breakpoint with a single test. Even with these fuels, for which extensive data were available to predict breakpoints and set the test temperatures in advance, two tests were generally inadequate to give a breakpoint that one would accept with confidence.

The standard deviations included in Table 38 are a rough measure of the agreement among raters on the position (temperature) for deposits matching each color code. These standard deviations indicate that the raters often had greater difficulty in identifying and agreeing upon the position of the Code 1 deposits than they did for the darker deposits. This is not too surprising, since the difference between Code 0 and Code 1 is not great.

The relatively good agreement among raters in identification of the breakpoint is an indication that the small tubes are at least no harder to rate visually than are the larger standard tubes. Nevertheless the one

standard deviation of about 20°F (AFFB-3, Run 88) indicates that there is still a great need for improvement. The disagreement among raters can be illustrated by the following list of individual 1-hour ratings of deposit inception temperatures:

	<u>Code 1</u>	<u>Code 2</u>	<u>Code 3</u>	<u>Code 4</u>
First rater	596	---	597	640
Second rater	597	---	---	598
Third rater	---	---	597	637
Fourth rater	---	592	638	640

Using the present breakpoint criterion of a Code 3 deposit or darker, the four operators would assign breakpoints ranging from 597 to 638°F, a spread of 41°F. The spread would be 42°F if the breakpoints were based on Code 4 deposits. In this particular test, a breakpoint based on "Code 1 or darker" or on "Code 2 or darker" would give less spread in breakpoint ratings—not more than 6°F in either case. This good agreement of Code 1 ratings is not typical; see, for instance Test 90 on AFFB-9, where the breakpoints based on Code 1 would range from 344 to 498°F, a spread of 154°F.

Since these rather large rater-to-rater deviations in breakpoint do exist, it is evident that the inherent precision of the JFTOT or any other coker cannot be evaluated until the color-rating problem can be removed or separated out. The CRC work planned for the JFTOT with a nonvisual rating device may resolve this problem.

No detailed analysis of precision has been made for the results presented here. These tests were a part of a larger CRC program, in which the results presumably will be extensive enough to permit a valid analysis.

(2) JFTOT Tests on Various Fuels

Several JP-4 and JP-7 fuels have been tested in the JFTOT in order to determine their thermal stability and at the same time accumulate more operating experience on the JFTOT. These fuels were of interest because of use in other Air Force contractual or in-house programs.

Two JFTOT cokers were used in this work. Coker No. 8 is a Model C JFTOT, and Coker No. 9 is a new Model B JFTOT. All tests were run with the system pressurized to 350 psig. Test fuel was prefiltered through Whatman No. 2V paper. A 0.45- μ in-line filter was installed ahead of the test section for all tests.

The results of these tests are shown in Table 39 (Appendix). The temperature values are uncorrected. The thermocouple calibration correction, if applied, would be +4°F for all tests on Coker No. 8 and +6°F for all tests on Coker No. 9.

For several of the tests listed in Table 39, the deposit inception points were beyond the range of the temperature profile as it was originally defined, and such inception points were determined by extrapolation. Subsequently, the temperature profile was redefined so as to cover the entire test surface.

Some of the problems identified previously did show up again in these tests. Five tests gave "post-peak" profile distributions, but this created a problem in interpretation for only one test (No. 6479, "DR" JP-4). Some of the tubes showed minor changes in deposit inception points following 24-hour storage, especially for Code 1 and 2 deposits.

For many of the tests on the "OF" JP-4 fuel and for one test on the 70-16 JP-7 fuel, the deposit pattern was exactly the reverse of that expected, the darkest deposits were at or near the fuel inlet, followed by progressively lighter deposits. Such reversals have been seen occasionally in the past, but generally for tubes having Code 1 and 2 deposits only. Here, for the "OF" fuel, the reversed deposit pattern included Code 3 deposits and showed up in most of the tests.

The tests on the three JP-7 fuels did not encounter any particular problems, but neither did they yield any specific breakpoint value. All of the fuels were stable at 700°F on the basis of the absence of Code 3 or darker deposits.

The tests on the "DR" and "HR" JP-4 fuels established breakpoints without any difficulty; the breakpoints were very sharp and repeatable. The best average values are 544°F for the "DR" fuel and 572°F for the "HR" fuel. The "HR" fuel also gave a very sharp breakpoint in filter plugging behavior between 500 and 525°F, with heavy plugging at all higher temperatures and little or no plugging at all lower temperatures. This sort of clear-cut behavior in filter plugging at these high temperatures is certainly the exception and not the rule. The JFTOT tests on these two JP-4 fuels illustrate its possible advantages for research purposes. These two fuels are evidently far better than the specification minimum level of thermal stability. Just how much better cannot be determined in gas-drive or research cokers, where the safe pressure limits are too low to permit satisfactory operation on JP-4 fuel at very high temperatures. We have operated gas-drive cokers at 280 psig, although the CRC-recommended limit is 210 psig. In the JFTOT unit, the system pressure of 350 psig in the present procedure permits the determination of breakpoints on these very stable JP-4 fuels. Even here, the vapor pressure may well be crowding or exceeding the system pressure at test temperatures of 550-575°F. If the JFTOT fuel system has a still higher safe pressure limit, this should be established so that its maximum capabilities can be used.

The "OF" JP-4 fuel caused very serious rating problems in these JFTOT tests. As already mentioned, most of the deposit patterns were reversed. Still worse, the breakpoint was elusive and could never be pinned down, even after 10 tests. The confusing situation is illustrated by the following:

T_{max} , °F	Breakpoint, °F	
	1-hr	24-hr
550	311	311
500	367	360
450	350	350
425	349	345 (3 tests)
415	>415	325
400	>400	>400 (3 tests)

The breakpoints obtained at a test temperature of 425°F appeared quite repeatable, averaging 347°F with extreme values of 335 and 354°F. These breakpoints still cannot be regarded as reliable; past experience has shown that so great a spread between test temperature and breakpoint usually gives unduly high breakpoints that will "float" downward in tests at successively lower temperatures. Here, however, a mere 25°F lowering of test temperature wiped out the breakpoint altogether. It is literally impossible to assign a breakpoint to this fuel, even after running 10 tests.

Since the "OF" JP-4 fuel had given a reversed-deposit pattern and a vanishing breakpoint in JFTOT tests, it was run in duplicate standard-coker tests at specification conditions of 300/400°F and in one test at 325/425°F. Code 3 deposits were observed in the test at 325°F fuel-out temperature and in one of the tests at 300°F fuel-out temperature. The deposits were abnormal, having a light greenish tint, and could be wiped off the tube readily and completely.

Thus, it appears that an "oddball" fuel can be difficult or impossible to rate properly for thermal stability in any test method thus far devised. It is not an answer to write off such fuels as freaks unworthy of attention, since they will appear in regular production as well as in experimental blends. If this particular "OF" JP-4 fuel can be saved in sufficient quantities for further tests, and if it does not change too much in storage, it could be a valuable reference fuel for use in thermal stability work.

(3) JFTOT Tests on Fuels from Storage and Compatibility Tests

The JFTOT was used, along with other fuel cokers, to evaluate the thermal stability of fuels from storage and compatibility tests on bladder-tank materials. The results of this work are presented in Section II-11.

e. Step-Test Method for JFTOT

As discussed in the preceding section, the original hope that a fuel breakpoint could be determined in a single JFTOT test has not been realized; a four-test series is more nearly typical. Any adaptation or modification of the apparatus or procedure to permit single-test determinations would be a very significant advance.

With standard fuel cokers, step-temperature tests have been proven to be quite practical in determining filter-plugging breakpoints. In such a test, the temperatures are raised according to a fixed schedule, and the breakpoint is detected in the usual manner by noting increase in pressure drop across the test filter. Preheater tube deposits can also be handled in step tests by using a transparent outer tube for the preheater, but the design and safety problems involved have prevented such methods from gaining acceptance.

The JFTOT is similar to the standard coker in that heater deposits cannot be rated until the end of the test, after disassembly of the test section. It was considered possible that deposit formation during the test could be followed by means of the power vs temperature curve. With a constant volumetric flow rate, the power required to maintain a given tube temperature will be affected by the mean temperature differential between the tube and the fuel, the volumetric specific heat of the fuel, and the overall heat transfer coefficient. The temperature differential and the specific heat should vary with temperature in accordance with smooth, continuous functions; the heat transfer coefficient for a clean tube should also follow a smooth curve. If enough deposits form on the tube to interfere with heat transfer, the effect might be detectable as a relatively sudden change in slope of the power vs temperature curve.

The JFTOT was not designed to measure heat transfer coefficients, so there was little reason to suppose that curves could be plotted with sufficient accuracy to detect deposit formation. Heat losses to the end connectors and to the atmosphere could well have major effects on the power/temperature curve. Nevertheless, the intriguing possibility of a single-test breakpoint determination seemed to warrant a brief study.

This possibility was investigated using a JP-8 fuel that had given a breakpoint of 573°F in a regular JFTOT test. The breakpoint in that test was very sharp. With a test temperature $T_{max} = 575^\circ\text{F}$, very dark colored deposits (Code 4 and darker) started at 573°F, just ahead of the maximum temperature point, and continued for 0.84 inch; there were no Code 1, 2, or 3 deposits. The fuel also plugged the test filter, starting at 60 minutes and reaching 10 in. Hg at 160 minutes.

Step tests were run on this fuel by starting at a temperature well below the expected breakpoint of 573°F and then increasing the setting of the temperature controller in 25°F increments. Three tests were run, differing in the temperature range covered and the length of time at each controller setting:

Test no.	Set point, °F		Time at each setting, min
	Start	Finish	
129	400	700	15
130	400	700	10
131	500	600	60

In each test, the wattage was recorded 5 minutes after reset and at the end of the period at the new set point; in Test 131, an intermediate reading at 30 minutes was added. The wattage readings at the end of each period should represent the best stabilized values.

Data from these tests are listed in Table 40 (Appendix), and the wattage readings at the end of each period are plotted in Figures 3-5, along with the filter pressure drops that were observed.

The most striking features of the data are the near linearity of the curves and the close agreement of wattage values for a given temperature setting. If one assumes that the effective mean fuel temperature increases linearly with tube temperature, then the effective temperature differential and the mean volumetric specific heat of

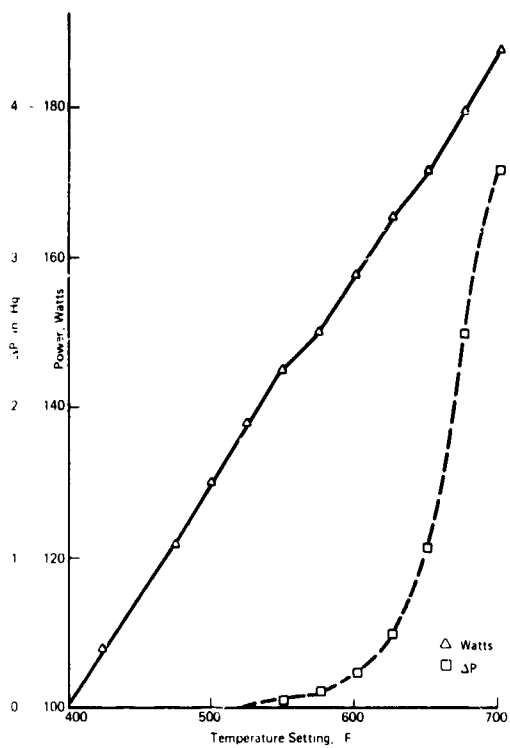


FIGURE 3. STABILIZED WATTAGE AND PRESSURE DROP VS TEMPERATURE SETTING IN JFTOT STEP TEST WITH 15-MINUTE PERIODS

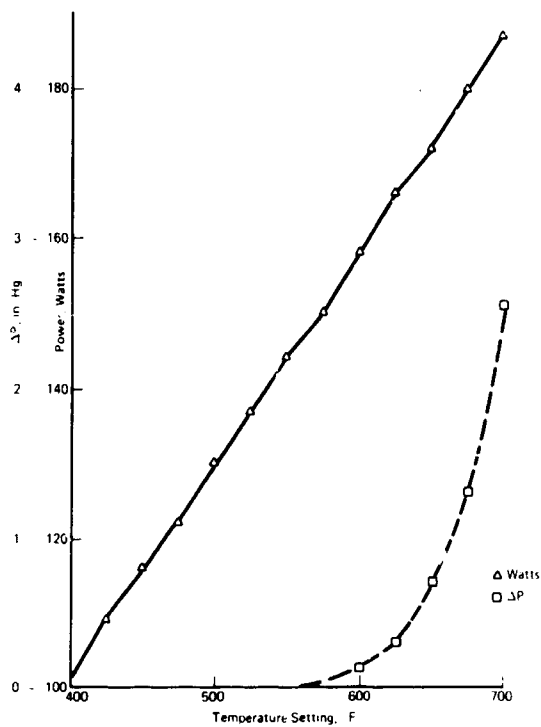


FIGURE 4. STABILIZED WATTAGE AND PRESSURE DROP VS TEMPERATURE SETTING IN JFTOT STEP TEST WITH 10-MINUTE PERIODS

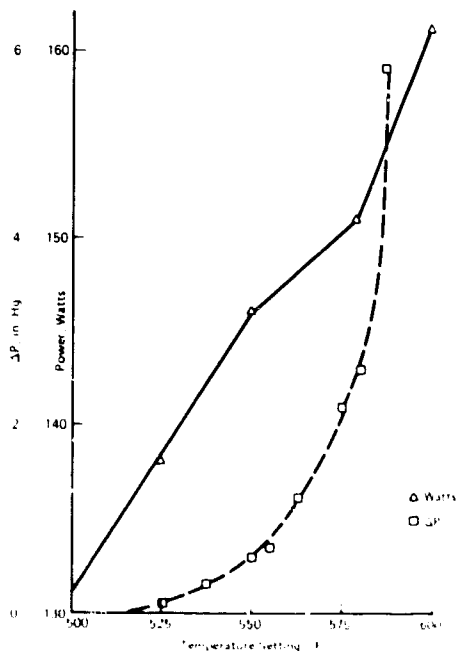


FIGURE 5. STABILIZED WATTAGE AND PRESSURE DROP VS TEMPERATURE SETTING IN JFTOT STEP TEST WITH 60-MINUTE PERIODS

the fuel will also be linear functions of tube temperature. The heat transfer coefficient, in the absence of deposits, will vary with fuel temperature in a complex manner because of the effects of fuel viscosity and flow velocity on the film coefficient. Finally, heat losses were expected to increase the relative power requirements at high temperatures and to cause erratic variations. The JFTOT test section is uninsulated and somewhat unprotected from air currents in the laboratory. In view of all of these possibilities, the near-linearity of the curves and the close agreement of wattage values are very surprising.

To illustrate the close repeatability of wattage values, the values in Tests 129 and 130 may be compared for each temperature setting, regardless of whether taken 5, 10, or 15 minutes after reset. The four wattage values for each temperature agree within 1 watt for 12 of the 13 temperatures. At one temperature (425°F) there is a 2-watt deviation.

The data that are of the most interest for present purposes are those at temperatures near the known breakpoint of 573°F. Each of the three runs shows a definite dip in the curve at 575°F. This can be illustrated best by the data on ΔW , the wattage required at a given temperature minus the wattage required 25°F lower. The following ΔW values refer to the wattage readings at the end of the period at a given temperature setting:

	Temperature, °F				
	<u>525</u>	<u>550</u>	<u>575</u>	<u>600</u>	<u>625</u>
ΔW , Test 129	8	7	5	8	8
ΔW , Test 130	7	7	6	8	8
ΔW , Test 131	7	8	5	10	

The differences are small but are believed to be significant, in view of the good repeatability of wattage values in Tests 129 and 130. In addition to the dip at 575°F, there is another definite dip at 650°F with an 8-8-6-8 pattern of ΔW values in Tests 129 and 130. These dips at 575 and 650°F probably represent changes in heat transfer coefficient caused by deposit formation.

In Test 130, which was run with 60-minute periods at each temperature setting, the break or dip at 575°F showed up very clearly. A surprising aspect of the data from this test was the increase in wattage requirement sometimes observed when holding a given temperature setting. The following wattages represent readings after 5, 30, and 60 minutes at each temperature:

500°F:	126, 130, 131
525°F:	138, 138, 138
550°F:	147, 146, 146
575°F:	151, 151, 151
600°F:	154, 157, 161

It is difficult to devise a theory to account for this behavior. The data at 500°F may be explained by incomplete thermal equilibrium 5 minutes after startup, but the data at 600°F cannot be explained in this way. Possibly, the behavior can be explained by initial deposition of "loose" or semiliquid material that subsequently condenses and becomes a better heat conductor.

The tubes from these tests were not color-rated in detail. It was noted that the tubes from Tests 129 and 130 had heavy deposits, starting further back from the fuel outlet than they did in the regular JFTOT test (1.4 in. for step tests, 1.04 in. for regular test). This should be expected because of the higher temperatures in the step tests. The tube from test 130 was not rated.

The filter-plugging data from the step tests line up well with each other and with those from the regular JFTOT test. In the step tests, the temperature at which plugging was first detected was related to the length of the period at each temperature step.

Step time, min	10	15	60
Plugging temperature, °F:			
Barely detectable	575	525	500
0.1 in. Hg or more	600	575	525

The data from these step tests are rather encouraging in that a wattage/temperature effect could be demonstrated in the vicinity of the known breakpoint. However, this fuel was deliberately selected as one having a sharp breakpoint; this sort of effect may be absent with other fuels. Also, even if similar effects do show up with other fuels, the JFTOT wattmeters are not sufficiently precise to make fine distinctions. The encouraging fact is that any effect could be observed at all.

Further work in this direction is planned.

f. Multiple Temperature Profiles

In the JFTOT procedure, the tube temperature profile is determined once during each test, 60 minutes after startup. This is done by switching the unit from automatic to manual control and moving the thermocouple to various positions in the tube and recording the temperatures. This temperature profile is used to translate deposit inception locations to inception temperatures. The selection of 60 minutes as the time for taking the profile may have been somewhat arbitrary. If the actual tube temperature profile is not changed significantly by deposit formation, any time during the test after temperatures have stabilized would be appropriate for taking the profile. In such a case, successive profiles taken at intervals during the test would differ only because of imprecision in the temperature determination, and variance would be simply a measure of repeatability. On the other hand, if the tube temperature profile is affected by deposit formation, then the variance would reflect not only the repeatability of temperature/position determination but also actual changes in the profile during the test. In this situation, again, definition of a "best" time to take the profile is difficult. Possibly one should assume that the earliest time when conditions have stabilized is the best time, since this will come nearest to the "clean-tube" condition.

Since the precision of deposit inception temperature and breakpoint data can be no better than that of the temperature profile, it would be desirable to obtain an independent measure of the precision of profile data, without the perturbing influence of deposit formation on temperature profile. A start on this problem has been made by repeating the profile determination at intervals through the test period. Such data will provide some measure of profile repeatability and may indicate a preferred time for determining the profile. Information on reproducibility of profiles among different JFTOT units may also be gained.

Multiple temperature profiles have been determined in 54 JFTOT tests, taking the data after 60, 120, 180, 240, and 290 minutes of test time. These data were obtained on a Model C JFTOT (Coker No. 8) and a new Model B JFTOT (Coker No. 9). The Model B unit was equipped with the latest temperature controller supplied for the JFTOT; the Model C unit had an older controller that had been modified so that its control characteristics approach that of the newer instrument. The temperature profiles were all taken in the normal manner. After switching from automatic to manual control, the temperature is recorded with the thermocouple in its normal position, 0.85 inch from the fuel outlet at the nominal position of maximum temperature. Then the thermocouple is moved to a position 0.2 inch from the fuel outlet, then 0.4, 0.6, 1.1, 1.4, 1.7, and 2.0 inches from the fuel outlet, recording the temperature at each point. Readings may also be taken at 2.2 and 2.35 inches from the fuel outlet. Finally, the thermocouple is moved back to the control position (0.85 inch from the fuel outlet), the temperature is recorded, and the unit is switched back to automatic control.

The multiple profile data are listed in Table 41 (Appendix). These data have been analyzed only in part, no attempt has been made thus far to relate temperature profile changes to deposit formation. Here we will present the results of preliminary analysis of the data.

For each thermocouple position in a given test, the five readings obtained in the five successive profile determinations have been used to calculate an average temperature and the corresponding standard deviation.* These deviations, which are entered in Table 41, have been inspected to determine what fraction (percentage) of them exceed certain values. These findings may be summarized as follows:

	Percentage exceeding		
	2°F	3°F	4°F
All standard deviations		13.3	4.6
Standard deviations for positions			
0.20-0.60 and 2.00-2.35 inches		15.9	
0.85-1.70 inches		10.7	
Standard deviations for T _{max}			
400-500°F	7.1	0.7	
501-600°F		11.0	3.8
601-700°F		25.3	8.9

A rather large number of the standard deviations exceed 3 and 4°F. A standard deviation on the order of 4 to 7°F appears rather high if viewed as a "repeatability standard deviation," but quite moderate if viewed as a result of error cumulated with actual changes in value. The deviations show some variation with position on the tube: The end sections account for more of the 3-and-up deviations than does the center section (0.85-1.70 inches). The center section is the more important in breakpoint ratings, since Code 3 deposits tend to occur there. The deviations are much less at low test temperatures than at high test temperatures. It has not yet been determined whether this reflects differences in error or differences in deposit formation.

These data do not appear suitable for any attempt to determine the reproducibility of profiles among different JFTOT units. Only two units were involved in this program, the temperature controllers were different on the two units, and parallel tests were rarely run in two units on the same fuel at the same conditions. A larger program with several units would be needed to investigate reproducibility of temperature profiles.

The five profiles are compared in a different manner in Table 42 (Appendix) in an attempt to determine how each of the five profiles deviates from the mean profile. For each temperature profile in a given test, temperature deviations from the mean profile for that test are listed. These deviations (absolute values) are then averaged for each profile in the test and listed in the table. These average deviations are in turn cumulated for all tests in five divisions according to profile determination time and averaged to give overall average deviations:

60-minute profiles	1.48°F
120-minute profiles	1.32°F
180-minute profiles	1.37°F
240-minute profiles	1.77°F
290-minute profiles	1.39°F

If the deviation of each profile from the test-mean profile is purely random, these overall deviations indicate there is little advantage of any one time over another for determining the profile. The lack of any clear trend in these overall deviations suggests that they may be indeed random. If there were a clear tendency for the profile to broaden as deposits form, then one would expect the intermediate-time (180-minute) profile in a given test to agree most closely with the mean for that test, and the 60-minute and 290-minute profiles to give the poorest agreement. If there is any such trend, it is not apparent in the overall average deviations.

*All standard deviations were calculated using Bessel's correction for small number of samples.

Further analysis of these data will be performed in an attempt to resolve the unanswered questions. Some of these, particularly those concerning the effect of deposit formation on temperature profile, are quite complex and may not be resolvable from the data at hand. These data were obtained in tests run for other purposes and hence do not provide all of the direct comparisons that are needed. For instance, the tests do not include enough replicate tests on the same fuel at the same test conditions to permit a direct correlation of deposit formation with change in temperature profile for one particular type of deposit and deposit pattern.

If the available data will not yield the desired information, consideration will be given to setting up a series of tests designed specifically for temperature profile investigation.

g. **Conclusions**

Further experience with the Alcor JFTOT has confirmed its advantages in operability and maintainability. Early problems with the temperature controllers have been resolved. The rating of tube deposits and the interpretation of results still present serious problems with some fuels, particularly the more stable ones. Even in favorable situations, single-test breakpoint determinations are infeasible. This test device is certainly not a cure for everything that has been wrong with thermal stability testing, but it does appear to represent a step in the right direction.

Future adoption of a 2.5-hour test period will increase the productivity of the unit for conventional fuel testing but will not be suitable for high-quality fuels unless means for operating above 700°F are devised. Future use of a nonvisual tube rating device may alleviate some of the present problems in rating tubes and interpreting results.

The JFTOT has been found very useful in studying a variety of fuel stability problems in this program, and it will be used to a greater extent in the future.

11. EFFECT OF FLEXIBLE-TANK LINERS ON JP-7 THERMAL STABILITY

a. **General**

One of the specialized problems in fuel/elastomer compatibility is the choice of materials for flexible fuel tanks or tank liners. The effects of the elastomer on the fuel are especially critical when storing high-quality fuels such as JP-7. Even minor amounts of contamination can degrade the fuel seriously. Especially severe are the effects on the thermal stability of the fuel.

In the current program, we are conducting long-term soak tests on a variety of elastomers that are used or proposed as liners for pillow tanks in jet fuel storage systems. Our evaluations have been concerned primarily with the thermal stability of the fuel. Along with this work, fuel samples from a full-size pillow tank have been checked periodically for changes in thermal stability.

b. **Long-Term Soak Tests**

(1) Outline of Program

These studies have consisted of soak tests in which various elastomer samples are suspended in JP-7 fuel and stored at 130°F and at indoor ambient temperature. The fuel is checked periodically for thermal stability using the Alcor JFTOT, and other tests and analyses are performed as needed. Storage periods up to one year have now been completed in one set of soak tests (Series I) and up to four months in another set (Series II). The program also includes evaluation of fuel samples from soak tests conducted in another laboratory.

(2) Soak Test Apparatus

Fuel containers for the soak tests were 15-gallon stainless steel "bain-maries" with loose fitting lids. For convenience in subsequent discussion, these will be called "pots." Stainless steel racks were designed and

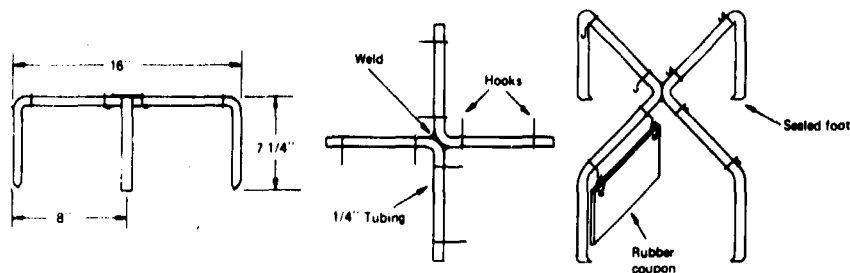


FIGURE 6. RACK FOR RUBBER COUPONS IN SOAK TESTS

fabricated; these are illustrated in Figure 6. Each rack fits into a pot and holds four 6 X 6-inch coupons of the test material in an upright, stable position in the test fuel. Any one rubber coupon can be removed from the test assembly without disturbing the rack or the other coupons.

(3) *Test Fuel*

A sufficient quantity of JP-7 fuel was segregated for each of the two test series. This was the fuel G identified in Section II-2-b. For each series, the fuel was mixed thoroughly to assure homogeneity; then 12.5 gallons were pumped to each sample pot through a 0.45- μ filter, using a fresh filter for each 12.5-gallon sample.

(4) *Test Materials*

The elastomers* used in this program were as follows:

Series I	ESS-690	(nitrile)
	80C29	(polyurethane)
	245-13	(epichlorohydrin)
	2361C	(polyurethane)
Series II	V-1	(fluorinated)
	N-1	(nylon)

Coupons, 6 X 6-inch with two holes, were cut from each material. For the 80C29 and 2361C materials, it was necessary to uncurl the coupons by storing them under a heavy weight. Before use, each coupon was washed with hot water and mild detergent, rinsed with tap water and then with distilled water, and press-dried between paper towels.

(5) *Assembly, Storage, Sampling, and Testing*

Each test assembly consisted of a sample pot with 12.5 gallons of fuel and a rack with four coupons. In each of the two series of tests, a "blank" assembly was included; this consisted of the pot, fuel, and rack without any coupons.

Duplicate assemblies were made up for each test material. One assembly was stored at indoor-ambient temperature (65-95°F), the other in a hot room nominally maintained at 130°F. Periodic failures of the hot room were followed by long-term shutdown. The Series I assemblies were exposed to 130°F conditions for 7 months, the Series II for 1.5 months. Subsequent storage of the "hot-room" assemblies was actually indoor-ambient storage. The hot-room sample storage and temperature history is shown in Figure 7.

*We have used the term "elastomer" loosely to denote any rubber, plastomer, or flexible plastic that can be used as a tank liner.

The initial ratio of test material surface to fuel volume was approximately 23.6 in²/gal, varying slightly from material to material depending on thickness. During the subsequent sampling, this ratio was maintained at 23.6 ± 3.1 in²/gal by removing one coupon at a time whenever necessary.* Fuel samples were scheduled to be drawn after 1, 2, 4, 6, and 12 months of storage. This schedule has been completed for the Series I tests, and the schedule through 6 months has been completed for the Series II tests.

It was planned to test each sample for thermal stability as soon as it was drawn. However, the heavy workload on the available JFTOT units made it necessary to hold these samples, in some cases several months. Such samples were stored in glass bottles at 40°F until they could be tested.

Thermal stability evaluations were performed in Alcor JFTOT units. In the evaluations of Series I samples, three different JFTOT units were used. All tests were carried out at 300 psig system pressure. For Series I samples, the JFTOT in-line filter was the original 17 μ; for Series II, it was 0.45 μ. Temperatures that are reported are uncorrected values; the thermocouple calibration corrections, if applied, would range from +4°F to +11°F. The temperature corrections based on the zinc standard were little different from the corresponding corrections based on the tin standard that is normally used.

(6) Thermal Stability Test Results and Discussion

Each fuel sample from the storage program was tested first at 700°F to determine an approximate breakpoint, then at lower temperatures if necessary to establish a reliable tube-deposit breakpoint with no more than 23°F spread between the test temperature (T_{max}) and the breakpoint. Floating breakpoints were encountered in the tests on the Series I storage samples, as were most of the other anomalies that have been described previously (Section II-10), including streak deposits, peacock deposits, post-peak profiles, and deposit inception points at or near the fuel inlet. As usual, these anomalous results appeared to occur at random. In the Series II tests, no such problems were encountered. This probably reflects the absence of any failing tests at 700°F with the Series II samples thus far tested rather than any difference in test equipment or techniques.

The available thermal stability data are summarized in Table 11. The breakpoints are the inception temperatures for Code 3 or darker deposits, based on 1-hour tube ratings, in tests with a T_{max} close to the breakpoint.

The base fuel, when stored without any elastomer, gave breakpoints in the 640-700°F range except for one unexplained value of 546°F obtained on the two-month hot-room sample. None of the samples gave any filter plugging in the JFTOT tests.

*After removal from the storage test assembly, each coupon was kept in fresh JP-7 fuel for subsequent examination and physical tests by another laboratory

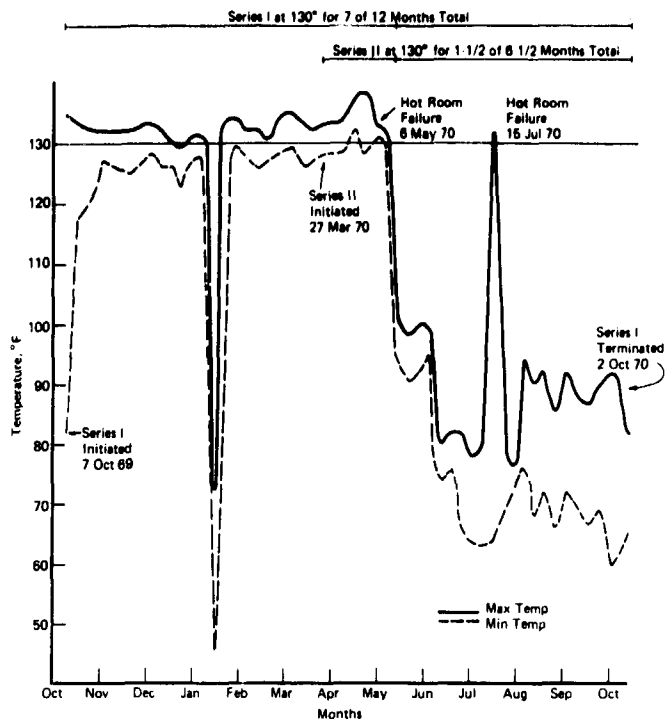


FIGURE 7. TEMPERATURE HISTORY OF HOT ROOM STORAGE SAMPLES, SERIES I AND II, OCTOBER 1969–OCTOBER 1970

TABLE 11. EFFECTS OF ELASTOMERS ON JP-7
FUEL THERMAL STABILITY

Alcor JFTOT fuel coker

Elastomer	Soak time, mo	Hot-room soak		Ambient soak	
		Breakpoint, °F	ΔP, in. Hg*	Breakpoint, °F	ΔP, in. Hg*
<i>Series I†</i>					
None	0	685	0.0	685	0.0
	1	664‡	0.0	639	0.0
	2	546	0.0		
	4	>700	0.0		
	6	646	0.0		
245-13	1	492	10.0/74	495	10.0/106
	2	491	10.0/66		
	4	491	10.0/39		
ESS-690	1	677	10.0/200**	494	10.0/210
	2	648	10.0/159**		
2361C	1	700	0.0	677	0.0
	2	612	0.0	677	0.1
	4	>700	8.5, 2.9		
	6	600	0.7, 10.0		
80C29	1	654	0.2	>700	0.0
	2	>700	0.3	>700	0.0
	4	668	3.0	>700	0.0
	6	634	0.0		
<i>Series II†</i>					
None	0	>700	0.0	>700	0.0
	1	>700	0.0	>700	0.0
V-1	1	>700	0.0	>700	0.0
	2‡‡	>700	0.0		
	4‡‡	>700	0.0		
N-1	1	>700	0.0	>700	0.0
	2‡‡	>700	0.0		
	4	>700	0.0		
<p>*At 300 min unless a different time is indicated; for example, 10.0/74 indicates a 10-in. pressure drop in 74 min. †Series I JFTOT tests with 17μ in-line filter, Series II with 0.45μ. ‡Only "failure" was a long, narrow streak of deposit. **Also plugged at test temperatures of 500-606°F. ††Hot-room storage periods included only 1.3 months at 130°F, balance at indoor-ambient temperature</p>					

The 245-13 epichlorohydrin and the ESS-690 nitrile rubber caused severe filter plugging in JFTOT tests on the stored fuel samples within only one month of storage at hot-room or ambient temperature. Tube-deposit breakpoints were quite low (490-500°F) in most cases, and the filter-plugging behavior persisted at low temperatures even when the tube-deposit breakpoint was high. This rapid degradation of fuel with these rubbers and the apparent lack of further degradation with additional storage time may reflect complete extraction of plasticizer or other ingredient within a very short time, or it might be explained by the presence of some ingredient having a very sharp decomposition temperature, so that the breakpoint is more or less independent of concentration in the fuel.

The poor results with the ESS-690 nitrile rubber had been expected, since earlier data⁽⁴⁾ had shown that plasticized Buna N and other low-temperature nitrile rubbers are inimical to JP-7 fuel stability. The epichlorohydrin rubber is more of an unknown quantity. The manufacturer has suggested that sulfur blisters formed in compounding may have been responsible for the poor performance of this particular sample.

The two polyurethane elastomers gave somewhat better results than the epichlorohydrin or nitrile rubber, but still showed some tendencies toward filter plugging. Ambient-temperature storage did not bring out these tendencies, at least in the first few months. Storage of these samples is being continued.

In the Series II tests, the limited data thus far available indicate that neither the V-1 fluorinated rubber nor the N-1 nylon liner material has any effect whatever on the thermal stability of the fuel. These tests are being continued. It should be noted that here the "hot-room" storage samples in Series II really represent only a little over a month at 130°F, with the balance of the storage at indoor-ambient temperature after the hot room became inoperable.

From the thermal stability test results thus far reported, it is evident that the nitrile and epichlorohydrin rubbers had rather drastic effects on JP-7 thermal stability, the polyurethanes were rather erratic but appear to be developing filter-plugging tendencies in the fuel, and the fluorinated rubber and the nylon appear to be inactive. It should be kept in mind that the ratio of elastomer surface to fuel volume in these tests is about the same as in a medium-size pillow tank*; therefore, these tests must be considered as simulative rather than accelerated. On this basis, the data now available are insufficient to judge the suitability of the polyurethane, fluorinated rubber, or nylon for the intended service. The nitrile and epichlorohydrin rubbers seem to be unsuitable for even short-term storage applications.

(7) Auxiliary Data

The stored fuels were checked for existent and potential gum contents whenever enough sample remained after the other test work. The available results are listed in Table 12. None of the stored samples had gum contents that ever approached JP-7 specification limits, which are 5 mg existent and 10 mg potential gum. For the stored samples, the existent and potential gum values for a given sample were quite similar. The gum contents do line up in a general way with the thermal stability results: Fuels stored without elastomer had essentially zero gum contents, the fuels with nitrile and epichlorohydrin rubbers gave values mostly in the 1-2 mg range, and the fuels with perfluorinated rubber and the nylon gave essentially zero values. The fuels with polyurethane rubbers gave values in the 0-1 mg range. All of these values are so low that close comparisons are impossible; the gum test is simply not that precise. Nevertheless, it is interesting that there is at least a general lineup with thermal stability results: evidently the fuel-extractables that are degrading the fuels' thermal stability are showing up in sufficient quantities to be detectable by the gum test.

In the Series I tests, the condition of the fuel and rubber samples was checked visually after three days of ambient and hot-room storage. The rubbers appeared the same as when installed; that is, the nitrile and epichlorohydrin coupons were flat, the polyurethane coupons were curled. The originally colorless fuel was yellowed slightly by the nitrile rubber and more markedly by the epichlorohydrin rubber. Contamination of the fuel evidently

*For example, in a pillow tank measuring 50 x 15 feet and containing 10,000 gallons, the ratio is 22.5 in²/gal, almost identical to that used in these tests.

TABLE 12. EFFECTS OF ELASTOMERS ON GUM CONTENTS OF JP-7 FUEL

Elastomer	Soak time, mo	Gum content (mg/100 ml) after hot-room soak		Gum content (mg/100 ml) after ambient soak	
		Existent	Potential	Existent	Potential
<i>Series I</i>					
None	0	0.2	---	0.2	---
	1	---	---	0.0	0.0
	2	---	---	0.0	0.0
	4	0.0	0.0	0.2	0.2
	6	0.0	0.0	0.0	0.0
	9	0.0	0.0	0.0	0.0
	12	0.0	0.0	---	---
245-13	1	---	---	2.4	2.4
	2	1.8	1.8	---	---
ESS-690	1	0.4	1.2	1.0	1.0
	2	0.4	1.2	---	---
2361C	1	0.2	1.0	1.0	1.0
	2	0.8	1.0	0.0	0.0
	4	0.4	0.4	0.0	0.2
	6	0.2	0.8	0.4	0.8
	9	0.2	1.4	0.6	1.0
	12	0.0	0.4	---	---
80C29	1	0.0	0.0	1.0	1.0
	2	---	---	0.0	0.0
	4	---	---	0.0	0.0
	6	0.0	0.0	0.2	0.2
	9	0.0	0.2	0.2	0.2
	12	0.2	0.4	---	---
<i>Series II</i>					
None	0	0.0	0.0	0.0	0.0
	1	0.0	0.0	0.0	---
V-1	1	0.0	0.0	0.0	---
	4	0.0	0.0	---	---
	6	0.0	0.2	---	---
N-1	1	0.0	0.0	0.2	0.2
	2	0.0	0.0	---	---
	4	0.0	0.0	---	---
	6	0.0	0.0	---	---

occurs rapidly with these rubbers. It is not known how fast this will show up as a thermal stability effect, since the first tests were not run until completion of one month's storage.

Samples of the Series I fuels were analyzed by gas chromatography and infrared spectroscopy. Neither the polyurethane nor the epichlorohydrin rubbers caused any detectable change in the base fuel. The fuel exposed to nitrile rubber gave an IR peak at 1735 cm^{-1} that was not found for the base fuel. This peak is characteristic of esters. Soxhlet extraction of a fresh sample of the nitrile rubber, using ethyl ether, gave a residue that was identified by infrared spectrum as dibutyl sebacate. Evidently this plasticizer was leached from the nitrile rubber in the JP-7 storage test. Effects of this type of plasticizer on fuel thermal stability are unknown. It had been established in earlier work⁽⁴⁾ that soluble lead and zinc compounds are present in fuels stored with certain nitrile rubbers, and more recent work reported here (Section II-3) indicates that very small amounts of these metals can cause serious degradation of thermal stability. In the elastomer-soak tests reported here, no effort was made to establish metal contents of the fuel, in view of the difficulties being experienced with metal analyses in another portion of this program. In the light of past experience, it appears probable that soluble metals are more important than rubber plasticizers in fuel degradation, but as yet we have no direct data to support this opinion.

(8) Fuel from Soak Test Conducted Elsewhere

Two samples of JP-7 fuel were supplied from soak tests conducted by a rubber producer. It is understood that the JP-7 used in these tests was comparable to that used in ours and that the rubber samples were modified formulations of the 245-13 epichlorohydrin rubber used in our tests. Exposure conditions for these outside tests are not known to us. The two rubber formulations were identified as WH15 and WH16. Fuel samples from these tests were furnished to us in tinned 1-gallon cans. The samples were sufficient for only one JFTOT test each. These were run with T_{max} 700°F , using the Model C unit with test conditions the same as for the Series I samples.

The data obtained in these tests are summarized in Table 13, along with comparable data (T_{max} 700°F) on the earlier sample 245-13 and on JP-7 without any exposure to rubber. It can be seen that the new samples of epichlorohydrin rubber did cause degradation of the JP-7 fuel, but not as much as did one month's exposure to

the previous sample. The single-test breakpoints are not extremely reliable, but they at least point out that the improvement over the early sample is not sufficient to eliminate the harmful effects. It is understood that the more recent samples were improved mainly by eliminating the sulfur blisters present in the earlier sample.

c. Storage Test in Pillow Tank

(1) Storage Conditions

The tank used in this study is a 10,000-gallon pillow tank with polyurethane liner. It was installed at Wright-Patterson AFB in 1969; previous history is unknown. The fuel is Shell JP-7, received in August 1969. This "base fuel" was sampled and tested. Then 3000 gallons of fuel was pumped into the tank as a rinse. This was allowed

TABLE 13. EFFECTS OF EPICHLOROHYDRIN RUBBERS ON JP-7 FUEL THERMAL STABILITY

*Alcor JFTOT, test temperature, 700°F
17 μ in-line filter*

Elastomer	Soak conditions	Breakpoint, $^{\circ}\text{F}$	ΔP , in. Hg
Compound WH15	Unknown	610	6.0
Compound WH16	Unknown	658	5.0
245-13	Hot room, 1 mo	597*	10.0 (65 min)
	Ambient, 1 mo	538	10.0 (51 min)
None	Hot room, 1 mo	680	0.0
	Ambient, 1 mo	673	0.1

*Test temperature, 665°F .

to stand for two weeks and then pumped out; a sample of rinse fuel was taken at that time. The tank was then filled with fuel and has remained essentially undisturbed since that time except for sampling operations. Samples were drawn monthly for thermal stability and other tests.

(2) *Thermal Stability Test Methods*

Gas-drive cokers, a research coker, and JFTOT cokers were used in these evaluations. The gas-drive and research coker tests were run within a few days after the samples were drawn. The JFTOT tests were often delayed by other commitments for the equipment; samples drawn for JFTOT testing were placed in amber glass jugs and held in cold storage (below 40°F) until ready for test.

Research coker tests were run with a fuel reservoir temperature of 300°F. The test pressure was matched to the preheater fuel-out temperature to minimize the possibility of fuel boiling and at the same time, to operate at the lowest pressure possible in order to minimize pump wear. A pressure of 150 psig was used for tests at fuel-out temperatures below 550°F, 180 psig for tests at 550°F, and 210 psig for tests above 550°F.

Gas-drive coker tests were run using the CRC flow configuration (Figure 1).

JFTOT tests were run with 300 psig system pressure. Test fuel was prefiltered through Whatman No. 2V paper, and a 17-μ in-line filter was used. In the early tests, attempts were made to run the JFTOT at temperatures above 700°F. Later tests were limited to 700°F. The early tests gave several instances of rating problems, such as floating breakpoints, post-peak profiles, and streak deposits. These problems were much less frequent in the later tests. Two tube-deposit failures in the later tests were characterized by thin, narrow streaks of deposit, and the tubes after test were found to be bent (bowed) rather more than usual. Both these tests were run in the new Model B unit; check tests in the Model C unit on the same fuel at the same conditions did not show either the excessive bending or the streak deposits. The outer tubes of both units were checked carefully and found to be identical. New outer tubes were installed in both JFTOT units and repeat tests were run, but the results were the same as before. The only difference in equipment that could be detected was a tighter fit between the heater tube and buss connectors in the new unit. This may impose more end-restraint on the tube when it is heated and thus distort the tube to a greater degree.

TABLE 14. EFFECT OF STORAGE IN
PILLOW TANK ON JP-7 FUEL
THERMAL STABILITY

Months storage	Heater breakpoint, °F, for Code 3		
	JFTOT coker	Gas-drive coker	Research coker, 300°F reservoir
0 (base)	686 715	>700	525 550
0 (rinse)	720 723	>700	525 550
1	>700	>700	575 600
2	>700	>700	550
3	>700	675 700	525 550
4	>700	650	550
5	>700	>700	525 550
6	>700	>700	525
7	>700	>700	525 550
8	>700	700	550
9	>700	700	550 575
10	>700	>700	550
11	>700	>700	525 550

Deposit inception points beyond the temperature profile limits were encountered in many tests, and these had to be estimated by extrapolation. As discussed earlier (Section II-10), deposits at or very near the cold fuel-inlet end do not really define an inception temperature, but merely mark the start of the heated section. Significance of inception temperatures based on such deposits is very doubtful at best. In these tests, only Code 1 and 2 deposits were involved, so there was no anomaly in breakpoint determinations.

(3) *Thermal Stability Test Results*

Thermal stability test results obtained in the three types of coker are summarized in Table 14. The breakpoints are expressed as a range of values whenever the pass-fail ratings were about equally divided at the indicated temperatures. Even the single-valued entries represent a certain amount of subjective interpretation of the data, since the series of tests on a given fuel would often include isolated instances of failure at a

temperature below the breakpoint or of passes .t the breakpoint or at higher temperatures. In assigning the JFTOT breakpoints, it was necessary to exclude numerous results in which the deposits were judged to be nonsignificant because of location or shape.

The breakpoints indicate that the JP-7 fuel has not changed in storage to any degree that can be detected by these tests.

Filter plugging data are not shown here, since the pressure drop was zero in almost all of the tests. One test in the gas-drive coker and one test in the research coker did show significant plugging, but these were isolated, nonrepeatable instances, probably reflecting either sample contamination or some idiosyncrasy of the particular coker test.

It is interesting to note that 11 months of storage in this pillow tank did not harm the thermal stability of this JP-7 fuel. In the laboratory soak tests described previously, using polyurethane rubbers at surface/volume ratios comparable to that of the pillow tank, some fuel degradation was observed after 6 months of hot-room storage but not after 2 to 4 months of indoor-ambient storage. It would be interesting to continue both the laboratory and the tank-storage programs to determine whether direct correlations can be obtained. Unfortunately, the hot-room test conditions were upset when the hot room became inoperable. Another difficulty in establishing any direct correlation is the lack of knowledge of the exact composition of the liner material in the pillow tank and of the previous history of the tank.

One point of difference between the laboratory and tank storage tests is the use of rinse fuel in the tank test. The rinse fuel itself did not drop in thermal stability, but there is still the presumption that it *may* have removed something that otherwise would have contributed to subsequent fuel deterioration.

That the rinse fuel did remove *something* is a fact, not a presumption. Data on WSIM values and gum contents of the test fuels are shown in Table 15. The rinse fuel WSIM was significantly lower than that of the base fuel or those of any later samples taken from the tank. The existent gum content of the rinse fuel was 0.6 mg/100 ml, which is slightly higher than the values of 0.0-0.2 obtained on the base fuel and all later samples from the tank.

Even though precise comparisons and correlations cannot be made between the laboratory and tank storage results on thermal stability, the data will provide very useful information for judging the suitability of this class of material for tank-liner service with JP-7 fuel.

TABLE 15. WSIM AND GUM CONTENTS OF JP-7 FUEL IN PILLOW-TANK STORAGE

Months storage	WSIM	Gum content, mg/100 ml	
		Existent	Potential
0 (base)	91	0.2	---
0 (rinse)	74	0.6	0.6
1	96	---	---
2	96	0.0	0.0
3	99	0.0	0.0
4	97	0.0	0.0
5	98	0.0	0.6
6	95	0.0	0.0
7	99	0.0	0.0
8	99	0.0	0.0
9	99	0.0	0.0
10	98	0.0	0.0
11	99	0.0	0.0

SECTION III FUEL LUBRICITY

1. BACKGROUND

The "lubricity" of jet fuels is a practical concern in operation of any fuel-system equipment in which the fuel acts as a lubricant. The work reported here deals with the effects of fuels on coefficient of friction, rather than effects on wear rates or on load-carrying ability. Interest in this particular fuel characteristic resulted from an Air Force field problem with sticking of servo valves in aircraft fuel controls. This was identified tentatively as a fuel lubricity problem, since there was no obvious evidence of valve silting or mechanical factors affecting the sticking. The problem first became evident in 1966, after discontinuing the use of corrosion inhibitors in JP-4 fuel. Once the corrosion inhibitors were restored, the problems diminished. Thus, the presumption was (and is) that these particular fuel-control malfunctions are related to fuel lubricity and that corrosion inhibitors (or at least certain corrosion inhibitors) do improve the lubricity of JP-4 fuel.

Another background item that should be mentioned is the imposition of a neutralization number limit in the JP-4 specification during the past year. This could have very definite effects on fuel lubricity in specific cases, but no direct information is available.

Investigation of the field problem has been hampered by the lack of any test device that might be expected to predict valve sticking. The lack of any suitable test has also hampered investigation of the antifriction characteristics of fuels and fuel additives. In this connection, the Air Force had funded a program, directed by the Coordinating Research Council (CRC), for the design and construction of a device to rate fuels in terms of this "lubricity"

characteristic. That program, which was carried out by the Bendix Corporation, resulted in the device known as the CRC Jet Fuel Lubricity Simulator. Acceptance tests on the simulator were performed by Bendix and reported by the Coordinating Research Council.⁽¹²⁾ The test section was then delivered to the Air Force. Two other test sections have been built by Bendix, one for General Electric Co. (Evendale, Ohio) and one retained by Bendix.

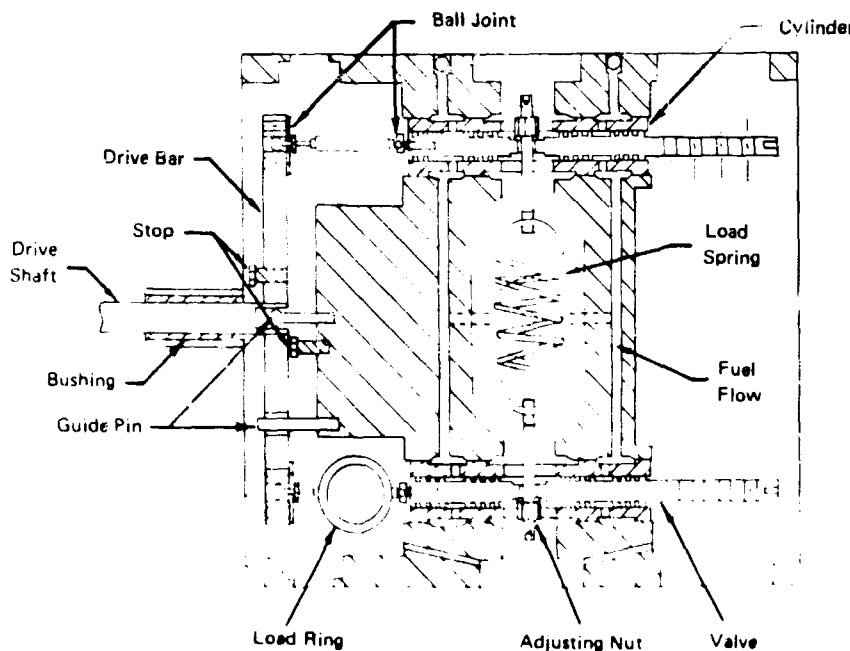


FIGURE 8. JET FUEL LUBRICITY
SIMULATOR TEST SECTION

The simulator test section as furnished by Bendix is illustrated in Figure 8. It consists of two aluminum cylinder valve sets with hard-faced barrel and lands, mounted in an aluminum block housing with passages for supplying test fuel to the cylinder valve sliding surfaces. In our work, the housing has been set flat, with both valves in the same horizontal plane. The valves are side-loaded by a calibrated spring connecting the two valves. Each

2. TEST APPARATUS

The simulator consists of two valve sets mounted in a housing with means for driving the valves in reciprocating motion, imposing a side load on the valves, and measuring axial force and valve position.

cylinder/valve set is matched; the components are not interchangeable. One end of each valve is connected via a load ring to the crossarm of a drive shaft. The axial force on each valve is measured by strain gages mounted on the load rings. Valve position is measured by means of a linear voltage differential transformer (LVDT), not shown in Figure 8. This instrument was not supplied with the test section.

The unit delivered to the Air Force and supplied to SwRI for evaluation consisted of a test section only, without any external drive, fuel supply system, or instrumentation. No specifications or description were given for such auxiliary equipment; hence, much of our initial effort was taken up in design, selection, procurement, assembly, and checkout of the auxiliary equipment. The three laboratories that have set up and operated lubricity simulators, Bendix, General Electric, and SwRI, have proceeded in somewhat different directions on this auxiliary equipment, and the overall apparatus cannot be regarded as fully standardized at present.

A diagram of the complete test assembly used in the work reported here is shown in Figure 9.

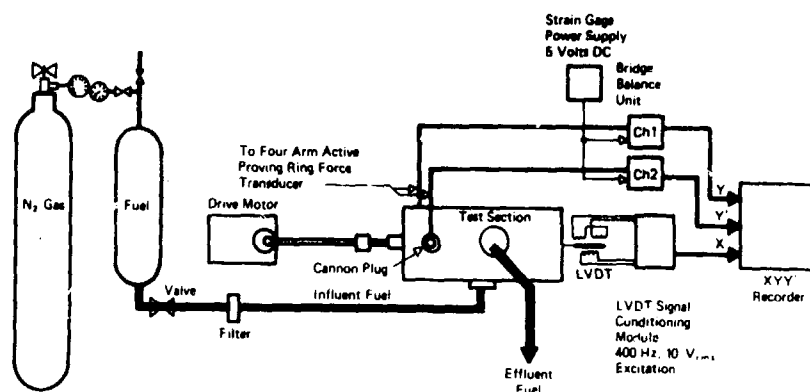


FIGURE 9. DIAGRAM OF JET FUEL LUBRICITY SIMULATOR TEST APPARATUS

In operation, the valves are moved back and forth by the drive system while pressurized fuel is fed to the sliding surfaces.

Signals representing axial force (load-ring strain gages) and valve position (LVDT) are fed through a demodulator circuit to an XYY recorder to give a continuous plot of axial force vs. valve position for each cylinder/valve assembly. For any given side load imposed on the valves by the load spring, the valve breakaway and running forces and the corresponding coefficients of friction should be a measure of the "lubricity" of the test fuel.

The fuel system consists of a nitrogen-pressurized fuel reservoir, an in-line filter (0.45- μ membrane), and the necessary valves and tubing. All fuel-wetted components are stainless steel. The original design was for a single fuel, as shown in Figure 9; later, a second fuel reservoir was added to facilitate switching from one test fuel to another.

The filtered fuel is fed into the bottom of the test section (center as shown in Figure 8), from which it flows through internal passages in the housing to the two inter-land spaces on each valve, through the land-cylinder clearances, and thence either to the center cavity or the end cavities of the housing. Oddly, almost all of the flow goes inward to the center cavity; no explanation has been found for this asymmetric flow. When the center cavity is filled with fuel, it overflows through a connection in one side of the housing (bottom as shown in Figure 8) and can be collected and timed to establish the fuel feed rate. These details are given because this configuration differs from those used by other laboratories. The test section could be operated in any position, and fuel flow could be reversed by pressurizing the entire housing. With the flow arrangement that we have used, the housing cavities are unpressurized and can be left open as needed to check fuel flow or leakage and to connect a position-indicating instrument to either valve.

The drive system used here includes a double-reduction, worm-gear motor with drive control delivering high torque at speeds down to 1/2 rpm, with precise speed control and provision for braking and reversing. The motor shaft is fitted with a drive crank (wheel) and connecting rod, which in turn is joined to the test-section drive shaft by means of a yoke. The crank radius is fixed at 0.14 in., giving a stroke of 0.28 in.

Instrumentation was selected so as to record axial force vs. position for each valve, or to give time-plots of any two variables. The axial force is determined by the outputs of strain gages on the two load-ring assemblies, calibration curves for which are given in Figure 10. Each load-ring response is linear, both in tension and

compression, over the range of application. Valve position is measured by a linear voltage differential transformer (LVDT) mounted on either valve at the end opposite the load ring. It is also possible to mount the LVDT on the drive shaft, but this would not give a good indication of valve position or travel. A calibration curve for the LVDT is shown in Figure 11. It will be noted that the deviation from linearity does not exceed 1.5% up to ± 0.15 in. displacement. This is entirely acceptable here, since the valve position measurements are used primarily to provide a reference axis for plotting the primary force values taken from the load-ring outputs.

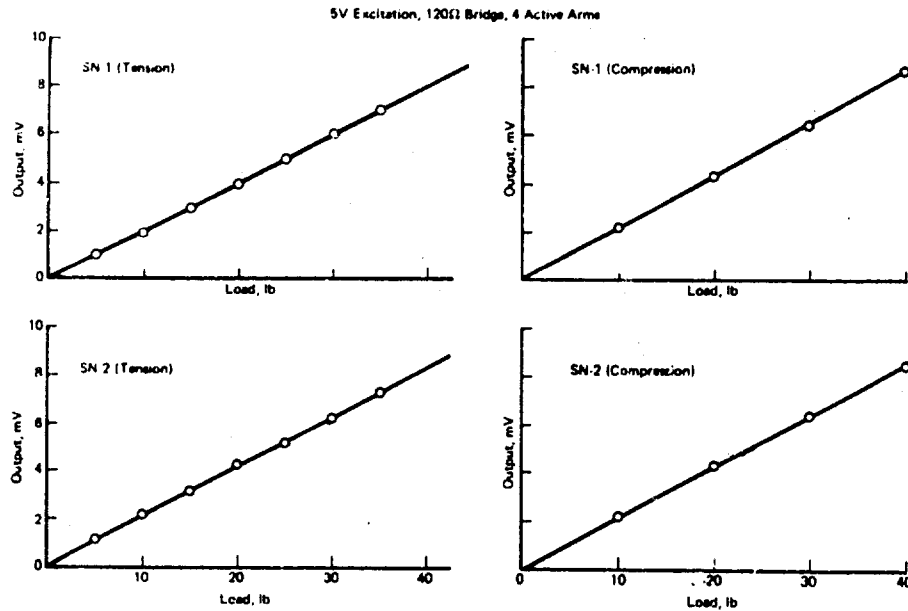


FIGURE 10. LOAD RING CALIBRATION

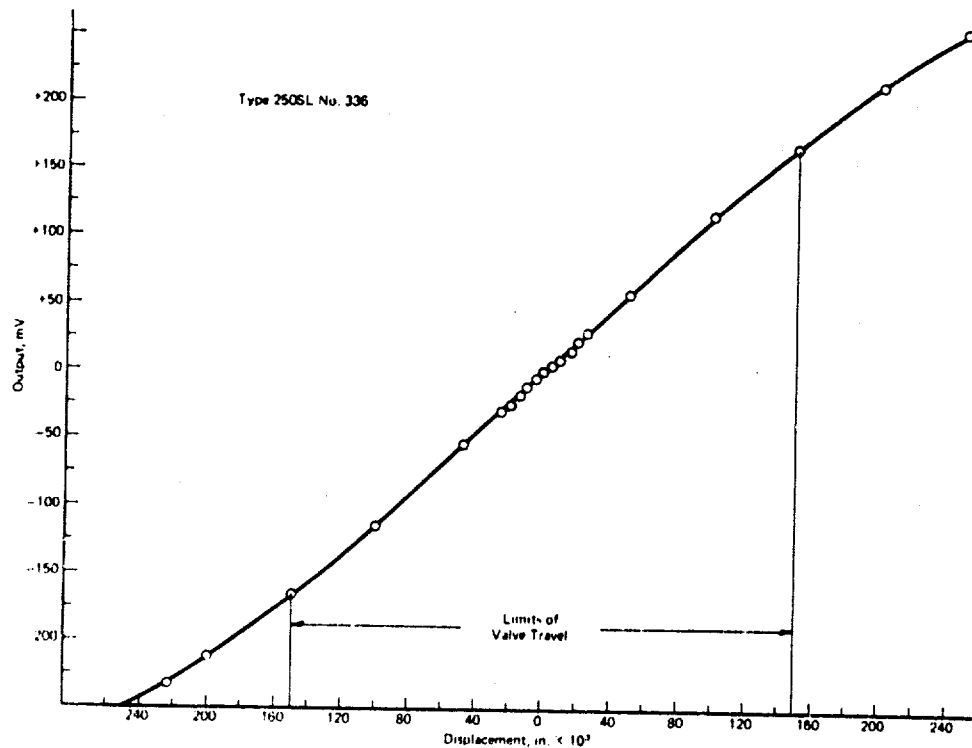


FIGURE 11. LVDT CALIBRATION

Signals from the LVDT and load rings are fed to a demodulator with digital indicator, the signal conditioning circuits of which have been matched to the characteristics of the strain gages and LVDT actually used. This indicator provides a convenient means of relating signal outputs to real values (force or position) while the simulator is in operation. However, the instrument has given a number of problems and has been returned to the manufacturer for correction. Meanwhile, a strain gage coupler, on loan, has been used to condition the simulator outputs.

The conditioned signals are fed to a two-pen, three-variable XYY' recorder that will generate two curves simultaneously from dc signals representing a single independent variable and two dependent variables. A built-in sweep generator provides a time base on the horizontal axis for plotting any two variables against time.

In the usual mode of operation, the position of one valve (LVDT output) is recorded on the X-axis, and the axial forces on the two valves (load-ring outputs) are recorded on the Y and Y' axes. This gives two traces, roughly rectangular, the heights of which (Y-direction) are proportional to the axial forces. Each trace shows a force peak at each end, representing the breakaway force. The peak-to-peak height is a measure of the average breakaway force. The average height of the rectangle (excluding the peaks) is a measure of the running force. These forces are converted to equivalent coefficients of friction by dividing by the side load on the valves. A typical plot of axial force vs position for a pair of valves is shown in Figure 12. The "push" direction occurs when the load rings are in compression and the "pull" direction results when the load rings are in tension.

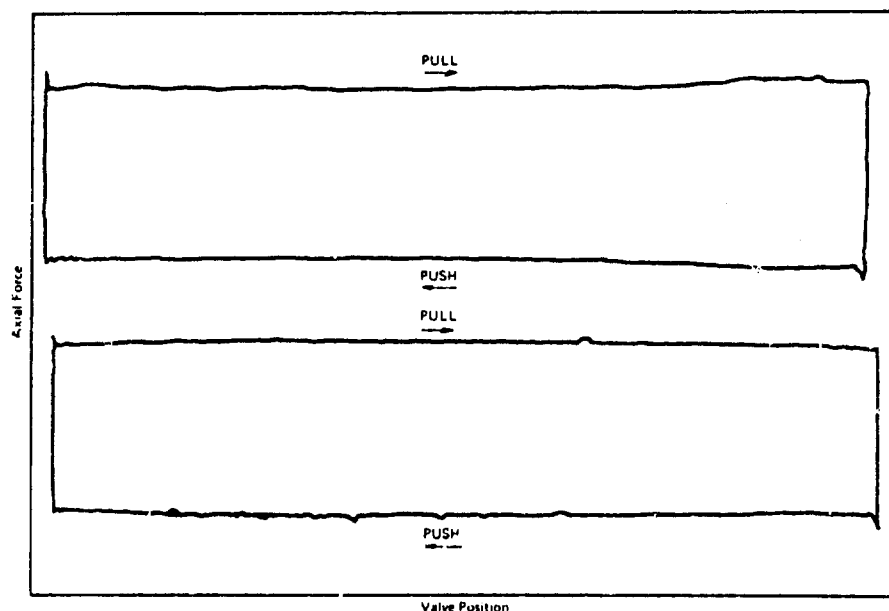


FIGURE 12. TYPICAL FIVE-CYCLE TRACE FOR TWO VALVES OF LUBRICITY SIMULATOR

The recorder may be set up to plot time on the X-axis, along with position and axial force for one valve on the Y and Y' axes (or, alternatively, axial force for both valves). This method of recording has been used primarily to analyze the motion-force relationships, to show the amount of "slop" in the linkages, and to run special experiments on breakaway forces with slow application of load.

In the initial stages of the lubricity simulator investigation, no one other than the manufacturer, Bendix, had any working experience with the simulator. Only limited background data were available, those having been obtained in the acceptance testing performed by Bendix. Hence, a rather thorough checkout and familiarization test series was required here. First, the test section was examined carefully to ensure that it did meet the specification requirements set forth by the CRC Fuel Lubricity Group and to determine that the critical dimensions were within the manufacturer's indicated tolerances. No discrepancies were found.

3. PRELIMINARY STUDIES

Isooctane was chosen as the test fluid for preliminary studies. This had been recommended by the CRC group as one of the fluids to be used in initial evaluations and had been used by Bendix in acceptance tests. Isooctane should represent a fluid with very little "lubricity," since it is relatively low in viscosity and free of nonhydrocarbon constituents. The isooctane used here is material conforming to TT-S-735, Type I, used without special treatment other than filtration through the in-line filter.

Before any testing, the individual components of the test section and the fuel system were rinsed thoroughly with isopropanol and then with acetone, and air-dried. As each cylinder/valve set was assembled, it was wet with the test fluid, isooctane. Pressurized fuel was fed to the test section and the center cavity was filled, after which flow rate was measured and adjusted.

For all of the work reported here, fuel flow rate was held at 3 ml/min, setting the drive pressure as needed. This flow rate is quite close to that used in some of the early work of General Electric and Bendix, where a fixed inlet pressure had been used.

For the work reported here, the load spring was placed in the center or "inboard" position of the valves, so as to apply a perpendicular force midway between the valve lands. This spring position is as shown in Figure 8. The valves can also be side-loaded in any one of four "outboard positions," as indicated by the holes in each valve stem. However, these outboard load positions have not been used in any of the work performed here.

In determining friction coefficients, the drive is started with fuel already flowing through the test section. A trace is plotted for five consecutive cycles, each cycle consisting of a crank revolution, i.e., a 0.28-in. valve stroke in each direction. When the simulator and associated instrumentation are operating properly, the five-cycle trace will appear almost as a single line. If the five cycles do not track properly, the simulator drive is left on and recording is repeated until a repeatable five-cycle trace is obtained.

The first study of coefficient of friction with isooctane was made with side loads of 5, 10, 15, and 20 lb and with speeds of 2, 3, and 5 cycles per minute. The results of these tests are shown in Table 16. It can be seen that the two valves differed significantly in the results obtained, and that there was a trend toward higher apparent coefficients of friction as the side load was increased. No significant effect of cycle rate could be detected. Averaging all results obtained with a given valve and a given side load (regardless of cycle rate), the following values are obtained for the mean coefficient of friction and the corresponding standard deviation:

	5 lb	10 lb	15 lb	20 lb
Breakaway				
3A	0.102(0.012)	0.213(0.023)	0.210(0.012)	0.228(0.004)
4A	0.127(0.010)	0.251(0.024)	0.259(0.012)	0.292(0.003)
Running				
3A	0.080(0.008)	0.176(0.016)	0.179(0.013)	0.196(0.004)
4A	0.101(0.009)	0.222(0.019)	0.230(0.015)	0.268(0.009)

The difference between the two valves is very apparent in this comparison. In all cases, comparing either breakaway or running friction at any given load, Valve 4A gave values some 20-35% higher than those given by Valve 3A, and the spread between the two valves was greater at the higher loads. Also, it can be seen that the major effect of increasing the side load was in the step from 5 to 10 lb; further increases in load gave only moderate increases in apparent coefficient of friction.

In examining the repeatability of these results, the schedule of testing must be taken into account. One series of runs at 5, 10, and 15 lb side load and all three cycle rates was made on one day. Four days later, this series was repeated, including one extra run at the 10-lb/2-cycle condition. On the following day, all runs at 20 lb side load

were made. It will be noted that the repeatability standard deviations were lower for the 20-lb runs than for those at lower side loads, particularly if compared in terms of percent of the mean, which was 1.3% for the 20-lb runs and 5-12% for the other runs. This difference may well reflect actual changes in the condition of the valves during the course of these tests; the 20-lb runs, all made on the same day, would be expected to be more repeatable than runs made on different days.

Another factor affecting the repeatability is error in the side load. Installation of the spring in the simulator is a rather awkward operation, and the adjustment to the desired extension (for a given load) must be made by trial and error setting of two retaining nuts. The spring furnished with the simulator has a force constant of about 65 lb/in., in line with original requirements for operation at side loads as high as 50 lb. With this spring, an error of only 0.001 in. when adjusting the extension will give a load error of 0.065 lb, which is 1.3% relative error with a 5-lb side load. Clearly, a lighter spring is needed for accurate setting of loads in the lower range. Such a spring was obtained recently but was not used in any of the tests reported here.

Referring again to Table 16, it will be noted that, for the runs at 5, 10, and 15-lb loads, the coefficient of friction was almost always higher in the second run than in the first run (made four days earlier). This could reflect merely the load spring setting inaccuracy just discussed. However, it is difficult to visualize enough error to account for individual deviations. For example, with Valve 4A at 5 lb and 2 cycles/min, the two breakaway friction values were 0.106 and 0.135. To attribute this deviation solely to load spring setting error would mean that there was a difference in true load of about 1.2 lb between the two tests, equivalent to about 0.018 in. spring adjustment. So large an error does not appear likely, even with the rather awkward means of adjustment that must be used.

The apparent friction trend from first to second run was uniformly upward, this argues against load spring setting error as the source of the discrepancy. It appears more likely that there was an actual change in the behavior of the fuel-lubricated sliding contact. This could be caused by trace residues of polar contaminants left in the apparatus even after the exhaustive cleaning, these residues gradually

TABLE 16. PRELIMINARY LUBRICITY SIMULATOR RESULTS ON ISOOCTANE

Flow rate: 3 ml/min
No. of cycles: 5
Temperature: 78 ± 3°F

Side load, lb	Coefficient of friction*					
	2 cyc/min		3 cyc/min		5 cyc/min	
	Bkw	Run	Bkw	Run	Bkw	Run
<i>Valve 3A</i>						
5	0.088	0.068	0.090	0.074	0.092	0.073
5	0.108	0.088	0.113	0.088	0.120	0.088
10	0.158	0.140	0.222	0.175	0.215	0.173
10	0.220	0.183	0.220	0.188	0.233	0.190
10	0.225	0.185				
15	0.194	0.167	0.201	0.165	0.196	0.166
15	0.222	0.194	0.222	0.195	0.220	0.188
20	0.225	0.195	0.233	0.198	0.235	0.203
20	0.223	0.193	0.225	0.193	0.230	0.192
<i>Valve 4A</i>						
5	0.106	0.088	0.120	0.093	0.135	0.095
5	0.135	0.113	0.135	0.106	0.130	0.108
10	0.194	0.180	0.265	0.218	0.265	0.218
10	0.265	0.230	0.255	0.235	0.263	0.243
10	0.255	0.230				
15	0.247	0.215	0.255	0.215	0.243	0.215
15	0.278	0.250	0.266	0.246	0.266	0.238
20	0.295	0.260	0.298	0.273	0.292	0.278
20	0.293	0.264	0.290	0.279	0.288	0.255

*Calculated by dividing axial force by side load, without any correction for "zero-side-load" axial force.

disappearing as the apparatus is run. Another possibility is the accumulation of small amounts of debris from wear and damage of the sliding surfaces, which would increase the apparent coefficient of friction. It should be remembered that the tests in question were run before the need for any break-in procedure had been established, so that timewise changes in the valve surfaces and in the apparent coefficient of friction were quite likely.

Another factor that could affect the test repeatability is temperature. No precise temperature control has been maintained, other than that the room was held between 75 and 80°F when tests were performed. Whenever severe temperature fluctuations were caused by heating or air conditioning failures, work was suspended in order to avoid possibly significant temperature effects of fuel viscosity or valve/cylinder geometry.

4. INITIAL EVALUATION OF FUELS

Following the preliminary work on isooctane, a brief attempt was made to determine whether the lubricity simulator could detect additive type and concentration in real fuels. For this work, a JP-4 fuel, produced by Cosden

TABLE 17. EFFECT OF CORROSION INHIBITORS ON BREAKAWAY FRICTION

Side load: 20 lb
 Cycle rate: 2 cycles/min
 No. of cycles: 5
 Temperature: 78 ± 3°F

Inhibitor, lb/Mbbl	Breakaway friction*, Valve 3A			Breakaway friction*, Valve 4A		
	0	4	16	0	4	16
Isooctane	0.225			0.295		
Mean	<u>0.223</u> 0.224			<u>0.293</u> 0.294		
Cosden JP-4 + inhibitor X	0.323	0.315	0.298	0.383	0.373	0.345
	0.290	<u>0.315</u>	0.290	0.373	<u>0.373</u>	0.348
	<u>0.295</u>		0.290	<u>0.383</u>		0.345
Mean	0.303	0.315	<u>0.293</u> 0.294	0.379	0.373	<u>0.343</u> 0.345
Cosden JP-4 + inhibitor Y	0.290	0.270	0.228	0.375	0.353	0.298
	0.298	0.273	0.230	0.368	0.360	0.310
	<u>0.298</u>	<u>0.278</u>	<u>0.240</u>	<u>0.375</u>	<u>0.363</u>	<u>0.315</u>
Mean	0.295	0.274	0.233	0.373	0.359	0.308

*Breakaway coefficient of friction calculated by dividing axial force by side load, without any correction for "zero-side-load" axial force.

Oil and Chemical Co., was furnished from Air Force stock. This fuel, which contains no corrosion inhibitor, was purchased in 1967, when corrosion inhibitors had been temporarily excluded from JP-4 fuel. For the tests reported here, this JP-4 fuel was blended with corrosion inhibitors X and Y, each at concentrations of 4 and 16 lb/1000 bbl, which represented current QPL minimum and maximum allowable use concentrations. These tests were conducted at a fuel flow rate of 3 ml/min, valve cycle rate 2 cycles/min, and side load 20 lb. Uninhibited JP-4 was run first, followed by JP-4 with 4 lb/Mbbl of Inhibitor X and then with 16 lb/Mbbl. Then the apparatus was flushed and rinsed thoroughly, after which the sequence of runs was repeated with JP-4 containing Inhibitor Y.

The results of these tests are listed in Table 17, along with values obtained on isooctane the day before this series was started. Oddly enough, all of the results of Cosden JP-4, with or without inhibitors, gave coefficients of

friction *higher* than those for the isooctane. This was completely unexpected and has not been explained satisfactorily. It will also be noted that the Inhibitor X gave little or no decrease in friction when added at the 4-lb level and only a moderate decrease when added at the 16-lb level. The effect of the Inhibitor Y was more pronounced.

It would not be safe to draw any conclusion from these tests as to the relative merits of the two additives in improving fuel lubricity. At the time these tests were run, considerable operating difficulty was being experienced with the simulator, and the reversal of the expected comparison with isooctane must cast doubt on all of the values.

5. FIRST INTERLABORATORY CHECKS

In an attempt to determine the reproducibility among the three prototype simulators, two different test fluid samples, each from the same bulk source, were supplied to General Electric, Bendix, and SwRI. The two fluids were isooctane and Bayol R-34, which had been recommended by the Test Fuels Panel of the CRC Lubricity Group. All laboratories conducted their tests in this program at a side load of 5 lb and a speed of 2 cycles/min. The following results were obtained:

	Breakaway coefficient of friction		
	Bendix	G.E.	SwRI
Isooctane	0.152	0.159	0.098 (3-B)
			0.108 (3-B)
			0.116 (4-B)
			0.135 (4-B)
Bayol R-34	0.170	0.197	0.338 (3-B)
		0.227	0.335 (4-B)

The Bendix and General Electric results were in reasonable agreement, but the SwRI results were lower on isooctane and far higher on Bayol R-34. Just why one laboratory should rate high on one fluid but lower on another is entirely unclear. All three laboratories agreed that Bayol R-34 gave higher friction values than did isooctane. This cannot be explained unless one assumes a large hydrodynamic component in the lubrication (unlikely under these slow-speed startup conditions) or insufficiently pure isooctane. All in all, this preliminary program did not lead to any satisfactory conclusions.

6. INDEPENDENT BREAKAWAY MEASUREMENTS

Following the attempt to establish reproducibility among laboratories, it suddenly became impossible to obtain any consistent results with any fluids. The valve operation became very noisy, and stick-slip behavior was evident, particularly at the lower cycle rates. The ensuing search for the causes finally developed into a complete shakedown of the entire apparatus. The demodulator instrument was returned to the manufacturer for service and recalibration, and a substitute was installed. Two unused cylinder/valve sets (Nos. 5 and 6) were placed in the simulator. It should be noted that no specific break-in procedures had been established at that time. No consistent results were obtained with the new cylinder/valve sets. In retrospect, it appears probable that the lack of any adequate break-in, plus operation with poor lubricants under fairly stringent conditions, damaged these valves sufficiently that operation was poor and consistent results could not be obtained.

During the course of this work, it was decided to determine whether the breakaway coefficient of friction could be determined by independent loading of one valve, having disconnected the drive system. This would eliminate the effects of vibration, valve-to-valve interactions (via the crossarm), and play in the linkages; in other words, it could provide a well-controlled means of recording the breakaway force with a gradual application of axial load at a fixed rate. In practice, since the simulator is designed with two valves, it is difficult to investigate one valve independently.

For this work, the drive-end cover plate, drive shaft, and crossarm were removed, and a load adaptor was mounted on each load ring, for connection to a variable weight (one-gallon bucket) by means of a flexible cable and pulley. With this setup, after hand-positioning both valves, establishing the flow rate of the test fluid (isooctane), and setting the side load, one valve would be end-loaded gradually by adding water to the bucket at a fixed rate. The axial load was plotted against time on the 'XYY' recorder, and the load at breakaway was taken as a measure of the static coefficient of friction.

The valves were hand-positioned in the cylinders and side-loaded to 2, 5, or 10 lb. The starting position for the valves was either "extended" or "midpoint," which would correspond in the regular drive configuration to the

TABLE 18. BREAKAWAY FRICTION WITH INDEPENDENT LOADING

Fluid: Isooctane
 Flow rate: 3 ml/min
 Temperature: 78 ± 3°F

Side load, lb	Starting position	Breakaway coefficient of friction					
		Valve 5A			Valve 6A		
		Result	Mean	Std dev	Result	Mean	Std dev
10	Extended	0.583	0.598	0.029	0.528	0.593	0.093
		0.623			0.503		
		0.628			0.743		
		0.558			0.598		
10	Midpoint	0.508	0.556	0.036	0.678	0.560	0.068
		0.573			0.533		
		0.540			0.523		
		0.603			0.508		
5	Extended	0.288	0.276	0.013	0.342	0.347	0.017
		0.288			0.362		
		0.258			0.322		
		0.268			0.362		
5	Midpoint	0.368	0.336	0.022	0.282	0.294	0.013
		0.308			0.302		
		0.328			0.282		
		0.338			0.312		
2	Extended	0.675	0.675		0.425	0.425	
		0.675			0.425		
2	Midpoint	0.715	0.715		0.445	0.445	
		0.715			0.415		

extreme end of the push stroke and to midstroke, respectively. The values that were obtained are shown in Table 18. It can be seen that the apparent coefficients of friction for breakaway with 2 or 10 lb side load are all in the 0.42-0.74 range, i.e., much higher than those obtained in the normal drive configuration. For the 5-lb side load, the coefficients of friction at breakaway are in the 0.28-0.37 range, which is still somewhat higher than we had considered "normal" for breakaway friction for isooctane. The reason for the higher values at 2 and 10 lb in comparison with 5 lb is not entirely clear. Normally, one would expect the coefficient of friction to remain constant or increase as the load is increased, as "lubrication" becomes poorer. The high values at the lowest load may merely reflect large relative errors introduced by "binding" of the valve, i.e., by the large relative effect of load self-imposed by imperfect fit of the valve and cylinder. At low applied loads, the true load is much greater than the applied load, but this is neglected in calculating the coefficient of friction. Unfortunately, there is no sound way of compensating for this error. In some of the subsequent work, a zero-applied-load breakaway force was determined, and this was subtracted from the

breakaway force with applied load to give a corrected force for use in calculating coefficient of friction. This method is not sound, since side-loading the valve changes the valve/cylinder geometry and hence the self-imposed drag.

The "independent breakaway" values, regardless of side load, are all considerably higher than those obtained under normal drive conditions. This can be explained by the difference in method of load application. In the independent breakaway tests, the valve is at rest for a relatively long period, amounting to several minutes. Load is applied very gradually, and there is no vibration. All of these conditions favor higher breakaway friction. Under normal drive conditions, some vibration is present, and the valve is at rest for only a few seconds. In theory, the rest period would be infinitesimal, assuming that the valve motion followed the theoretical sine curve. In practice, the curve of valve displacement vs. time has flats at each extreme position, representing play in the linkages. Most of this probably comes from the ball-joints that connect up the load rings; these joints develop considerable looseness, whether from wear or from cold flow of the Teflon inserts, and it has been necessary to replace them several times. The motion curves have not yet been analyzed to calculate the actual amount of play, but it is often enough to account for some 5-10% of the total elapsed time being spent at rest at the ends of the stroke. Then, after the play is taken up, the valve is set into motion quite rapidly, since the drive shaft is already moving. This sort of breakaway is quite different from either a true sine-curve reversal with

infinitesimal dwell time or from a breakaway obtained in the independent gradual-loading tests. Hence, it is not at all surprising that the independent measurements gave completely different (higher) values than did the measurements made with the simulator drive system.

In many of the tests with gradual loading, the normal straight line on the plot of axial force vs. time would show an intermediate break, in which the valve evidently slipped slightly from its original position but was hung up almost immediately; thereafter, the straight line continued up to the final breakaway and travel.

Returning to the data of Table 18, it will be noted that the repeatability standard deviation for the runs at 5 lb side load was significantly less than in the runs at 10 lb. At the same time, valve-to-valve and position-to-position deviations (based on the means) were less in the 10-lb runs than in the 5-lb runs. These data did not furnish any real basis for a choice of side load for further work. The 2 lb side load is considered too low to give any meaningful results, in view of the large relative effects of internal binding loads (note also the large valve-to-valve variations at the 2 lb load). The 5 lb load was chosen for further work primarily because of our belief that higher loads, with poor lubricants, can lead to damage of the test surfaces and hence aggravate the uncontrollable effects of mechanical factors.

7. INTERLABORATORY CHECKS WITH SAME VALVES

In order to determine whether individual valve characteristics or other factors were the primary cause of the poor reproducibility observed earlier, a test program was set up using the General Electric and SwRI lubricity simulators with the same cylinder/valve sets to be used in both simulators. The agreed test conditions were:

Side load (center position)	5 lb
Cycle rate	5 cycles/min
Stroke length	0.28 in.
Flow rate	3 ml/min

General Electric furnished two new cylinder/valve sets for this work, identified as GE-5 and GE-6. General Electric also furnished a common stock of isooctane and oleic acid for the test fluids. Basically, the program plan consisted of a break-in run of 600 cycles in the G.E. simulator using isooctane plus 50 mg/liter of oleic acid as the test fluid, at the end of which friction would be determined. They would then switch to straight isooctane by their normal fuel-change procedure and determine the new friction value. The cylinder/valve sets would then be removed and transferred to the SwRI simulator, where essentially the same sequence as that performed by G.E. would be repeated.

It should be mentioned that, prior to this work, G.E. had come to the conclusion that the valves and cylinders should never be removed from the housing except when absolutely necessary, in order to avoid possible damage to the brittle hard-faced surfaces. Hence, they would switch from one test fuel to another without removing the valves. We had gone to the other extreme, and had made it a point to disassemble and solvent-clean the entire apparatus, including valves, cylinders, housing, fuel tank and filter, whenever test fuel was changed. For this program, however, we installed a second fuel supply tank, connected to the main fuel supply line just ahead of the in-line filter, to permit changing test fuels without complete disassembly.

So far as we can determine, the G.E. and SwRI drive systems were very comparable at the time this program was run. Instrumentation differed somewhat. G.E. handled their outputs on a high-speed two-pen recorder, plotting position and axial force (vs. time) for one valve only, rather than 'XXX' plotting for both valves. This difference in recording instruments should not have any effect on the results obtained, assuming that both instruments have a sufficiently fast response to pick up the actual maximum breakaway force. At the slow valve travel rates that are used in this work, instrument response time does not appear to be critical.

When G.E. gave Valves GE-5 and GE-6 a break-in run on isooctane plus oleic acid, they found that the friction stabilized after some 550-600 cycles; values of 0.150 for breakaway and 0.140 for running coefficient of friction* were obtained after 600 cycles. This refers only to Valve GE-5; they did not obtain data on the other valve. They next switched the fuel feed to straight isooctane and ran for 50 more cycles, finding no change in the friction values. These values are recorded in Table 19, along with the results subsequently obtained in our tests.

The cylinder/valve sets were then removed from the G.E. simulator and transferred to the SwRI simulator, which had been solvent-cleaned before this operation. The cylinders and valves were installed in the housing in the same configuration used in the G.E. simulator.

In the initial SwRI tests (see Table 19), isooctane plus oleic acid was run for 600 cycles. Operation was stable, and it was noted that the valves appeared to function more smoothly than those sets we had been using previously. The repeatability standard deviation ranged from about 2.5 to 5% of the respective means, which is considered quite good for this type of extended run. The mean values for Valve GE-5 were 0.164 and 0.151 (breakaway and running) which are quite close to the values of 0.150 and 0.140 obtained by G.E. Valve GE-6 (which was not instrumented in the G.E. tests) gave values of 0.137 and 0.122 here.

No clear trend in friction values, either upward or downward, was observed in the 600 cycles on isooctane plus oleic acid. If anything, the trend was upward, but it was obscured by high initial values and one out-of-line final value. The high initial values suggest that a certain amount of break-in is necessary after reinstalling the valves, even though every effort is made to reinstall them in the same position.

After completing the runs with isooctane plus oleic acid, all fluid was removed from the test housing and fuel lines by suction. The filter housing was drained, and a new filter membrane was installed. Then the system was switched over to the second fuel supply tank, containing straight isooctane, and all fuel lines were flushed twice with approximately 100-ml portions of the new test fluid. Flow through the simulator was reestablished at 3 ml/min, and the drive system was turned on.

The values obtained in 175 cycles (35 minutes) of operation on straight isooctane were considerably higher than those obtained on isooctane plus oleic acid, contrary to the results reported by G.E. Also, it was noted that the friction values continued to rise during these 175 cycles, reaching final values of 0.261/0.206 for Valve GE-5 and 0.233/0.183 for GE-6. This suggests very strongly that traces of the oleic acid were still contaminating the working surfaces, and that the friction might well have continued to rise if additional cycles had been run.

This sequence was repeated, with 61 cycles on isooctane/oleic acid followed by 60 cycles on straight isooctane. Again, the friction values on isooctane/oleic were in the 0.14-0.16 range for GE-5 and 0.12-0.14 for GE-6. Subsequent cycles on straight isooctane gave the same pattern as before: rising friction values, significantly higher than on isooctane/oleic acid.

Since the G.E. results did not show any difference between isooctane with or without oleic acid, it appears logical to ascribe the discrepancies between the two laboratories' results to differences in the fuel supply system configuration and the flushing procedures that were used. In the SwRI tests, all excess fluid was removed from the housing and fuel lines before switching fuels, and the fuel lines were flushed with new fuel before completing the hookup. In the G.E. tests, the switch of fuels was made without any extra flushing or removal of the previous fuel. Even with the flushing procedures used in the SwRI tests, the coefficient of friction with isooctane was continuing to rise, although at a decreasing rate, after 175 cycles of operation. This suggests the need for completely independent fuel supply systems if it is desired to go directly from one fuel to another in testing. Such systems would meet at a three-way valve mounted immediately at the inlet to the test section. Consideration should also be given to the use of an intermediate flushing solvent. It appears undesirable to disassemble the cylinder/valve sets each time fuel is changed, since this can easily lead to damage and is time-consuming. However, some alternative, thorough

*In G.E.'s tests, a zero-load axial force was subtracted from each axial force before calculating the coefficients of friction. The same procedure was used in the SwRI tests to ensure comparability of results.

TABLE 19. INTERLABORATORY CHECKS USING SAME VALVES IN LUBRICITY SIMULATOR

Flow rate: 3 ml/min
 Side load: 5 lb
 Cycle rate: 5 cycles/min
 No. of cycles: 5 (in SwRI tests)
 Temperature: 78 ± 3°F (in SwRI tests)

Lab	Test fluid*	Cumulative cycles on valves		Coefficient [†] of friction [‡] with indicated valve			
		Series	By lab	Breakaway		Running	
				GE-5	GE-6	GE-5	GE-6
G.E.	Isooctane + oleic acid	600	600	0.150		0.140	
G.E.	Isooctane	50	650	0.150		0.140	
SwRI	Isooctane + oleic acid	5	5	0.171	0.143	0.158	0.129
		55	55	0.145	0.127	0.142	0.119
		110	110	0.154	0.134	0.146	0.122
		205	205	0.165	0.137	0.153	0.128
		300	300	0.241 [‡]	0.164 [‡]	0.228 [‡]	0.157 [‡]
		351	351	0.168	0.144	0.153	0.127
		400	400	0.164	0.137	0.149	0.122
		454	454	0.166	0.137	0.150	0.122
		500	500	0.167	0.136	0.151	0.122
		551	551	0.168	0.139	0.153	0.122
		600	600	<u>0.170</u>	<u>0.140</u>	<u>0.153</u>	<u>0.108</u>
		Mean			0.164	0.137	0.151
	Std deviation			0.008	0.004	0.004	0.006
SwRI	Isooctane	30	630	0.224	0.213	0.176	0.149
		55	655	0.234	0.217	0.189	0.174
		70	670	0.233	0.219	0.189	0.175
		100	700	0.247	0.223	0.199	0.172
		150	750	0.255	0.231	0.202	0.181
		175	775	0.261	0.233	0.206	0.183
SwRI	Isooctane + oleic acid	11	786	0.164	0.145	0.134	0.122
		36	811	0.160	0.131	0.131	0.114
		61	836	0.165	0.135	0.138	0.117
SwRI	Isooctane	10	846	0.209	0.169	0.175	0.140
		35	871	0.223	0.187	0.192	0.156
		60	896	0.237	0.199	0.198	0.165

*Oleic acid concentration 50 mg/liter. Materials supplied by G.E.
[†]Breakaway force corrected by subtracting no load breakaway force.
[‡]Fuel supply exhausted during recording cycle; results excluded in calculating means and standard deviations.

method of cleaning the apparatus between fuels will be necessary in the development of a practical test procedure.

Apart from these indications on cleanup procedures, this program has illustrated the desirability of a definite break-in procedure to be applied to each cylinder/valve set. We attribute the smoothness of operation of the valves used in this program and the repeatable and comparable results to the fact that the valves were broken in with a fluid having (presumably) good lubricity, at the same conditions at which the tests were run.

These particular two cylinder/valve sets gave reasonably close agreement of results. It now remains to establish whether other sets will give comparable results on the same two fluids.

8. MECHANICAL AND OPERATING PROBLEMS

Several problems in the operation and maintenance of the lubricity simulator have been mentioned at various points in this discussion. It should be noted that these problems have taken up a good deal of time in our work with the simulator, and that some of the problems have not yet been solved. Therefore, we will list here some of the major problems that have been encountered and indicate the status of their solutions.

Of primary importance is the valve-to-valve variation in apparent coefficient of friction. Such differences are probably inherent in normal manufacturing procedures and cannot be eliminated at a reasonable cost. If these differences prove to be sufficiently large, the lubricity simulator can function only as a relative rating device. In this respect, the situation is analogous to that with the Ryder gear machine for synthetic lubricants, where the use of a reference oil and a relative rating system have become necessary.

During our operation of the simulator, we have observed considerable damage to the valve land surfaces. Damage to the barrel surfaces is also very probable, but is more difficult to detect visually. Damage of the lands shows up as scratches on the surfaces and nicks on the edges. We do not feel that minor damage of this sort should affect friction values to any great extent, *except* that the presence of wear debris in the working valve will probably cause a drag that is not part of the inherent "friction" of the valve. The hard-coating used in manufacturing these valves is a proprietary process; evidently, it is not possible to exercise quantitative control on the surface hardness. The coating is very hard, extremely brittle, and susceptible to mechanical damage either in rough handling during installation or in operation under unfavorable conditions such as large loads and a poor lubricant. From this point of view, once the valves are broken in and functioning satisfactorily, it is very desirable to leave them undisturbed.

One of the major operating problems during this program was poor operation of the valves, as evidenced by audible noise and "chatter" that could be detected on the XYY' plots, particularly at low valve speeds. This may be attributed partly to the valves themselves and partly to the drive system. Chatter and stick-slip phenomena, whenever they occurred, were always more severe on the push stroke than on the pull stroke. Chatter can be reduced by tightening up on the drive system, but it cannot be eliminated from a given set of valves once it has appeared. We believe that the key to avoiding this problem is proper break-in of the valves and restricting the operations to relatively low loads.

End-play in the drive system has caused much concern. The play develops in the ball-joint connectors for the load rings. As discussed previously, play in the ball joints affects the motion curve of the valves and does introduce an uncontrollable factor into the start of valve motion. The greater the play in the ball joints, the greater the speed of force application and valve startup after the slack is taken up; it is logical to believe that such variations will affect the breakaway force. This is a design deficiency for which we have not found any solution, other than replacing the ball joints when the play is judged excessive.

Side-play develops in the drive system because of wear of the drive-shaft bushing. The rate and severity of such wear will depend on the side loads imposed by the drive system and by the two valves. The drive system is not standardized and is different in each laboratory. No problems of this nature have been reported by Bendix or General Electric. The excessive wear that we have encountered may reflect the nature of our particular drive system, or it may simply reflect the rather large amount of test operation that this rig has undergone. In any case, when the

drive-shaft bushing became worn, the entire drive-shaft assembly developed a sidewise wobble so severe as to be quite apparent to the eye when the rig was operating. The geometrical relationships of the drive-shaft assembly can be visualized by reference to Figure 8. The drive shaft is centered by means of the bushing and by a centering pin, mounted on the housing block, which extends into a hole in the end of the drive shaft. The pin/hole arrangement is grease-lubricated and has not shown excessive wear. In addition to the bushing and centering pin, further control of drive-shaft motion is exercised by a second pin mounted in the housing block and extending through an oval slot in the crossarm. This pin-slot arrangement is likewise grease-lubricated and has not shown excessive wear. The pin/slot pair restricts the drive shaft and crossarm from rotation around its own axis. With this combination of restrictions (bushing, centering pin, and pin/slot guide), only the bushing and centering pin are restraining the drive shaft from wobbling either sidewise or up-and-down. Since off-center loads from the drive crank are up-and-down, it would be thought that wear and play would be in this direction. Actually, the worn bushing permitted mainly sidewise wobble, probably because of the unbalanced loads from the two valves. The bushing supplied with the simulator became very badly worn and was replaced with a bushing of the same dimensions, made of oil-impregnated bronze. This has functioned fairly well, but nevertheless has developed some side play. It would be desirable to redesign the drive-shaft restraint system for more positive alignment, possibly by adding a yoke and outboard bushing.

With the simulator drive arrangement as it now exists, the two valves do affect each other's operation. When the direction of motion is reversed, one valve will always break away before the other; when it breaks, the force on the other valve suddenly increases and then decreases as the slack in the first valve is taken up. This behavior can be seen quite clearly by watching the two pens as they trace force vs. position for the two valves. It is not clear whether the breakaway force recorded for the second valve will be affected by the breakaway of the first valve, but it seems reasonable to expect that some effect would exist. Such interactions are aggravated, of course, by any sidewise play in the drive shaft, and could be minimized by providing restraints to keep the drive shaft in better alignment. However, in retrospect, it would have been simpler to have started with a single-valve design, possibly with dead weight loading.

As mentioned previously, the load spring supplied with the simulator was designed for loads up to 50 lb, hence is rather inaccurate at the 5-lb load adopted for most of the work performed here. A lighter spring has been obtained and is being used at present. Consideration has also been given to dead-weight loading, but this would be difficult to accomplish without changing the present configuration.

Some of the cylinder/valve sets that have been used in the simulator show very definite irregularities: i.e., there will be a "hump" in the force trace at one specific location. In some cases, such an irregularity has persisted over many hundreds of operating cycles; in other cases, an irregularity will disappear or shift position after a few cycles of operation. These phenomena may be related to chips of the hard-coating material that are broken loose either in installation or during operation under severe conditions.

The only clear indications of valve-land wear are found on the innermost edges of the inner two lands, i.e., those nearest to the side-load point. No wear can be detected on the other 14 lands. This is evidence that the valve is bending appreciably under the influence of the side load. This is not too surprising, since the diameter of the aluminum valve stem and the inter-land grooves is only about 0.44 inch. Approximate calculations have indicated that deflections with 10 to 20-lb side loads would exceed the valve/cylinder clearances (were the valve not restrained). These considerations add weight to the arguments in favor of operating at side loads as low as possible and loading at the center position rather than at an outboard position.

From a practical viewpoint, it is undesirable to reduce the side load to around 2 lb, even though this would minimize valve damage and unequal loading of the lands. At such a low applied load, the self-binding forces are large in relation to the applied load. For example, with a 2-lb applied side load and a breakaway force of 0.4 lb, the apparent coefficient of friction would be 0.2. However, with essentially no applied load (with the spring in place but tightened only enough to position the valves), the breakaway force may typically be around 0.15 lb. With the spring entirely loosened, the breakaway force will generally be at least this high. This is 38% of the total breakaway load (0.4 lb) at 2 lb side load—a major factor affecting the apparent coefficient of friction. It would be desirable to apply some sort of correction. However, it is unrealistic to subtract a "zero-load" value as a correction, since valve geometry will change when the load is applied.

Valve-stem bending may also be a partial explanation of why the major portion of the fuel flow goes inward, toward the center-load point. This question has not been resolved, and further work is planned.

9. FUTURE WORK

It is planned to modify the fuel supply system to make each of the two fuel supply lines entirely separate and independent, joining only at the entrance to the simulator. Once this has been done, criteria will be established for flushing procedures when changing test fuels. Specifically, it will be determined how much flushing and subsequent running time are needed to restore the coefficient of friction to the "clean isooctane" level after the system has been contaminated with a polar additive. In the event that the requirements for flushing and operating prove to be excessive, use of an intermediate flushing solvent will be evaluated.

Along with this, it is planned to standardize procedures for valve installation and break in, standardize the operating schedules in relation to when the traces are recorded, and continue to accumulate data on isooctane to define repeatability and valve-to-valve reproducibility, both short-term and long-term.

As soon as possible, evaluation of presently qualified corrosion inhibitors and other "lubricity" additives will be started. The choice of test solvent offers some problems here, since some of the presently qualified corrosion inhibitors have limited solubility in isooctane. In terms of actual performance in the field, a "real" fuel would be desirable as a base stock, but the well-known difficulties with maintaining a standard fuel for any test will apply with even greater force to lubricity work. One possible choice as a reference fluid would be a blend of a highly refined distillate and an aromatic hydrocarbon, such as the Bayol/toluene blend used as a standard fluid in the water separator. In this connection, it will be desirable to investigate the effect of treating the reference fluids to remove traces of polar impurities.

It should be noted here that, in relation to the overall fuel lubricity problem, the lubricity simulator was designed to investigate just one aspect—the effect of fuel characteristics on valve sticking or similar problems. There is no reason to believe that the simulator will give any information that is of value in predicting, for example, pump wear rates. Numerous other test devices have been developed and evaluated for prediction of wear rates, and there is now considerable interest in use of these devices to evaluate fuels. In the current program, there are no plans to broaden the investigation into this area unless critical field problems develop in the near future.

SECTION IV

FUEL CORROSION INHIBITORS

1. BACKGROUND

Corrosion inhibitors used in military fuels are currently covered by Specification MIL-I-25017B, issued in 1962. For some time, it has been desired to issue a revised specification and a new Qualified Products List (QPL), in view of the many new products that have come on the market, the rather ancient status of some of the original qualifications, and certain inconsistencies and questions as to test methods used in MIL-I-25017B. Work in this direction was hampered by problems with the rusting test that is used to define the "relative effective concentration" (REC), which is in effect the minimum permissible blending concentration.

The Air Force had issued a proposed specification, MIL-I-25017C draft dated January 1968. This proposed specification included a rusting test very similar to that specified in MIL-I-25017B, except that the test period of 20 hours in MIL-I-25017B was shortened to 5 hours in the 1968 proposed MIL-I-25017C. At the time this problem was assigned to SwRI for investigation, in 1969, it had become evident that there were serious problems with the repeatability of the rusting test results, and most of our work to date has been concentrated on developing and defining a complete test procedure that will give good repeatability. Along with this, numerous problems and inconsistencies in other test methods of the specification have been investigated, and new or modified test methods have been proposed.

As reported previously⁽²⁾, substitution of distilled water for synthetic sea water in the rusting test did not solve the problem of poor repeatability. Here we are summarizing the developments and results of the past year and the status of the investigation and the proposed specification.

2. STATUS OF SPECIFICATION DEVELOPMENT

After completing the investigations and testing that are described in the following sections, a number of specification changes were recommended to the Aero Propulsion Laboratory. Test data were obtained in accordance with the proposed new specification on samples of corrosion inhibitors that had been submitted for qualification from 1968 through early 1970. These data, along with a draft of the proposed new specification, were furnished to the respective inhibitor manufacturers for comment.

Meanwhile, it became evident that the long time lapse between submittal of some of the samples and final testing did introduce ambiguity into the results. Accordingly, the Aero Propulsion Laboratory requested a fresh sample of each product from the interested manufacturers. The test program on these samples was started in November 1970 and has been completed since the time of writing this report. These results and the recommended test procedures have been used as the basis for a new specification MIL-I-25017C dated 8 March 1971 and a corresponding Qualified Products List that have been issued by the Air Force.

3. STATUS OF INDIVIDUAL TEST METHODS

a. Relative Effective Concentration (Rusting Test)

After an extensive investigation, which is described in Section IV-4, a rusting test procedure and a corresponding method of establishing the relative effective concentration (REC) were chosen and written up in detail. These are included in the new specification MIL-I-25017C. Briefly, the REC is established by testing a given inhibitor, blended in depolarized isooctane at integral-value concentrations such as 3, 4, 5, 6, and 7 lb/1000 bbl. The lowest concentration giving a passing result is defined as the REC. The test specimen is a standard-size spindle (per ASTM D 665) but is made of SAE-1020 hot-rolled steel. The test specimen is finish-ground with 400-grit abrasive cloth and cleaned with isopropanol before testing. The test blend of inhibitor in isooctane is prepared by direct blending and is used without any preliminary water washing. The test water is a synthetic, medium-hard water. The

test specimen is immersed for 5 hours at 100°F in a stirred mixture of the test blend and test water. After test, the specimen is examined without magnification. Only the center section of the specimen is rated, and failure of a given test is defined as rusting of this section to the extent of six or more rust spots of any size or any rust spot 1 mm in diameter or larger. Duplicate tests are run on each inhibitor at each concentration; in case of disagreement (one pass, one fail), two additional tests are run, and both must pass if the inhibitor is to be considered passing at this concentration.

This rusting test procedure was chosen because of its advantages in repeatability of results in comparison with the other procedures investigated.

Test results by this procedure are summarized in Table 20 for inhibitors that had been submitted for qualification. It should be emphasized that these are *not* QPL test results, as later samples have been submitted on all inhibitors.

TABLE 20. RUSTING TEST RESULTS BY RECOMMENDED SPECIFICATION PROCEDURE*

Inhibitor	Concentration, lb/1000 bbl															
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
A			1P 1F	<u>4P</u> 2F	1P											
B			1P 3F	<u>4P</u> 2P	2P	1P										
C			1P 3F	<u>4P</u> 2F	<u>5P</u> 2F	1P										
D		4P 3F	<u>6P</u> 3F	2P	1P	1P	1P	1P	1P							
E					1F					3F	<u>6P</u> <u>1F</u>	3F	1P		1P	
F		2F	2F	<u>4P</u>		1P										
G		2F	<u>4P</u>	2P	1P	1P										
H		2F	2P 5F	<u>5P</u>	2P	1P	1P									

*Procedure recommended for June 1970 draft of Proposed MIL-C-25017C. Entries in table indicate number of individual passing (P) and failing (F) tests. Underlined values define the REC.

It will be noted that, in all cases, a clear-cut REC could be established from these data. For several of the inhibitors, more tests were run at the REC than the maximum of four required by the specification procedure. This was done to make sure there were no ambiguous cases in which further testing might have cast doubt on the REC value.

b. Maximum Allowable Concentration

Determination of the maximum allowable concentration (MAC) involves the REC, the ash content of the inhibitor, and separometer results on blends of the inhibitor. The recommended method of determining MAC as specified in the new MIL-I-25017C is basically the same as in older specifications, differing only in that some ambiguities have been eliminated.

Based on the most recent data available on eight inhibitors, the separometer results (WSIM values) were one of the limiting factors in establishing the MAC for six of the eight inhibitors. These six cases of WSIM limitation included three that were also limited by the "4 X REC" level and one that was also limited by the 20 lb/1000 bbl maximum. Thus, in two cases out of eight, the WSIM was the sole limiting factor determining the MAC. Ash content has not been a limiting factor in any of the inhibitors tested.

c. Water Separometer Index Modified (WSIM)

As already indicated, the WSIM enters into the determination of maximum allowable concentration, at the time of product qualification. Under the old specifications, that was the end of it, since acceptance tests did not require determination of maximum allowable concentration, hence did not require WSIM values. We do not feel that this is a desirable situation, since even fairly minor and inadvertent changes in inhibitor composition as it is produced might change the effects on fuel/water separation quite drastically. Even though the WSIM values are known to suffer from poor repeatability and doubtful value in prediction of behavior of fuels in the field, they are at least a safeguard against the use of some completely unsuitable additive or fuel component.

Based on this reasoning, we have recommended that acceptance tests for corrosion inhibitor lots should include WSIM tests at the maximum allowable concentration. This will apply to Government procurements only, since commercial purchases of MIL-I-25017 inhibitor are not subject to acceptance tests. It is recognized that inclusion of a WSIM requirement in lot acceptance tests may cause difficulty. For six inhibitors that have been tested, the WSIM was a limiting factor in establishing the maximum allowable concentration; hence, test-to-test variations could result in a failing WSIM test in lot acceptance, even though the inhibitor lot were absolutely identical with the qualification sample.

In using the WSIM values to establish the maximum allowable concentration, three tests have been run at each concentration, and the results averaged. In accordance with the recommended specification, the inhibitors were tested for WSIM at 2 lb/1000 bbl concentration increments from 6 lb/1000 bbl upward, the range of present interest in establishing maximum allowable concentration.

Whether or not the 70-WSIM limit is feasible in lot acceptance tests can be determined only after procurements have been made on this basis.

d. Ash Content

The ash methods used, recommended, and specified for corrosion inhibitors have gone through a number of changes. The basic difficulty occurs with phosphorus-containing inhibitors, which cannot be ashed completely or reproducibly in Vycor or porcelain dishes. Such additives can be ashed accurately in platinum dishes but tend to etch or embrittle the dishes. Dilution of the inhibitor with an ashless white oil or refined distillate has been suggested as a means of minimizing attack on platinum.

Since the inhibitors under consideration do include a number with high phosphorus contents, we encountered several instances of very poor repeatability when using the Vycor dishes specified in the 1968 draft of the revised specification. It was found necessary to specify platinum dishes to obtain satisfactory repeatability. Our experience has not shown any really severe problems in corrosion of the platinum dishes.

Using platinum dishes, all of the inhibitors that have been tested gave ash contents of 0.01% or less. For these low-ash materials, the recommended limit for lot acceptance tests is 0.04% maximum, to allow for normal scatter of results plus manufacturing tolerance.

e Identification Tests

In previous specifications, physical and chemical property ranges were left entirely to the supplier, so far as can be determined. This resulted in a rather jumbled situation, in which each product listed on the QPL carried its own set of tests and property limits, hardly any two of which were comparable. Some of the property ranges cited by manufacturers and listed on the QPL were so wide as to be of no value whatever for either identification or quality control. Also, test methods were widely divergent and in many cases unspecified.

In an effort to alleviate this situation, we have spelled out certain recommended properties and corresponding test methods for use in lot acceptance and for identification. Actual ranges of properties will be established at the time of qualification by agreement between the manufacturer and the Air Force. Tentative limits have been drafted for each product and transmitted to the respective manufacturers for comment. These limits are based uniformly on the following criteria:

Specific gravity	±0.010
Viscosity, cs/100	±20%
Flash point	±15°F
pH	±1.0
Neut. no.	±5% with additional allowance for ASTM reproducibility limits

Each range selected will necessarily include the value actually obtained on the qualification sample. However, the qualification value will not necessarily be the midpoint of the range selected, since manufacturing experience will dictate the most practical limits.

4. STEPS IN DEVELOPMENT OF RUSTING TEST

Development of the rusting test into the presently recommended form went through five more-or-less distinct steps and resulted in five distinct test procedures, these we have identified, with no great originality, as Methods I, II, III, IV, and V. They are all 5-hour tests, differing in preliminary water extraction of test blend, specimen finish, specimen cleanup method, and composition of the test water. The test procedures and the results obtained with each are summarized in Table 21.

Method I, used in the initial work by SwRI, is essentially the same as the 1968 method except that the test water has been changed from sea water to distilled water. The results shown in Table 21 for Method I are a summary of those reported previously⁽²⁾, with some updating. As noted a year ago in the referenced report, there was often no sharp transition from passing tests to failing tests as the concentration of a given inhibitor was decreased, and definition of the REC was further confounded by occasional "passes" at concentrations far below the nominal REC.

Method II was derived from Method I by eliminating the pretest extraction step, in which the inhibitor/iso-octane test blend is washed with distilled water, separated, and then used in the rusting test. Such an extraction procedure has been a part of the MIL-I-25017 procedure for a number of years. It serves a valid purpose, to prevent the qualification of materials that are preferentially water-soluble. Nevertheless, as shown by later results, this water extraction step can actually *impure* some inhibitors to an extent that probably would not be realized in actual service. Thus, this question of the need and desirability of a preliminary water extraction is quite complex and cannot be resolved by laboratory work alone.

For the purposes of this program, the primary aim has been improvement of test repeatability. The water extraction step is an added operation that is not well defined in existing test procedures and can introduce uncontrolled inhibitor depletion in any case. In general, one would expect to improve the test repeatability by

TABLE 21. COMPARISON OF RUSTING TEST PROCEDURES

Method modification:	I	II	III	IV	V
Blending method:	Conc	Conc	Conc	Direct	Direct
Blend water-extracted:	Yes	No	Yes	No	No
Specimen finish (grit):	240	240	400	400	400
Wiped with isopropanol:	No	No	No	Yes	Yes
Test water used:	Distilled	Distilled	Distilled	Distilled	Type B*
REC, lb/1000 bbl:					
Inhibitor A	4.5	<2.5†	>8	4†	4
B	3.0		2.0	>10	3
C	4.5		6.5	4†	5
D	18-20	8-10	10-12‡	5	3
E	>20	20†	20†		11
F	3.5		5.0	5†	4
G	2.5		4.0	2†	2
H	3.0	2.0†	1.5	>9	3
*Type B synthetic, medium-hard water is distilled water with the following added salts: NaHCO ₃ , 164 mg/liter; CaCl ₂ · 2H ₂ O, 132 mg/liter; MgSO ₄ · 7H ₂ O, 82 mg/liter. †Tentative; based on only one or two tests. ‡For >50% passes; other inhibitor REC's by Method III are based on >70% passes.					

eliminating the water extraction step. However, this change by itself (from Method I to Method II) did not improve the situation for the one inhibitor that was investigated thoroughly; there was still a marked scatter of pass-fail results on Inhibitor D, and it was still impossible to assign an unambiguous REC to this inhibitor.

Method III differs from Method I only in the specimen finish, which is 400-grit instead of the normal 240-grit. Here again, repeatability for Inhibitor D was not improved by this change, and poor repeatability was encountered for other inhibitors as well. If anything, Method III appeared to be the least precise of the three procedures.

Although neither the finer polish nor elimination of the water extraction did any visible good, the presumption still exists that these changes are in the right direction. The finer surface seems to be easier to reproduce, is far easier to clean properly before tests, and is far easier to rate after test. The water extraction step, apart from its broader merits or demerits, is a source of manipulative error, so that one should expect it to have an adverse effect on test repeatability.

Further steps were taken toward eliminating manipulative errors by direct-blending the test solutions rather than using intermediate concentrates or stock solutions. This should eliminate any uncertainty in final test-bler 1 concentrations caused by partial additive insolubility or by surface adsorption losses of inhibitor in the transfer and blending steps. With the direct-injection blending method, the blend is prepared by adding isooctane and the inhibitor to the test beaker itself.

Finally, a specimen cleanup was introduced after the final polishing operation. This cleanup consists of rinsing with isopropanol and wiping dry. Specimen cleanup is discussed further in Section IV-5.

Method IV, then, incorporated all of the changes considered desirable: Inhibitors were direct blended, water extraction was eliminated, and specimens were fine-polished and cleaned. The repeatability of this method was never really established, since two inhibitors (B and H) very unexpectedly failed to protect at concentrations of 9 to

10 lb/1000 bbl or even higher. These two inhibitors had been consistently among the better performers in all earlier test work, and at least one of them has been used in pipeline protection for some time.

It was found that the failures on Inhibitor H were specific to the use of distilled water in the rusting test (Inhibitor B was not investigated fully). Under these same general test conditions, Inhibitor H was found to perform very satisfactorily with synthetic sea water (regular or diluted), with blends of water and fuel system icing inhibitor, or with a synthetic, medium-hard water originally defined for use in filter-separator testing.⁽¹³⁾ Further, the performance of Inhibitor H with distilled water as the test water could be brought back to its "normal" level by preliminary water extraction of the test blend. All of these facts pointed to the presence of some corrosive, water-soluble constituent in this inhibitor, conceivably one that had developed in the sample of inhibitor during the two years it had been on hand. The presence of a water-soluble acidic constituent was confirmed by extracting concentrates of the inhibitor, yielding extracts with pH in the neighborhood of 5.

It was concluded tentatively that the lack of any buffering capacity when using distilled water as the test water was responsible for the failures of these two inhibitors. Assuming that these samples contained traces of inorganic acid, the use of distilled water could give a test medium of a type that would not be encountered in normal service. Even when the water present in service is condensate derived either from the atmosphere or from fuel-dissolved water, it will be contaminated by contact with metal surfaces, rust, and the fuel itself, and will be far from "pure" in comparison with laboratory-grade distilled water. Although no one test water can be chosen as the most "realistic" in terms of widely divergent service conditions, distilled water is probably near the bottom of the list.

Method V, which uses synthetic, medium-hard water, is the method finally recommended for specification use. In determining REC's by Method V for all eight inhibitors in this program, no anomalies or reversals were noted. The repeatability of the test results can be judged by referring to Table 20. Clear-cut REC values were established for all inhibitors with a minimum number of tests. Extra tests were run in most cases to try to find reversals, but none were found.

In comparing the results obtained by the different test methods, it will be noted that the changes in test procedure, although they might appear relatively minor, often gave drastic reversals in the order of ranking the inhibitors. For example, compare Inhibitors B and D in Methods I and IV:

	<u>B</u>	<u>D</u>
REC, Method I	3.0	18-20
REC, Method IV	>10	5

It happened that both of these inhibitors have seen considerable pipeline service. Test failures at concentrations above 10 lb/1000 bbl are difficult to reconcile with reported performance. In the case of Inhibitor B, as previously noted, this poor performance was specific for tests with distilled water, without preliminary water extraction of the test blend. No such specific condition could be ascribed to the poor results on Inhibitor D by Method I.

In the absence of any reliable and comprehensive field data, it is not really possible to say that the lineup of inhibitors by one test method is any more "correct" than it is by another. The final choice of method was made mainly on the basis of repeatability.

In view of the many reversals of inhibitor rankings, one can argue that no one test method can give any valid correlation with field performance in general. This is almost a truism, but it is not easy to work out a practical series of tests that would cover even the most important variations in field conditions. For example, such a series of tests would surely have to include test waters varying in pH, chloride content, and possibly contents of other ions and of fuel system icing inhibitor, as well as tests with and without a preliminary water extraction. Other variations would soon bring the test sequence beyond the bounds of reason for routine use. Nevertheless, it should be kept in mind that use of a single test does have definite limitations.

5. INVESTIGATION OF SPECIFIC TEST PARAMETERS

a. Blending of Test Solutions

Direct injection of the additive to the final test blend, using micropipets, was adopted to eliminate the variable of insolubility in concentrated stock solutions and possible adsorption of the inhibitor on glass surfaces in the blending and transfer steps. Partial insolubility in isooctane is a definite problem with some inhibitors. In retrospect, we feel that the direct injection is unduly time-consuming and possibly unnecessary; however, it has been retained in the final test version, since extensive testing would be required to prove it unnecessary.

We do feel that any future rusting test method should incorporate a base solvent more closely approximating the solvent properties of jet fuels than does isooctane. Bayol-toluene blends would be one possibility.

b. Isooctane Depolarization

One aspect of the test which has received only cursory examination by us is the isooctane depolarization procedure. We ran one set of tests without depolarizing the isooctane. No significant difference in results was noted between this test and those run using depolarized isooctane.

The real need or lack of need for depolarization could be proven only by testing a large number of different batches of isooctane, some of which could conceivably contain interfering impurities. In any case, inclusion of the depolarization step in future testing is dependent on the solvent chosen for future work and the purity obtainable.

If depolarization of the solvent seems wise, the present depolarization procedure needs to be more closely controlled to be more effective. As presently specified, the procedure probably removes only water and the other very polar impurities from the isooctane. Presently the silica gel is used as received. The activity of column chromatographic adsorbents such as silica gel is very much dependent on moisture content. Adsorbents for column chromatography are usually activated at a high temperature and stored either at an elevated temperature or tightly stoppered to prevent moisture pickup. Also, impurity levels in the silica gel and its mesh size can materially affect its separating properties. Thus, closer attention to these factors is desirable in specifying the depolarization procedure. It would also be desirable to use a more efficient apparatus for the depolarization, e.g., a column rather than the separatory funnel that is used at present.

c. Apparatus Modifications

During the course of this program, several small changes were made in the actual rusting test apparatus specified in ASTM D 665, and additional improvements could be made.

The beaker cover diagram given in D 665 shows a groove having one tapered side. In our work, we found that the dimensions of some beakers varied so that the cover did not lie flat. Since the specimen holder is supported by the cover, the cover being askew also causes the specimen to tilt rather than being perfectly vertical. We, therefore, had the cover groove slightly enlarged and machined with straight edges so that the covers would fit evenly on all beakers.

The ASTM procedure also calls for placing a thermometer in each beaker and allowing it to stay there during the entire test. We placed a thermometer in the last beaker only, and that thermometer was left in place only until the test solution had reached the temperature of the oil bath surrounding the beakers. The thermometer was always removed before stirring was begun, and only the bath temperature was monitored during the entire test. Leaving a thermometer in each beaker throughout the test (as in the standard ASTM procedure) would cause a different dispersion pattern and solution movement in three of the beakers as compared to the other three. The cover configuration has the thermometer hole 90° clockwise from the specimen hole. Since the apparatus is designed so that the stirrers in the alternate positions are belt driven in opposite directions, the thermometer would be expected to cause a different flow pattern, probably with less water droplets impinging on the specimen, in those

positions where the stirrer rotation is counterclockwise. In our later testing, no thermometers were in place after the specimen was inserted in the test blend; a cork was placed in the thermometer hole of each cover during the test.

If the same type of apparatus is to be employed in future rust test development, additional modifications would be warranted. Among these are: (1) inclusion of a stirrer in the oil bath to prevent temperature gradients, (2) replacement of the present stirrer chuck assemblies (preferably by better-designed units) to limit stirrer wobble and expedite more exact stirrer placement, (3) better control of beaker dimensions. Standard beakers vary widely in curvature of the bottom, which affects the paddle position in relation to the water-isooctane interface and hence could affect the water dispersion pattern. We do know from certain experimental results that specimen geometry and placement *are* very critical factors; it seems equally reasonable that beaker geometry and stirrer placement are equally critical. In any beaker-stirrer test of this type, it would appear advisable to use a container having closely controlled dimensions—something that is not feasible with stock-item glass beakers.

d. Specimen Cleanup

Our experience with the rusting test leads us to believe that one of the most important factors in obtaining reproducible results with any of the method variations is the specimen cleanup before test. In all of our early test work, we had followed the Air Force procedure (MIL-I-25017C draft, January 1968) in which the specimen is merely wiped with a dry paper towel after the final polishing.* In some cases, rusted test specimens, when examined under a microscope, showed rust spots that had originated around clumps of particulate matter. This indicated that incomplete removal of "dusts" could well be responsible for erratic test results.

Various specimen cleanup techniques were tried at various times throughout the testing period. One of these was vapor degreasing over toluene, followed by immersion in boiling methanol, and again vapor degreasing over toluene. No positive improvement in results was noted. Other solvents, including Stoddard solvent, isooctane, and methanol were tried both as rinses and as swabs. To facilitate handling the specimens in these procedures, the methacrylate specimen holders were replaced by poly(tetrafluoroethylene), which is much more resistant to heat and solvents. The PTFE holder is allowed but not required in ASTM D 665-60.

The only cleanup which was found to be consistently effective was rinsing the specimen with isopropanol, then thoroughly wiping it with a dry paper tissue. It has been suggested that the polishing step may induce some magnetism in the specimen; this would cause metallic particles to be difficult to dislodge from the specimen surface. This may explain why the present cleanup, using firm pressure during the paper tissue wipe of the specimen, is more effective than vapor degreasing. In this respect, it would be interesting to try demagnetizing the specimens after polish to see if this would aid the cleanup.

The isopropanol seems more effective than less polar solvents such as toluene in the cleanup, but we cannot offer any sound explanation for this. It may be speculated that the isopropanol is more of a "general-purpose" solvent in that it will aid in removing traces of resinous materials left by the abrasive cloth, as well as any minute amounts of perspiration residues that may get onto the specimen despite the extreme precautions that are taken.

The use of isopropanol for the specimen cleanup was started at the same time high relative humidities were encountered in the laboratory (Spring, 1970), and a number of unexpected test failures were observed. It appears that the isopropanol rinse does aggravate such troubles. This should be expected with any volatile solvent and particularly with one that is volatile and water-miscible, since the chances for water condensation on the specimen are increased with this type of solvent. When the steel specimen is in the "clean" state, even very brief contact with water or water-solvent mixture will cause rust initiation. Evidently, some heating of the specimen or solvent is necessary. A practical procedure was worked out, in which the specimen is heated before rinsing and the cleanup is carried out under a heat lamp.

*ASTM D 665-60 specifies light wiping with cloth or tissue, or brushing with a camel-hair brush. In older versions of the test, simply tapping the specimen on a hard surface was considered adequate, and this technique apparently is being retained as an acceptable alternate in the current NACE version of the test.

e. Specimen Finishing

The very fine finish (400 grit) that has been recommended does offer advantages in ease of plating after test. Probably more important, the smoother surface is much easier to clean up before test and to tell when it is clean. This is true whether the specimen is dry-wiped, rapped, solvent-wiped, or vapor-degreased.

A much coarser specimen finish (100 grit) is used in the test currently recommended by a NACE committee for fuels and fuel-soluble corrosion inhibitors.

The 400-grit finish recommended for the MIL-I-25017 test was introduced along with a number of other changes, and there is no definite proof that the finer finish per se does improve the repeatability of results.

6. FUTURE PROGRAMS

a. Storage Stability of Inhibitors

Stability of corrosion inhibitors in prolonged storage has been of some concern to the Air Force, since drummed inhibitor may be purchased for use in Air Force pipelines in quantities for a year or more of operation; hence, the total storage period of the inhibitor, from manufacture to use, may be as much as several years. Existing corrosion inhibitor specifications do not have any storage stability requirement or method of evaluation.

In the test program described here, most of the inhibitors were received in 1968 and 1969, and testing was not completed until 1970. Meanwhile, the inhibitors had been stored at room temperature. Visibly, some of them had changed by settling out of solid material, sometimes very difficult to redisperse. Other possible changes during storage are loss of solvent and chemical changes such as oxidation and hydrolysis. One example of possible change in storage was shown by Inhibitor H. As discussed previously, eliminating the water extraction step when going from Method III to Method IV had a very harmful effect on this inhibitor's performance. Some five months earlier, eliminating the water extraction step when going from Method I to Method II had *not* harmed this material's performance. Traces of a fairly acidic material were found in the aged sample of inhibitor, and it can be theorized that the product had deteriorated in storage. This theory is supported by chemical analyses, the results of which cannot be reported here because of proprietary considerations.

In view of possible deterioration of inhibitor samples, fresh samples have been obtained by the Air Force for qualification testing.

It is planned to check the stability of each corrosion inhibitor by a storage test in which samples will be held under outdoor-ambient conditions for 6-12 months, then checked for REC level and other physical and chemical properties.

b. Effects of Corrosion Inhibitors on Fuel Thermal Stability

In the existing corrosion inhibitor specification, there is no evaluation of effects on fuel thermal stability. This is controlled indirectly by the thermal stability requirement in the specification for JP-4 fuel. Any corrosion inhibitor that was extremely bad in this respect would not find much of a market for use in JP-4 fuel. Generally one will expect the qualified corrosion inhibitors to improve the filter-plugging behavior of fuels if there is any effect at all. Corrosion inhibitors may have adverse effects on preheater deposits in the thermal stability test, but this effect does not often show up at the relatively mild test conditions used testing JP-4.

In view of the numerous new products offered as corrosion inhibitors, it appears desirable to obtain at least minimum information on thermal stability effect. It is planned to test each corrosion inhibitor at the maximum allowable concentration in some selected JP-4 fuel, probably at the normal specification test conditions of 300-400°F. Ideally, one would select a JP-4 fuel having a preheater breakpoint around 325°F, so as to give a more

critical test of the additive's effect. It is not often easy to locate such a fuel when it is wanted. Actually, if a more elaborate evaluation of effects on thermal stability were wanted, it would be necessary to use base fuels having greater stability and lower volatility than JP-4.

c. Improvement of Rusting Test

Unless a completely new rusting test is developed, certain improvements are needed in the present test. One of the primary needs is for a better method of rating the test specimens; this is one of the weakest points of the present test method. Lighting conditions and the visual acuity of the observer are critical. Closer standardization of the lighting conditions will be relatively simple, and it is planned to develop some recommendations along this line. Standardization of the observer is much more difficult. It may be more feasible to consider microscopic scanning of the specimens (at relatively low power) to identify corrosion spots, pits, and stains, and to develop rating criteria on this basis. Some work along this line is planned, on a low-priority basis.

The present system of pass-fail ratings does not give maximum information on inhibitor performance, although it may be adequate for specification purposes. A graded rating system for test spindles has been developed by NACE Committee T-3P, as follows:

Rating	Test surface rusted
A	None
B++	Less than 0.1% (equivalent to 2 or 3 spots, not larger than 1 mm diameter)
B+	Less than 5%
B	5-25%
C	25-50%
D	50-75%
E	75-100%

In MIL-I-25017 testing, some sort of rust severity rating would be useful in establishing REC's in case of doubt. Such a rating would also be useful in studying the effects of test conditions on severity level and repeatability of ratings, as well as in product development. Some work is planned in developing a rust-severity rating that will distinguish between the borderline "failures" often observed and the clear cases of rusting, in other words, a rating scale with several gradations at the trace-rust level. Again, this is not any easy problem, no visual-rating system is possible without subjective interpretation.

Perhaps a better line of approach will be an adjustment of test conditions such that rusting will become more apparent. Many of the steps that have been taken in the prior development in the interest of repeatability have tended to make the rusting less severe. Quite possibly a return to a more corrosive test medium would simplify the rating problems and might be accomplished without doing violence to the precision of results.

d. Investigation of New Types of Functional Tests

In view of the wide use and acceptance of the spindle test for corrosion inhibitors, there is little pressure to investigate completely new functional tests. Such tests might be different types of rusting tests, or they might be derived from measurements of electrochemical behavior. An example of the latter is the techniques developed by Nestor⁽¹⁴⁾, measuring polarization currents to determine protection.

Investigation of any new technique is hampered by the lack of any readily available, reliable field data on comparative performance of different corrosion inhibitors. The lack of such data reflects partly the virtual impossibility of obtaining accurate, side-by-side comparisons in large-scale operating situations and partly the restrictions imposed by proprietary considerations. Nevertheless, some attempt will be made to accumulate and evaluate such data as may be made available.

No large-scale efforts of any kind are planned in the area of performance tests for corrosion inhibitors. It should be kept in mind that across-the-board, mandatory use of corrosion inhibitors in Air Force jet fuel is currently justified by lubricity problems rather than any widespread rusting problems. If it were not for the fuel lubricity problems, the use of corrosion inhibitors could be restricted to fuels moving by pipeline. Since the emphasis is on lubricity in the current program, any studies of functional tests for corrosion prevention will necessarily be quite limited.

SECTION V

FUEL AND LUBRICANT ANALYSIS AND TESTING

1. GENERAL

In the area of fuel and lubricant analysis and testing, major efforts have been devoted to continuing improvements in the gas chromatographic analysis of lubricants and to a variety of short-term projects evaluating the effects of fuel additives and methods of fuel analysis and inspection. Analytical support services have been provided for other activities within the contractual effort and in-house activities of the Propulsion Laboratory. Only the analytical and developmental work of independent interest is reported here.

2. LUBRICANT ANALYSIS

The standardized operating parameters for gas chromatographic "fingerprinting" of synthetic lubricants were reported in last year's Technical Report.⁽²⁾

The use of OV-17 as the liquid phase for "fingerprinting" has been continued; among those liquid phases having the highest temperature capabilities, the OV-17 produces the best overall resolution of components among a variety of synthetic lubricants. Our files contain many chromatograms of numerous types of oils that were obtained using this liquid phase. This experience and background are of great help in identification work. The higher temperature capability of certain newly marketed liquid phases (discussed later in this section) may be of considerable value in future work, but we have not yet used such liquid phases to any great extent for ordinary identification work.

Various used lubricants, both from operating aircraft and from engine tests, have been chromatographed. The used oils from operating aircraft have been examined primarily to estimate the relative proportions of the five types of MIL-L-7808G oils presently qualified. A typical chromatogram of such an unknown oil mixture is shown in Figure 13. Estimates of the mixture composition have been based on comparison of the peak heights of individual late-eluting components of each of the two or more lubricants in the mixture. By relating these to known mixtures of similar composition, Figure 14, we believe that estimates of the mixtures' composition accurate to $\pm 10\%$ (absolute) can be achieved. More exact quantification of such mixtures in used lubricants would be difficult; the more volatile components of each type of lubricant are depleted in service, as illustrated by comparison of Figures 13 and 14, whereas the standard mixtures used for comparison are necessarily unused lubricants. This difference introduces an initial error of varying magnitude. The present accuracy has been acceptable for solving most field identification problems.

Development work in the area of lubricant analysis has been concentrated primarily in the areas of achieving better resolution and investigating new column materials.

Diisooctyl adipate (DIOA) is listed by Gunderson⁽¹⁵⁾ as being one component of synthetic lubricants and is used in MIL-L-7808 type oils. Technical grade samples of DIOA chromatographed using our standardized operating parameters with OV-17 columns produced a broad multicomponent peak having five partially resolved components, as illustrated in Figure 15. This was compared with a sample of pure DIOA* obtained from Applied Science Laboratories, Inc. This "pure DIOA" gives only one sharp peak under the same conditions, as shown in Figure 16. This rules out the possibility that degradation of the technical DIOA during chromatography was responsible for the multicomponent peak.

Several liquid phases were tested to determine whether better resolution of the components of the technical DIOA could be achieved. It was found that Apiezon L gave the best resolution of the liquid

*This particular material had been obtained several years ago for use as a liquid phase in chromatography. The structure of the principal ester in this sample has not been identified. The term "diisooctyl adipate" is sometimes applied loosely to the 2-ethylhexyl ester.

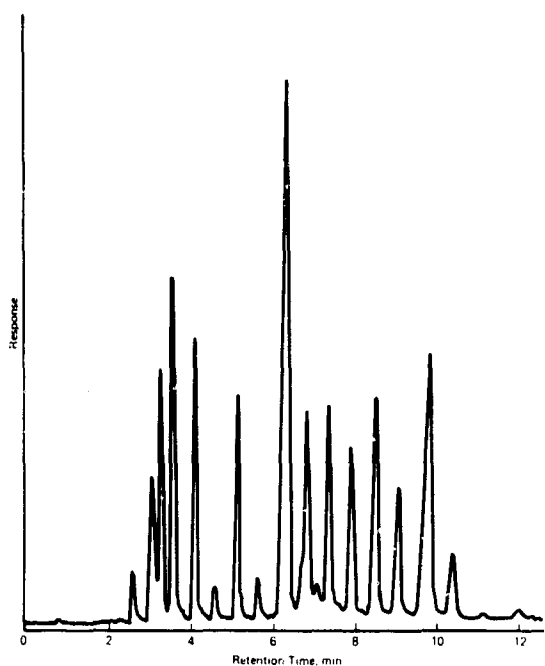


FIGURE 13. USED MIXTURE OF SYNTHETIC LUBRICANTS

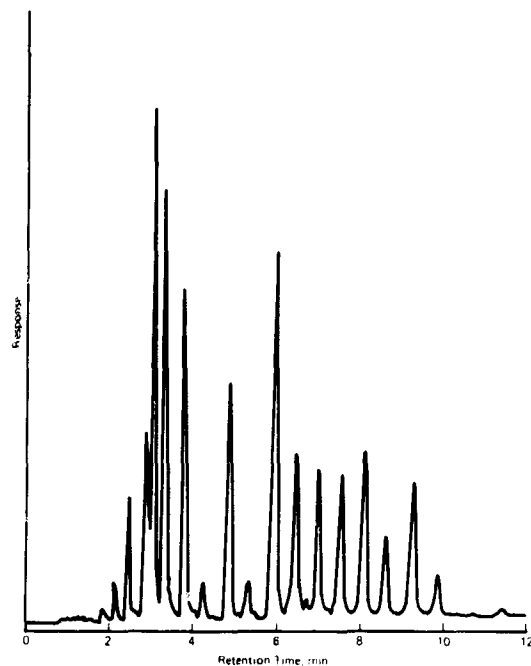


FIGURE 14. KNOWN MIXTURE OF UNUSED SYNTHETIC LUBRICANTS

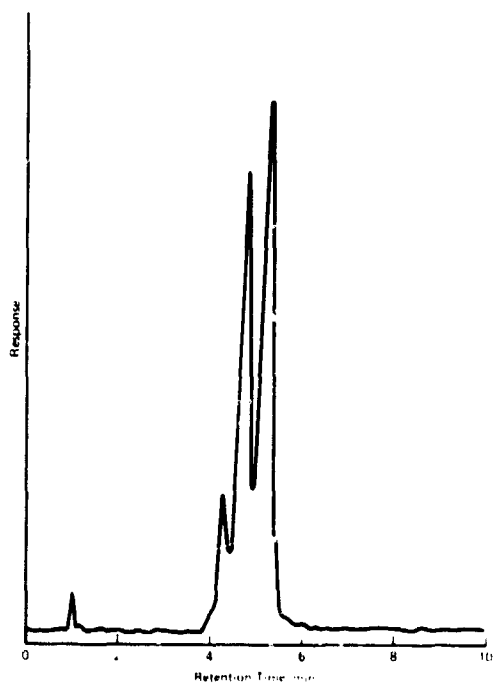


FIGURE 15. TECHNICAL DIISOOCTYL ADIPATE CHROMATOGRAPHED ON OV-17 AT 240-320°C, 10°/MIN

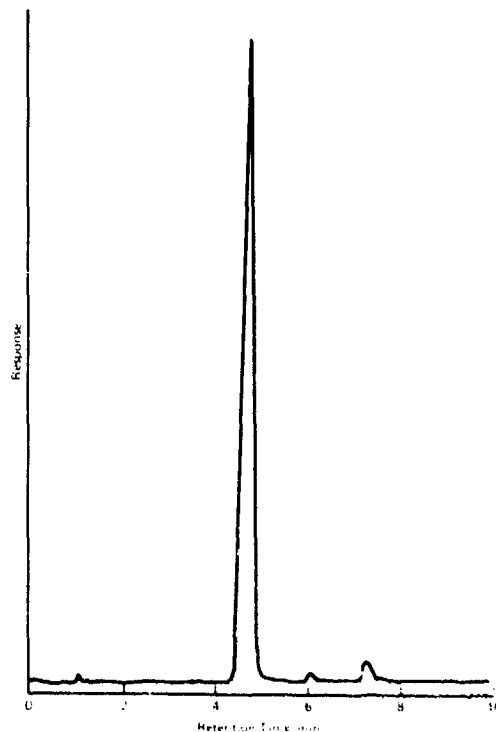


FIGURE 16. PURIFIED DIISOOCTYL ADIPATE CHROMATOGRAPHED ON OV-17 AT 240-320°C, 10°/MIN

phases tested. Other liquid phases giving better resolution than OV-17 for columns of comparable length were OV-225, Versamid 900, and JXR. Poorer resolution was given by OV-210, DC-560, SP-400, SE-30, and OV-1.

Better resolution only served to indicate the true complexity of the technical DIOA. The best resolution (Figure 17) indicated that at least eight components were present. Gunderson⁽¹⁵⁾ states that the common process

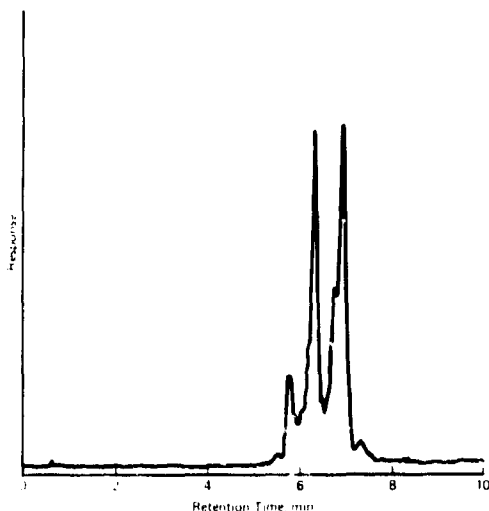


FIGURE 17. TECHNICAL DIISOCTYL ADIPATE CHROMATOGRAPHED ON APIEZON L AT 240-320°C, 10³/MIN

for producing a typical isooctyl alcohol results in the following isomer distribution: (a) 20% 3,4-dimethyl-1-hexanol, (b) 30% 3,5-dimethyl-1-hexanol, (c) 30% 4,5-dimethyl-1-hexanol, (d) 15% 3-methyl-1-heptanol and 5-methyl-1-heptanol, and (e) 5% unidentified. Esterification of the five identified isomeric alcohols with adipic acid could lead to 15 isomers of diisooctyl adipate, using all possible binary combinations in the diester molecule. The unidentified 5% (e) may well contain additional isomers of isooctyl alcohol, leading to still more isomeric diesters.

Work was suspended on attempting further resolution of the individual isomers using packed columns. Complete separation of all isomers of diisooctyl adipate would require a considerable expansion of our present capabilities, and there appears to be little practical value in attempting the complete separation, so far as the present goals are concerned.

Aside from the investigation just described, new liquid phases have been evaluated as they became commercially available.

The first of these new liquid phases to be investigated during the past year was SP-400. It has temperature capabilities equivalent to those of OV-17; values of 300°C are conservatively listed, but it has reportedly been used up to 375°C. Since the SP-400 is more polar, it has superior resolution for certain synthetic lubricant components. Although it offers no clear advantage over OV-17 for routine identification work, it furnishes the capability of changing the relative elution order of various components to supplement OV-17 for complex separations.

A new liquid phase, Dexsil 300 GC, was introduced in July 1970 by Analabs, Inc. This liquid phase, a polycarboranesiloxane, is reported to be stable to 500°C. In preliminary investigations of this material, we have prepared packed columns that indicate it may well be as satisfactory as OV-17 for chromatographing MIL-L-7808 type synthetic lubricants. A support-coated open tubular (SCOT) column utilizing the Dexsil 300 GC has been ordered; this should offer

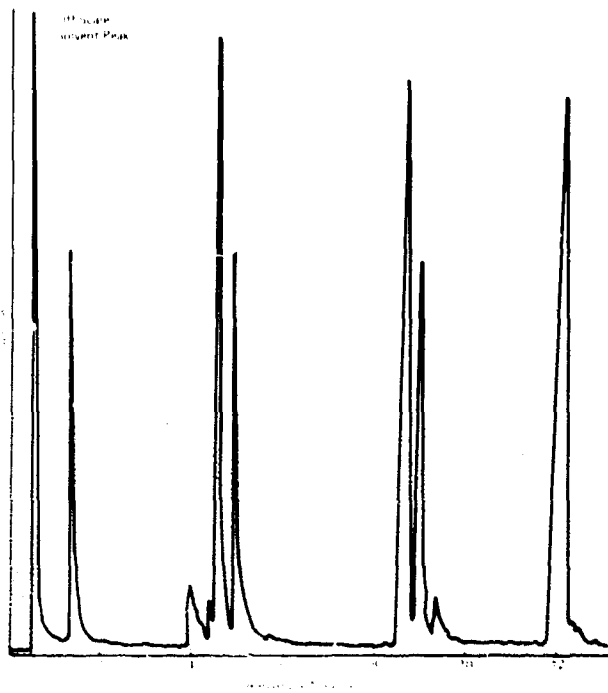


FIGURE 18. CHROMATOGRAPHIC SEPARATION OF THREE-, FOUR-, FIVE-, AND SIX-RING POLYPHENYL ETHERS ON DEXSIL 300 GC

resolution superior to that of packed columns and still offer the capability of operating above 300°C, where OV-17 is rapidly bled off SCOT columns.

We have used the packed Dexsil 300 GC column to separate 3- to 6-ring polyphenyl ethers, as shown in Figure 18. Until this high-temperature liquid phase became available, gas chromatography of polyphenyl ethers was generally considered to be unfruitful.

3. ANALYSIS FOR CONTENT OF FUEL SYSTEM ICING INHIBITOR (FSII)

a. General

Various techniques have been applied in determining FSII contents of jet fuels, all based on water-extracting the FSII from the fuel and then determining the FSII content of the extract. Four such test methods have been examined critically for application to JP-4 fuel:

Freeze point method of the California Oil Company⁽¹⁶⁾

Seiscor differential refractometer method, FS-791b Method 5340.1, with SwRI modifications⁽¹⁷⁾

Colorimetric method with ceric ammonium nitrate, developed by the National Research Council of Canada⁽¹⁸⁾

Iodometric titration, FS-791b Method 5327.3

Standard fuel/FSII blends were prepared for this study. Since the currently used FSII does not contain any glycerol, straight 2-methoxyethanol (ACS reagent grade) was used for this work. Blends containing 0.02 to 0.20% by volume of 2-methoxyethanol (FSII) were prepared, using a nonadditive JP-4 as the base stock. This JP-4, although reported to have no additives, gave appreciable blank values in each of the four methods: 0.017% FSII by iodometric titration, 0.006% by the colorimetric and freeze point methods, and 0.005% by the refractometer method. We suspect that these values were caused not by actual FSII in the base fuel, but by interfering water-soluble components of the fuel. It would have been possible to water-extract the base fuel before use to eliminate these interferences, but it was felt that the use of a "real" JP-4 would give a more realistic indication of the performance of the test methods.

b. Freeze Point Method

In this procedure, an 800-ml fuel sample is extracted with 10 ml of water, and the freezing point of the extract is determined. The FSII content of the fuel is read from a calibration curve. The thermometer used in our freezing point determinations was an ASTM 63F type, range 18-88°F, 0.2°F graduations, 0.2°F maximum scale error. The particular thermometer that was used was found to read 32.2°F when used to determine the freezing point of pure water by the same method used for the extracts. This error was not taken into account in subsequent work, since here we were concerned mainly with scatter of results rather than absolute values. If maximum accuracy were desired, the thermometer error would have to be established over the entire range of interest and applied as a correction. A thermometer error of 0.2°F corresponds to an error in absolute FSII content of about 0.01%.

A series of determinations was performed on the standard fuel/FSII blends to establish a calibration curve, which is shown in Figure 19. This is a straight line, fitted graphically. This calibration curve is in surprisingly good agreement with that given in the original article describing the test method.⁽¹⁶⁾ Deviations between the two curves are not greater than 0.01%, as illustrated by the following FSII contents read from the end-portions of the two curves:

F.P., °F	% FSII from curve	
	Original	SwRI
26.8	0.18	0.177
26.9	0.18	0.171
27.0	0.17	0.168
27.1	0.17	0.164
27.2	0.17	0.161
29.7	0.08	0.077
29.8	0.08	0.074
29.9	0.08	0.070
30.0	0.07	0.066

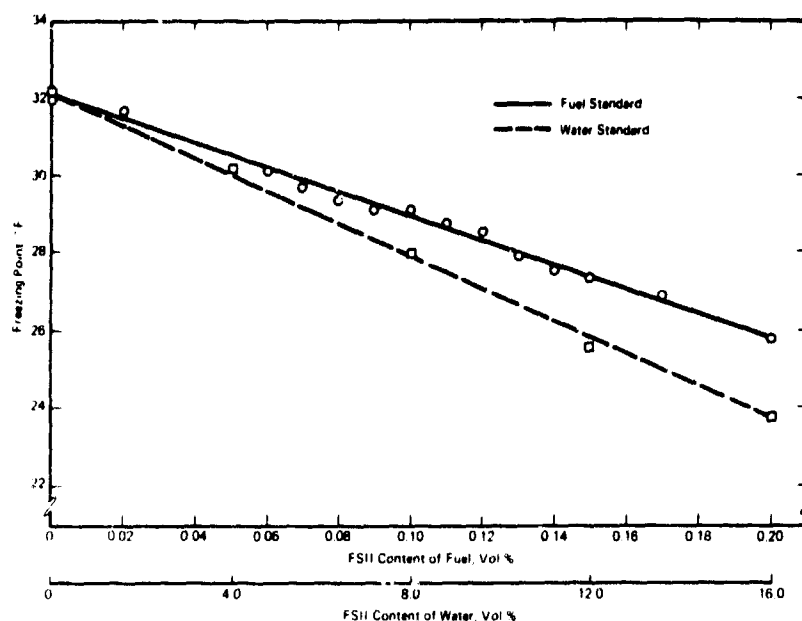


FIGURE 19. FREEZE POINT CALIBRATION CURVES FOR FSII CONTENT

This agreement is extremely good when it is considered that the original curves were established with a different fuel, with an FSII composition containing 2% glycerol, and with a thermometer that may have had different error characteristics than the one used here. The close match of the two curves may be fortuitous.

Deviations of the individual points from the calibration curve are on the order of 0.008% or less in the middle portion of the curve between 0.06 and 0.14% FSII. Individual deviations up to about 0.015% are observed in the end portions of the curve. These deviations give a rough qualitative measure of repeatability.

Because of the limited amount of water used for extraction in the freeze point method, any calibration curve must be based on water extracts from known fuel/FSII samples, rather than on direct water/FSII mixtures. A curve based on water/FSII mixtures is shown in Figure 19 for comparison. The use of such a curve, without any correction for incomplete extraction of FSII from the test samples, would give FSII concentrations approximately 75% of the true values. This apparent extraction efficiency of 75% is close to the theoretical extraction efficiencies, which are 71.4 to 73.6% (depending on original FSII concentration) for a phase distribution coefficient of 200/1.

c. Seiscor Differential Refractometer Method

As defined in FS-791b Method 5340.1, this method consists of extracting 800 ml of fuel with 50 ml of water and measuring the difference in refractive indices of the extract and of pure water, read directly on the dial of the differential refractometer. A calibration curve is required to relate fuel FSII content to dial reading. As the test procedure is written, FSII/water mixtures are used to establish the calibration curve, and no allowance is made for incomplete extraction of FSII from test samples. As we had demonstrated in previous work⁽¹⁷⁾, this gives results that are 8-12% below the true FSII contents. The test method modifications that were developed in the previous work were used here. The principal modification is the use of fuel/FSII blends (extracted) to establish the calibration curve.

Calibration-curve data obtained with known fuel/FSII blends are shown in Figure 20. The scatter was greater than expected, and a second series of samples was run, again showing considerable scatter but no unidirectional trend as compared to the first set. Both sets of results were used to plot a straight-line calibration curve. Maximum deviations were on the order of 0.012%, and some of these were in the middle portion of the calibration curve, i.e., in the normal range of use concentrations.

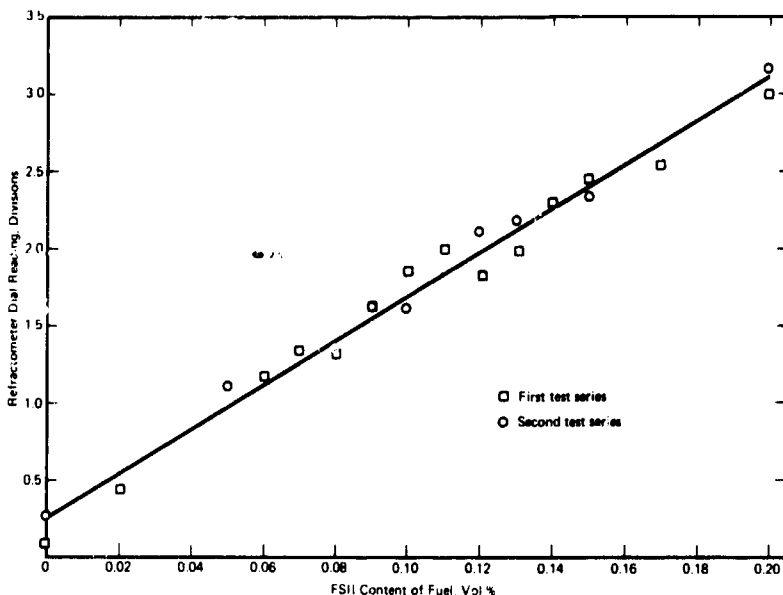


FIGURE 20. REFRACTOMETER CALIBRATION CURVE FOR FSII CONTENT

It should be mentioned that these data were obtained by an operator without previous experience in this test method. Thus, these results are comparable in this respect to the results obtained by the freeze point and colorimetric method, which had not been performed before by any of the laboratory personnel. It is probable that the scatter of results can be reduced in any of these test methods as experience is gained.

d. Colorimetric Method

In this test procedure, 100 ml of fuel is extracted twice with 10-ml portions of water. A 15-ml portion of the combined extract is mixed with 15 ml of hexanitrate ammonium cerate (ceric ammonium nitrate) color reagent solution. A portion of the mixture is filtered into a spectrophotometer cell, and the absorbance at 550 μ is measured immediately.

The calibration curve obtained in this manner is shown in Figure 21. An excellent straight-line fit was obtained, with deviations no greater than 0.004% throughout. However, repeat tests a day later on the same

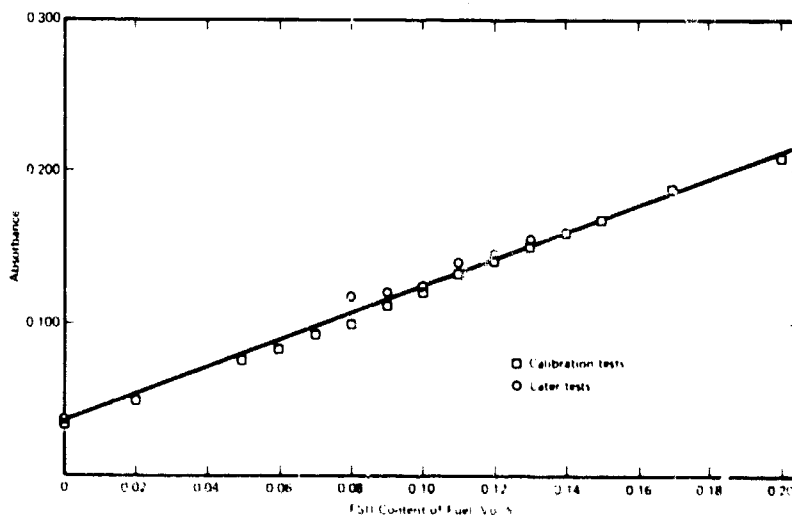


FIGURE 21. COLORIMETRIC CALIBRATION CURVE FOR FSII CONTENT

fuel/FSII blends gave appreciably greater absorbances, as indicated in Figure 21. One of these results deviates from the original calibration line by 0.013% FSII. The reason for the greater absorbances in the repeat tests is not known.

In all of these tests, absorbance of the test solution was measured versus distilled water; thus, the reagent blank itself absorbs with no FSII present. No correction has been made for reagent-blank absorbance, which accounts for the calibration curve not passing through the origin.

In this colorimetric method, extraction of the FSII from the test fuel sample is accomplished with two successive portions of water, each 10% of the fuel volume. The theoretical extraction efficiency is about 99.75% under these conditions. Therefore, it is sufficiently accurate to use water/FSII blends to establish the calibration curve rather than to run actual fuel extractions.

As an alternate to the spectrophotometric determination of absorbance, Gardner⁽¹⁸⁾ has suggested visual estimation of FSII content by matching the color with permanent potassium dichromate color standards. Tests were run, using the exact procedure outlined by Gardner, on blends with FSII contents of 0, 0.05, 0.10, and 0.15%. The increasing FSII content in this series was very evident from the colors developed. However, matching these colors with the permanent potassium dichromate standards was not at all satisfactory. The FSII extracts had a more brownish hue than the dichromate solutions and appeared darker than the concentrations which they were to match. In our tests, estimation even to $\pm 0.05\%$ FSII would not be feasible.

e. Iodometric Titration

This method, FS-791b Method 5327.3, has been the recognized method for determining FSII contents of jet fuels for a number of years. In this method, a fuel sample is extracted with an equal volume of water, and the extract is reacted with excess dichromate solution (oxidizing the FSII), and the excess dichromate is titrated iodometrically with sodium thiosulfate. Water/FSII blends are used as standards, although in principle it should be possible to use the dichromate as the primary standard and calculate the FSII contents from the dichromate consumption, assuming theoretical stoichiometry.

The 1/1 extraction ratio used in this method gives a theoretical extraction efficiency of about 99.5%, so the use of water/FSII blends as standards should introduce no significant error.

Seven of the fuel/FSII standard blends were submitted to an Air Force quality control laboratory (SAOQLA) for analysis by Method 5327.3, with the following results for FSII content (vol. %):

Added:	0.02	0.05	0.09	0.10	0.13	0.14	0.20
Found:	0.025	0.045	0.068	0.104	0.124	0.129	0.190

The deviation for the 0.09% FSII blend was excessive (0.021%). Of the other deviations, the greatest was 0.011%.

No attempt was made to identify the most probable causes of the deviations.

f. Comparison of Precision of Methods

Although the data that were obtained do not permit a sound statistical determination of precision, it is of some interest to compare the results obtained by the four methods with the known FSII contents of the standard blends. In the case of the three methods employing calibration curves (freeze point, differential refractometer, and colorimetric), this is done by taking the freezing point, R.I., or absorbance actually obtained on a given sample, then referring to the corresponding calibration curve, and reading the hypothetical FSII content. Here, of course, deviations merely represent the scatter in the data used to construct the calibration curves. Such a comparison of the four methods is shown in Table 22. These data illustrate the pattern noted previously at various points in this discussion.

Iodometric titration: One deviation 0.021%, others up to 0.011%
 Freeze point: Deviations up to 0.008% in range of interest
 Refractometer: Deviations up to 0.012%
 Colorimetric: Deviations up to 0.04% in first series, up to 0.013% in repeat series

TABLE 22. FSII CONTENT OF JP-4 FUEL BY VARIOUS METHODS

Actual FSII content, vol %	Indicated FSII content, vol %			
	Iodometric titration	Freeze point*	Colorimetric*	Refractometer*
0.02	0.025	0.014	0.021	0.008
0.05	0.045	---	0.048	0.057
0.06	---	0.060	0.058	0.063
0.07	---	0.072	0.068	0.075
0.08	---	0.087	0.076	0.073
0.09	0.068	0.094	0.090	0.094
0.10	0.104	0.094	0.100	0.110
0.11	---	0.107	0.112	0.120
0.12	---	0.114	0.122	0.108
0.13	0.124	0.134	0.133	0.120
0.14	0.129	0.148	0.143	0.142
0.15	---	0.154	0.153	0.153
0.17	---	0.171	0.173	0.161
0.20	0.190	0.208	0.198	0.193

*Data are for the same results used as calibration points.

It can be seen that errors on the order of 0.01% are to be expected in all of these methods under the conditions in which they were run here. Based on the rather scanty data obtained in these tests, there does not appear to be any outstanding advantage for any one method in terms of precision. The data are being examined further to attempt to develop statistically valid comparisons of repeatability.

g. Interferences and Difficulties in Techniques

All of the methods of FSII determination are subject to interference from other water-extractable substances in the fuel. In the freeze point method, any extractable would lower the freezing point and cause unduly high FSII results. In the refractometer method, other water extractables could cause either positive or negative interference, depending on their refractive indices. The colorimetric (ceric ammonium nitrate) method is dependent on reaction with hydroxyl groups, and any water-extractable compounds with a hydroxyl group would interfere to give high results. In the iodometric method, any readily oxidizable material extracted into the water would give positive interference; presence of an oxidizer in the water-extractables could in theory give negative interference, but this situation is most unlikely.

In general, it is considered unlikely that interfering, water-extractable substances will be present in normal fuels in concentrations high enough to perturb the FSII results significantly. Freak fuels or contaminated batches may well present difficulties in any FSII analysis.

One difficulty common to all FSII methods is the dependence on a quantitative or at least reproducible extraction of the FSII from the fuel by water. The extraction conditions are probably more critical whenever the water/fuel ratio is small, since then the extraction cannot be quantitative, and the results must vary with the

contacting efficiency and the partition coefficient. These in turn depend on fuel composition and extraction temperature. From this point of view, the freeze point and refractometer methods, with theoretical extraction efficiencies near 72 and 93%, respectively, are less desirable than the colorimetric and iodometric titration methods, with near-100% theoretical extraction.

Reproducibility of extraction procedures is also harmed by poor separation and the presence of emulsions. Improved techniques for contacting and separation would be desirable for all methods of FSII analysis.

h. Conclusions

Selection of a method for FSII analysis will probably be based mainly on convenience. No great differences in precision of the different methods could be noted in the limited amount of testing performed here.

The freeze point method is probably most suitable for use in the field with limited laboratory facilities; the equipment and procedures are simple. A supply of ice must be available. Care must be taken that a calibrated thermometer of good accuracy is used, that the mercury column in the thermometer does not separate, that the ice bath temperature is sufficiently low, and that a freezing point calibration curve is obtained on the basis of known blends of fuel and FSII of the types actually in use.

The refractometer method appears simple but actually requires great attention to detail and development of operator technique.

The colorimetric method using spectrophotometric measurements should receive further evaluation. This method is probably faster than titration, if time for standardization of solutions is taken into account. Preliminary results on the visual color-comparison version of the colorimetric method indicated poor discrimination of FSII concentration level.

The rather poor accuracy of the standard titration method that was observed here is contrary to most past experience and should be investigated further.

4. DETERMINATION OF FUEL VAPOR CONCENTRATIONS IN TANKS

a. General

A rapid analytical method was needed for determining the amounts of JP-4 fuel vapors in simulated fuel tank atmospheres, in support of a program being conducted by the Fire Protection Branch of the Fuels and Lubrication Division of the Aero Propulsion Laboratory. Here we will describe the analytical apparatus and techniques that were developed and will give illustrative results only. Detailed results have been reported to the Fire Protection Branch.

b. Apparatus and Procedures

Since only a measure of the total vapor concentration was desired, no separation or identification of individual hydrocarbons was needed. The simplest approach appeared to be the use of a gas chromatograph with flame ionization detector. Since the individual hydrocarbons in the vapor may differ in magnitude of detector response per unit weight, it is necessary to have a known standard of similar vapor composition in order to quantify the results. The standard chosen was fuel-saturated air at 100°F, adjusted to 760 mm pressure. The temperature of 100°F was selected since this was the temperature used in most of the fuel tank tests. It was expected that this standard sample will have a higher vapor concentration than would be encountered in any of the fuel tank test samples.

The fuel-saturated air (FSA) was injected into the chromatograph each day to serve as a 100% FSA standard. This daily calibration was required to compensate for variations in detector response that may have occurred because of deposit formation on the detector electrodes or because of variations in the hydrogen and air

flow rates. The fuel vapor contents of the tank test samples were expressed as percentages of the vapor content of the 100% FSA fuel standard.

The chromatographic column was relatively short and nonpolar and was used at high temperature, since there was no interest in separating individual components, but only in measuring total response in terms of total peak height or peak area. The vapor standard or test sample was introduced into the column using a 250 μ l gas-sampling valve.

The 100%-FSA standards were prepared by bubbling dry air through a column of fresh JP-4 fuel, using the apparatus shown in Figure 22.

In order to check the linearity of detector response, over the concentration range of interest, it was necessary to prepare air mixtures at various fuel-saturation levels. This was done by means of the apparatus shown in Figure 23. Analysis of these samples showed that the response was not linear, as indicated by the curves in Figure 24. Corrections derived from these curves were applied to all sample results.

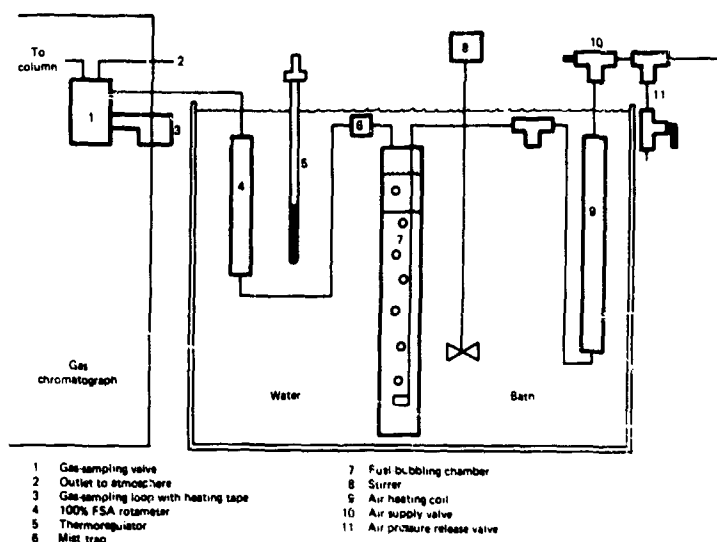


FIGURE 22. FUEL-BUBBLING APPARATUS

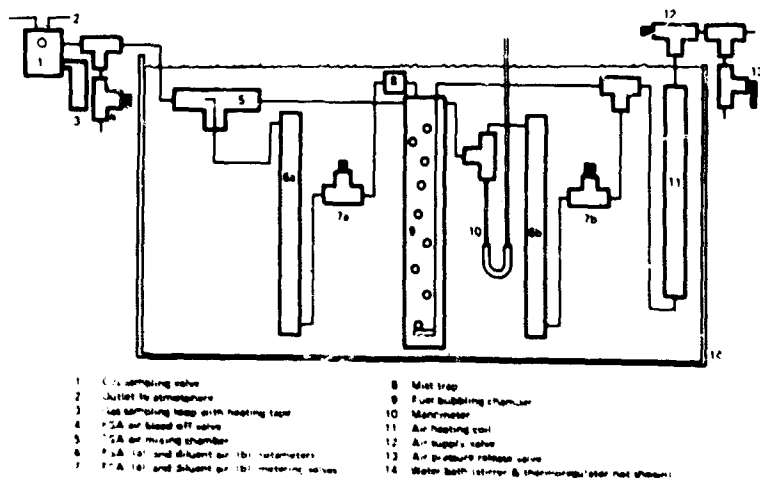


FIGURE 23. FSA-AIR MIXING APPARATUS

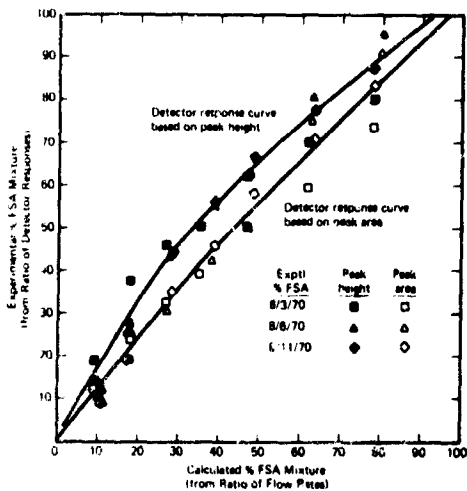


FIGURE 24. CALIBRATION CURVE FOR NONLINEAR DETECTOR RESPONSE

nonreproducible. Later, an oily contaminant was discovered when the gas-sampling valve was disassembled. Heavy components of the vapors would be adsorbed by this oily contaminant, and their slow elution was responsible for the peak tailing. Large amounts of these heavy components could be picked up during the rather prolonged operations on FSA standards. Because of this peak tailing, measurement of peak areas did not give any meaningful results in the early work, and reported results were based on peak height only. After eliminating the contamination problem, reported results were based on peak areas.

This situation is illustrated by the following values for corrected average % FSA in tank-test samples:

Early work, peak area	9	6	11	11	10	13	19	19	16
peak height	17	15	21	21	22	25	38	39	37
Later work, peak area	44	44	33	35	34	33			
peak height	46	46	30	33	30	29			

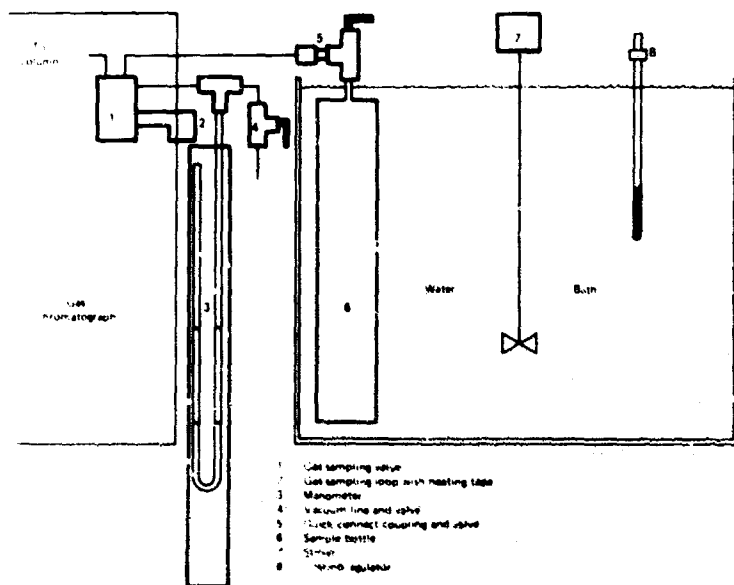


FIGURE 25. SAMPLE BOTTLE APPARATUS

Sample bottles containing tank test samples were brought to the chromatograph, and the contents were analyzed using a vacuum gas-sampling system as indicated in Figure 25. The sample transfer was effected by drawing a vacuum on the system, then opening the sample bottle and filling the system with the sample gas. A manometer in the system measured the sample pressure differential from atmospheric. The gas chromatographic response was corrected for sample size variations caused by pressure differences and was compared with the same day's FSA standard to give the experimental % FSA by volume. Experimental values for two or three samplings from each bottle were averaged and corrected for the nonlinearity of response (Figure 24).

c. Discussion of Results

Early results on test samples were based on the peak height response relative to that of the FSA standard. It was noted in this early work that peak tailing was excessive and

In the early work, the average values for % FSA based on peak area were considerably lower than those based on peak height. This is consistent with the role of the oily contaminant, since vapor buildup in the contaminant and the consequent peak tailing would affect the prolonged operations with standard FSA more than they would affect the shorter operations on test samples. In the data shown for the earlier operations, the apparent % FSA in the test samples, based on peak area, was only about half of that based on peak height; the peak-height data are clearly the more reliable here. In the later operations, the area- and height-based values are in good agreement, and, in general, the area-based values are considered the more reliable. In the later work, random

comparisons of peak height- and area-based values (not shown here) indicated similarly good agreement.

The calibration data (Figure 24), gathered to check the linearity of detector response, show greater scatter than is desirable in calibration data. Nevertheless, the data indicate clearly that response is nonlinear, and the corrected values based on these curves should be more reliable than uncorrected values. More exact calibration could be achieved but would have required extensive changes in the apparatus design. Deviations in rotameter flow rates from calibrated values were noted; these undoubtedly accounted for some error. Possible inefficiency of the mixing chamber could account for error. Also, unexplainable changes in the 100% FSA responses occurred in two of the three calibration runs. It would have taken considerably more time to resolve these questions than was available during this work.

Another question that might merit further investigation is the depletion of lighter hydrocarbons from the JP-4 fuel used in preparing the 100% FSA and known mixtures for calibration. Trends toward decrease in peak height and increases in tailing were sometimes noted when successive samples of 100% FSA were taken. For more accurate work, it would be desirable to design and use a flow-through apparatus where fresh fuel was introduced constantly into the vapor production chamber.

Attempts were made to produce 100% FSA by static saturation, using 100 ml of JP-4 fuel in a sample bottle of the same type used for test samples. This bottle was held at 100°F and shaken periodically, drawing off small samples of the atmosphere for chromatographing. Oddly enough, samples of the bottled atmosphere gave only about half the peak area response of the 100% FSA produced by bubbling air through a column of fuel at 100°F. Both approaches should be expected to produce approximately the same fuel-vapor content; the reason or reasons for the discrepancy could not be determined. In the absence of any contrary indications, the method giving the higher apparent hydrocarbon contents (peak areas) must be considered the better source of "fuel-saturated air." Hence, the FSA prepared in the bubbling apparatus was used as the standard.

It is difficult to estimate the accuracy and precision of the measurements, since the techniques were being developed and improved as the analyses were being made. Also, there were numerous interrelated and partially unresolved variables in these analyses. We believe that the later results using peak areas are probably within 10 percentage units of their "true" % FSA concentration. However, this is only an estimate based on the consideration of possible errors.

Much more work would be needed to perfect the method of analysis and resolve the variables still remaining.

5. DETERMINATION OF OXYGEN CONTENT OF FUELS

Oxygen determinations on fuels have been continued in support of Air Force studies involving the Advanced Fuel System Simulator Rig at Wright-Patterson AFB. Previous results obtained by SwRI and details of the gas chromatographic method of analysis were discussed in last year's annual report⁽²⁾.

Test results for a JP-7 fuel, AFFB-13-69, are shown in Table 23. This fuel was tested in December 1969. No further oxygen analyses have been run during the reporting period.

The oxygen-content results for air-saturated hexane have been included in the tabulated data to indicate the day-to-day repeatability of the method for a liquid sample. Three to six hexane injections were made with each day's set of fuel samples. The initial value is for the first hexane injection each day; comparison with the daily and final means indicates that fair repeatability is obtained in these analyses.

6. EFFECTS OF FUEL DYES ON FUEL PROPERTIES

Several oil-soluble dyes have been proposed for use in JP-4 fuel for special purposes. It was desired to determine the effects of these dyes on fuel properties, in particular the contamination level, thermal stability, and water-separating characteristics.

TABLE 23. OXYGEN ANALYSIS OF AFFB-13-69 FUEL FROM SIMULATOR

Oxygen content in ppm (wt)

Test no.	Incoming fuel, t = 0 min* 3	Start cruise t = 25 min*				End cruise t = 105 min*				Peak descent*	Air-saturated hexane	
		4	5	6	7	4	5	6	7		Initial value†	Mean value‡
9.080	68	20	18	17	19	11	13	10	5	<2	122	129
9.082	72	19	17	15	16	10	8	11	4	<2	115	120
9.087	80	19	20	19	19	12	10	10	5	<2	118	122
9.089	79	18	18	19	18	11	10	10	8	<2	117	121
9.094	79	21	18	16	19	10	9	8	5	<2	115	120
9.096	81	20	15	16	16	7	8	10	5	<2	124	127
9.099	51	18	20	19	18	8	8	7	2	<2	122	124
9.101	68	17	13	16	14	7	11	9	2	<2	116	118
9.105	56	17	17	18	17	8	10	10	4	<2	121	124
9.106	51	17	16	16	17	5	8	7	4	<2	110	120
9.110	77	22	17	15	17	8	11	10	3	<2	125	126
9.123	71	17	15	15	15	9	9	16	8	<2	125	125
9.132	70	18	16	12	16	9	9	8	6	<2	121	129
9.137	61	17	13	15	13	8	8	11	11	<2	118	125
9.139	76	19	19	16	16	9	9	9	6	<2	122	133
9.141	75	24	20	22	20	13	13	13	16	<2	126	128
Average	70	19	17	17	17	9	10	10	6	<2	120	124

*Numbers in subheading represent valve numbers.
†Value of initial injections with incoming fuel.
‡Average of all injections for the test.

The three dyes evaluated were Automate Yellow No. 662, Automate Blue SF, and Automate Red BSF. These were blended and tested in a non-additive JP-4 fuel selected from tank-farm storage. This base fuel conformed to all JP-4 specification requirements. The fuel's content of aromatic hydrocarbons was 8.5%, which is somewhat low but not at all unusual. By way of comparison, the Bureau of Mines 1969 average for JP-4 fuels were 11.5% aromatics; the 1967 average for Air Force procurements of JP-4 was 12.1% aromatics, with some 30% of the samples having less than 10% aromatics, and 10% of the samples having less than 7.5% aromatics.

The base fuel was transferred from the storage tank to a precleaned, epoxy-lined, 55-gallon drum, then through 0.45 μ filters to four precleaned, unlined, 10-gallon steel pails. Container precleaning was performed with filtered toluene/acetone/isopropanol trisolvant. Three of the fuel samples were blended with test dye at a concentration of 2 fl oz/100 gal (about 140 mg/liter); the fourth was used as a control sample. Immediately after blending, samples were drawn for initial tests. The pails were then closed with lug-lock lids and stored for three months at 70-90°F, after which the fuels were again tested.

The tests that were run included particulate contaminant (ASTM D 2276), silting index (FS 791b, Method 5350) WSIM (ASTM D 2550, modified), emulsion characteristics (ASTM D 1401, modified), and thermal stability (ASTM D 1660). The WSIM tests could not be run in the usual manner because of the deep colors of the fuels, which made it impossible to set the light output meter at 100 when operating on clean fuel at the start of the test. Therefore, the meter was simply set at the highest reading possible, and changes caused by water entrainment were noted. The values obtained cannot be correlated with conventional WSIM values. Because of these difficulties, the water-separating properties of the fuels after storage were evaluated by a modified emulsion test of the type

TABLE 24. EFFECTS OF DYES ON FUEL PROPERTIES

Dye*	Months storage	Silting index, SI,	Solids, mg/liter	Emulsion test, ml fuel/water/emuls at 0 minutes	WSIM meter reading		Fuel coker, std, 300/400°F	
					Initial	Final	Tube rating, unwiped/wiped	Filter ΔP, in. Hg
None	0	2.1	---	---	100	99	1/1	0.3
	3	0.12	0.2	40/40/0	100	98	1/1	0.6
Blue	0	2.3	1.8	---	13	5	---	---
	3	33.2†	12.4	40/37/3	---	---	1/1	1.2
Red	0	1.5	1.2	---	40	19	---	---
	3	3.1	3.7	40/39/1	---	---	1/1	3.7
Yellow	0	0.13	0.4	---	66	40	---	---
	3	2.7	2.0	40/39/1	---	---	1/1‡	0.0

*Tested at 2 fl oz/100 gal in non-additive JP-4; Automate Yellow No. 662, Automate Blue SF, and Automate Red BSF.
 †SI₂.
 ‡Warmup time 20.7 minutes; other tests 15-20 minutes.

applied to steam-turbine lubricating oils, in which the test sample is agitated with water under prescribed conditions, and the condition of the two layers and the interface is noted periodically. As applied to fuels, we ran the test at 100°F instead of the 130°F specified for oils and merely noted the condition of the mixture at "zero minutes," i.e., within 10-20 seconds after stopping the stirrer, since settling was generally so rapid as to preclude long-time observations.

The results obtained are listed in Table 24. It will be noted that all of the dyes had adverse effects on fuel contamination level and water separation characteristics.

The silting index results on the freshly blended samples showed no significant effects of the dyes, but the 3-month results showed severe plugging problems with the blue dye and slight adverse effects from the other dyes. Contents of particulate matter (solids) showed the same general trend, with the blue dye contributing rather huge amounts of insoluble material. The level of 12.4 mg/liter insoluble matter amounts to nearly 10% of the total amount of blue dye added.

The thermal stability of the dyed and undyed fuel samples was determined by standard coker tests at 300/400°F. All fuels were prefiltered through Whatman 2V paper immediately before testing. These fuel coker test results (Table 24) show that the dyes had no effect on preheater tube deposits; Code 1 color ratings were obtained in all tests. The blue and red dyes did cause filter plugging in the coker tests; for the red dye, this plugging amounted to 3.7 in. Hg, above the specification limit of 3.0 in. The plugging effects of the dyes cannot be regarded as extremely severe, since the base fuel and control fuel also gave some plugging. It appears probable that the plugging caused by the dyes in the coker tests is a reflection of insoluble matter not removed by the Whatman prefiltration, rather than any degradation of thermal stability.

From the WSIM results, all one can say is that the dyed fuels did entrain some water during test, as evidenced by the drops in meter readings. The emulsion tests likewise showed some adverse effects of the dyes. There is no way to relate these data to conventional test results or to performance, but it is clear that water-separating characteristics should be a matter of concern in considering dyes for use in the field.

The insolubility problems on these dyes were somewhat unexpected. The fuel that was used as a base stock was not at all atypical; the aromatics content was only slightly below average. The absence of FSII (fuel system icing inhibitor) might be an explanation for the very poor solubility characteristics. Use of the normal content of this

additive should increase a fuel's solvency for many nonhydrocarbon compounds such as dyes. However, from a practical point of view, FSII may be present in varying amounts, and one should not rely on it as a solvent for a secondary additive.

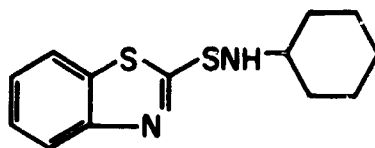
It is concluded that the red and blue dyes are not sufficiently soluble in JP-4 fuel for field use, and that there may be a solubility problem with the yellow dye. All of these materials appear to have some adverse effect on fuel water-separating characteristics.

7. COAGULATING AGENTS FOR PARTICULATE MATTER

a. Background

There are indications in the Soviet literature⁽¹⁹⁻²¹⁾ that considerable work has been performed on fuel additives intended to coagulate finely divided particulate matter, so that it can be removed more easily by settling or filtration. Among the compounds mentioned most are sulfenamide derivatives of 2-mercaptobenzothiazole; esters of diethylene glycol, glutaric acid, and C₁₀-C₁₂ aliphatic alcohols; and the octadecyl-amide of γ -hydroxybutyric acid. It is stated that the best of the compounds tried is N-cyclohexyl-2-mercaptobenzothiazolesulfenamide, which is designated TsBSA additive in the Soviet literature. Most of the test work cited has been concerned with field tests at airports, where concentrations of 0.002-0.005% (20-50 ppm) are said to improve the efficiency of fuel filtration and to aid in the settling of fuel-suspended matter in storage tanks.

There is an anomaly in the chemical name assigned to this compound, since the 2-position in the benzothiazole cannot be occupied by both a mercapto and a sulfenamide group. The compound is thought to be N-cyclohexyl-2-benzothiazolesulfenamide:



This compound is prepared from 2-mercaptobenzothiazole and cyclohexylamine, which may account for the confusion in nomenclature. A further indication that the formula shown does represent the material cited in the Soviet literature is their abbreviation TsBSA, which would correspond to N-tsiklogeksil-2-benzotiazolsul'fenamid.

This compound has been patented by Monsanto⁽²²⁾ along with other similar compounds as vulcanization accelerators; the melting point of this compound is reported as 97.5°C. No other references to chemical or physical properties have been found.

This compound was found to be available from the Aldrich Chemical Company, and a sample was obtained for evaluation. No information on purity of this material was available; from its behavior in solubility tests, it appears to contain substantial amounts of impurities.

This material, which we will designate here as "CBSA," was evaluated for effectiveness in improving the filtration efficiency when removing finely dispersed inorganic solid contaminants from fuel.

b. Solubility Studies

Preliminary checks had indicated that the CBSA was quite insoluble in fuel. Further checks on solubility behavior were made by stirring 100 mg of the CBSA with a limited amount of fuel and then diluting successively with more fuel to attempt to determine a solubility limit. The solubilities of CBSA in 2-methoxyethanol and in toluene were checked in the same general manner. The following observations were made:

JP-5 fuel	Not completely soluble at 20-50 ppm
2-Methoxyethanol	Soluble at 5.6 g/liter
Toluene	Mostly soluble at 5 g/liter

In the 5 g/liter blend of CBSA in toluene, the small amount of residual undissolved material would not dissolve when more toluene was added. The insoluble material, amounting to 2.6% of the original CBSA, was recovered and compared with the original material by pressing with KBr to obtain infrared spectra. The differences in spectra confirmed that the toluene-insoluble material is an impurity.

For preparing fuel blends, the CBSA can be predissolved in 2-methoxyethanol at a concentration of 5 g/liter and then added to the fuel. A final blend containing 20 ppm (wt) of the CBSA will contain about 0.3% (vol) of 2-methoxyethanol, which is twice the maximum amount of this material allowed for use as a fuel system icing inhibitor. Blends prepared in this manner apparently do not reprecipitate any significant amount of CBSA when the concentrate is diluted with fuel, as evidenced by a check on one blend in JP-5 fuel with 20 ppm CBSA and 0.30% FSII.* Out of 15 mg of CBSA added, only 0.06 mg was recovered by filtering the fuel blend.

If toluene is used to predissolve the CBSA in making fuel blends, a small portion of the CBSA will remain insoluble in the toluene and may or may not dissolve when the fuel is added.

c. Coagulation Tests

Each check on coagulating efficiency consisted of a pair of tests on 1-liter fuel samples, one with CBSA and the other without. Nonadditive base fuels were used, with corrosion inhibitors added in most tests to increase the dispersity of the solid contaminants that were added. In order to provide a good test of coagulation or the lack thereof, the test mixtures were filtered quantitatively through 0.8- μ silver membrane filters. The solid contaminant used in most of this work was red iron oxide, Fisher I-116, which is one of the materials used as standard contaminants in filter-separator testing. The particle size of this material is mostly in the submicron range, and, if it is thoroughly dispersed in a fuel, it will be retained very little by a 0.8- μ silver membrane filter.† Thus, in these tests, the degree of recovery of the suspended contaminant is a measure of the agglomeration of the particles of solid contaminant.

The first coagulation tests were run by prefiltering the base fuel (0.45 μ), blending in the corrosion inhibitor, adding the solid contaminant, mixing at high speed for 3 to 5 minutes, adding the CBSA to one of the two test samples, and stirring for 7 to 10 minutes, after which the samples were each filtered through a 0.8- μ silver membrane filter. In this sequence, any insoluble portion of the CBSA will show up on the test filter along with the recovered solid contaminant; hence, the results obtained for contaminant recovery are ambiguous.

For subsequent tests, the order of operations was changed so that the corrosion inhibitor was blended first, then the CBSA was added to one of the two test samples, and then these blends were prefiltered (0.45 μ), after which the solid contaminant was added and mixed, and the final mixtures were each filtered through 0.8- μ silver membrane filter. In all of the later tests, the CBSA was predissolved in a solvent to facilitate blending with the fuel. Solvents used were toluene, 2-methoxyethanol, and the then-current fuel system icing inhibitor, which contained 99.6% 2-methoxyethanol and 0.4% glycerol.

The test results are listed chronologically in Table 25. It will be noted that the apparent filtration recovery of red iron oxide in the first test with the CBSA was over 100%, reflecting the presence of insoluble matter from the CBSA itself. There did appear to be some coagulation of the red iron oxide in this test, judging by the filter colors with and without the CBSA. When these tests were repeated with prefiltration to remove the insoluble portion of the CBSA, there was no significant coagulating effect, judging by either the weights of iron oxide recovered or by the filter colors. Quite possibly the true concentration of CBSA in the solution was far below the added 20 ppm.

When the CBSA was predissolved in FSII, a very marked coagulating effect was observed; the CBSA raised the recovery from 20 to 92%, and the increased recovery was very evident from the filter colors. This single

*Fuel system icing inhibitor containing 99.6% 2-methoxyethanol, 0.4% glycerol.

†A nonmetallic 0.8- μ membrane filter of the type used in ASTM D 2276 will retain this iron oxide quantitatively. Apparently the difference in behavior is due to electrification effects of the nonmetallic filter.

TABLE 25. COAGULATION TEST RESULTS

CBSA* ppm (wt)	Solids added, mg†	Solids recovered		Filter appearance	Remarks
		mg	%		
<i>JP-4 with 6 lb/Mbbl corrosion inhibitor "X"</i>					
0	2.00	1.10	55	Pink	Recovery includes insol CBSA
20	2.00	2.35	118	Red	
0	4.00	3.00	75	Lt red	Insol CBSA prefiltered
20	4.00	2.70	68	Lt red	
<i>JP-5 with 6 lb/Mbbl "X" and 0.3% FSII‡</i>					
0	4.00	0.80	20	Pink	
20	4.00	3.70	92	Red	
<i>JP-5 with 16 lb/Mbbl "Y" and 3% toluene</i>					
0	4.00	3.18	80	Red	
50	4.00	3.39	85	Red	
<i>JP-5 with 6 lb/Mbbl "X" and 0.3% 2-methoxyethanol</i>					
0	4.03	3.27	81	Lt red	Settled 5 min before filtering
20	3.97	3.27	82	Lt red	Settled 5 min before filtering
<i>JP-5 with 0.3% 2-methoxyethanol, no corrosion inhibitor</i>					
0	3.96	3.41	86	Dark red	Settled 5 min before filtering
20	3.95	3.65	92	Dark red	Settled 5 min before filtering
<i>JP-5 with 16 lb/Mbbl "Y" and 3% toluene; contaminant fine AC test dust</i>					
0	4.36	2.65	61	Lt brown	Settled 5 min before filtering
50	4.11	2.55	62	Lt brown	Settled 5 min before filtering
<p>*N-cyclohexyl-2-benzothiazoleulfenamide. †Solids Fisher I-116 red iron oxide unless otherwise indicated; added to 1 liter of test fuel. ‡99.67 2-methoxyethanol, 0.47 glycerol.</p>					

favorable result was never duplicated in later work. In all of the remaining tests, regardless of the corrosion inhibitor, solvent, or solid contaminant that was used, the CBSA gave no significant effect whatever. In fact, the results with and without CBSA were in very close agreement.

In some of the later tests, the "settling period" between the end of agitation with solid contaminant and the start of the filtration was standardized at 5 minutes. Previously, this had not been standardized but was generally much shorter. In all cases, any material that settled out was transferred quantitatively to the filter. In these tests with controlled schedule, it will be observed that the corrosion inhibitor "X" did have some effect in increasing the dispersity of the red iron oxide as evidenced by the lower recoveries, but the effect was not great. The CBSA was completely ineffective in coagulation. The same applied to tests with fine AC dust as the solid contaminant, which gave surprisingly low recoveries in comparison with the red iron oxide. Although the fine AC dust is not nearly as fine as the I-116 red iron oxide in ultimate particle size, the AC dust is much easier to disperse in fuel. Here, no special efforts were made to obtain ultimate-particle dispersions with either dust, and the iron oxide very likely remained in large aggregates despite the presence of corrosion inhibitor and the violent stirring. The resistance of the iron oxide to dispersal will depend on a number of factors, particularly its moisture content. This rather brief attempt at evaluating coagulant action did not include any rigorous control on the condition of the red iron oxide, nor did it include any independent measurement of the dispersity of the mixtures. In view of the results obtained, it is fairly certain that dispersal to ultimate particle size was *not* effected in most cases.

In the tests in which a 5-minute settling period was maintained between the end of agitation and the start of filtration, appreciable sedimentation resulted both with and without the CBSA additive.

Based on indications from the Soviet literature, it appeared possible that short periods of contact between the CBSA and the solid particles are insufficient to effect any coagulating actions, and therefore, these tests did not evaluate its full capabilities. One pair of qualitative tests was run with a high concentration of fine AC dust (100 mg/liter) in JP-5 fuel with 16 lb/Mbbl of corrosion inhibitor "Y", in which the test samples were stirred and checked visually at intervals for dispersity, judged by the amount of residue settling out and the appearance of the supernatant fuel. It was found that the degree of dispersity increased up to about three hours of agitation and then did not change, so far as could be determined visually. Settling times of 10 minutes were sufficient to rate the samples. The amounts of residue and the appearance of the supernatant fuels were not affected by the presence or absence of the CBSA coagulant additive.

Naturally, it is possible that much longer contact times, on the order of days, are necessary for the additive to function properly. No study was made of contact times longer than a few hours.

The net result of this study is one observation of a positive coagulating effect of the CBSA, which could not be duplicated. If any more work on coagulant additives is to be performed, it is clear that any artificial dispersions of solid materials that are used must be prepared and controlled in a more rigorous manner. Another possible approach is taking samples from a large batch of fuel, containing relatively large amounts of suspended material, and testing the effect of CBSA in coagulating the particulate contaminants. Such an approach is more realistic than can be achieved with laboratory prepared dispersions and should be given serious consideration for any further work along this line.

Of the materials other than CBSA listed in the Soviet literature as coagulating agents for fuels, none appears to be available from U.S. sources.

8. EFFECT OF CORROSION INHIBITORS ON LEAD

a. Background

Field problems with failures of certain aircraft fuel pumps had been traced tentatively to freezeup of lead-coated bronze surfaces. This condition was often accompanied by whitish deposits on the metal surfaces. It appeared desirable to learn whether any of the commonly used types of corrosion inhibitors have any unusual interactions with lead.

Previous attempts along this line had met with some qualitative success, indicating that metal loss and/or deposit formation could be attributed in some cases to corrosion inhibitors containing organic acids. The investigation of fuel/inhibitor blends at actual working concentrations is hampered by the presence of nonhydrocarbon fuel constituents and also by the exceedingly low contents of inhibitor in the fuels. In a static laboratory test it is almost impossible to arrange for a metal test surface to "see" as much fuel and inhibitor as it will in service. For instance, a small surface area in a fuel pump may experience the passage of (say) 10,000 gallons of fuel within a relatively short time. If this fuel contains 6 lb/1000 bbl of a corrosion inhibitor, the few square inches of metal surface may in effect be exposed to more than a pound of corrosion inhibitor.

Since this situation is not amenable to duplication in the laboratory, it was decided to accelerate fuel/metal interactions by using rather high concentrations of corrosion inhibitors and other material in static exposure tests.

b. Test Procedure

The materials taken for test were oleic acid, naphthenic acids, and two MIL-I-25017 corrosion inhibitors. Identification of these materials and total acid numbers on the samples used in this work are as follows:

	Total acid no., mg KOH/g
Inhibitor X, carboxylic acid type	94
Inhibitor Y, amine phosphate type	138
Oleic acid, Eastman Technical Grade	199
Naphthenic acids, MCB Technical Grade	223

Blends were prepared from each of the two corrosion inhibitors in toluene (ACS Reagent) at a concentration of 1000 lb/1000 bbl (2.85 g/liter). Oleic acid was blended in toluene at 1.34 g/liter and naphthenic acids at 1.18 g/liter in order to match the blend acid number with that of the Inhibitor X blend.

Lead strips, each having approximately 4 in.² of surface area, were cut from 4-lb "chemical lead" sheet of the type used in lead corrosion testing of synthetic lubricants. This is a fairly pure lead with a moderately high copper content, giving it better strength and corrosion resistance than those of very pure lead.

The lead strips were polished with steel wool, cleaned, and weighed. Each strip was placed on edge in a bottle containing 100 ml of test blend. One strip was placed in a bottle of pure toluene as a control. The test assemblies were stored at room temperature and observed periodically. After 26 days, the strips were removed, rinsed with petroleum ether, dried, and weighed. Subsequently, they were rinsed with methanol, dried, and reweighed.

c. Results and Discussion:

Within two days of immersion, orange-yellow deposits started to build up on the lead strip in the Inhibitor X test blend. These deposits had a clear and "gummy" appearance rather than powdery. The strips in the oleic and naphthenic acid solutions gradually took on a lighter but duller appearance than the original strips. The strips immersed in the Inhibitor Y blend or the straight toluene showed only a gradual darkening.

When the strips were removed after 26 days, the Inhibitor D blend had accumulated a rather large amount of yellow-orange gummy material on the bottom of the bottle, as well as on the lead strip. In solvent-rinsing this specimen (and the others), no attempt was made to physically remove the deposits. Little gum was removed by either petroleum ether or methanol. After the strip was allowed to dry, the gummy deposit hardened into a hard, varnish-like material covering the entire strip.

No precipitates were noted in the sample bottles that had contained the other test blends. The lead strips from the oleic and naphthenic acid solutions showed some whitish or whitish-yellow granular deposits after solvent-rinsing. The strips from the Inhibitor Y blend and from the straight toluene showed only slight surface darkening.

The cumulative weight losses of the lead strips after solvent-rinsing, in comparison with the pretest weights, were as follows:

	Weight change, mg	
	Petr ether rinse	Methanol rinse
Inhibitor X	-2.3	-2.8
Inhibitor Y	+0.4	+0.4
Oleic Acid	-84.7	-84.3
Naphthenic acid	-83.5	-83.5
No additive	+0.4	+0.6

The lead weight losses observed with the oleic and naphthenic acid blends are in line with theoretical values based on acid number. The lead-dissolving capability of each of these blends is 99 mg if the lead is assumed to form the basic soap, or 49.5 mg if the lead is assumed to form the normal soap.

The lead strips in the straight toluene or in the Inhibitor Y blend showed only very slight weight gains, which, along with their appearance, indicated only normal formation of an oxidized film.

The lead strip in the Inhibitor X solution showed a slight weight loss in spite of the heavy deposits. Lead removal is clearly indicated. In view of the rather large amounts of insoluble precipitate in the sample bottle, it is probable that metal removal was quite extensive. Attempts were made to remove the varnish-like deposit from the lead strip in order to determine the actual metal weight loss. However, no known solvent had any effect on the deposit.

These results indicate that Inhibitor Y has no effect on lead, at least at normal room temperature. Inhibitor X reacts with lead rather rapidly at room temperature when the inhibitor is present in very high concentration, and the reaction products, presumably lead soaps, are gummy or varnish-like materials that are insoluble in toluene and other solvents.

Whether or not such deposits could accumulate in fuel pumps operating on fuels with normal concentrations of inhibitor, and whether such deposits could result in pump freezups, are still matters for speculation.

If such a link is definitely established, it would be desirable to investigate in more detail the effects of fuel corrosion inhibitors and other additives on nonferrous metals, and to develop an appropriate corrosion test procedure to guard against harmful effects.

9. EFFECT OF SAMPLE CANS ON WATER SEPAROMETER RESULTS

a. Background

It has been known for some time that the results of water separator tests are very sensitive to fuel contamination, in particular contamination from the sample container. Ordinary tinned cans, the usual container for fuel samples, are not clean enough for samples intended for separator tests. Such cans may be precleaned with solvents or may be used repeatedly in fuel-sample service in order to remove the fuel-soluble contaminants that influence WSIM results. However, from a practical point of view, either precleaning or reuse may be impossible or highly undesirable.

In connection with an Air Force fuel quality control program, it was desired to check the suitability of commercially available, phenolic-lined, 1-gallon cans as WSIM sample containers. These cans were identified as Vulcan Part 1 BGSN-572-200. Ordinary tinned 1-gallon cans used for comparison were obtained from local stock and were identified as E.J.B. Part DSA 400-68-C-5402.

b. Storage and Test Procedures

Test fuel was chosen by sampling several storage tanks in the Area B fuel farm containing "in-flight" JP-4 fuel, that is, fuel containing all inhibitors. Several of the samples were found to have WSIM values below 85, but it appeared later that these low values were caused by sample-line contamination. The fuel finally selected, from Tank F-10, had a very high WSIM, averaging 97. Although fuel of this quality may not be typical of JP-4, it will serve as a sensitive indicator of contamination and hence was considered very suitable for use in this program. This test fuel met all MIL-T-5624G specification requirements.

An unlined 55-gallon steel drum was rinsed three times with several gallons of test fuel and then filled. The drummed fuel was sampled in amber glass jugs (thoroughly precleaned with particle-free solvents) and tested for initial WSIM rating. Samples of the drummed fuel were also drawn into the 1-gallon test cans as described in the following paragraphs. The remaining drummed fuel was kept in storage and resampled for WSIM rating after 4 weeks.

The phenolic-lined cans were precleaned by rinsing twice with 1 to 2 pints of test fuel and then filled to within about 1/2 in. of the top. The cans were closed with metal innerseals and caps and then held in storage.

One group of tinned cans was precleaned and filled in the same manner as the phenolic-lined cans. Another group of tinned cans was precleaned by rinsing thoroughly with chloroform and then with acetone. Each rinse was about 75-100 ml and was accomplished by means of a wash bottle, directing the solvent stream toward the can seams and spout in order to remove as much foreign matter as possible. As discussed later, there was evidence that this treatment did not remove all of the contaminants. After each rinse, the can with solvent was shaken and drained. Three rinses with chloroform were performed, then three with acetone. The cans were then air-dried and filled with test fuel.

All of the fuel samples, in the test cans and in the drum, were held in storage at approximately 50-60 F, all samples being exposed to the same temperature conditions.

Separometer tests in this program were run by a single operator with a single instrument. Current ASTM procedure was followed, with minor modifications. Coalescer disks from a single batch were used in these tests, and all disks were airflow checked before use to verify their acceptability.

TABLE 26. EFFECT OF STORAGE IN CANS ON FUEL WSIM

Storage containers	Weeks storage	WSIM results	Mean WSIM	ASTM repeatability range
Unlined steel drum	0	97, 97, 97	97	95-99
	4	88, 93, 96	92	89-95
Phenolic-lined cans, fuel-rinsed	1	94, 94, 93	94	92-96
	2	94, 96, 91	94	92-96
	3	97, 94, 97	96	94-98
	4	89, 88, 87	88	84-92
Tinned cans, fuel-rinsed	4	65, 59, 68	64	53-75
Tinned cans, solvent-rinsed	4	83, 86, 79	83	77-90

c. Separometer Test Results

The WSIM values that were obtained are listed in Table 26. Each storage condition and storage time is represented by three separometer test results. The means of the three results are listed, along with ranges derived from ASTM repeatability criteria. It will be noted that, in almost all cases, all three values were within the ASTM repeatability range, or at most 1 WSIM unit beyond the range.

d. Condition of Cans Before and After Storage

The phenolic-lined cans, as received, had thinly coated areas. In most cans, a very thin strip of bare metal was exposed along the seam. Also, on the bottom, a minute area of bare metal was

exposed on the edges of the letters and numerals stamped into the metal. The area around the spout of these cans was thinly coated in most cases. All of these breaks or deficiencies in the lining were judged to be very minor. Assuming that the base metal is clean, such defects should not be any factor in fuel contamination.

One of the phenolic-lined cans that had contained fuel for 4 weeks was sectioned and examined after test. There was no apparent softening or deterioration of the phenolic coating, nor was there any visible residue from the fuel.

The tinned cans, as received, were found to contain a black residue and some small metal particles that could be removed from the seam areas by using a cotton swab. It appears probable that the black material consisted of decomposed soldering flux. Also detected was a brown, resinous material around the spout.

Tinned cans that had contained fuel for 4 weeks were reexamined. Cans with both types of precleaning (fuel or solvents) were included in this examination. It was found that the black residue and the small metal particles adhering to it were still present, and could be removed by swabbing more easily and more completely than from new cans. Apparently the fuel had either removed some "binder" from the contaminant or had softened it.

e. Discussion of Results

The drummed fuel showed a 5-point drop in WSIM during the 4 weeks of storage; this is barely significant.

The fuel stored in phenolic-lined cans showed no significant WSIM changes during the first three weeks. The 4-week sample gave lower WSIM values, the decrease being barely significant.

As expected, the fuel stored in tinned cans that were prerinsed with fuel did drop drastically in WSIM, below the specification minimum of 70.

The results on the fuel stored in solvent-rinsed tinned cans are rather ambiguous. The average WSIM value after 4 weeks was 83. However, when comparing this with the results on fuel stored in phenolic-lined cans it will be noted that there is only 5 points difference in the mean, and that the corresponding repeatability ranges overlap by a considerable amount. Looking at it another way, the highest WSIM value obtained on the fuels stored in solvent-rinsed tinned cans (86) is only one point below the lowest WSIM value obtained on the fuels stored in phenolic-lined cans (87).

Part of the over-all drop in WSIM values during storage could be caused either by "aging" of the fuel or by differences in separometer rating level during the 4-week period. In any case, the drop in WSIM values for the drummed fuel was barely significant.

From these results, it can be concluded tentatively that these phenolic-lined cans, precleaned only by rinsing with test fuel, are at least as good as solvent-cleaned tinned cans with respect to lack of effect on WSIM values. The phenolic-lined cans should be suitable containers for quality-control or exchange samples, particularly if the period between filling and testing can be minimized.

These results also indicate that even very thorough rinsing with solvents cannot give a complete cleanup of ordinary tinned cans. The effect of the residues on WSIM values in these tests is somewhat ambiguous but at least it is clear that cans cleaned in this manner cannot be regarded as "safe." The presence of visible residues in the cans after fuel storage is evidence of the incompleteness of cleaning.

No attempt was made to identify the residues in the cans before or after fuel storage. If soldering flux is responsible, it could be detected by ASTM method D 2546-66T.

10. FUEL DEMULSIBILITY TESTS

In view of the well-known deficiencies of the water separometer test, there is considerable interest in investigating other possible methods of rating the water-separating characteristics of jet fuels. Ideally, such a test should be simple, have good precision, and correlate well with field performance of filter-separators.

A very simple test, still used for jet fuels, is the "water reaction test," in which a mixture of fuel with buffered phosphate solution is hand-shaken in a graduated cylinder and allowed to settle, after which the condition of the interface is observed. Unfortunately, this test is not sensitive enough to pick up the harmful effects of even such materials as formerly qualified amine sulfonate type corrosion inhibitors, which were very detrimental to filter-separator operation when used at effective concentrations.

A standardized demulsibility test that is widely used for steam turbine oils is ASTM D 1401. It is similar in principle to the water reaction test. A mixture of 40 ml of oil and 40 ml of water (normally distilled water) is agitated and then allowed to settle, after which the presence or absence of emulsion is recorded. However, the agitation is mechanical and is relatively well standardized in comparison with that of the water reaction test. The agitation specified in the demulsibility test consists of a 5-minute stirring period with a paddle turning inside the graduated cylinder at 1500 rpm. This provides rather more violent agitation than can be achieved by hand shaking. When testing oils by D 1401, the oil/water mixture is maintained in a bath at 130°F during the 5-minute stirring and the subsequent period of settling and observation. Observations are made every 5 minutes up to a maximum of 60 minutes, and the time required for reduction of the emulsion volume to 3 ml is taken as a measure of the oil's emulsifying characteristics.

In our initial attempts to apply this test method to fuels, the only modification that was made was the obvious one of reducing the test temperature to 100°F, to reduce fuel evaporation losses and fire hazard. This test was applied to certain dyed fuels, and did give some indications of harmful effects of the dyes on fuel demulsibility. These results, along with subsequent results from modified demulsibility tests, are listed in Table 27.

In this test as applied to fuels, the layers separate rather rapidly, so that equilibrium may be attained in 5 minutes or less. Therefore, we attempted to take an "immediate" reading as soon as possible after stopping the stirrer, wiping the paddle, and taking the graduate out of the bath—generally about 10 to 20 seconds after stopping the stirrer. It will be appreciated that these "immediate" observations, on a rapidly settling system, are not expected to be especially repeatable.

Three corrosion inhibitors, AFA-1, RP-2, and Unicor M, when tested in this manner at normal working concentrations, failed to give any emulsion persisting as long as 5 minutes. Of these three inhibitors two (RP-2 and Unicor M) have rather drastic effects in lowering the WSIM value of fuels, but the AFA-1 has little effect. This difference in WSIM was reflected to some extent in single-element filter-separator test results reported previously by SwRI.⁽¹⁷⁾

Further work on the demulsibility test was concentrated on blends containing Petronate L, a sodium petroleum sulfonate of the type used in soluble oils. It had been reported previously⁽¹³⁾ that this sulfonate was very harmful to filter-separator operation when present in the fuel at concentrations as low as 0.05-0.20 mg/liter. The 0.05 mg/liter concentration did not give any correspondingly drastic effect on fuel WSIM.

Initial demulsibility tests on Petronate L in JP-5 fuel (Table 27) failed to show any emulsifying characteristics whatever. Increasing the stirring time to 20 minutes resulted in measurable amounts of emulsion at the time of "immediate" observation, but this did not always persist for as much as 5 minutes. Changing the base stock to isooctane or Bayol/toluene did not change this situation significantly.

A combination of a 20-minute stirring time and a test temperature of 80°F appeared to accentuate the emulsification characteristics. Meanwhile, various fuel-water ratios had been tried, on the supposition that the phase ratio might be critical in determining emulsification characteristics. There were scattered, exploratory runs, and the results do not permit any generalizations as to the effect of phase ratio.

TABLE 27. RESULTS OF MODIFIED D 1401 DEMULSIBILITY TESTS

Base fuel*	Additive* and concn, mg/liter	Test temp, °F	Fuel-water ratio	Stirring time, min	Emulsion volume, ml		Emulsion vanish time, min	Remarks
					Immed	5-min		
JP-4	None	100	1/1	5	0	0	---	
JP-4	Dyes 140	100	1/1	5	1-10	0-8	5-30	7 tests
Bay-tol	AFA-1 34	100	1/1	5	0-1	0	<5	2 tests
JP-5	RP-2 20	100	7/1	5	1	0	<5	
JP-5	Uni M 26	100	7/1	5	1	0	<5	
JP-5	Uni M 26	100	3/1	5	6	0	<1	
JP-5	Pet L 0.20	100	7/1	5	0	0	---	
JP-5	Pet L 0.05	100	7/1	5	0	0	---	
JP-5	Pet L 0.10	100	7/1	5	0	0	---	
JP-5	Pet L 0.10	100	7/1	20	2	0	2	
JP-5	Pet L 0.10	100	1/1	20	5	0	2	
JP-5	Pet L 0.05	100	7/1	20	4	1	7	
Isooctane	Pet L 0.10	100	7/1	20	4	1	20	
Bay-tol	Pet L 0.10	100	7/1	20	2	0	1	
Bay-tol	Pet L 0.10	80	7/1	20	4-7	2-5	10-60	3 tests
Bay-tol	Pet L 0.20	80	7/1	20	8	4	>35	1-ml emulsion at 30 min
Bay-tol	Pet L 0.20	80	400/1	20	0	0	---	
Bay-tol	Pet L 0.20	80	80/1	20	<1	<1	85	
Bay-tol	Pet L 0.10	80	1/1	20	24-29	2	>60	2-ml emulsion at 3 min (2 tests)
Bay-tol	Pet L 0.05	80	1/1	20	20	1	15	
Bay-tol	Pet L 0.20	80	1/1	20	17	2	>20	2-ml emulsion at 3 min
Bay-tol	None	80	80/1	20	<1	<1	44	
Bay-tol	Pet L 0.20	Amb	1/1	4†	0	0	---	
Bay-tol	Pet L 0.20	Amb	79/1	2†	0	0	---	
Bay-tol	None	Amb	1/1	2†	0	0	---	
Bay-tol	Pet L 0.20	Amb	80/1	1‡	0	0	---	Cloudy fuel layer
Bay-tol	Pet L 0.20	Amb	1/1	1‡	0	0	---	Cloudy middle layer, 20 ml
Bay-tol	Pet L 0.20	Amb	1/1	2‡	0	0	---	Cloudy fuel and water layers
Bay-tol	Pet L 0.20	Amb	4/1	1-5‡	0	0	---	Cloudy fuel, partly cloudy water
Bay-tol	Pet L 0.20	Amb	80/1	10-20‡	0	0	---	

*Base fuel uninhibited JP-4 or JP-5, isooctane reference fuel, or 85/15 blend of Bayol R-34 and toluene. Additive oil-soluble Automate dye (blue, red, yellow), corrosion inhibitor AFA-1, RP-2, or Unicor M, or Petronate L petroleum sulfonate.
†In hand shaker.
‡In high-speed malted milk mixer, ca 17,000 rpm.

Considering only the last three tests on Petronate L in Bayol/toluene with 20 minutes of stirring at 80°F with a 1/1 phase ratio (Table 27), it will be noted that large volumes of emulsion were seen in the "immediate" observations, with 1-2 ml remaining after 5 minutes. Comparing the three concentrations of Petronate L tested under these conditions, the observations were as follows:

		Immediate emulsion, ml	5-min emulsion, ml	Emulsion vanish time, min
Petronate L,	0.05 mg/liter	20	1	15
	0.10 mg/liter	24-29	2	>60
	0.20 mg/liter	17	2	>20

Here the lower concentration of Petronate L clearly had less effect on water separation than did the two higher concentrations, which had equivalent effects so far as can be determined from these data.

It must be noted that the next test that was run, on Bayol/toluene without any additive, showed traces of very persistent emulsion. This would indicate contamination of the test fluid, possibly by carryover in the apparatus. In the standard ASTM procedure, the stirring paddle is cleaned between runs merely by wiping with solvent, and it appears quite possible that traces of a potent emulsifier such as the Petronate L could carry over from test to test.

A few tests were attempted with hand-shaking of the test mixture, without any success in producing emulsions. The use of a high-speed malted milk mixer produced measurable amounts of emulsion but introduced some problems in transfer of the fuel to the graduates after mixing and in warmup of the fuel/water mixture when mixing was prolonged. The mixtures prepared in this manner tended to maintain cloudy fuel or water layers, rather than any detectable amounts of "emulsion."

These preliminary results are very interesting in that they illustrate the possibility of producing measurable emulsions by mechanical agitation in the standard ASTM emulsion test apparatus. This is far from development of a valid or repeatable test. Even though the results are only moderately encouraging, further studies along this line appear to be in order, in view of the rather poor situation with the present separometer test.

11. MISCELLANEOUS EVALUATIONS

a. Fuel Antioxidant Evaluation

A new antioxidant proposed for use in JP-4 and other fuels has been evaluated in a long-term storage test. Specification testing of the fuel showed no detrimental effect from the antioxidant before or after storage. No detailed evaluations were made of the antioxidant performance of the material.

b. Effects of FSII on Polyurethane Foam

Soak tests have been run to compare the effects of FSII solutions, with and without the glycerol component, on open-cell polyurethane foam intended for use in aircraft fuel tanks. Aqueous solutions containing 25% FSII were used to simulate water bottoms encountered in fuel tanks. At the time these tests were run, the FSII consisted of 99.6% 2-methoxyethanol and 0.4% glycerol. This formulation was compared with straight 2-methoxyethanol, which has since become the new standard FSII.

Samples of the foam were stored at 130°F in 25% "old FSII" aqueous solution, 25% "new FSII" aqueous solution, and straight water. After periods of one to four months, none of the foam samples showed any obvious deterioration. After six months, all three samples had become so brittle that they would crumble at a touch.

A more detailed and critical evaluation has been reported by Scribner and Gandee⁽²³⁾, who concluded that the severe degradation of polyurethane foam previously noted with certain lots of ethylene glycol monomethyl ether (2-methoxyethanol) was caused by the presence of lead and/or tin at ppm levels of concentration. The presence of these metals was traced to storage of the 2-methoxyethanol in cans having soldered seams. Deterioration of the foam occurred whether or not the 2-methoxyethanol was formulated with glycerol.

In the SwRI work reported here, the 2-methoxyethanol and glycerol were both ACS reagent grade, furnished in glass containers and hence presumably free of any significant amounts of lead and tin. The only possible source of any contaminant metal was the metal lids of the glass jars used in the foam exposure tests. The lids of all three jars rusted during the tests, even though plastic liners were used, and internal condensation of liquid on the lids resulted in some contamination of the solutions. This contamination, presumably iron, may have had something to do with the deterioration of the foam that was observed after six months of exposure. The work of Scribner and Gandee did not show any pronounced effect of iron, but they pointed out that several metals (including lead, tin, and iron) did not show the foam-degrading effect when used in buffered

TABLE 28. BOILING POINT DISTRIBUTION
BY ASTM D 2887-70T

Sample: D. Reed; WPAFB B-9

Run #1 of 2		Date: 8/30/70					
Tray #2		Manual data entry 95					
% Off	Deg F	% Off	Deg F	% Off	Deg F	% Off	Deg F
IBP	83						
1	83	26	244	51	317	76	392
2	103	27	248	52	321	77	392
3	103	28	251	53	325	78	392
4	139	29	251	54	325	79	401
5	147	30	251	55	330	80	404
6	147	31	258	56	330	81	408
7	157	32	258	57	336	82	408
8	162	33	264	58	336	83	416
9	166	34	264	59	341	84	420
10	166	35	269	60	341	85	424
11	176	36	269	61	347	86	424
12	189	37	278	62	350	87	430
13	197	38	278	63	350	88	434
14	201	39	283	64	350	89	441
15	201	40	283	65	357	90	447
16	207	41	289	66	357	91	452
17	207	42	289	67	364	92	457
18	213	43	295	68	367	93	462
19	213	44	295	69	367	94	471
20	219	45	300	70	373	95	482
21	219	46	300	71	377	96	490
22	224	47	306	72	377	97	501
23	224	48	309	73	384	98	517
24	231	49	309	74	388	99	539
25	237	50	309	75	388	FBP	561

Run #2 of 2		Date: 8/30/70					
Tray #2		Manual data entry 95					
IBP	83						
1	83	26	245	51	315	76	393
2	103	27	249	52	321	77	393
3	103	28	252	53	325	78	398
4	139	29	252	54	325	79	398
5	147	30	252	55	330	80	405
6	147	31	258	56	330	81	408
7	158	32	258	57	336	82	412
8	162	33	264	58	336	83	412
9	166	34	267	59	341	84	420
10	166	35	267	60	341	85	424
11	177	36	267	61	347	86	424
12	190	37	278	62	350	87	430
13	197	38	278	63	350	88	435
14	202	39	284	64	350	89	441
15	202	40	284	65	358	90	447
16	207	41	290	66	361	91	453
17	207	42	293	67	361	92	458
18	213	43	293	68	367	93	463
19	218	44	293	69	367	94	471
20	218	45	301	70	374	95	482
21	218	46	301	71	377	96	491
22	225	47	306	72	377	97	501
23	225	48	310	73	385	98	516
24	232	49	310	74	388	99	538
25	238	50	315	75	388	FBP	559

solutions. Evidently, any critical evaluation of the effect of iron on the degradation of polyurethane foam in 2-methoxyethanol would require a rather extensive investigation.

c. Plastic-Lined Tank Evaluation

Fuel tanks lined with ABS (acrylonitrile-butadiene-styrene) plastic were investigated for deterioration during storage of isooctane-toluene mixtures. After six months of storage, the isooctane-toluene mixture was found to be contaminated with traces of a nonvolatile material. This was found to be identical to a plastic component obtained in the laboratory by Soxhlet extraction of a sample of the ABS.

d. Gas Chromatography for Simulated Distillation

Recent developments in this area have been followed closely, with some thought of setting up the necessary equipment and evaluating the technique for Air Force applications.

The major published work in this area is that of Green.⁽²⁴⁾ ASTM Committee D-2 has developed a method for "Boiling Range Distribution of Petroleum Fractions by Gas Chromatography" that is appearing in the 1970 Book of Standards as an ASTM Tentative Method.

The advantages of the gas chromatographic method over an ASTM D 86 are in more precise determination of initial and final boiling points, more detailed distribution data, and more precise prediction of true-boiling-point distillation curves. If fully automated, it also has significant advantages in operating costs in comparison with nonautomated distillation apparatus.

As applied to JP-4 fuels the simulated distillation apparatus requires subambient temperature operation and control. This adds to the cost and has made it infeasible to investigate the technique with existing equipment.

Outfitting the present equipment for occasional analyses could be accomplished for about \$6,000. A completely automated system, including computer, is marketed by Hewlett-Packard for \$33,000. It was concluded that the present interests do not justify these costs.

As a matter of interest, we are including here data sheets illustrating the type of analyses that can be produced. Table 28 is a reproduction of computer printouts furnished by Hewlett-Packard from analyses of duplicate samples of a JP-4 fuel.

SECTION VI

OPTICAL TECHNIQUES FOR CHEMICAL ANALYSIS

1. GENERAL

As reported previously⁽²⁵⁾, preliminary studies of the Kerr effect phase shift had demonstrated in principle the feasibility of applying this phenomenon to chemical analysis, particularly the analysis of synthetic lubricants. Further development of this technique into a practical tool would still require a considerable amount of work. It was decided to devote efforts instead to other approaches for which the groundwork was already available.

Results from a preliminary study of fluorescence spectra have shown this approach to be a highly sensitive and selective analytical tool for certain components of synthetic lubricants. The results from this work have been presented to the Air Force in an informal technical report.

Subsequently, several samples of synthetic lubricant formulations were furnished by the Aero Propulsion Laboratory for a study of how various techniques could be applied to identification of materials and detection of batch variations.

These samples were studied by nuclear magnetic resonance (NMR), optical absorption, fluorescence, and phosphorescence spectroscopy, with two objectives in mind. The first objective was to identify, using various spectroscopic techniques, the different lubricant formulations of current interest. The second objective was to identify spectral variations in different batches of the same formulations.

2. SAMPLES AND SPECTROSCOPIC TECHNIQUES

The synthetic lubricants that were studied included two pairs of samples, each pair representing two different batches of a given formulation, and four other samples representing different formulations. The pairs are identified as E-1 and E-2 (here further termed "a" batches) and F-1 and F-2 (here termed "b" batches). The four other formulations were identified as A, B, C, and D.

Spectra were run on these samples using a Varian A-60 (60 MHz) NMR spectrometer, a Cary 14 spectrophotometer, and a Baird Atomic SF-100 fluorescence/phosphorescence spectrophotometer. The spectra were taken in standard ways, the NMR spectra being from pure or neat samples, the optical absorption spectra being from 10.0-0.02% (v/v) dilutions in spectroquality solvents, and the luminescence spectra from 0.1% and 0.01% (v/v) dilutions in low-background ethanol or cyclohexane.

3. NMR SPECTRA

The NMR spectra consist of rf absorption as a function of magnetic field, referenced to tetramethylsilane (0 cps). The magnetic field is given in cps through the relation $\nu(\text{cps}) = \gamma H$ (gauss), where $\gamma = 4257.7$.

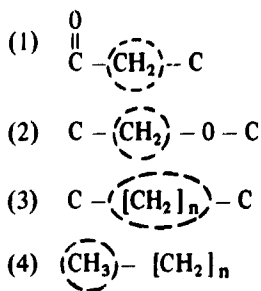
The NMR spectra of the formulations A-D are superficially similar consisting of four major absorption regions.

- (1) \approx 240 cps
- (2) \approx 135 cps
- (3) \approx 75 cps
- (4) \approx 55 cps

Of these regions, A and D show the most dramatic differences between formulations, with C differing in region (2) as well.

These spectra have been broadly interpreted as follows:

TABLE 29. NMR INTEGRALS (IN %)



Formulation or batch	Spectral region			
	(1)	(2)	(3)	(4)
A	9.13	11.6	54.8	25.0
B	9.86	12.7	53.5	22.5
C	7.75	9.48	45.7	36.6
D	8.0	10.3	57.7	23.1
E-1	12.8	14.5	51.7	20.6
E-2*	12.6	13.8	52.1	21.1
F-1	10.6	10.9	48.1	34.8
F-2†	10.7	11.1	48.8	34.4

where the circled group is thought to be responsible for absorption in the region indicated.

In order to facilitate comparison of these assignments with known concentrations of esters, the integrals of the absorption spectra have been measured as well. These have been tabulated for the different regions as Table 29. In Table 29, 10% means that 10% of the protons resonating in the region covered are of that type. Aromatic protons were not included in the integrals.

The NMR spectra of the "a" batches show very small differences except for a new band at ≈ 195 cps. E-1 has a very small peak in this region, and E-2 a larger one. Finally, an old sample of E-2 has a considerably larger peak in this region. The "a" batches do not correlate exactly to any of the first four formulations, but are most similar [significant differences show in region (1)] to D. The "b" batches are virtually identical to C.

4. VISIBLE AND UV ABSORPTION

The absorption spectra were taken on the Cary 14 spectrophotometer as optical density ($-\log_{10} T$, $T = I/I_0$) vs wavelength in $m\mu$. The lubricant samples were diluted 1:200 v/v in cyclohexane and measured in quartz cells with 1 cm path length for visible, 1 mm for UV. The spectrometer was zeroed against air (-0.03 D.U. correction factor), and all spectra were run vs air. The differential spectra were measured by adjusting the concentration to give appreciable slit opening at the absorption peak of interest with maximum gain setting on the spectrometer. The relatively large baseline drift precluded use of the 0.0-0.1 slide wire, so all spectra were taken with the 0.0-1.0, 1.0-2.0 slide wire.

The formulations A and D have very similar spectra. B appears to differ from A and D in relative concentrations of the substance responsible for the major UV peak. C has a UV absorption system that is superficially similar to A and D but the highest energy band is narrower and shifted from the same band in A and D. Also, C has a prominent absorption system in the visible. This band is highly structured.

The batches "a" and "b" have a completely different absorption spectrum from the other four formulations.

5. FLUORESCENCE

The fluorescence spectra were taken by exciting the solution (usually 1:1000 v/v dilution of lubricant) at a particular wavelength and scanning the emitted light. The exciting wavelength was then changed and the emission spectrum obtained again. The spectra are uncorrected for the spectra response of lamp and detector and hence show shifts toward longer wavelengths. The lamp is a 150 watt Zenon arc; the detector is an RCA 1P28 photomultiplier. Each spectrum is standardized with respect to gain changes via arc shifts, voltage drops, etc by measuring the fluorescence of a solution of rhodamine (Dupont Rhodamine 5GDN Extra, Lot 375, 0.03% w/v in H_2O , 1:20 v/v dilution). This standard was stored overnight in water to prevent evaporation and changed periodically.

The spectra generally showed at least two fluorescent compounds. Since A and D had very similar optical spectra, they were treated as batches in the luminescence work. Their spectra show three species. Again, the spectra

were very similar, quantitative differences showing up in two emissions. B shows three species in emission; a shoulder and a shape change in the second band.

6. PHOSPHORESCENCE

The phosphorescence spectra were taken from 1:100 v/v dilutions of the lubricant in ethanol. The samples were put in 1 mm ID (nominal) quartz cells and frozen in liquid nitrogen. The tail of the LN₂ dewar was inserted in the rotating can chopper of the phosphorimetry attachment made for the Baird Atomic Fluorispec. In the spectra presented here, no attempt was made to deoxygenate the samples or to use the phosphorescence lifetime to increase resolution. As in fluorescence, the exciting wavelength was set and noted, and the spectrum of the emitted light was scanned.

Again, the formulations A and D were treated as batches because of the great similarity in their optical spectra. Two emission peaks were overlapping. A third emission appears with near UV excitation. The spectra show some shape differences suggesting an extra component in A. Mainly, the two formulations appeared identical, neglecting quantitative differences.

The phosphorescence spectrum of B showed three emission peaks. Formulation B is distinguishable from A and D on the basis of the visible emission, this having approximately twice the intensity in B.

The phosphorescence spectrum of C is distinctive, emission peaks being shifted from the others. Also, an additional narrow band appears.

The phosphorescence spectrum of the "a" and "b" batches showed the most distinctive differences in species which emit at the same wavelength. Hence these spectra were run in the reverse manner, setting the emission wavelength and scanning the excitation spectrum. These spectra show large quantitative differences in two peaks.

7. DISCUSSION

The work presented here had two goals. The first goal was to establish the feasibility of "fingerprinting" the various formulations for quick identification. The second was to establish whether or not the various techniques could be used to detect batch-to-batch variations and would, therefore, be useful in quality control. The NMR

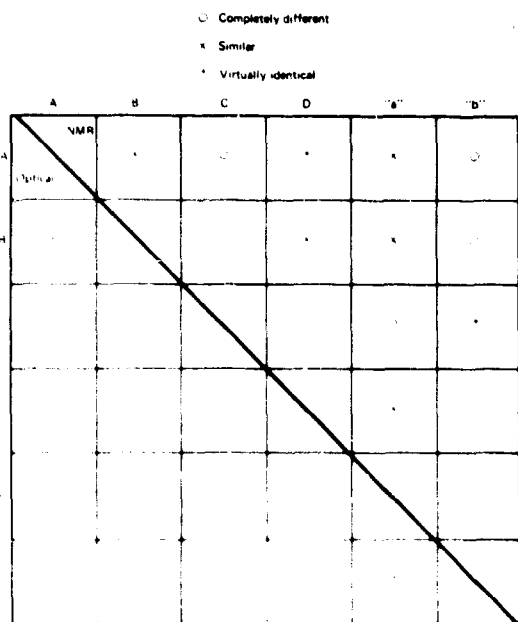


FIGURE 26. LUBRICANT FINGERPRINTING BY NMR AND OPTICAL SPECTROSCOPY

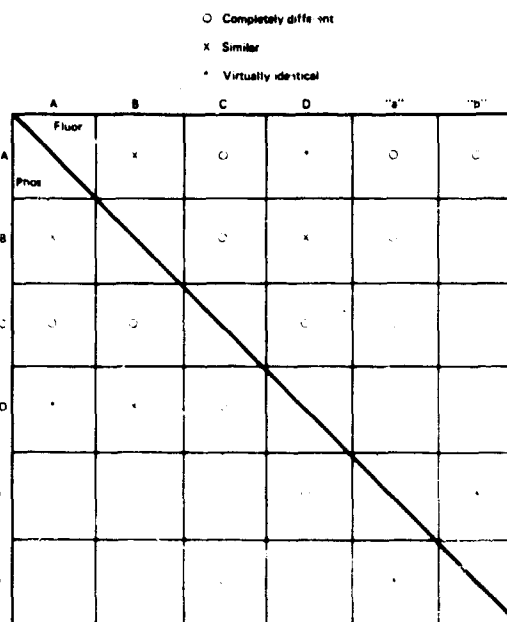


FIGURE 27. LUBRICANT FINGERPRINTING BY FLUORESCENCE AND PHOSPHORESCENCE

spectra obtained indicate that NMR is suitable for "fingerprinting" unless C or the "b" batches are involved. In such an application, the NMR should be followed by optical absorption to establish firmly the identification. Also, the differences between A and D in region (1) should be checked on several samples of each formulation to establish firmly the identification. A summary of the qualitative aspects of the spectra is given in Figure 26.

The qualitative features of optical (visible and UV) absorption spectra as a fingerprint technique are also summarized in Figure 26. Here, A and D are indistinguishable, requiring NMR verification, and the "a" and "b" difference is small.

TABLE 30. DETECTION OF LUBRICANT BATCH VARIATION

	NMR	Optical absorption differential	After UV irradiation	Fluorescence	Phosphorescence
"a" E-1 E-2	O	*	O	X	X
"b" F-1 F-2	8	O	X	X	X
O—Completely different. X—Similar. *Virtually identical.					

In Figure 27, the "fingerprinting" characteristics of fluorescence and phosphorescence are shown. The general trend is with the optical absorption data, so that these techniques appear to offer no current advantage in fingerprinting. However, since a single additive is likely to dominate the absorption spectrum, a change in another additive at a later date could be shown up in fluorescence or the phosphorescence and not in absorption. Our experience with the batches indicates this order in selectivity:

1. Phosphorescence
2. Fluorescence
3. Optical absorption

The results of our investigation into batch variations is given in Table 30. The NMR shows a new peak in one of the "a" batches, and the optical absorption shows differences after light and heat treatment. The fluorescence shows an extra peak in E-2, apparently confirming the NMR observation of an extra component in E-2. Phosphorescence shows quantitative differences in the spectra of both "a" and "b" batches.

8. CONCLUSIONS

For current formulations, the use of NMR and optical absorption together provides a positive means of identifying the various formulations, i.e., "fingerprinting." The two techniques provide a method of investigating both base stock and additives. However, since the optical absorption usually allows observation of only one or two major additives, there may be a need to use more selective techniques at a later date.

The observation of a new peak via NMR in the batch investigation is encouraging, as is the quantitative variation observed in the optical absorption and luminescence. Confirmatory work on these observations is under way.

SECTION VII

TURBINE ENGINE INSTRUMENTATION AND CONTROL

A research and development program for dynamic testing and control loop evaluation of a propulsion system has been defined. As a result of a systems study, the methods of approach, potential problem areas, required instrumentation, and required major sub-systems have been established. The primary work effort has been directed toward the development of an instrumentation and data acquisition system which will allow a J-85-7 test engine to be interfaced with an IBM 1800 process control computer. This will provide great flexibility for performing dynamic engine testing and control loop evaluations.

In order to evaluate control loop concepts more comprehensively, it is necessary to look more into the details of the engine operation by monitoring and examining many thermodynamic, aerodynamic and mechanical variables. It is with this need in mind that a multichannel data acquisition and external control system is being implemented. Data are to be collected from the engine using numerous sensors which provide analog readout of the monitored variable. As desired, these data may be (a) processed and examined on line, (b) taped for future examination, (c) used to implement some new pure analog control loop concept, (d) coded into digital signals and fed into the IBM 1800, and used to implement some hybrid control concept using the IBM 1800 as part of the control loop. Ideally, any combination of the above data handling methods may be implemented, as desired, so the system offers an immense amount of flexibility for future dynamics and control studies.

The instrumentation for the J-85 engine will consist basically of numerous static and total-pressure probes (with both dynamic and quasi-steady capability), hot wires, and thermocouples. This basic monitoring system will be supplemented by fluidic temperature sensors for turbine inlet temperature measurement, plus other miscellaneous probes as required. A detailed plan of transducer location on the engine has been formulated, and a number of probes have already been built.

The high-frequency pressure transducers will be short-coupled Kistlers and Kulites. Water-cooled jackets will be used on those probes placed in hot-gas flows. Additional high-frequency data will be obtained from hot wires placed in the first, third, fifth and seventh stages of the compressor. Temperatures will be measured with standard and special purpose thermocouples.

High-frequency data will be FM multiplexed and stored on magnetic tape. The Honeywell 7600 and Ampex FR 1600 wideband tape recorders will be incorporated into the system which will allow constant bandwidth operation. Tape speed compensation techniques will also be employed.

The central building block of the instrumentation system will be an IBM 1800 process control computer operated in the supervisory mode. The low-frequency data will be time division multiplexed, and high-frequency data will be FM multiplexed stored on magnetic tape for subsequent data reduction by the 1800 and other special purpose gear. It is anticipated that the 1800 can take over the function of the fuel controller of the J-85 engine for special hybrid control investigations. This will allow an operator to program arbitrary control schedules (i.e., control strategies) which will be, in general, dependent upon new state-of-the-art sensors, a performance index, and possibly some optimization index. The computer can be programmed to initiate the control function.

The variable geometry components have been isolated from the mechanical fuel control. A servo loop allowing external control of the J-85 inlet guide vane angle has been designed and the system transfer function has been experimentally derived. In order to actuate the bleed valves, independent of the fuel control, the hydraulic actuators normally used on the engine will be incorporated in another hydraulic servo loop similar to the one used for the inlet guide vanes.

During the first year of this contract, effort was concentrated on developing flow instrumentation for compressors, and particularly on developing and evaluating improved sensors. The second-year effort has been directed toward the development of a facility to do meaningful research in the area of engine dynamics.

Detailed information on these developments will be presented separately in a Technical Report describing accomplishments to date in the field of turbine engine instrumentation and control.

SECTION VIII

INFORMATION RETRIEVAL SYSTEM

1. GENERAL

SwRI has continued to operate and broaden the information retrieval system which is maintained as a part of this program. The information system was an outgrowth of an obvious need to centralize and conveniently catalog, for ready reference, the mass of technical literature of interest to the Fuels, Lubrication, and Fire Protection Branches of the Fuels and Lubrication Division of the Aero Propulsion Laboratory. The retrieval system provides the APL engineering staff, and other groups authorized access to the system, with a rapid and convenient means of obtaining documents contained in the system. Further, it eliminates the necessity for each engineer to establish and maintain his personnel reference and information file, which often becomes both cumbersome and inadequate. Currently, the system holdings stand in excess of 8,000 documents, of which some 2,000 are on microform. Because much of the technical literature is now available only on microform, suitable microfilm and microfiche readers and printers are conveniently located for system users and are maintained as part of the information system responsibility.

The information retrieval system is established on the Concept Coordination principle. That is, each document placed in the system is assigned an accession number, and by use of the Royal Keydex punch card system, each accession number is related to index terms and key words, which in turn allow the speedy recovery of all documents having a common subject relationship. In addition, a title index, author index, report number index, and a contract number index are maintained to provide additional means of locating directly a specific document.

The information system consists primarily of technical reports embracing or relating to fuels, lubricants, and hazards studies. A limited number of journal articles considered of significance in the subject areas have also been placed in the information system, but books have been excluded. As a further aid to the system user, an abstract file is readily available wherein each system holding has been abstracted for quick assessment of the documents' subject matter.

The information retrieval system contains classified documents up to and including Secret, and appropriate security measures have been established for the handling of these documents. These procedures have been set forth to enable properly authorized persons convenient access to classified documents, while at the same time providing for compliance with all applicable security regulations.

2. SYSTEM OPERATION

The major retrieval system activity in 1970 was accomplishing the total incorporation of the "Fire Protection" documents in the central system. For a time, the users in this group felt that their needs could best be served by a separate handling of the Fire Protection literature, and initially, these documents were maintained separately. It was later agreed that, in order for the Fire Protection Branch to avail itself of all the services the information retrieval system could provide, it would be necessary to handle these documents within the system in a manner paralleling the handling of the Fuels and Lubrication documents. Consequently, the task of assimilating close to 1000 Fire Protection documents was begun, while at the same time continued attention was given to the acquisition of new documents in all three technical areas.

The complete incorporation and centralizing of the Fire Protection documents has already been found to be both efficient and effective. The only remaining operation is completing the Keydex file for these documents, which will be accomplished in the very near future.

To date, the information retrieval system has satisfactorily filled the needs for information and specific documents in the Fuels, Lubrication, and Fire Protection Branches. Further technical literature in these areas of interest is reviewed on a continuing basis, and any document considered of interest is promptly ordered. Naturally, documents requested by staff members of the branches are also obtained promptly.

Periodically, a list of new acquisitions is circulated among the APL engineering staff to assist them in keeping abreast of documents on hand. Almost all of the requests for general information or specific documents have been filled from system holdings; in those few cases when requested material was not in the system, immediate action was taken to obtain it.

For the past year (excluding the large transfer of Fire Protection documents), system acquisition rate has averaged approximately 30 documents per month. The general coverage of the technical areas is considered most adequate at this time, and no significant changes are contemplated in the system itself or in the method of handling.

APPENDIX

TABLE 31. FUEL COKER TESTS ON VARIOUS FUELS

Test temperatures for research coker are reservoir/preheater/filter

Test temperatures for other cokers are preheater/filter

Fuel paper-filtered for standard coker, 0.45 micron for others

All gas-drive coker tests in cokers 1 and 2 with CRC configuration

Fuel	Coker type	Test temp, °F	Tube rating		Filter* ΔP, in. Hg	Coker test no.	Date tested
			Unwiped	(Wiped)			
JP-7 Fuel G	Research	300/500/600	2	(2)	0.0	6016	11 Mar 70
JP-7 Fuel G	Gas-drive	625/700	<3	(2)	0.0	6066	27 Mar 70
		625/700	2	(2)	0.0	6063	27 Mar 70
		650/700	<4	(<4)	0.0	6053	25 Mar 70
		650/700	2	(2)	0.0	6054	25 Mar 70
		650/700	2	(2)	0.0	6057	26 Mar 70
		650/700	<4	(3)	0.0	6060	26 Mar 70
		700/700	>4	(>4)	0.0	6049	24 Mar 70
		700/700	>4	(>4)	2.5	6051	24 Mar 70
		AFFB-12-68	Research	300/500/600	2	(2)	0.0
JP-8 Fuel B	Standard	300/400	1	(1)	0.2	5875	14 Jan 70
		300/400	1	(1)	0.3	5879	15 Jan 70
JP-7 Fuel C	Research	300/500/600	2	(2)	0.8	6011	10 Mar 70
PF-1	Research	300/500/600	<3	(<3)	0.0	6000	5 Mar 70
PF-1	Gas-drive	625/700	2	(2)	0.0	6150	1 May 70
		625/700	2	(2)	0.0	6151	4 May 70
		650/700	3	(2)	0.0	6140	27 Apr 70
		650/700	2	(2)	0.0	6144	28 Apr 70
		650/700	3	(2)	0.0	6147	30 Apr 70
		675/700	4	(3)	0.0	6146	29 Apr 70
PF-1A	Research	300/500/600	2	(2)	0.0	6004	6 Mar 70
PF-1A	Gas-drive	600/700	2	(2)	0.0	5809	9 Dec 69
		625/700	4	(3)	0.0	5807	8 Dec 69
		625/700	1	(1)	0.0	5810	9 Dec 69
		650/700	2	(2)	0.0	5806	8 Dec 69
		650/700	2	(2)	0.0	5812	10 Dec 69

TABLE 31. FUEL COKER TESTS ON VARIOUS FUELS (Cont'd)

Fuel	Coker type	Test temp. °F	Tube rating		Filter* ΔP, in. Hg	Coker test no.	Date tested
			Unwiped	(Wiped)			
PF-1A (cont'd)		675/700	1	(1)	0.0	5814	10 Dec 69
		675/700	3	(<3)	0.0	5819	12 Dec 69
		675/700	2	(1)	0.0	5818	15 Dec 69
		675/700	2	(1)	0.0	5821	15 Dec 69
		700/700	3	(3)	0.0	5815	11 Dec 69
		700/700	<3	(<3)	0.0	5816	11 Dec 69
JP-4 Fuel H	Standard	300/400	1	(1)	6.7	5966	20 Feb 70
		300/400	1	(1)	3.8†	5973	25 Feb 70
JP-4 Fuel H	Gas-drive	250/350	2	(2)	0.1	6018	12 Mar 70
		275/375	1	(1)	0.3	6020	12 Mar 70
		275/375	2	(2)	0.3	6023	13 Mar 70
		275/375	2	(2)	1.1	6047	23 Mar 70
		300/400	1	(2)	16.9	6017	11 Mar 70
		300/400	1	(1)	0.4	6022	13 Mar 70
		300/400	2	(2)	4.8	6041	20 Mar 70
		300/400	1	(1)	2.4	6045	23 Mar 70
		350/450	1	(1)	8.9	6014	11 Mar 70
		JP-6 Fuel D	Standard	425/525	2	(1)	1.2
JP-5 Fuel E	Standard	300/400	1	(1)	1.1	5959	18 Feb 70
		400/500‡	4	(4)	25.0	5954	17 Feb 70
JP-4 Fuel F	Standard	300/400	1	(1)	0.4	5923	3 Feb 70
		300/400	1	(1)	0.7	6385	3 Aug 70
JP-4 SA	Standard	300/400	>4	(>4)	2.4	5991	3 Mar 70
JP-4 SA	Gas-drive	300/400	1	(1)	0.0	5975	26 Feb 70
		350/450	1	(1)	0.1	5974	26 Feb 70
		350/450	1	(1)	1.3	5987	2 Mar 70
		375/475	2	(1)	25.0/106	5981	27 Feb 70
		375/475	1	(1)	25.0/151	5986	2 Mar 70
		400/500	2	(2)	25.0/52	5971	25 Feb 70
		400/500	1	(1)	25.0/51	5978	27 Feb 70
		450/550	>4	(>4)	25.0/47	5972	25 Feb 70

TABLE 31. FUEL COKER TESTS ON VARIOUS FUELS (Cont'd)

Fuel	Coker type	Test temp, °F	Tube rating		Filter* ΔP, in. Hg	Coker test no.	Date tested		
			Unwiped	(Wiped)					
JP-7, 70-2546	Research	300/500/600	2	(2)	0.0	6172	14 May 70		
JP-7, 70-16	Research	300/500/600	1	(1)	0.0	6169	13 May 70		
JP-7, 70-14, 13	Research	300/500/600	2	(1)	2.9	5952	16 Feb 70		
JP-7, JFA5	Research	300/500/600	2	(2)	0.0	5967	24 Feb 70		
JP-7, NRSX-1	Research	300/500/600	1	(1)	0.0	6224	8 Jun 70		
JP-7, NRSX-2	Research	300/500/600	2	(2)	0.0	6234	10 Jun 70		
AFFB-13-69, #6	Standard	350/450	2	(1)	1.0	5723	21 Oct 69		
		375/475	1	(1)	2.3	5722	20 Oct 69		
		400/500**	2	(2)	13.0	5719	15 Oct 69		
		425/525	4	(4)	15.8	5721	17 Oct 69		
		425/525	2	(2)	2.5	5724	22 Oct 69		
		425/525	2	(2)	1.6	5725	23 Oct 69		
		425/525	<3	(1)	3.8	5742	31 Oct 69		
		450/550	3	(<3)	25.0/264	5720	16 Oct 69		
		AFFB-13-69, #7	Standard	325/425	2	(1)	0.4	5799	3 Dec 69
				350/450	2	(1)	2.1	5796	2 Dec 69
350/450	2			(1)	0.3	5801	4 Dec 69		
350/450	2			(1)	0.3	5817	11 Dec 69		
375/475	2			(1)	4.0	5791	1 Dec 69		
375/475	2			(1)	0.5	5805	5 Dec 69		
375/475	2			(1)	0.4	5820	12 Dec 69		
400/500	2			(1)	1.6	5808	8 Dec 69		
425/525	2			(1)	8.3	5782	25 Nov 69		
425/525	<3			(2)	0.7	5811	9 Dec 69		
AFFB-10-67	Research	450/550	<3	(1)	2.3	5785	26 Nov 69		
		450/550	<3	(1)	1.0	5787	28 Nov 69		
		450/550	<3	(2)	0.9	5813	10 Dec 69		
		300/375/475	1	(1)	0.2	5726	23 Oct 69		
		300/375/475	1	(1)	0.2	5729	27 Oct 69		
		300/375/475	1	(1)	0.0	5735	29 Oct 69		
		300/375/475	1	(1)	0.1	5741	31 Oct 69		

TABLE 31. FUEL COKER TESTS ON VARIOUS FUELS (Cont'd)

Fuel	Coker type	Test temp, °F	Tube rating		Filter* ΔP, in. Hg	Coker test no.	Date tested
			Unwiped	(Wiped)			
AFFB-10-67 (cont'd)		300/400/500	1	(1)	0.0	5727	24 Oct 69
		300/400/500	1	(1)	0.0	5731	28 Oct 69
		300/400/500	1	(1)	0.0	5740	30 Oct 69
		300/400/500	1	(1)	0.1	5743	3 Nov 69
TS, 70A, 77A, 79A	Standard	450/550	2	(1)	0.4	5884	16 Jan 70
		450/550	2	(1)	0.3	5888	19 Jan 70
		450/550	2	(2)	7.2	5928	5 Feb 70
		450/550	2	(2)	7.5	5931	6 Feb 70
TS, 82A, 86A, 87A	Standard	425/525	4	(4)	0.4	5869	13 Jan 70
		450/550	4	(4)	0.6	5868	12 Jan 70
TS, 82A, 86A, 87A	Gas-drive	375/475	1	(1)	0.0	5864	9 Jan 70
		400/500	1	(1)	0.0	5865	9 Jan 70
		400/500	2	(1)	0.0	5867	12 Jan 70
		425/525	4	(4)	0.0	5861	8 Jan 70
		425/525	1	(1)	0.0	5866	12 Jan 70
		425/525	<4	(1)	0.0	5870	13 Jan 70
		425/525	4	(2)	0.0	5871	13 Jan 70
		450/550	4	(3)	0.0	5863	8 Jan 70
		475/575	4	(2)	0.0	5858	7 Jan 70
		500/600	4	(2)	0.0	5859	7 Jan 70
		575/675++	4	(4)	0.0	5855	6 Jan 70
		600/700++	<4	(2)	0.0	5856	6 Jan 70
		675/700++	>4	(>4)	0.0	5853	5 Jan 70
675/700++	>4	(>4)	0.0	5854	5 Jan 70		

*Pressure drop at 300 minutes unless otherwise noted.
†Prefiltration of test fuel for this test was by 0.45μ Millipore instead of Whatman 2V.
‡Preheater warm-up time 101 minutes.
§Preheater warm-up time 41 minutes.
¶Fuel boiled; test pressure 270 psi.

TABLE 32. STANDARD COKER RATINGS ON FUELS FOR JFTOT PROGRAM

Run no.	Test temp. °F	Filter* ΔP, in. Hg	Rating	Deposit inception ratings, 1-hr (and 24-hr)			
				1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-3-64 fuel</i>							
5736	450†/550	9.7	Code 1, °F	239† (242†)	550 (540)	432 (544)	434 (240)
			Code 2, °F	603 (604)	584 (553)	579 (590)	582 (586)
			Code 3, °F	---	603 (604)	602	---
			Code 4, °F	---	---	---	---
			Max code	<3 (2)	3 (3)	3 (2)	2 (2)
5750	450/550	25.0/279	Code 1, °F	203† (216†)	209 (203)	203† (375)	196 (196)
			Code 2, °F	567† (567†)	568 (568)	563 (568)	569†
			Code 3, °F	---	---	---	---
			Code 4, °F	---	---	---	---
			Max code	2 (3)	<3 (2)	<3 (2)	2 (3)
5758	450/550	24.3	Code 1, °F	239 (242†)	379 (355)	242 (196)	196 (196)
			Code 2, °F	601 (577)	526 (531)	531 (531)	579 (565)
			Code 3, °F	---	---	---	---
			Code 4, °F	---	---	---	---
			Max code	<3 (<3)	2 (<3)	<3 (<3)	2 (2)
5769	450**/550	25.0/282	Code 1, °F	501 (203)	203 (196††)	203 (196)	203 (203)
			Code 2, °F	546 (547)	502 (502)	430† (429†)	357† (538)
			Code 3, °F	---	---	---	---
			Code 4, °F	---	---	---	---
			Max code	2 (2)	<3 (2)	<3 (<3)	2 (2)
<i>AFFB-4-64 fuel</i>							
5748	325/425	0.0	Code 1, °F	380 (383)	382 (338†)	---	382 (383)
			Code 2, °F	450 (330†)	---	427 (383)	324*** (326***)
			Code 3, °F	---	---	319*** (335***)	---
			Code 4, °F	---	---	---	---
			Max code	2 (2)	<2 (2)	3 (3)	2 (2)

TABLE 32. STANDARD COKER RATINGS ON FUELS FOR JFTOT PROGRAM (Cont'd)

Run no.	Test temp., °F	Filter* ΔP, in. Hg	Rating	Deposit inception ratings, 1-hr (and 24-hr)			
				1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-4-64 fuel (cont'd)</i>							
5732	350/450	0.3	Code 1, °F	329 (379)	335 (356)	335 (350)	163 (163)
			Code 2, °F	459 (456)	454 (424)	449 (477)	471 (479)
			Code 3, °F	---	---	---	---
			Code 4, °F	483 (493)	482 (483)	483 (483)	483 (482)
			Max code	4 (4)	4 (4)	4 (4)	4 (4)
5760	350/450	0.4	Code 1, °F	329 (382)	323 (290)	323 (290)	323 (290)
			Code 2, °F	444 (451)	421 (368)	449 (413)	446 (454)
			Code 3, °F	480 (475)	454 (451)	475 (475)	---
			Code 4, °F	---	---	---	475 (475†)
			Max code	3 (3)	<4 (<4)	3 (<4)	4 (4)
5773	350/450	0.3	Code 1, °F	240 (163)	254 (163)	163 (168)	338 (379)
			Code 2, °F	388 (445)	382 (447)	272 (300)	269††† (268†††)
			Code 3, °F	---	---	---	---
			Code 4, °F	477	469	---	---
			Max code	>4 (<3)	4 (2)	3 (3)	<3 (<3)
<i>AFFB-8-67 fuel</i>							
5754	400†/500	0.0	Code 1, °F	338 (338)	181 (181)	334 (338)	338 (336)
			Code 2, °F	---	---	---	---
			Code 3, °F	---	---	---	---
			Code 4, °F	1 (1)	1 (1)	1 (1)	1 (1)
			Max code	3 (3)	3 (3)	2 (3)	<4 (<4)
5728	425/525	1.3	Code 1, °F	547 (570)	475† (469†)	570	570 (570)
			Code 2, °F	566 (573)	573 (573)	570 (189)	572 (573)
			Code 3, °F	571 (572)	572 (572)	---	572 (572)
			Code 4, °F	---	---	---	---
			Max code	3 (3)	3 (3)	2 (3)	<4 (<4)

TABLE 32. STANDARD COKER RATINGS ON FUELS FOR JFTOT PROGRAM (Cont'd)

Run no.	Test temp., °F	Filter* ΔP, in. Hg	Rating	Deposit inception ratings, 1-hr (and 24-hr)			
				1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-8-67 fuel (cont'd)</i>							
5744	425/525	1.6	Code 1, °F	538 (481)	537 (539)	478 (478)	566 (569)
			Code 2, °F	(546)	---	569 (568)	---
5763	425/525	5.6	Code 3, °F	---	---	---	---
			Code 4, °F	570 (570)	570 (570)	570 (570)	570 (570)
			Max code	>4 (>4)	>4 (>4)	4 (>4)	4 (>4)
			Code 1, °F	236 (236)	195 (195)	195 (380)	189 (189)
			Code 2, °F	400 (419)	380 (499)	380 (508)	523 (520)
			Code 3, °F	523	523 (520)	520 (520)	534 (525)
			Code 4, °F	531 (524)	534 (534)	535 (532)	538 (542)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
<i>AFFB-9-67 fuel</i>							
5746	375/475	5.0	Code 1, °F	383 (389)	435 (367)	439 (386)	377 (389)
			Code 2, °F	---	---	---	---
5730	400/500	22.4	Code 3, °F	---	---	---	---
			Code 4, °F	---	---	---	---
			Max code	1 (1)	<2 (1)	<2 (1)	1 (2)
			Code 1, °F	502 (475)	505 (494)	449 (406)	450 (443)
			Code 2, °F	516 (541)	512 (512)	505 (494)	518 (509)
			Code 3, °F	---	541 (541)	541	541 (541)
			Code 4, °F	542 (542)	542 (542)	542 (542)	542 (542)
			Max code	4 (>4)	>4 (>4)	4 (>4)	>4 (>4)
5756	400/500	20.6	Code 1, °F	474 (475)	210 (374)	369 (385)	449 (403)
			Code 2, °F	390*** (318****)	187**** (425)	532 (516)	422*** (527)
			Code 3, °F	---	---	538 (533)	533 (540)
			Code 4, °F	533 (540)	533 (533)	540 (540)	540 (540)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)

TABLE 32. STANDARD COKER RATINGS ON FUELS FOR JFTOT PROGRAM (Cont'd)

Run no.	Test temp., °F	Filter* ΔP in. Hg	Rating	Deposit inception ratings, 1-hr (and 24-hr)			
				1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-9-67 fuel (cont'd)</i>							
5766	400/500	25.0/184.6	Code 1, °F	479 (483)	479 (472)	374 (383)	403 (438)
			Code 2, °F	---	534 (526)	508 (513)	529 (533)
			Code 3, °F	---	540 (535)	535 (533)	540 (540)
			Code 4, °F	542 (541)	541 (540)	541 (540)	541 (541)
			Max code	4 (4)	>4 (>4)	4 (4)	4 (4)
<i>RAF-174-63 fuel†††</i>							
5739	375/475	0.0	Code 1, °F	402 ---	415 (410)	399 (410)	411 (418)
			Code 2, °F	---	462 (451)	439 (449)	458 (457)
			Code 3, °F	427 ---	---	474 (498)	---
			Code 4, °F	480 (499)	488 ---	---	---
			Max code	4 (4)	4 (3)	3 (3)	<3 (<3)
5752	375/475	0.0	Code 1, °F	429 ---	172 (172)	440 (424)	172 (172)
			Code 2, °F	---	488 (478)	462 (466)	440† (467)
			Code 3, °F	492 (502)	---	511 (504)	483 (483)
			Code 4, °F	---	---	---	---
			Max code	3 (3)	<3 (4)	3 (3)	3 (3)
5762	375/475	0.0	Code 1, °F	361 (355)	358 (361)	367 (359)	226 (361)
			Code 2, °F	429 (386)	---	426 (399)	---
			Code 3, °F	457 (429)	449 (443)	450 (428)	432 (462)
			Code 4, °F	---	---	---	---
			Max code	3 (<4)	<4 (<4)	3 (4)	<4 (3)
5778	375/475	0.0	Code 1, °F	172 (338)	336 (336)	328 (331)	342 (355)
			Code 2, °F	371 ---	423 (424)	398 (389)	467 (457)
			Code 3, °F	---	490 (479)	475 (413)	---
			Code 4, °F	481 (480)	---	---	490 (471)
			Max code	>4 (4)	3 (3)	3 (<4)	4 (4)

TABLE 32. STANDARD COKER RATINGS ON FUELS FOR JFTOT PROGRAM (Cont'd)

Footnotes

- *Filter ΔP at 300 min unless otherwise indicated.
- †Preheater on temperature at 27 min.
- ‡Intermittent deposit; the deposit conforming to this code number does not extend uninterrupted to the next highest inception point (or to the tube end), but alternates with a deposit conforming to a lower deposit code number.
- **Preheater on temperature at 21 min.
- †† The 30-min and 24-hr ratings were done by different raters.
- ‡‡ "Secondary" inception point at 450°F. See Note ***.
- ***Deposit inception point is at a position further downstream, and hence at a lower temperature, than the inception point for a deposit conforming to the next lowest code number.
- ††† "Secondary" inception point at 479°F. See Note ***.
- ‡‡‡ "Secondary" inception point at 473°F. See Note ***.
- **** "Secondary" inception point at 518°F. See Note ***.
- †††† Peacocking made rating difficult for all tests on this fuel.

TABLE 33. AVERAGE STANDARD COKER RATINGS ON FUELS FOR JFTOT PROGRAM

All temperatures are mean values based on the results of 4 raters unless otherwise indicated

Run no.	Test temp. °F	Average deposit inception temperature, °F, for color rating as indicated								Composite heater breakpoint, °F		Filter* ΔP, in. Hg
		1-hr rating				24-hr rating				1-hr	24-hr	
		1	2	3	4	1	2	3	4			
AFFB-3-64 fuel												
5736	450/550	414	587	602†	---	392	583	604‡	---	602†	604‡	9.7
	Std dev. °F	128.9	10.9	1.0	---	173.8	21.6	---	---	1.0	---	
5750	450/550	203	567	---	---	248	568**	512†	---	---	512†	25.0/279
	Std dev. °F	5.3	2.6	---	---	84.7	0.6	130.1	---	---	130.1	
5758	450/550	264	559	---	---	247	551	---	---	---	---	24.3
	Std dev. °F	79.5	36.7	---	---	75.0	23.6	---	---	---	---	
5769	450/550	278	459	---	---	200	504	---	---	---	---	25.0/282
	Std dev. °F	149.0	83.0	---	---	4.1	53.6	---	---	---	---	
AFFB-4-64 fuel												
5748	325/425	381**	400**	319‡	---	368**	369	335‡	---	319‡	335‡	0.0
	Std dev. °F	1.2	67.1	---	---	26.0	52.2	---	---	---	---	
5732	350/450	290	458	---	483	312	459	482**	483	483	482	0.3
	Std dev. °F	85.0	9.4	---	0.6	100.1	25.5	0.0	0.6	0.6	0.0	
5760	350/450	325	440	470**	475‡	313	422	467**	475**	471	469	0.4
	Std dev. °F	3.0	12.8	11.2	---	46.0	40.2	13.8	---	11.6	12.0	
5773	350/450	249	328	481‡	473†	218	365	481‡	---	476**	481‡	0.3
	Std dev. °F	71.7	58.4	---	5.6	107.2	94.4	---	---	6.1	---	
AFFB-8-67 fuel												
5754	400/500	298	---	---	---	298	---	---	---	---	---	0.0
	Std dev. °F	77.8	---	---	---	78.2	---	---	---	---	---	
5728	425/525	541	570	572**	---	536**	477	572	---	572**	572	1.3
	Std dev. °F	45.0	3.1	0.7	---	47.4	192.0	0.6	---	0.7	0.6	
5744	425/525	530	570†	---	570	517	557†	---	570	570	570	1.6
	Std dev. °F	37.0	1.0	---	0.0	44.7	15.5	---	0.0	0.0	0.0	
5763	425/525	204	421	525	535	250	487	522**	533	525	522	5.6
	Std dev. °F	21.7	68.8	6.2	6.5	89.1	45.8	2.9	7.4	6.2	2.4	
AFFB-9-67 fuel												
5746	375/475	408	---	---	---	383	475‡	---	---	---	---	5.0
	Std dev. °F	33.0	---	---	---	10.6	---	---	---	---	---	
5730	400/500	476	513	541**	542	454	514	541†	542	541	542	22.4
	Std dev. °F	31.2	5.7	0.0	0.0	38.6	19.6	0.0	0.0	0.6	0.8	
5756	400/500	376	383	536†	536	409	447	536**	538	534	535	20.6
	Std dev. °F	119.1	144.0	3.6	4.1	45.4	97.1	3.6	3.5	2.5	3.3	
5766	400/500	434	524**	538**	541	444	527	536**	540	539	537	25.0/184.6
	Std dev. °F	53.6	13.8	2.9	---	44.9	10.2	3.6	1.0	3.0	3.9	
RAF-174-63 fuel††												
5739	375/475	407	453**	450†	484†	413**	455	498†	499‡	463**	498**	0.0
	Std dev. °F	7.5	12.3	33.2	5.6	4.6	5.9	1.0	---	32.0	1.0	
5752	375/475	303	463**	495**	---	256**	474	496**	502‡	495**	498	0.0
	Std dev. °F	151.6	24.0	14.3	---	129.5	9.2	11.6	---	14.3	9.9	
5762	375/475	328	428†	447	---	359	405**	440	446‡	447	440	0.0
	Std dev. °F	68.1	2.2	10.6	---	2.8	18.5	15.9	---	10.6	15.9	
5778	375/475	204	415	482†	486†	340	423**	451**	476†	484	456	0.0
	Std dev. °F	81.9	40.8	10.6	6.4	10.4	34.0	34.3	6.4	7.3	29.7	

* Filter ΔP at 100 min unless otherwise indicated.
† Temperature based on the results of two raters; the other two raters saw no such deposit.
‡ Temperature based on the results of one rater; the other three raters saw no such deposit.
** Temperature based on the results of three raters; one rater saw no such deposit.
†† Peacocking made rating difficult for all tests on this fuel.

TABLE 34. REPEATABILITY OF STANDARD COKER TESTS ON FUELS FOR JFTOT PROGRAM

Number of tests	Test temp, °F	Multiple test ratings									
		Average deposit inception temperature, °F, for color rating as indicated								Composite heater breakpoint, °F	
		1-hr rating				24-hr rating				1-hr	24-hr
		1	2	3	4	1	2	3	4		
<i>AFFB-3-64 fuel</i>											
4	450/550	290	543	603	---	271	550	543	---	603	543
Mul std dev, °F		123.9	65.6	0.7	---	119.0	42.7	106.2	---	0.7	106.1
Avg std dev, °F		90.7	33.3	1.0	---	84.4	24.8	130.1	---	1.0	130.1
Ratio (avg/mul X 100)		73%	51%	70%	---	71%	58%	123%	---	143%	123%
<i>AFFB-4-64 fuel</i>											
3	350/450	288	409	473	478	281	415	475	481	477	476
Mul std dev, °F		66.5	70.0	12.6	5.4	92.7	68.4	11.2	3.5	8.7	10.0
Avg std dev, °F		40.2	37.0	11.2	3.1	69.8	53.1	6.9	0.6	6.1	6.0
Ratio (avg/mul X 100)		60%	53%	89%	57%	75%	78%	62%	17%	70%	60%
<i>AFFB-8-67 fuel</i>											
3	425/525	425	510	545	552	425	497	551	552	554	555
Mul std dev, °F		166.4	86.7	25.3	19.1	151.6	118.5	27.1	20.4	23.3	24.1
Avg std dev, °F		45.4	24.3	3.5	3.2	64.8	84.4	1.8	3.7	2.3	1.0
Ratio (avg/mul X 100)		27%	28%	14%	17%	43%	71%	7%	18%	10%	4%
<i>AFFB-9-67 fuel</i>											
3	400/500	429	468	539	540	436	496	537	540	538	538
Mul std dev, °F		82.4	104.4	3.3	3.3	43.9	63.7	3.6	2.4	3.7	3.8
Avg std dev, °F		59.2	54.5	2.2	1.4	34.9	42.3	2.4	1.5	2.0	2.7
Ratio (avg/mul X 100)		72%	52%	17%	42%	79%	66%	67%	63%	54%	71%
<i>RAF-174-63 fuel</i>											
4	375/475	333	439	467	485	343	443	467	480	471	471
Mul std dev, °F		94.7	32.2	26.4	5.0	79.0	32.1	32.1	22.8	25.6	30.9
Avg std dev, °F		77.3	19.8	17.2	6.0	41.8	16.9	15.7	6.4	16.0	14.1
Ratio (avg/mul X 100)		82%	61%	65%	120%	53%	53%	49%	28%	63%	46%

TABLE 35. THERMAL STABILITY RATINGS IN ERDCO PRECISION COKER

Run no.	T _{max} , °F	Filter ΔP, in. Hg	Deposit inception ratings, 30-min (and 24-hr)				
			Rating	1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-4 fuel</i>							
9	455	0.2	Code 1, °F	368 (368)	368 --	368 (368)	368 --
			Code 2, °F	-- --	399 (368)	457 --	459 (368)
			Code 3, °F	456 (457)	-- (461)	-- --	-- (454)
			Code 4, °F	-- --	-- --	-- --	-- --
			Max code	3 (3)	2 (3)	2 (1)	2 (3)
15	455	0.0	Code 1, °F	-- (368)	368 (368)	368 (368)	441 (432)
			Code 2, °F	368 (436)	432 (430)	-- --	456 (456)
			Code 3, °F	-- --	-- --	-- --	-- --
			Code 4, °F	-- --	-- --	-- --	-- --
			Max code	2 (2)	2 (2)	1 (1)	2 (2)
3	435	0.2	Code 1, °F	352 (352)	-- --	352 (352)	352 --
			Code 2, °F	407 (383)	352 (352)	436 (435)	-- (352)
			Code 3, °F	-- --	-- --	-- --	-- --
			Code 4, °F	-- --	-- --	-- --	-- --
			Max code	2 (2)	2 (2)	2 (2)	1 (2)
12	435	0.1	Code 1, °F	351 (351)	-- (351)	351 (351)	351 (351)
			Code 2, °F	413 (413)	351 (417)	-- (442)	438 (436)
			Code 3, °F	-- --	-- --	-- --	-- --
			Code 4, °F	-- --	-- --	-- --	-- --
			Max code	2 (2)	2 (2)	1 (2)	2 (2)
<i>AFFB-8 fuel</i>							
7	540	0.0	Code 1, °F	-- --	-- --	428 (428)	-- --
			Code 2, °F	428 (428)	428 (428)	-- --	428 (428)
			Code 3, °F	527 (527)	529 (529)	529 (529)	525 (525)
			Code 4, °F	-- --	-- --	-- --	-- --
			Max code	3 (3)	3 (3)	3 (3)	3 (3)
13	540	0.0	Code 1, °F	428 (428)	465 (428)	428 (428)	525 (525)
			Code 2, °F	525 (525)	528 (525)	-- --	546 (546)
			Code 3, °F	-- --	-- --	-- --	-- --
			Code 4, °F	-- --	-- --	-- --	-- --
			Max code	2 (2)	2 (2)	1 (1)	2 (2)
2	520	0.9	Code 1, °F	-- --	-- --	-- --	-- --
			Code 2, °F	415 (415)	415 (415)	419 (419)	415 (479)
			Code 3, °F	522 (522)	522 (522)	-- --	522 (522)
			Code 4, °F	-- --	-- --	-- --	-- --
			Max code	3 (3)	3 (3)	2 (2)	3 (3)

TABLE 35. THERMAL STABILITY RATINGS IN ERDCO PRECISION COKER (Cont'd)

Run no.	T _{max} , °F	Filter ΔP, in. Hg	Deposit inception ratings, 30-min (and 24-hr)				
			Rating	1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-8 fuel (cont'd)</i>							
20	520	0.0	Code 1, °F	415 (415)	--- ---	--- (415)	--- (517)
			Code 2, °F	520 (496)	415 (415)	525 ---	522 ---
			Code 3, °F	--- ---	--- ---	--- ---	--- ---
			Code 4, °F	--- ---	--- ---	--- ---	--- ---
			Max code	2 (2)	2 (2)	2 (1)	---
<i>AFFB-3 fuel</i>							
6	585	0.0	Code 1, °F	--- ---	--- ---	461 (461)	461 (461)
			Code 2, °F	461 (461)	461 (461)	549 ---	500 (589)
			Code 3, °F	--- ---	546 (549)	--- ---	--- ---
			Code 4, °F	--- ---	--- ---	--- ---	--- ---
			Max code	2 (2)	3 (3)	2 (1)	2 (2)
19	585	0.0	Code 1, °F	--- (461)	--- ---	549 (540)	461 (461)
			Code 2, °F	461 (555)	461 (461)	--- (585)	--- ---
			Code 3, °F	--- ---	--- ---	--- ---	--- ---
			Code 4, °F	--- ---	--- ---	--- ---	--- ---
			Max code	2 (2)	2 (2)	1 (2)	1 (1)
1	565	0.5	Code 1, °F	--- ---	--- ---	447 (447)	--- (504)
			Code 2, °F	447 (447)	551 (447)	--- ---	553 ---
			Code 3, °F	--- ---	--- ---	--- ---	--- ---
			Code 4, °F	--- ---	--- ---	--- ---	--- ---
			Max code	2 (2)	2 (2)	1 (1)	2 (1)
14	565	0.0	Code 1, °F	--- (447)	447 (447)	447 (447)	560 (560)
			Code 2, °F	447 (511)	536 (559)	--- ---	--- ---
			Code 3, °F	--- ---	--- ---	--- ---	--- ---
			Code 4, °F	--- ---	--- ---	--- ---	--- ---
			Max code	2 (2)	2 (2)	1 (1)	1 (1)
<i>AFFB-9 fuel</i>							
10	520	9.6	Code 1, °F	415 (415)	415 (415)	415 (415)	415 (415)
			Code 2, °F	--- ---	--- ---	459 ---	--- ---
			Code 3, °F	--- ---	--- ---	--- ---	--- ---
			Code 4, °F	472 (472)	472 (472)	515 (479)	466 (466)
			Max code	4 (4)	4 (4)	4 (4)	4 (4)
11	520	4.5	Code 1, °F	--- (415)	--- ---	415 (415)	415 (415)
			Code 2, °F	415 ---	415 (415)	--- ---	--- (449)
			Code 3, °F	--- ---	--- ---	--- ---	482 (482)
			Code 4, °F	489 (489)	513 (499)	489 (489)	--- ---
			Max code	4 (4)	4 (4)	4 (4)	3 (3)

TABLE 35. THERMAL STABILITY RATINGS IN ERDCO PRECISION COKER (Cont'd)

Run no.	T _{max} , °F	Filter ΔP, in. Hg	Deposit inception ratings, 30-min (and 24-hr)				
			Rating	1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-9 fuel (cont'd)</i>							
8	500	0.0	Code 1, °F	399	399 (399)	399 ...
			Code 2, °F	... (399)	399 (399)	433 (399)
			Code 3, °F	500 (500)	500 (503)	458 (502)
			Code 4, °F	502 (502)
			Max code	3 (3)	3 (3)	3 (3)	4 (4)
18	500	10.0	Code 1, °F	399 (399)	399 (399)	465 (449)
			Code 2, °F	433 ...	399 (399)
			Code 3, °F
			Code 4, °F	480 (480)	442 (452)	480 (480)	477 (477)
			Max code	4 (4)	4 (4)	4 (4)	4 (4)
<i>RAF-174 fuel</i>							
5	485	10.0	Code 1, °F	480 (480)	480 ...	388 (388)
			Code 2, °F	388 (388)	388 (388)
			Code 3, °F
			Code 4, °F	484 (484)	480 (484)	476 (484)	482 (482)
			Max code	4 (4)	4 (4)	4 (4)	4 (4)
16	485	10.7	Code 1, °F	388 (388)	388 (388)	388 (388)	388 (388)
			Code 2, °F	421 (486)
			Code 3, °F
			Code 4, °F	487 (487)	487 (487)	488 (488)	487 (487)
			Max code	4 (4)	4 (4)	4 (4)	4 (4)
4	465	1.2	Code 1, °F	375 ...	375 (375)
			Code 2, °F	375 (375)	390 (472)	386 (466)	375 (375)
			Code 3, °F	470 (471)	472 (472)	470 ...
			Code 4, °F
			Max code	3 (3)	3 (3)	2 (2)	3 (2)
17	465	0.6	Code 1, °F	375 (375)	... (435)
			Code 2, °F	375 (375)	375 (375)	465 ...
			Code 3, °F
			Code 4, °F
			Max code	2 (2)	2 (2)	1 (2)	2 (1)

TABLE 36. AVERAGE THERMAL STABILITY RATINGS IN ERDCO PRECISION COKER

All temperatures are mean values based on the results of 4 raters unless otherwise indicated

Run no.	T _{max} , °F	Average deposit inception temperature, °F, for color rating as indicated								Composite heater breakpoint, °F		Filter ΔP, in. Hg
		30-min rating				24-hr rating				30-min	24-hr	
		1	2	3	4	1	2	3	4			
AFFB-3												
6	585	461*	493	546†	---	461*	504‡	549†	---	546	549	0.0
	Std dev, °F	0.0	41.8	---	---	0.0	73.8	---	---	---	---	
19	585	505*	461*	---	---	487‡	534‡	---	---	>585	>585	0.0
	Std dev, °F	8.5	0.0	---	---	45.6	64.7	---	---	---	---	
1	565	447†	517‡	---	---	475*	447*	---	---	>565	>565	0.5
	Std dev, °F	---	60.6	---	---	---	0.0	---	---	---	---	
14	565	485	492*	---	---	475	535*	---	---	>565	>565	0.0
	Std dev, °F	65.3	62.9	---	---	56.5	33.9	---	---	---	---	
AFFB-4												
9	455	368	438‡	456†	---	368*	368*	457‡	---	456	457	0.2
	Std dev, °F	0.0	34.0	---	---	0.0	0.0	3.6	---	---	3.6	
15	455	392‡	419‡	---	---	384	441‡	---	---	>455	>455	0.0
	Std dev, °F	42.1	45.4	---	---	32.0	13.6	---	---	---	---	
3	435	352‡	398‡	---	---	352*	380	---	---	>435	>435	0.2
	Std dev, °F	0.0	42.6	---	---	0.0	39.1	---	---	---	---	
12	435	351‡	401‡	---	---	351	427	---	---	>435	>435	0.1
	Std dev, °F	0.0	44.8	---	---	0.0	14.2	---	---	---	---	
AFFB-8												
7	540	428†	428‡	528	---	428†	428‡	528	---	528	528	0.0
	Std dev, °F	---	0.0	2.0	---	---	0.0	2.0	---	2.0	2.0	
13	540	462	533‡	---	---	452	532‡	---	---	>540	>540	0.0
	Std dev, °F	45.7	11.4	---	---	48.5	12.1	---	---	---	---	
2	520	---	416	522‡	---	---	432	522‡	---	522	522	0.9
	Std dev, °F	---	2.0	0.0	---	---	31.4	0.0	---	0.0	0.0	
20	520	415†	496	---	---	449‡	456	---	---	>520	>520	0.0
	Std dev, °F	---	53.7	---	---	58.9	57.3	---	---	---	---	

TABLE 36. AVERAGE THERMAL STABILITY RATINGS IN ERDCO PRECISION COKER (Cont'd)

Run no.	T _{max} , °F	Average deposit inception temperature, °F, for color rating as indicated								Composite heater breakpoint, °F		Filter ΔP, in. Hg
		30-min rating				24-hr rating				30-min	24-hr	
		1	2	3	4	1	2	3	4			
<i>AFFB-9</i>												
10	520	415	459†	---	481	415	---	---	472	481	472	9.6
	Std dev, °F	0.0	0.0	---	22.7	0.0	---	---	5.3	22.7	5.3	
11	520	415*	415*	482†	497‡	415‡	432*	482†	492‡	493	490	4.5
	Std dev, °F	0.0	0.0	---	13.8	0.0	24.0	---	5.7	13.6	6.9	
8	500	399‡	416*	486‡	502†	399†	399‡	502‡	502†	490	502	0.0
	Std dev, °F	0.0	24.0	24.2	0.0	---	0.0	1.5	0.0	21.4	1.3	
18	500	421‡	416*	---	470	416‡	399†	---	472	470	472	10.0
	Std dev, °F	38.1	24.0	---	18.6	29.0	0.0	---	13.5	18.6	13.5	
<i>RAF-174</i>												
5	485	449‡	388*	---	480	438*	388*	---	484	480	484	10.0
	Std dev, °F	53.2	0.0	---	3.4	65.0	0.0	---	1.0	3.4	1.0	
16	485	388	421†	---	487	388	486†	---	487	487	487	10.7
	Std dev, °F	0.0	---	---	0.5	0.0	---	---	0.5	0.5	0.5	
4	465	375*	382	471‡	---	375†	422	472*	---	471	472	1.2
	Std dev, °F	0.0	7.7	1.2	---	0.0	47.0	0.7	---	1.2	0.7	
17	465	375†	405‡	---	---	405*	375*	---	---	>465	>465	0.6
	Std dev, °F	---	63.7	---	---	42.4	0.0	---	---	---	---	
*Temperature based on the results of two raters; the other two raters saw no such deposit. †Temperature based on the results of one rater; the other three raters saw no such deposit. ‡Temperature based on the results of three raters; the other rater saw no such deposit.												

TABLE 37. RATINGS WITH ALCOR JFTOT IN CRC EVALUATION TESTS

Test pressure 300 psi for all tests; Dutch-weave 17μ in-line filter

Run no.	T _{max} , °F	Filter ΔP, in. Hg	Deposit inception ratings, 1-hr (and 24-hr)				
			Rating	1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-3 fuel</i>							
96	660	10.0	Code 1, °F	--- ---	--- (613)	613 (613)	--- ---
			Code 2, °F	--- ---	--- (614)	--- ---	--- ---
			Code 3, °F	--- ---	614 (615)	--- ---	--- ---
			Code 4, °F	614 (614)	618 (618)	614 (614)	612 (612)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
88	640	1.4	Code 1, °F	596 (592)	597 (596)	--- (597)	--- ---
			Code 2, °F	--- (592)	--- ---	--- ---	592 (592)
			Code 3, °F	597 (596)	--- ---	597 ---	638 ---
			Code 4, °F	640 (640)	598 (597)	637 (598)	640 (638)
			Max code	4 (4)	>4 (>4)	>4 (>4)	>4 (>4)
<i>AFFB-4 fuel</i>							
91*	520	3.0	Code 1, °F	429 (435)	P (P)	407 (386)	436 (436)
			Code 2, °F	485 (483)	P (P)	463 (445)	P (P)
			Code 3, °F	495 (485)	491 (501)	501 (501)	494 (491)
			Code 4, °F	507 (505)	501 (508)	511 (511)	505 (507)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
99	500	10.0	Code 1, °F	412 (412)	405 (400)	400 (390)	412 (390)
			Code 2, °F	--- ---	492 (492)	--- ---	--- ---
			Code 3, °F	--- (492)	495 (495)	495 (492)	--- ---
			Code 4, °F	495 (499)	498 (497)	498 (498)	492 (492)
			Max code	4 (4)	4 (4)	>4 (>4)	>4 (>4)
<i>AFFB-8 fuel</i>							
98*	570	10.0	Code 1, °F	567 (567)	567 (568)	569 (567)	566 (566)
			Code 2, °F	--- ---	--- ---	--- ---	--- ---
			Code 3, °F	--- ---	--- ---	--- ---	--- ---
			Code 4, °F	569 (568)	568 (569)	569 (568)	568 (568)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
92†	550	10.0	Code 1, °F	--- (537)	536 (537)	--- (493)	539 ---
			Code 2, °F	539 ---	--- ---	--- ---	537 (535)
			Code 3, °F	537 (535)	--- ---	538 (534)	--- (534)
			Code 4, °F	--- ---	534 (534)	--- ---	534 ---
			Max code	<4 (<4)	4 (4)	<4 (3)	4 (3)

TABLE 37. RATINGS WITH ALCOR JFTOT IN CPC EVALUATION TESTS (Cont'd)

Run no.	T _{max} , °F	Filter ΔP, in. Hg	Deposit inception ratings, 1-hr (and 24-hr)				
			Rating	1st rater	2nd rater	3rd rater	4th rater
<i>AFFB-9 fuel</i>							
90*	535	10.0	Code 1, °F	395 (395)	344 (395)	495 (495)
			Code 2, °F	493 (493)	493 (493)	498 (498)
			Code 3, °F	504 (504)	498	498 (498)
			Code 4, °F	510 (512)	505 (504)	504 (504)	505 (505)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
97	515	10.0	Code 1, °F	474	485 (485)
			Code 2, °F	496 ..	496 (504)
			Code 3, °F	504 (496)	506 (506)	506 (507)	... (504)
			Code 4, °F	508 (511)	507 (509)	507 (508)	485 (508)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
<i>RAF-174 fuel</i>							
94‡	565	0.0	Code 1, °F	506 (506)	495 (495)	506 (506)	506 (513)
			Code 2, °F
			Code 3, °F
			Code 4, °F	513 (513)	518 (518)	519 (513)	513 (520)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
93‡	545	0.0	Code 1, °F	505 (504)	505 (505)	505 (505)	... (505)
			Code 2, °F
			Code 3, °F
			Code 4, °F	509 (508)	509 (508)	509 (510)	509 (508)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
95‡	500	0.0	Code 1, °F	495 (496)	496 (495)	497 (497)	496 (495)
			Code 2, °F
			Code 3, °F
			Code 4, °F	497 (498)	498 (498)	498 (499)	497 (498)
			Max code	>4 (>4)	>4 (>4)	>4 (>4)	>4 (>4)
<p>*Peacocking noted on the heater tube. "P" denotes peacocking rating. If no "P" is given, then peacocking occurred on a region of the tube beyond the maximum rating.</p> <p>‡The deposit color for this test was beyond the tube's hottest point, but was within 0.2 in. of the hottest point.</p> <p>‡Heavy peacocking noted on the heater tube.</p>							

TABLE 38. AVERAGE RATINGS WITH ALCOR JFTOT IN CRC EVALUATION TESTS

All temperatures are mean values based on the results of 4 raters unless otherwise indicated

Run no.	T _{max} , °F	Average deposit inception temperature, °F, for color rating as indicated								Composite heater breakpoint, °F		Filter ΔP, in. Hg
		1-hr rating				24-hr rating				1-hr	24-hr	
		1	2	3	4	1	2	3	4			
<i>AFFB-3 fuel</i>												
96	660	613*	---	614*	614	613†	614*	615*	614	614	614	10.0
	Std dev. °F	---	---	---	2.6	0.0	---	---	2.6	1.2	1.3	
88	640	596†	572*	610‡	629	595‡	592†	596*	618	608	607	1.4
	Std dev. °F	1.0	---	23.7	20.5	2.6	0.0	---	24.0	20.3	20.5	
<i>AFFB-4 fuel</i>												
91**	520	424‡	474†	495	506	419‡	464†	494	508	495	494	3.0
	Std dev. °F	15.1	15.6	4.2	4.2	28.6	25.9	7.9	2.5	4.2	7.9	
99	500	407	492*	495†	496	398	492*	493‡	496	494	493	10.0
	Std dev. °F	5.8	---	0.0	2.9	10.4	---	1.7	3.2	2.0	1.5	
<i>AFFB-8 fuel</i>												
98**	570	567	---	---	568	567	---	---	568	568	568	10.0
	Std dev. °F	1.3	---	---	0.8	0.8	---	---	0.6	0.8	0.6	
92††	550	538†	538†	538†	534†	522‡	535*	534‡	534*	536	534	10.0
	Std dev. °F	2.2	1.4	1.0	0.0	25.4	---	0.7	---	2.1	0.6	
<i>AFFB-9 fuel</i>												
90**	535	411‡	495‡	500‡	506	428‡	495‡	501†	506	501	502	10.0
	Std dev. °F	76.8	2.9	3.5	2.7	57.7	2.9	4.2	3.9	3.4	3.1	
97	515	480†	496†	505‡	502	485*	504*	503	509	500	503	10.0
	Std dev. °F	7.8	0.0	1.2	11.2	---	---	5.0	1.4	10.2	5.0	
<i>RAF-174 fuel</i>												
94‡‡	565	503	---	---	516	505	---	---	516	516	516	0.0
	Std dev. °F	5.5	---	---	3.2	7.4	---	---	3.6	3.2	3.6	
93‡‡	545	505	---	---	509	505	---	---	508	509	508	0.0
	Std dev. °F	0.0	---	---	0.0	0.6	---	---	1.2	0.0	1.2	
95‡‡	500	496	---	---	498	496	---	---	498	498	498	0.0
	Std dev. °F	0.8	---	---	0.8	1.0	---	---	0.6	0.8	0.6	
* Temperature based on the results of one rater; the other three raters saw no such deposit † Temperature based on the results of two raters; the other two raters saw no such deposit ‡ Temperature based on the results of three raters; one rater saw no such deposit. ** Peacocking occurred on the heater tube for this test. †† The deposit color for this test was beyond the tube's hottest point, but was within 0.2 in. of the hottest point. ‡‡ Heavy peacocking occurred on the heater tube for this test.												

TABLE 39. THERMAL STABILITY OF TURBINE ENGINE FUELS IN ALCOR JFTOT

Test Pressure 350 psi
Dutch-weave 17 μ in-line filter followed by 0.45 μ filter

Run no.	I _{max} °F	Heater temperature, °F, for first color rating as indicated								Heater breakpoint, °F		Filter* Δ P in Hg	Coker no.	Date tested
		1-hr rating				24-hr rating				1-hr	24-hr			
		1	2	3	4	1	2	3	4					
<i>70-16 JP-7 fuel</i>														
6360	700	555	395‡	555	395‡	>700	>700	0.0	8	28 Jul 70
6377**	700	...	364	364	>700	>700	0.0	8	29 Jul 70
<i>70-17 JP-7 fuel</i>														
6378	700	467	680††	467	>700	>700	0.0	8	31 Jul 70
6386	700	476	697	399	>700	>700	0.0	8	3 Aug 70
<i>70-18 JP-7 fuel</i>														
6390	700	600	692	476	692	>700	>700	0.0	8	4 Aug 70
6393	700	...	467	390	>700	>700	0.0	8	5 Aug 70
<i>OF JP-4</i>														
6437	550	...	397‡	311‡	397‡	311‡	475††	311	311	10.0/169	9	21 Aug 70
6421	800	488‡	390‡	367‡	...	488‡	390‡	360‡	...	367	360	0.15	9	17 Aug 70
6417	450	...	310	350	295	350	...	350	350	0.0	9	14 Aug 70
6425	425	...	394‡	345‡	394‡	345‡	...	345	345	0.0	9	18 Aug 70
6441	425	300	405‡	348‡	...	300	423‡	335‡	...	348	335	0.0	9	20 Aug 70
6440	325	...	384‡	354‡	384‡	354‡	...	354	354	0.0	8	24 Aug 70
6447	415	415‡	344‡	415‡	357‡	325†	...	>415	325	0.0	8	26 Aug 70
6412	400	...	240	240	>400	>400	0.0	9	13 Aug 70
6429	400	217‡‡	335	217‡‡	335	>400	>400	0.0	9	19 Aug 70
6443	400	357‡	236‡	357‡	236‡	>400	>400	0.0	8	25 Aug 70
<i>DR JP-4</i>														
6475	560	...	385	...	560	...	385	...	560	560	560	0.0	8	4 Sep 70
6467	550	425	...	544	540	425	...	549	547	540	547	0.0	8	2 Sep 70
6476	550	425	503	538	486	548	535††	548	548	0.0	9	4 Sep 70
6471	540	...	482	482	>540	>540	0.0	8	3 Sep 70
6479	540	...	473	...	410††	...	473	...	410††	++	++	0.0	8	8 Sep 70
6480	540	...	480	480	>540	>540	0.0	9	8 Sep 70
6477	530	510	519	>530	>530	0.0	9	3 Sep 70
6468	525	565‡‡	480	480	>525	>525	0.05	9	2 Sep 70
6463	500	586	456	>500	>500	0.0	8	1 Sep 70
<i>HR JP-4</i>														
6486	575	445	571	445	571	571	571	10.0/67	9	28 Aug 70
6460	575	304‡‡	469	...	574	304‡‡	490	...	571	574	571	10.0/62	9	31 Aug 70
6457	550	465	530	445	538	>550	>550	10.0/118	9	27 Aug 70
6464	575	...	267‡‡	262‡‡	>575	>575	10.0/128	9	1 Sep 70
6445	500	584‡	587	254‡‡	287	>500	>500	0.9	9	26 Aug 70
6444	450	...	300	306	>450	>450	0.0	9	25 Aug 70
6440	425	...	236	234	323††	>425	>425	0.0	9	24 Aug 70

* Filter Δ P in Hg at 300 ml/min flow, unless otherwise indicated.
 †† Fuel system OK, but water coolant supply system failed.
 ‡‡ Fuel system OK, but air after the fuel inlet end of the tube (lower code deposits occur upstream closer to the I_{max} point) or fuel system OK, but air after coolant supply system failed.
 † Fuel system OK, but an air filter was used in breakpoint determination.
 ‡ Fuel system OK, but an air filter was used in breakpoint determination.
 ††† Fuel system OK, but the I_{max} point data not used in breakpoint determination.
 ‡‡‡ Fuel system OK, but the I_{max} point data not used in breakpoint determination.

TABLE 40. STEP-TEMPERATURE READINGS FOR ALCOR JFTOT

Test time, min	T _{max} , °F	Wattage reading at indicated time interval following warm-up to T _{max}				ΔP, in. Hg at 15 min following warm-up to T _{max}	Volts		
		Watts 5 min	ΔW*	Watts 15 min	ΔW*				
<i>Test no. 129</i>									
0	70	---	---	---	---	---	60		
1.1	400	105	---	---	---	0.0	60		
15	400	100	5	100	---	0.0	70		
30	425	107	7	108	8	0.0	70		
45	450	115	8	115	7	0.0	70		
60	475	122	7	122	7	0.0	70		
75	500	131	9	130	8	0.0	70		
90	525	138	7	138	8	0.01	70		
105	550	145	7	145	7	0.05	80		
120	575	150	5	150	5	0.1	80		
135	600	158	8	158	8	0.23	80		
150	625	165	7	166	8	0.47	80		
165	650	172	7	172	6	1.07	80		
180	675	180	8	180	8	2.5	80		
195	700	188	8	188	8	3.6	90		
Test time, min	T _{max} , °F	Wattage reading at indicated time interval following warm-up to T _{max}				ΔP, in. Hg at 10 min following warm-up to T _{max}	Volts		
		Watts 5 min	ΔW*	Watts 10 min	ΔW*				
<i>Test no. 130</i>									
0	72	---	---	---	---	---	60		
1.1	400	107	---	---	---	---	60		
10	400	101	6	101	---	0.0	60		
20	425	109	8	109	8	0.0	60		
30	450	116	7	116	7	0.0	64		
40	475	123	7	122	6	0.0	66		
50	500	130	7	130	8	0.0	70		
60	525	137	7	137	7	0.0	70		
70	550	144	7	144	7	0.0	70		
80	575	150	6	150	6	0.04	80		
99	600	158	8	158	8	0.12	80		
100	625	166	8	166	8	0.3	80		
110	650	172	6	172	6	0.7	80		
120	675	180	8	180	8	1.3	81		
130	700	187	7	187	7	2.55	84		
Test time, min	T _{max} , °F	Wattage reading at indicated time interval following warm-up to T _{max}						ΔP, in. Hg at 15 min following warm-up to T _{max}	Volts
		Watts 5 min	ΔW*	Watts 30 min	ΔW*	Watts 60 min	ΔW*		
<i>Test no. 131</i>									
0	72	---	---	---	---	---	---	0.0	80
5	500	126	---	---	---	---	---	0.0	80
30	500	---	---	130	---	---	---	0.0	80
60	500	---	---	---	---	131	---	0.01	80
65	525	138	12	---	---	---	---	0.01	80
90	525	---	---	138	5	---	---	0.05	80
120	525	---	---	---	---	138	7	0.1	80
125	550	147	9	---	---	---	---	0.15	80
150	550	---	---	146	8	---	---	0.3	80
180	550	---	---	---	---	146	8	0.58	80
185	575	151	4	---	---	---	---	0.67	80
210	575	---	---	151	5	---	---	1.2	80
240	575	---	---	---	---	151	5	2.15	80
245	600	154	3	---	---	---	---	2.57	80
270	600	---	---	157	6	---	---	5.8	80
300	600	---	---	---	---	161	10	---	80

*Wattage difference between reading at indicated test time and prior wattage reading

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
<i>T_{max} 400°F</i>													
60	6443	8	400	317	359	386	393	369	330	277	---	206	397
120			400	317	361	387	395	371	350	279	---	209	400
180			400	317	359	386	393	369	329	278	---	208	397
240			400	316	360	388	395	372	331	280	---	210	400
290			400	318	359	387	394	370	331	280	---	209	399
Average temp., °F			400	317	360	387	394	370	330	279	---	208	399
Std dev., °F			0.0	0.7	1.0	0.9	1.0	1.3	0.9	1.3	---	1.6	1.6
<i>T_{max} 415°F</i>													
60	6412	9	400	322	362	385	392	370	332	280	238	---	400
120			400	321	363	387	393	371	333	283	240	---	402
180			400	320	362	384	392	370	333	283	240	---	400
240			400	322	361	386	393	372	336	283	243	---	401
290			400	323	360	386	393	370	334	283	243	---	400
Average temp., °F			400	322	362	386	393	371	334	282	241	---	401
Std dev., °F			0.0	1.2	1.2	1.2	0.7	1.0	1.6	1.2	2.2	---	1.0
<i>T_{max} 415°F</i>													
60	6429	9	400	320	362	387	394	373	335	280	238	---	401
120			400	323	360	386	394	373	336	282	239	---	401
180			400	320	360	385	393	373	335	282	238	---	401
240			400	321	363	387	396	375	338	283	240	---	402
290			400	323	361	387	394	374	336	284	239	---	401
Average temp., °F			400	321	361	386	393	373	336	282	239	---	401
Std dev., °F			0.0	1.6	1.3	1.0	1.1	1.2	1.2	1.5	0.9	---	0.5
<i>T_{max} 415°F</i>													
60	6447	8	415	327	372	400	410	386	344	291	---	215	414
120			415	326	372	401	411	386	346	293	---	217	414
180			415	326	373	398	407	384	344	290	---	218	413
240			415	329	373	400	413	388	346	294	---	218	415
290			415	328	371	400	410	387	347	293	---	220	414
Average temp., °F			415	327	372	400	410	386	345	292	---	218	414
Std dev., °F			0.0	1.3	0.9	0.9	2.2	1.5	1.4	1.1	---	1.9	0.7
<i>T_{max} 425°F</i>													
60	6440	8	425	337	384	412	420	397	354	299	---	220	423
120			425	337	382	411	418	393	352	297	---	221	424
180			425	338	383	413	419	393	351	299	---	222	425
240			425	336	385	410	419	393	350	298	---	224	423
290			425	339	383	412	420	393	353	297	---	223	427
Average temp., °F			425	337	383	412	419	394	352	298	---	222	424
Std dev., °F			0.0	1.2	1.2	1.2	0.9	1.5	1.6	1.0	---	1.6	1.7

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 425°F (cont'd)</i>													
60	6425	9	425	345	385	412	418	394	353	300	255	...	426
120			425	343	387	411	417	393	354	298	253	...	426
180			425	340	385	413	420	395	354	300	255	...	425
240			425	345	389	416	420	393	353	297	256	...	425
290			425	343	385	412	416	393	354	301	254	...	425
Average temp, °F			425	343	386	413	418	394	354	299	255	...	425
Std dev, °F			0.0	1.6	1.8	1.9	1.6	1.0	0.7	1.6	1.2	...	0.7
60	6431	9	425	337	383	409	418	393	355	300	...	356	426
120			425	339	382	410	418	394	353	300	...	255	426
180			425	338	382	411	418	393	354	299	...	255	425
240			425	339	382	410	419	395	355	301	...	257	425
290*		
Average temp, °F			425	338	382	410	418	394	354	300	...	256	426
Std dev, °F			0.0	1.0	0.2	0.2	0.2	1.0	1.0	0.2	...	1.0	0.2
60	6441	9	425	346	388	412	415	390	346	290	246	...	425
120			425	345	387	413	417	391	348	293	248	...	425
180			425	346	387	412	416	390	347	294	248	...	426
240			425	346	388	414	417	392	349	294	250	...	427
290			425	344	387	412	416	390	349	292	251	...	425
Average temp, °F			425	345	387	413	416	391	348	293	249	...	426
Std dev, °F			0.0	1.0	0.7	1.0	0.9	1.0	1.3	1.7	2.0	...	1.0
<i>T_{max} 450°F</i>													
60	6417	9	450	363	410	438	443	416	370	310	262	...	451
120			450	363	408	437	441	414	370	312	262	...	450
180			450	361	410	438	442	413	370	312	263	...	450
240			450	362	409	439	440	415	372	313	263	...	450
290			450	363	409	435	442	415	373	314	263	...	449
Average temp, °F			450	362	409	437	442	415	371	312	263	...	450
Std dev, °F			0.0	1.0	0.9	1.3	1.2	1.2	1.4	1.2	0.7	...	0.7
60	6444	9	450	358	408	435	440	414	370	308	256	...	449
120			450	360	406	435	442	413	368	310	258	...	450
180			450	361	408	435	441	414	370	310	258	...	450
240			450	360	406	436	442	414	370	310	260	...	450
290			450	362	407	435	443	414	371	313	262	...	450
Average temp, °F			450	360	407	435	442	414	370	310	259	...	450
Std dev, °F			0.0	1.5	1.0	0.5	1.2	0.5	1.1	1.8	2.3	...	0.7

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 500°F</i>													
60	6345	8	500	384	446	479	492	462	414	348	---	266	500
120			500	384	446	486	498	470	418	353	---	271	503
180			500	386	446	483	496	465	417	350	---	271	501
240			500	386	447	481	496	466	417	352	---	268	501
290			500	386	446	480	492	464	416	348	---	267	500
Average temp, °F			500	385	446	482	495	465	416	350	---	269	501
Std dev, °F			0.0	1.1	0.5	2.8	2.7	3.1	1.6	2.3	---	2.3	1.2
60	6463	8	500	402	456	486	491	456	403	338	---	247	499
120			500	403	455	487	491	458	406	339	---	248	500
180			500	401	453	485	489	455	402	337	---	248	499
240			500	405	455	486	491	458	405	338	---	250	500
290			500	401	454	484	489	457	405	337	---	251	498
Average temp, °F			500	402	455	486	490	457	404	338	---	249	499
Std dev, °F			0.0	1.7	1.2	1.2	1.1	1.3	1.6	0.9	---	1.6	0.9
60	6448	9	500	401	453	486	490	456	404	333	282	---	500
120			500	403	454	485	490	458	406	340	283	---	500
180			500	404	452	484	488	459	408	339	284	---	501
240			500	401	453	483	488	458	407	341	285	---	500
290			500	403	454	486	490	460	407	342	286	---	498
Average temp, °F			500	402	453	485	489	458	406	340	284	---	500
Std dev, °F			0.0	1.4	0.9	1.3	1.1	1.5	1.6	1.6	1.6	---	1.1
60	6421	9	500	407	456	487	488	459	409	340	292	---	501
120			500	406	455	485	487	457	407	342	291	---	500
180			500	404	458	489	490	460	409	343	291	---	502
240			500	408	459	488	489	461	412	345	295	---	500
290			500	407	454	485	488	461	410	346	295	---	501
Average temp, °F			500	406	456	487	488	460	409	343	293	---	501
Std dev, °F			0.0	1.6	2.1	1.8	1.2	1.7	1.9	2.4	1.6	---	0.9
<i>T_{max} 510°F</i>													
60	6328	8	510	389	452	489	504	473	420	354	---	268	508
120			510	392	453	492	505	474	423	359	---	265	510
180			510	394	451	492	504	475	423	359	---	272	510
240			510	387	448	486	503	475	424	360	---	265	507
290			510	390	452	493	504	475	425	361	---	277	508
Average temp, °F			510	390	451	490	504	474	423	359	---	269	509
Std dev, °F			0.0	2.7	1.9	2.9	0.7	1.0	1.6	2.7	---	5.1	1.9

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 510°F (cont'd)</i>													
60	6337	8	510	395	457	492	503	471	417	349	---	268	507
120			510	393	454	493	505	472	420	352	---	271	512
180			510	396	456	493	507	473	422	353	---	270	512
240			510	395	453	487	499	471	418	353	---	262	508
290			510	394	453	492	507	474	421	357	---	274	512
Average temp, °F			510	395	455	491	504	472	420	353	---	269	510
Std dev, °F			0.0	1.9	2.3	2.5	3.3	1.3	2.1	2.9	---	4.5	2.5
60	6342	8	510	392	451	491	504	474	422	356	---	271	508
120			510	392	454	493	506	476	424	357	---	278	507
180			510	392	453	491	505	474	423	357	---	275	509
240			510	392	453	491	506	475	426	358	---	275	510
290			510	394	453	492	508	480	428	362	---	277	510
Average temp, °F			510	392	453	492	506	476	425	358	---	275	509
Std dev, °F			0.0	1.0	1.1	1.0	1.5	2.5	2.4	2.3	---	2.5	1.3
60	6349	8	510	392	451	487	501	473	419	354	---	257	507
120			510	393	454	492	504	473	423	355	---	265	509
180			510	391	453	490	503	471	420	353	---	261	510
240			510	391	452	483	497	468	418	351	---	257	509
290			510	393	453	489	501	469	417	353	---	259	509
Average temp, °F			510	392	453	489	501	471	419	353	---	260	509
Std dev, °F			0.0	1.1	1.2	1.9	2.7	2.3	2.3	0.9	---	3.3	1.1
<i>T_{max} 525°F</i>													
60	6325	8	525	414	475	512	519	489	431	359	---	271	525
120			525	410	472	507	517	483	427	359	---	276	525
180			525	413	473	511	520	484	431	360	---	277	525
240			525	406	470	505	517	484	430	362	---	275	525
290			525	410	469	510	520	487	432	362	---	272	525
Average temp, °F			525	411	472	509	519	485	430	360	---	274	525
Std dev, °F			0.0	3.1	2.4	2.9	1.6	2.8	1.9	1.6	---	2.6	0.0
60	6332	8	525	403	468	508	521	489	432	366	---	273	523
120			525	406	470	507	520	489	435	366	---	281	525
180			525	404	465	506	519	489	438	368	---	286	525
240			525	404	463	504	518	488	438	370	---	285	525
290			525	402	467	507	519	489	441	370	---	284	522
Average temp, °F			525	404	467	506	519	489	437	368	---	282	524
Std dev, °F			0.0	1.5	2.7	1.6	1.2	0.5	3.7	2.0	---	5.3	1.4

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 525°F (cont'd)</i>													
60	6339	8	525	399	467	506	521	490	437	369	---	278	523
120			525	399	465	505	517	489	435	367	---	282	523
180			525	402	467	505	517	487	435	367	---	284	523
240			525	401	467	503	511	487	434	368	---	282	523
290			525	399	468	507	522	493	443	373	---	285	522
Average temp., °F			525	400	467	505	518	489	437	369	---	282	523
Std dev., °F			0.0	1.4	1.1	1.5	4.2	2.5	3.6	2.5	---	2.7	0.5
60	6350	8	525	406	470	507	516	482	428	357	---	263	525
120			525	407	470	507	516	482	429	357	---	265	526
180			525	410	471	508	515	478	425	356	---	272	524
240			525	413	476	510	514	479	424	352	---	265	524
290			525	414	477	509	514	478	426	354	---	267	524
Average temp., °F			525	410	473	508	515	480	426	355	---	266	525
Std dev., °F			0.0	3.4	3.4	1.3	1.0	1.8	1.9	2.2	---	3.6	1.0
60	6464	9	525	420	478	510	512	478	423	352	292	---	524
120			525	422	477	509	510	481	426	354	301	---	525
180			525	418	478	511	516	480	426	355	297	---	527
240			525	421	478	512	515	483	425	356	301	---	527
290			525	417	474	508	513	480	427	356	302	---	525
Average temp., °F			525	420	477	510	513	480	425	355	299	---	526
Std dev., °F			0.0	2.1	1.7	1.6	2.3	1.9	1.6	1.7	4.2	---	1.4
60	6468	9	525	424	480	510	515	480	425	355	295	---	525
120			525	421	479	511	515	482	428	358	300	---	527
180			525	420	476	509	511	479	426	356	301	---	525
240			525	425	479	509	514	483	428	358	300	---	526
290			525	426	477	511	514	483	432	360	298	---	526
Average temp., °F			525	423	478	510	514	481	428	357	299	---	526
Std dev., °F			0.0	2.5	1.6	1.0	1.6	1.9	2.7	2.0	2.4	---	0.9
<i>T_{max} 530°F</i>													
60	6472	9	530	426	482	515	528	485	432	360	306	---	530
120			530	426	484	518	524	488	433	362	308	---	532
180			530	426	481	516	524	489	435	364	308	---	530
240			530	427	482	515	522	488	435	362	306	---	532
290			530	426	480	512	518	485	431	363	308	---	529
Average temp., °F			530	426	482	515	523	487	433	362	307	---	531
Std dev., °F			0.0	0.5	1.5	2.2	3.6	1.9	1.8	1.5	1.1	---	1.4

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet. °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 540°F</i>													
60	6471	8	540	430	488	525	532	498	442	372	---	270	538
120			540	430	485	520	526	494	440	369	---	269	537
180			540	428	484	522	527	495	442	372	---	272	540
240			540	426	486	519	530	496	441	372	---	274	538
290			540	428	484	520	528	498	442	373	---	276	538
Average temp, °F			540	428	485	521	529	496	441	372	---	272	538
Std dev, °F			0.0	1.7	1.7	2.4	2.4	1.8	1.0	2.5	---	2.9	1.1
60	6479	8	540	427	485	521	527	493	438	370	---	267	537
120			540	432	493	526	532	497	441	368	---	269	542
180			540	430	490	526	532	498	442	370	---	269	541
240			540	426	487	522	530	496	443	371	---	269	538
290			540	429	488	522	530	497	441	373	---	272	540
Average temp, °F			540	429	489	523	530	496	441	370	---	269	540
Std dev, °F			0.0	2.4	3.7	2.4	2.1	1.9	1.9	1.9	---	1.8	2.1
60	6480	9	540	433	492	527	527	492	437	364	304	---	542
120			540	429	490	525	525	492	438	366	305	---	541
180			540	429	492	527	529	493	439	367	303	---	541
240			540	426	489	523	526	494	436	363	305	---	539
290			540	424	483	522	526	490	434	361	302	---	537
Average temp, °F			540	428	489	525	527	492	437	364	304	---	540
Std dev, °F			0.0	3.4	3.7	2.3	1.6	1.5	1.9	2.4	1.3	---	2.0
<i>T_{max} 550°F</i>													
60	6319	8	550	421	493	530	540	507	451	381	---	272	548
120			550	425	490	534	544	506	448	378	---	286	548
180			550	421	486	529	541	507	453	381	---	275	548
240			550	425	491	529	542	506	451	381	---	294	550
290			550	428	483	527	539	507	463	390	---	281	555
Average temp, °F			550	424	489	530	541	507	453	382	---	282	550
Std dev, °F			0.0	3.0	4.0	2.6	1.9	0.7	2.9	4.6	---	8.8	3.0
60	6437	9	550	446	501	534	535	501	447	371	311	---	550
120			550	447	501	537	539	504	448	374	315	---	552
180			550	446	503	535	538	504	449	374	315	---	549
240			550	448	503	536	540	508	453	376	319	---	553
290			550	447	502	535	541	507	451	376	317	---	550
Average temp, °F			550	447	502	535	539	505	450	374	315	---	551
Std dev, °F			0.0	0.9	1.0	1.2	2.3	2.8	2.4	2.1	2.9	---	1.6

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 550°F (cont'd)</i>													
60	6452	9	550	442	498	534	538	503	445	370	305	---	550
120			550	440	502	534	539	504	447	370	307	---	552
180			550	438	496	532	540	506	447	372	309	---	548
240			550	436	498	533	538	507	448	373	310	---	548
290			550	439	499	534	539	508	448	374	311	---	548
Average temp, °F			550	439	499	533	539	506	447	372	308	---	549
Std dev, °F			0.0	2.2	2.2	1.0	0.9	2.1	1.2	1.8	2.6	---	1.8
60	6476	9	550	438	498	535	538	503	446	375	308	---	551
120			550	438	498	536	540	505	448	376	309	---	551
180			550	436	496	537	539	503	449	373	310	---	550
240			550	438	494	532	537	502	448	376	310	---	550
290			550	438	500	532	539	505	449	377	312	---	550
Average temp, °F			550	438	497	534	539	504	448	375	310	---	550
Std dev, °F			0.0	0.7	2.3	2.3	1.2	1.4	1.2	1.6	1.5	---	0.7
<i>T_{max} 560°F</i>													
60	6475	8	560	442	501	539	549	515	459	385	---	275	560
120			560	440	505	542	551	518	459	383	---	278	560
180			560	437	501	542	556	521	465	387	---	276	563
240			560	439	501	539	550	516	460	384	---	276	560
290			560	438	504	540	552	515	460	385	---	278	558
Average temp, °F			560	439	502	540	552	517	461	385	---	277	560
Std dev, °F			0.0	1.9	2.0	1.6	2.7	2.5	2.5	1.5	---	1.4	1.8
<i>T_{max} 575°F</i>													
60	6456	9	575	465	525	560	564	524	465	383	322	---	577
120			575	463	526	562	566	526	467	388	332	---	579
180			575	464	524	560	562	522	465	386	324	---	575
240			575	463	522	558	559	522	464	386	325	---	575
290			575	465	524	560	558	523	465	386	322	---	575
Average temp, °F			575	464	524	560	561	523	465	386	325	---	576
Std dev, °F			0.0	1.0	1.5	1.4	3.3	1.7	1.1	1.8	4.1	---	1.8
60	6460	9	575	460	520	560	562	526	466	390	333	---	576
120			575	461	520	559	564	525	468	391	328	---	576
180			575	462	521	561	565	525	466	389	329	---	578
240			575	461	519	559	560	524	465	389	324	---	575
290			575	458	522	559	564	526	470	390	330	---	575
Average temp, °F			575	460	520	560	563	525	467	390	329	---	576
Std dev, °F			0.0	1.6	1.2	1.0	2.0	0.9	2.0	0.9	3.3	---	1.2

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 610°F</i>													
60	6362	8	610	485	559	593	594	553	487	406	---	310	607
120			610	488	555	594	595	550	495	413	---	312	607
180			610	482	557	595	596	552	493	409	---	312	608
240			610	476	557	597	599	558	496	413	---	318	608
290			610	479	556	597	597	556	494	410	---	313	610
Average temp, °F			610	482	557	595	596	554	493	410	---	313	608
Std dev, °F			0.0	4.7	1.5	1.8	1.9	3.2	3.4	3.0	---	3.0	1.2
<i>T_{max} 635°F</i>													
60	6360	8	635	499	583	623	623	579	511	419	---	319	635
120			635	505	588	618	617	576	510	423	---	333	637
180			635	508	586	621	620	574	503	417	---	324	637
240			635	508	582	620	623	581	512	425	---	323	637
290			635	509	589	623	620	571	506	420	---	323	638
Average temp, °F			635	506	586	621	621	576	508	421	---	324	637
Std dev, °F			0.0	4.0	3.1	2.1	2.5	4.0	3.7	3.2	---	5.2	1.1
<i>T_{max} 650°F</i>													
60	6357	8	650	503	583	623	636	595	522	435	---	321	652
120			650	505	587	631	636	592	522	432	---	324	650
180			650	504	584	630	635	587	515	424	---	323	647
240			650	509	589	634	638	590	522	436	---	330	652
290			650	502	588	633	639	595	523	436	---	331	651
Average temp, °F			650	505	586	632	637	592	521	433	---	326	650
Std dev, °F			0.0	2.5	2.6	1.6	1.6	3.4	3.3	5.1	---	4.5	2.1
60	6459	8	650	517	587	630	637	594	524	437	---	315	648
120			650	518	589	631	640	595	527	442	---	316	651
180			650	514	583	627	635	591	523	437	---	313	647
240			650	516	587	628	636	593	523	438	---	318	648
290			650	513	587	631	639	595	530	442	---	316	648
Average temp, °F			650	516	587	629	637	594	525	439	---	316	648
Std dev, °F			0.0	2.1	2.2	1.9	2.1	1.7	3.1	2.6	---	1.9	1.6
60	6652	8	650	500	579	625	633	592	521	428	358	277	647
120			650	498	579	626	634	591	521	430	357	278	648
180			650	498	580	628	636	594	522	432	357	278	650
240			650	503	583	629	638	596	525	434	359	279	652
290			650	502	582	630	639	596	524	434	360	280	652
Average temp, °F			650	500	581	627	636	594	523	432	358	278	650
Std dev, °F			0.0	2.3	1.9	2.2	2.5	2.3	1.9	2.4	1.3	1.2	2.3

TABLE 39. THERMAL STABILITY OF TURBINE ENGINE FUELS IN ALCOR JFTOT

Test Pressure 350 psi
Dutch-weave 17μ in-line filter followed by 0.45μ filter

Run no.	T _{max} , °F	Heater temperature, °F, for first color rating as indicated								Heater breakpoint, °F		Filter ΔP in. Hg	Coker no.	Date tested
		1-hr rating				24-hr rating				1-hr	24-hr			
		1	2	3	4	1	2	3	4					
70-16 JP-7 fuel														
6366†	700	555	395‡	—	—	555	395‡	—	—	>700	>700	0.0	8	28 Jul 70
6372**	700	—	364	—	—	—	364	—	—	>700	>700	0.0	8	29 Jul 70
70-17 JP-7 fuel														
6378	700	467	680††	—	—	—	467	—	—	>700	>700	0.0	8	31 Jul 70
6386	700	476	697	—	—	—	399	—	—	>700	>700	0.0	8	3 Aug 70
70-18 JP-7 fuel														
6390	700	600	692	—	—	476	692	—	—	>700	>700	0.0	8	4 Aug 70
6393	700	—	467	—	—	—	390	—	—	>700	>700	0.0	8	5 Aug 70
OF JP-4														
6437	550	—	397‡	311‡	—	—	397‡	311‡	475††	311	311	10.0/169	9	21 Aug 70
6421	500	488‡	390‡	367‡	—	488‡	390‡	360‡	—	367	360	0.15	9	17 Aug 70
6417	450	—	310	350	—	—	295	350	—	350	350	0.0	9	14 Aug 70
6425	425	—	394‡	345‡	—	—	394‡	345‡	—	345	345	0.0	9	18 Aug 70
6431	425	300	405‡	348‡	—	300	423‡	335‡	—	348	335	0.0	9	20 Aug 70
6440	125	—	384‡	354‡	—	—	384‡	354‡	—	354	354	0.0	8	24 Aug 70
6447	415	415‡	344‡	—	—	415‡	357‡	325†	—	>415	325	0.0	8	26 Aug 70
6412	400	—	240	—	—	—	240	—	—	>400	>400	0.0	9	13 Aug 70
6429	400	217‡†	335	—	—	217‡†	335	—	—	>400	>400	0.0	9	19 Aug 70
6443	400	357‡	236‡	—	—	357‡	236‡	—	—	>400	>400	0.0	8	25 Aug 70
DR JP-4														
6475	560	—	385	—	560	—	385	—	560	560	560	0.0	8	4 Sep 70
6467	550	425	—	544	540	425	—	549	547	540	547	0.0	8	2 Sep 70
6476	550	428	503	548	—	—	486	548	535††	548	548	0.0	9	4 Sep 70
6471	540	—	482	—	—	—	482	—	—	>540	>540	0.0	8	3 Sep 70
6479	540	—	473	—	410††	—	473	—	410††	††	††	0.0	8	8 Sep 70
6480	540	—	480	—	—	—	480	—	—	>540	>540	0.0	9	8 Sep 70
6472	530	519	—	—	—	519	—	—	—	>530	>530	0.0	9	3 Sep 70
6468	525	265‡†	480	—	—	480	—	—	—	>525	>525	0.05	9	2 Sep 70
6463	500	456	—	—	—	456	—	—	—	>500	>500	0.0	8	1 Sep 70
HR JP-4														
6456	575	445	—	—	571	445	—	—	571	571	571	10.0/67	9	28 Aug 70
6460	575	304‡†	469	—	574	304‡†	490	—	571	574	571	10.0/62	9	31 Aug 70
6452	550	468	530	—	—	445	538	—	—	>550	>550	10.0/118	9	27 Aug 70
6464	525	—	262‡†	—	—	—	262‡†	—	—	>525	>525	10.0/128	9	1 Sep 70
6448	500	254‡†	282	—	—	254‡†	282	—	—	>500	>500	0.9	9	26 Aug 70
6444	450	—	290	—	—	—	308	—	—	>450	>450	0.0	9	25 Aug 70
6441	425	254	323††	—	—	254	323††	—	—	>425	>425	0.0	9	24 Aug 70

† Filter pressure drop (ΔP) at 300 minutes unless otherwise indicated.
 ‡ Test duration 284 minutes, water coolant supply system failed.
 † Maximum deposit occurs at the fuel inlet end of the tube; lower end deposits occur upstream closer to the T_{max} point.
 †† Test duration 280 minutes, water coolant supply system failed.
 ††† Peak deposit more than 0.2 inch beyond the T_{max} point data not used in breakpoint determination.
 †††† Deposit inception point beyond range of profile, temperature determined by interpolation.

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F											
			Inches from fuel outlet											
0.85 0.20 0.40 0.60 1.10 1.40 1.70 2.00 2.20 2.35 0.65														
$T_{max} 700^{\circ}F$														
60	6372	8	700	548	638	684	684	632	557	466	---	347	701	
120			700	551	641	685	689	635	559	463	---	352	703	
180			700	552	636	679	684	636	560	466	---	354	697	
240			700	547	632	684	691	652	578	485	---	351	702	
290			---	---	---	---	---	---	---	---	---	---	---	---
Average temp, °F			700	550	637	683	687	639	564	470	---	351	701	
Std dev, °F			0.0	2.4	3.7	2.7	3.6	8.5	9.7	10.1	---	2.9	2.6	
60	6378	8	700	548	635	680	682	636	561	467	---	344	699	
120			700	556	640	687	687	638	562	468	---	349	703	
180			700	555	637	686	688	639	566	471	---	349	701	
240			700	551	640	683	685	635	563	468	---	354	697	
290			700	550	637	680	689	639	566	469	---	353	701	
Average temp, °F			700	552	638	683	686	637	564	469	---	350	700	
Std dev, °F			0.0	3.3	2.2	3.3	2.8	1.9	2.3	1.6	---	4.0	2.3	
60	6386	8	700	550	624	676	687	640	569	475	---	342	698	
120			700	547	631	678	691	647	572	477	---	346	701	
180			700	549	629	678	687	639	567	475	---	358	700	
240			700	546	627	672	686	641	571	479	---	341	698	
290			700	553	631	679	688	641	572	479	---	345	700	
Average temp, °F			700	549	628	678	688	642	570	477	---	346	699	
Std dev, °F			0.0	2.7	3.0	3.2	1.9	3.1	2.2	2.0	---	6.7	1.4	
60	6390	8	700	543	627	678	689	642	572	476	---	332	701	
120			700	546	627	678	689	644	570	474	---	332	699	
180			700	543	627	676	687	641	572	478	---	335	700	
240			700	542	629	678	687	643	573	480	---	334	701	
290			700	540	623	678	689	642	573	482	---	336	698	
Average temp, °F			700	543	627	678	688	642	572	478	---	334	700	
Std dev, °F			0.0	2.2	2.2	1.0	1.1	1.2	1.2	3.1	---	1.8	1.3	
60	6393	8	700	541	626	678	687	640	565	467	---	332	701	
120			700	533	622	672	683	633	560	465	---	330	698	
180			700	544	625	679	685	640	563	467	---	336	700	
240			700	540	628	679	689	641	569	470	---	336	701	
290			700	539	623	676	686	640	564	469	---	333	700	
Average temp, °F			700	539	625	677	686	639	564	468	---	333	700	
Std dev, °F			0.0	4.0	2.4	2.8	2.2	3.3	3.3	2.0	---	2.6	1.2	

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 400°F</i>													
60	6443	8	400	317	359	386	393	369	330	277	---	206	397
120			400	317	361	387	395	371	350	279	---	209	400
180			400	317	359	386	393	369	329	278	---	208	397
240			400	316	360	388	395	372	331	280	---	210	400
290			400	318	359	387	394	370	331	280	---	209	399
Average temp., °F			400	317	360	387	394	370	330	279	---	208	399
Std dev., °F			0.0	0.7	1.0	0.9	1.0	1.3	0.9	1.3	---	1.6	1.6
60	6412	9	400	322	362	385	392	370	332	280	238	---	400
120			400	321	363	387	393	371	333	283	240	---	402
180			400	320	362	384	392	370	333	283	240	---	400
240			400	322	361	386	393	372	336	283	243	---	401
290			400	323	360	386	393	370	334	283	243	---	400
Average temp., °F			400	322	362	386	393	371	334	282	241	---	401
Std dev., °F			0.0	1.2	1.2	1.2	0.7	1.0	1.6	1.2	2.2	---	1.0
60	6429	9	400	320	362	387	394	372	335	280	238	---	401
120			400	323	360	386	394	373	336	282	239	---	401
180			400	320	360	385	393	373	335	282	238	---	401
240			400	321	363	387	396	375	338	283	240	---	402
290			400	323	361	387	394	374	336	284	239	---	401
Average temp., °F			400	321	361	386	393	373	336	282	239	---	401
Std dev., °F			0.0	1.6	1.3	1.0	1.1	1.2	1.2	1.5	0.9	---	0.5
<i>T_{max} 415°F</i>													
60	6447	8	415	327	372	400	410	386	344	291	---	215	414
120			415	326	372	401	411	386	346	293	---	217	414
180			415	326	373	398	407	384	344	290	---	218	413
240			415	329	373	400	413	388	346	294	---	218	415
290			415	328	371	400	410	387	347	293	---	220	414
Average temp., °F			415	327	372	400	410	386	345	292	---	218	414
Std dev., °F			0.0	1.3	0.9	0.9	2.2	1.5	1.4	1.1	---	1.9	0.7
<i>T_{max} 425°F</i>													
60	6440	8	425	337	384	412	420	397	354	299	---	220	423
120			425	337	382	411	418	393	352	297	---	221	424
180			425	338	383	413	419	393	351	299	---	222	425
240			425	336	385	410	419	393	350	298	---	224	423
290			425	339	383	412	420	393	353	297	---	223	427
Average temp., °F			425	337	383	412	419	394	352	298	---	222	424
Std dev., °F			0.0	1.2	1.2	1.2	0.9	1.5	1.6	1.0	---	1.6	1.7

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT

Test time, min	Test no.	Coke no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 400°F</i>													
60	6443	8	0.0	0.0	1.0	1.0	1.0	1.0	0.0	2.0	---	2.0	2.0
120			0.0	0.0	1.0	0.0	1.0	1.0	0.0	0.0	---	1.0	1.0
180			0.0	0.0	1.0	1.0	1.0	1.0	1.0	1.0	---	0.0	2.0
240			0.0	1.0	0.0	1.0	1.0	2.0	1.0	1.0	---	2.0	1.0
290			0.0	1.0	1.0	0.0	0.0	0.0	1.0	1.0	---	1.0	0.0
Average dev at 60 min = 1.0			Average dev at 180 min = 0.8					Average dev at 290 min = 0.5					
Average dev at 120 min = 0.5			Average dev at 240 min = 1.0										
60	6412	9	0.0	0.0	0.0	1.0	1.0	1.0	2.0	2.0	3.0	---	1.0
120			0.0	1.0	1.0	1.0	0.0	0.0	1.0	1.0	1.0	---	1.0
180			0.0	2.0	0.0	2.0	1.0	1.0	1.0	1.0	1.0	---	1.0
240			0.0	0.0	1.0	0.0	0.0	1.0	2.0	1.0	2.0	---	0.0
290			0.0	1.0	2.0	0.0	0.0	1.0	0.0	1.0	2.0	---	1.0
Average dev at 60 min = 1.1			Average dev at 180 min = 1.0					Average dev at 290 min = 0.8					
Average dev at 120 min = 0.7			Average dev at 240 min = 0.7										
60	6424	9	0.0	0.0	0.0	1.0	1.0	1.0	2.0	2.0	3.0	---	1.0
120			0.0	1.0	1.0	1.0	0.0	0.0	1.0	1.0	1.0	---	1.0
180			0.0	2.0	0.0	2.0	1.0	1.0	1.0	1.0	1.0	---	1.0
240			0.0	0.0	1.0	0.0	0.0	1.0	2.0	1.0	2.0	---	0.0
290			0.0	1.0	2.0	0.0	0.0	1.0	0.0	1.0	2.0	---	1.0
Average dev at 60 min = 1.1			Average dev at 180 min = 1.0					Average dev at 290 min = 0.8					
Average dev at 120 min = 0.7			Average dev at 240 min = 0.7										
<i>T_{max} 415°F</i>													
60	6447	8	0.0	0.0	0.0	0.0	0.0	0.0	1.0	1.0	---	3.0	0.0
120			0.0	1.0	0.0	1.0	1.0	0.0	1.0	1.0	---	1.0	0.0
180			0.0	1.0	1.0	2.0	3.0	2.0	1.0	2.0	---	0.0	1.0
240			0.0	2.0	1.0	0.0	3.0	2.0	1.0	2.0	---	0.0	1.0
290			0.0	1.0	1.0	0.0	0.0	1.0	2.0	1.0	---	2.0	0.0
Average dev at 60 min = 0.5			Average dev at 180 min = 1.30					Average dev at 290 min = 0.8					
Average dev at 120 min = 0.6			Average dev at 240 min = 1.20										
<i>T_{max} 425°F</i>													
60	6440	8	0.0	0.0	1.0	0.0	1.0	3.0	2.0	1.0	---	2.0	1.0
120			0.0	0.0	1.0	1.0	1.0	1.0	0.0	1.0	---	1.0	0.0
180			0.0	1.0	0.0	1.0	0.0	1.0	1.0	1.0	---	0.0	1.0
240			0.0	1.0	2.0	2.0	0.0	1.0	2.0	0.0	---	2.0	1.0
290			0.0	2.0	0.0	0.0	1.0	1.0	1.0	1.0	---	1.0	3.0
Average dev at 60 min = 1.10			Average dev at 180 min = 0.6					Average dev at 290 min = 1.00					
Average dev at 120 min = 0.60			Average dev at 240 min = 1.10										

TABLE 41. MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 500°F</i>													
60	6345	8	500	384	446	479	492	462	414	348	---	266	500
120			500	384	446	486	498	470	418	353	---	271	503
180			500	386	446	483	496	465	417	350	---	271	501
240			500	386	447	481	496	466	417	352	---	268	501
290			500	386	446	480	492	464	416	348	---	267	500
Average temp., °F			500	385	446	482	495	465	416	350	---	269	501
Std dev., °F			0.0	1.1	0.5	2.8	2.7	3.1	1.6	2.3	---	2.3	1.2
60	6463	8	500	402	456	486	491	456	403	338	---	247	499
120			500	403	455	487	491	458	406	339	---	248	500
180			500	401	453	485	489	455	402	337	---	248	499
240			500	405	455	486	491	458	405	338	---	250	500
290			500	401	454	484	489	457	405	337	---	251	498
Average temp., °F			500	402	455	486	490	457	404	338	---	249	499
Std dev., °F			0.0	1.7	1.2	1.2	1.1	1.3	1.6	0.9	---	1.6	0.9
60	6448	9	500	401	453	486	490	456	404	338	282	---	500
120			500	403	454	485	490	458	406	340	283	---	500
180			500	404	452	484	488	459	408	339	284	---	501
240			500	401	453	483	488	458	407	341	285	---	500
290			500	403	454	486	490	460	407	342	286	---	498
Average temp., °F			500	402	453	485	489	458	406	340	284	---	500
Std dev., °F			0.0	1.4	0.9	1.3	1.1	1.5	1.6	1.6	1.6	---	1.1
60	6421	9	500	407	456	487	488	459	409	340	292	---	501
120			500	406	455	485	487	457	407	342	291	---	500
180			500	404	458	489	490	460	409	343	291	---	502
240			500	408	459	488	489	461	412	345	295	---	500
290			500	407	454	485	488	461	410	346	295	---	501
Average temp., °F			500	406	456	487	488	460	409	343	293	---	501
Std dev., °F			0.0	1.6	2.1	1.8	1.2	1.7	1.9	2.4	1.6	---	0.9
<i>T_{max} 510°F</i>													
60	6328	8	510	389	452	489	504	473	420	354	---	268	508
120			510	392	453	492	505	474	423	359	---	265	510
180			510	394	451	492	504	475	423	359	---	272	510
240			510	387	448	486	503	475	424	360	---	265	507
290			510	390	452	493	504	475	425	361	---	277	508
Average temp., °F			510	390	451	490	504	474	423	359	---	269	509
Std dev., °F			0.0	2.7	1.9	2.9	0.7	1.0	1.6	2.7	---	5.1	1.9

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no	Coker no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F											
			Inches from fuel outlet											
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85	
<i>T_{max} 500°F</i>														
60	6345	8	0.0	1.0	0.0	3.0	3.0	3.0	2.0	2.0	---	3.0	1.0	
120			0.0	1.0	0.0	4.0	3.0	5.0	2.0	3.0	---	2.0	2.0	
180			0.0	1.0	0.0	1.0	1.0	0.0	1.0	0.0	---	2.0	0.0	
240			0.0	1.0	1.0	1.0	1.0	1.0	1.0	2.0	---	1.0	0.0	
290			0.0	1.0	0.0	2.0	3.0	1.0	0.0	2.0	---	2.0	1.0	
Average dev at 60 min = 1.80			Average dev at 180 min = 0.60					Average dev at 290 min = 1.20						
Average dev at 120 min = 2.20			Average dev at 240 min = 0.90											
60	6463	8	0.0	0.0	1.0	0.0	1.0	1.0	1.0	0.0	---	2.0	0.0	
120			0.0	1.0	0.0	1.0	1.0	1.0	1.0	1.0	---	1.0	1.0	
180			0.0	1.0	2.0	1.0	1.0	2.0	2.0	1.0	---	1.0	0.0	
240			0.0	3.0	0.0	0.0	1.0	1.0	1.0	0.0	---	1.0	1.0	
290			0.0	1.0	1.0	2.0	1.0	0.0	0.0	1.0	---	2.0	1.0	
Average dev at 60 min = 0.60			Average dev at 180 min = 1.10					Average dev at 290 min = 0.90						
Average dev at 120 min = 0.80			Average dev at 240 min = 0.80											
60	6448	9	0.0	1.0	0.0	1.0	1.0	2.0	2.0	2.0	2.0	---	0.0	
120			0.0	1.0	1.0	0.0	1.0	0.0	0.0	0.0	1.0	---	0.0	
180			0.0	2.0	1.0	1.0	1.0	1.0	2.0	1.0	0.0	---	1.0	
240			0.0	1.0	0.0	2.0	1.0	0.0	1.0	1.0	1.0	---	0.0	
290			0.0	1.0	1.0	1.0	1.0	2.0	1.0	2.0	2.0	---	2.0	
Average dev at 60 min = 1.10			Average dev at 180 min = 1.00					Average dev at 290 min = 1.30						
Average dev at 120 min = 0.40			Average dev at 240 min = 0.70											
60	6421	9	0.0	1.0	0.0	0.0	0.0	1.0	0.0	3.0	1.0	---	0.0	
120			0.0	0.0	1.0	2.0	1.0	3.0	2.0	1.0	2.0	---	1.0	
180			0.0	2.0	2.0	2.0	2.0	0.0	0.0	0.0	2.0	---	1.0	
240			0.0	2.0	3.0	1.0	1.0	1.0	3.0	2.0	2.0	---	1.0	
290			0.0	1.0	2.0	2.0	0.0	1.0	1.0	3.0	3.0	---	0.0	
Average dev at 60 min = 0.60			Average dev at 180 min = 1.10					Average dev at 290 min = 1.20						
Average dev at 120 min = 1.30			Average dev at 240 min = 1.60											
<i>T_{max} 510°F</i>														
60	6328	8	0.0	1.0	1.0	1.0	0.0	1.0	3.0	5.0	---	1.0	1.0	
120			0.0	2.0	2.0	2.0	1.0	0.0	0.0	0.0	---	4.0	1.0	
180			0.0	4.0	0.0	2.0	0.0	1.0	0.0	0.0	---	3.0	1.0	
240			0.0	3.0	3.0	4.0	1.0	1.0	1.0	1.0	---	4.0	2.0	
290			0.0	0.0	1.0	3.0	0.0	1.0	2.0	2.0	---	8.0	1.0	
Average dev at 60 min = 1.40			Average dev at 180 min = 1.10					Average dev at 290 min = 1.80						
Average dev at 120 min = 1.20			Average dev at 240 min = 2.00											

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 510°F (cont'd)</i>													
60	6337	8	0.0	0.0	2.0	1.0	1.0	1.0	3.0	4.0	---	1.0	3.0
120			0.0	2.0	1.0	2.0	1.0	0.0	0.0	1.0	---	2.0	2.0
180			0.0	1.0	1.0	2.0	3.0	1.0	2.0	0.0	---	1.0	2.0
240			0.0	0.0	2.0	4.0	5.0	1.0	2.0	0.0	---	7.0	2.0
290			0.0	1.0	2.0	1.0	3.0	2.0	1.0	4.0	---	5.0	2.0
Average dev at 60 min = 1.60			Average dev at 180 min = 1.30					Average dev at 290 min = 2.10					
Average dev at 120 min = 1.10			Average dev at 240 min = 2.30										
60	6342	8	0.0	0.0	2.0	1.0	2.0	2.0	3.0	2.0	---	4.0	1.0
120			0.0	0.0	1.0	1.0	0.0	1.0	1.0	1.0	---	3.0	2.0
180			0.0	0.0	0.0	1.0	1.0	2.0	2.0	1.0	---	0.0	0.0
240			0.0	0.0	0.0	1.0	0.0	1.0	1.0	0.0	---	0.0	1.0
290			0.0	2.0	0.0	0.0	2.0	4.0	3.0	4.0	---	2.0	1.0
Average dev at 60 min = 1.70			Average dev at 180 min = 0.70					Average dev at 290 min = 1.80					
Average dev at 120 min = 1.00			Average dev at 240 min = 0.40										
60	6349	8	0.0	1.0	2.0	2.0	0.0	2.0	0.0	1.0	---	3.0	2.0
120			0.0	1.0	1.0	3.0	3.0	2.0	4.0	2.0	---	5.0	0.0
180			0.0	1.0	0.0	1.0	2.0	0.0	1.0	0.0	---	1.0	1.0
240			0.0	1.0	1.0	1.0	4.0	3.0	1.0	2.0	---	3.0	0.0
290			0.0	1.0	0.0	0.0	0.0	2.0	2.0	0.0	---	1.0	0.0
Average dev at 60 min = 1.30			Average dev at 180 min = 0.70					Average dev at 290 min = 0.60					
Average dev at 120 min = 2.10			Average dev at 240 min = 1.60										
<i>T_{max} 525°F</i>													
60	6325	8	0.0	3.0	3.0	3.0	0.0	4.0	1.0	1.0	---	3.0	0.0
120			0.0	1.0	0.0	2.0	2.0	2.0	3.0	1.0	---	2.0	0.0
180			0.0	2.0	1.0	2.0	1.0	1.0	1.0	0.0	---	3.0	0.0
240			0.0	5.0	2.0	6.0	2.0	1.0	0.0	2.0	---	1.0	0.0
290			0.0	1.0	3.0	1.0	1.0	2.0	2.0	2.0	---	2.0	0.0
Average dev at 60 min = 1.80			Average dev at 180 min = 1.10					Average dev at 290 min = 1.40					
Average dev at 120 min = 1.30			Average dev at 240 min = 1.90										
60	6332	8	0.0	1.0	1.0	2.0	2.0	0.0	5.0	2.0	---	9.0	1.0
120			0.0	2.0	3.0	1.0	1.0	0.0	2.0	2.0	---	1.0	1.0
180			0.0	0.0	2.0	0.0	0.0	0.0	1.0	0.0	---	4.0	1.0
240			0.0	0.0	4.0	2.0	1.0	1.0	1.0	2.0	---	3.0	1.0
290			0.0	2.0	0.0	1.0	0.0	0.0	4.0	2.0	---	2.0	2.0
Average dev at 60 min = 2.30			Average dev at 180 min = 0.80					Average dev at 290 min = 1.30					
Average dev at 120 min = 1.30			Average dev at 240 min = 1.50										

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 525°F (cont'd)</i>													
60	6339	8	0.0	1.0	0.0	1.0	3.0	1.0	0.0	0.0	---	4.0	0.0
120			0.0	1.0	2.0	0.0	1.0	0.0	2.0	2.0	---	0.0	0.0
180			0.0	2.0	0.0	0.0	1.0	2.0	2.0	2.0	---	2.0	0.0
240			0.0	1.0	0.0	2.0	7.0	2.0	3.0	1.0	---	0.0	0.0
290			0.0	1.0	1.0	2.0	4.0	4.0	6.0	4.0	---	3.0	1.0
Average dev at 60 min = 1.00			Average dev at 180 min = 1.10					Average dev at 290 min = 2.60					
Average dev at 120 min = 0.80			Average dev at 240 min = 1.60										
60	6350	8	0.0	4.0	3.0	1.0	1.0	2.0	2.0	2.0	---	3.0	0.0
120			0.0	3.0	3.0	1.0	1.0	2.0	3.0	2.0	---	1.0	1.0
180			0.0	0.0	2.0	0.0	0.0	2.0	1.0	1.0	---	6.0	1.0
240			0.0	3.0	3.0	2.0	1.0	1.0	2.0	3.0	---	1.0	1.0
290			0.0	4.0	4.0	1.0	1.0	2.0	0.0	1.0	---	1.0	1.0
Average dev at 60 min = 1.80			Average dev at 180 min = 1.30					Average dev at 290 min = 1.50					
Average dev at 120 min = 1.70			Average dev at 240 min = 1.70										
60	6464	9	0.0	0.0	1.0	0.0	1.0	2.0	2.0	3.0	7.0	---	2.0
120			0.0	2.0	0.0	1.0	3.0	1.0	1.0	1.0	2.0	---	1.0
180			0.0	2.0	1.0	1.0	3.0	0.0	1.0	0.0	2.0	---	1.0
240			0.0	3.0	1.0	2.0	2.0	3.0	0.0	1.0	2.0	---	1.0
290			0.0	1.0	3.0	2.0	0.0	0.0	2.0	1.0	3.0	---	1.0
Average dev at 60 min = 1.80			Average dev at 180 min = 1.10					Average dev at 290 min = 1.30					
Average dev at 120 min = 1.20			Average dev at 240 min = 1.50										
60	6468	9	0.0	1.0	2.0	0.0	1.0	1.0	3.0	2.0	4.0	---	1.0
120			0.0	2.0	1.0	1.0	1.0	1.0	0.0	1.0	1.0	---	1.0
180			0.0	3.0	2.0	1.0	3.0	2.0	2.0	1.0	2.0	---	1.0
240			0.0	2.0	1.0	1.0	0.0	2.0	0.0	1.0	1.0	---	0.0
290			0.0	3.0	1.0	1.0	0.0	2.0	4.0	3.0	1.0	---	0.0
Average dev at 60 min = 1.50			Average dev at 180 min = 1.70					Average dev at 290 min = 1.50					
Average dev at 120 min = 0.90			Average dev at 240 min = 0.80										
<i>T_{max} 530°F</i>													
60	6472	9	0.0	0.0	0.0	0.0	5.0	2.0	1.0	2.0	1.0	---	1.0
120			0.0	0.0	2.0	3.0	1.0	1.0	0.0	0.0	1.0	---	1.0
180			0.0	0.0	1.0	1.0	1.0	2.0	2.0	2.0	1.0	---	1.0
240			0.0	1.0	0.0	0.0	1.0	1.0	2.0	0.0	1.0	---	1.0
290			0.0	0.0	2.0	3.0	5.0	2.0	2.0	1.0	1.0	---	2.0
Average dev at 60 min = 1.20			Average dev at 180 min = 1.10					Average dev at 290 min = 1.80					
Average dev at 120 min = 0.90			Average dev at 240 min = 0.70										

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 540°F</i>													
60	6471	8	0.0	2.0	3.0	4.0	3.0	2.0	1.0	0.0	---	2.0	0.0
120			0.0	2.0	0.0	1.0	3.0	2.0	1.0	3.0	---	3.0	1.0
180			0.0	0.0	0.0	1.0	2.0	1.0	1.0	0.0	---	0.0	2.0
240			0.0	2.0	0.0	2.0	1.0	0.0	0.0	0.0	---	2.0	0.0
290			0.0	0.0	0.0	1.0	1.0	2.0	1.0	1.0	---	4.0	0.0
Average dev at 60 min = 1.70			Average dev at 180 min = 0.70					Average dev at 290 min = 1.00					
Average dev at 120 min = 1.60			Average dev at 240 min = 0.70										
60	6479	8	0.0	2.0	4.0	2.0	3.0	3.0	3.0	3.0	---	2.0	3.0
120			0.0	3.0	4.0	3.0	2.0	1.0	1.0	0.0	---	0.0	2.0
180			0.0	1.0	1.0	3.0	2.0	2.0	2.0	1.0	---	0.0	1.0
240			0.0	3.0	2.0	1.0	0.0	0.0	0.0	2.0	---	0.0	2.0
290			0.0	0.0	1.0	1.0	0.0	1.0	1.0	1.0	---	3.0	0.0
Average dev at 60 min = 2.50			Average dev at 180 min = 1.30					Average dev at 290 min = 0.70					
Average dev at 120 min = 1.60			Average dev at 240 min = 1.00										
60	6480	9	0.0	5.0	3.0	2.0	0.0	0.0	0.0	0.0	0.0	---	2.0
120			0.0	1.0	1.0	0.0	2.0	0.0	1.0	2.0	1.0	---	1.0
180			0.0	1.0	3.0	2.0	2.0	1.0	2.0	3.0	1.0	---	1.0
240			0.0	2.0	0.0	2.0	1.0	2.0	1.0	1.0	1.0	---	1.0
290			0.0	2.0	6.0	3.0	1.0	0.0	3.0	3.0	2.0	---	3.0
Average dev at 60 min = 1.20			Average dev at 180 min = 1.60					Average dev at 290 min = 2.30					
Average dev at 120 min = 0.90			Average dev at 240 min = 1.10										
<i>T_{max} 550°F</i>													
60	6319	8	0.0	3.0	4.0	0.0	1.0	0.0	2.0	1.0	---	10.0	2.0
120			0.0	1.0	1.0	4.0	3.0	1.0	5.0	4.0	---	4.0	2.0
180			0.0	3.0	3.0	1.0	0.0	0.0	0.0	1.0	---	7.0	2.0
240			0.0	1.0	2.0	1.0	1.0	1.0	2.0	1.0	---	12.0	0.0
290			0.0	4.0	6.0	3.0	2.0	0.0	10.0	8.0	---	1.0	5.0
Average dev at 60 min = 2.30			Average dev at 180 min = 1.70					Average dev at 290 min = 3.90					
Average dev at 120 min = 2.50			Average dev at 240 min = 2.10										
60	6437	9	0.0	1.0	1.0	1.0	4.0	4.0	3.0	3.0	4.0	---	1.0
120			0.0	0.0	1.0	2.0	0.0	1.0	2.0	0.0	0.0	---	1.0
180			0.0	1.0	1.0	0.0	1.0	1.0	1.0	0.0	0.0	---	2.0
240			0.0	1.0	1.0	1.0	1.0	3.0	3.0	2.0	4.0	---	2.0
290			0.0	0.0	0.0	0.0	2.0	2.0	1.0	2.0	2.0	---	1.0
Average dev at 60 min = 2.20			Average dev at 180 min = 0.70					Average dev at 290 min = 1.0					
Average dev at 120 min = 0.70			Average dev at 240 min = 1.80										

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 550°F (cont'd)</i>													
60	6452	9	0.0	3.0	1.0	1.0	1.0	3.0	2.0	2.0	3.0	---	9.0
120			0.0	1.0	3.0	1.0	0.0	2.0	0.0	2.0	1.0	---	3.0
180			0.0	1.0	3.0	1.0	1.0	0.0	0.0	0.0	1.0	---	1.0
240			0.0	3.0	1.0	0.0	1.0	1.0	1.0	1.0	2.0	---	1.0
290			0.0	0.0	0.0	1.0	0.0	2.0	1.0	2.0	3.0	---	1.0
Average dev at 60 min = 2.50			Average dev at 180 min = 0.80					Average dev at 290 min = 1.10					
Average dev at 120 min = 1.30			Average dev at 240 min = 1.10										
60	6476	9	0.0	0.0	1.0	1.0	1.0	1.0	2.0	0.0	2.0	---	1.0
120			0.0	0.0	1.0	2.0	1.0	1.0	0.0	1.0	1.0	---	1.0
180			0.0	2.0	1.0	3.0	0.0	1.0	1.0	2.0	0.0	---	0.0
240			0.0	0.0	2.0	2.0	2.0	2.0	0.0	1.0	0.0	---	0.0
290			0.0	0.0	3.0	2.0	0.0	1.0	1.0	2.0	2.0	---	0.0
Average dev at 60 min = 0.90			Average dev at 180 min = 1.00					Average dev at 290 min = 1.10					
Average dev at 120 min = 0.80			Average dev at 240 min = 0.90										
<i>T_{max} 560°F</i>													
60	6475	8	0.0	3.0	1.0	1.0	3.0	2.0	2.0	0.0	---	2.0	0.0
120			0.0	1.0	3.0	2.0	1.0	1.0	2.0	2.0	---	1.0	0.0
180			0.0	2.0	1.0	2.0	4.0	4.0	4.0	2.0	---	1.0	3.0
240			0.0	0.0	1.0	1.0	2.0	1.0	1.0	1.0	---	1.0	0.0
290			0.0	1.0	2.0	0.0	0.0	2.0	1.0	0.0	---	1.0	2.0
Average dev at 60 min = 1.40			Average dev at 180 min = 2.30					Average dev at 290 min = 0.90					
Average dev at 120 min = 1.30			Average dev at 240 min = 0.80										
<i>T_{max} 575°F</i>													
60	6456	9	0.0	1.0	1.0	0.0	3.0	1.0	0.0	3.0	3.0	---	1.0
120			0.0	1.0	2.0	2.0	5.0	3.0	2.0	2.0	7.0	---	3.0
180			0.0	0.0	0.0	0.0	1.0	1.0	0.0	0.0	1.0	---	1.0
240			0.0	1.0	2.0	2.0	2.0	1.0	1.0	0.0	0.0	---	1.0
290			0.0	1.0	0.0	0.0	3.0	0.0	0.0	0.0	3.0	---	1.0
Average dev at 60 min = 1.30			Average dev at 180 min = 0.40					Average dev at 290 min = 0.80					
Average dev at 120 min = 2.70			Average dev at 240 min = 1.00										
60	6460	9	0.0	0.0	0.0	0.0	1.0	1.0	1.0	0.0	4.0	---	0.0
120			0.0	1.0	0.0	1.0	1.0	0.0	1.0	1.0	1.0	---	0.0
180			0.0	2.0	1.0	1.0	2.0	0.0	1.0	1.0	0.0	---	2.0
240			0.0	1.0	1.0	1.0	3.0	1.0	2.0	1.0	5.0	---	1.0
290			0.0	2.0	2.0	1.0	1.0	1.0	3.0	0.0	1.0	---	1.0
Average dev at 60 min = 0.70			Average dev at 180 min = 10.0					Average dev at 290 min = 1.20					
Average dev at 120 min = 0.60			Average dev at 240 min = 15.0										

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 610°F</i>													
60	6362	8	0.0	3.0	2.0	2.0	2.0	1.0	6.0	4.0	---	3.0	1.0
120			0.0	6.0	2.0	1.0	1.0	4.0	2.0	3.0	---	1.0	1.0
180			0.0	0.0	0.0	0.0	0.0	2.0	0.0	1.0	---	1.0	0.0
240			0.0	6.0	0.0	2.0	3.0	4.0	3.0	3.0	---	6.0	0.0
290			0.0	3.0	1.0	2.0	1.0	2.0	1.0	0.0	---	0.0	2.0
Average dev at 60 min = 2.40			Average dev at 180 min = 0.40					Average dev at 290 min = 1.20					
Average dev at 120 min = 2.10			Average dev at 240 min = 2.60										
<i>T_{max} 635°F</i>													
60	6360	8	0.0	7.0	3.0	2.0	2.0	3.0	3.0	2.0	---	5.0	2.0
120			0.0	1.0	2.0	3.0	4.0	0.0	2.0	2.0	---	9.0	0.0
180			0.0	2.0	0.0	0.0	1.0	2.0	5.0	4.0	---	0.0	0.0
240			0.0	2.0	4.0	1.0	2.0	5.0	4.0	4.0	---	1.0	0.0
290			0.0	3.0	3.0	2.0	1.0	5.0	2.0	1.0	---	1.0	1.0
Average dev at 60 min = 2.90			Average dev at 180 min = 1.40					Average dev at 290 min = 1.90					
Average dev at 120 min = 2.30			Average dev at 240 min = 2.30										
<i>T_{max} 650°F</i>													
60	6357	8	0.0	2.0	3.0	1.0	1.0	3.0	1.0	2.0	---	5.0	2.0
120			0.0	0.0	1.0	1.0	1.0	0.0	1.0	1.0	---	2.0	0.0
180			0.0	1.0	2.0	2.0	2.0	5.0	6.0	9.0	---	3.0	3.0
240			0.0	4.0	3.0	2.0	1.0	2.0	1.0	3.0	---	4.0	2.0
290			0.0	3.0	2.0	1.0	2.0	3.0	2.0	3.0	---	5.0	1.0
Average dev at 60 min = 2.00			Average dev at 180 min = 3.30					Average dev at 290 min = 2.20					
Average dev at 120 min = 0.70			Average dev at 240 min = 2.20										
60	6459	8	0.0	1.0	0.0	1.0	0.0	0.0	1.0	2.0	---	1.0	0.0
120			0.0	2.0	2.0	2.0	3.0	1.0	2.0	3.0	---	0.0	3.0
180			0.0	2.0	4.0	2.0	2.0	3.0	2.0	2.0	---	3.0	1.0
240			0.0	0.0	0.0	1.0	1.0	1.0	2.0	1.0	---	2.0	0.0
290			0.0	3.0	0.0	2.0	2.0	1.0	3.0	3.0	---	0.0	0.0
Average dev at 60 min = 0.60			Average dev at 180 min = 2.10					Average dev at 290 min = 1.40					
Average dev at 120 min = 1.80			Average dev at 240 min = 0.80										
60	6652	8	0.0	0.0	2.0	2.0	3.0	2.0	2.0	4.0	0.0	1.0	3.0
120			0.0	2.0	2.0	1.0	2.0	3.0	2.0	2.0	1.0	0.0	2.0
180			0.0	2.0	1.0	1.0	0.0	0.0	1.0	0.0	1.0	0.0	0.0
240			0.0	3.0	2.0	2.0	2.0	2.0	2.0	2.0	1.0	1.0	2.0
290			0.0	2.0	1.0	3.0	3.0	2.0	1.0	2.0	2.0	2.0	2.0
Average dev at 60 min = 1.90			Average dev at 180 min = 0.60					Average dev at 290 min = 2.00					
Average dev at 120 min = 1.70			Average dev at 240 min = 1.90										

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coke: no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F											
			Inches from fuel outlet											
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85	
$T_{max} 665^{\circ}F$														
60	6508	9	0.0	0.0	2.0	0.0	1.0	1.0	0.0	1.0	---	1.0	1.0	
120			0.0	1.0	2.0	0.0	1.0	1.0	2.0	2.0	---	0.0	1.0	
180			0.0	2.0	2.0	1.0	1.0	1.0	0.0	1.0	---	1.0	2.0	
240			0.0	3.0	2.0	1.0	1.0	1.0	1.0	1.0	---	1.0	3.0	
290			0.0	1.0	3.0	1.0	0.0	1.0	2.0	1.0	---	1.0	1.0	
Average dev at 60 min = 0.70			Average dev at 180 min = 1.10					Average dev at 290 min = 1.10						
Average dev at 120 min = 1.00			Average dev at 240 min = 1.40											
$T_{max} 675^{\circ}F$														
60	6509	8	0.0	0.0	1.0	1.0	3.0	3.0	3.0	2.0	---	2.0	0.0	
120			0.0	2.0	1.0	2.0	1.0	4.0	1.0	0.0	---	0.0	0.0	
180			0.0	3.0	1.0	0.0	1.0	1.0	2.0	1.0	---	0.0	1.0	
240			0.0	3.0	3.0	3.0	2.0	2.0	1.0	1.0	---	1.0	3.0	
290			0.0	0.0	2.0	3.0	5.0	4.0	4.0	3.0	---	3.0	2.0	
Average dev at 60 min = 1.50			Average dev at 180 min = 1.00					Average dev at 290 min = 2.60						
Average dev at 120 min = 1.10			Average dev at 240 min = 1.90											
$T_{max} 700^{\circ}F$														
60	6314	8	0.0	0.0	3.0	4.0	3.0	2.0	1.0	3.0	---	0.0	1.0	
120			0.0	2.0	3.0	4.0	6.0	5.0	3.0	3.0	---	2.0	3.0	
180			0.0	2.0	4.0	1.0	0.0	0.0	2.0	0.0	---	1.0	1.0	
240			0.0	5.0	3.0	2.0	3.0	3.0	1.0	3.0	---	3.0	3.0	
290			0.0	3.0	1.0	2.0	1.0	0.0	3.0	1.0	---	2.0	1.0	
Average dev at 60 min = 1.70			Average dev at 180 min = 1.10					Average dev at 290 min = 1.40						
Average dev at 120 min = 3.10			Average dev at 240 min = 2.60											
60	6354	8	0.0	4.0	1.0	2.0	1.0	1.0	1.0	1.0	---	6.0	1.0	
120			0.0	1.0	4.0	1.0	0.0	0.0	2.0	4.0	---	3.0	2.0	
180			0.0	1.0	3.0	1.0	2.0	6.0	3.0	9.0	---	3.0	3.0	
240			0.0	2.0	5.0	0.0	3.0	3.0	7.0	6.0	---	10.0	0.0	
290			0.0	0.0	5.0	2.0	2.0	6.0	9.0	11.0	---	1.0	2.0	
Average dev at 60 min = 1.80			Average dev at 180 min = 3.10					Average dev at 290 min = 3.80						
Average dev at 120 min = 1.70			Average dev at 240 min = 3.60											
60	6366	8	0.0	5.0	0.0	2.0	2.0	0.0	1.0	3.0	---	4.0	2.0	
120			0.0	5.0	1.0	1.0	0.0	2.0	1.0	7.0	---	1.0	1.0	
180			0.0	1.0	0.0	0.0	1.0	3.0	2.0	6.0	---	1.0	2.0	
240			0.0	7.0	3.0	2.0	2.0	0.0	2.0	4.0	---	6.0	1.0	
290			---	---	---	---	---	---	---	---	---	---	---	
Average dev at 60 min = 1.90			Average dev at 180 min = 1.60					Average dev at 290 min = --						
Average dev at 120 min = 1.90			Average dev at 240 min = 2.70											

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F										
			Inches from fuel outlet										
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85
<i>T_{max} 700°F (cont'd)</i>													
60	6372	8	0.0	2.0	1.0	1.0	3.0	7.0	7.0	4.0	---	4.0	0.0
120			0.0	1.0	4.0	2.0	2.0	2.0	5.0	7.0	---	1.0	2.0
180			0.0	2.0	1.0	4.0	3.0	3.0	4.0	4.0	---	3.0	4.0
240			0.0	3.0	5.0	1.0	4.0	13.0	14.0	15.0	---	0.0	1.0
290			---	---	---	---	---	---	---	---	---	---	---
Average dev at 60 min = 2.90			Average dev at 180 min = 2.80					Average dev at 290 min = --					
Average dev at 120 min = 2.60			Average dev at 240 min = 5.60										
60	6378	8	0.0	4.0	3.0	3.0	4.0	1.0	3.0	2.0	---	6.0	1.0
120			0.0	4.0	2.0	4.0	1.0	1.0	2.0	1.0	---	1.0	3.0
180			0.0	3.0	1.0	3.0	2.0	2.0	2.0	2.0	---	1.0	1.0
240			0.0	1.0	2.0	3.0	1.0	2.0	1.0	1.0	---	4.0	3.0
290			0.0	2.0	1.0	3.0	3.0	2.0	2.0	0.0	---	3.0	1.0
Average dev at 60 min = 2.70			Average dev at 180 min = 1.70					Average dev at 290 min = 1.70					
Average dev at 120 min = 1.90			Average dev at 240 min = 1.50										
60	6386	8	0.0	1.0	4.0	2.0	1.0	2.0	1.0	2.0	---	4.0	1.0
120			0.0	2.0	3.0	0.0	3.0	5.0	2.0	0.0	---	0.0	2.0
180			0.0	0.0	1.0	0.0	1.0	3.0	3.0	2.0	---	12.0	1.0
240			0.0	3.0	1.0	6.0	2.0	1.0	1.0	2.0	---	5.0	1.0
290			0.0	4.0	3.0	1.0	0.0	1.0	2.0	2.0	---	1.0	1.0
Average dev at 60 min = 1.80			Average dev at 180 min = 2.30					Average dev at 290 min = 1.50					
Average dev at 120 min = 1.70			Average dev at 240 min = 2.20										
60	6390	8	0.0	0.0	0.0	0.0	1.0	0.0	0.0	2.0	---	2.0	1.0
120			0.0	3.0	2.0	0.0	1.0	2.0	2.0	4.0	---	2.0	1.0
180			0.0	0.0	0.0	2.0	1.0	1.0	0.0	0.0	---	1.0	0.0
240			0.0	1.0	2.0	0.0	1.0	1.0	1.0	2.0	---	0.0	1.0
290			0.0	3.0	4.0	0.0	0.0	2.0	1.0	4.0	---	1.0	2.0
Average dev at 60 min = 0.60			Average dev at 180 min = 0.50					Average dev at 290 min = 1.70					
Average dev at 120 min = 1.50			Average dev at 240 min = 0.90										
60	6393	8	0.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	---	1.0	1.0
120			0.0	6.0	3.0	5.0	3.0	6.0	4.0	3.0	---	3.0	2.0
180			0.0	5.0	0.0	2.0	1.0	1.0	1.0	1.0	---	3.0	0.0
240			0.0	1.0	3.0	2.0	3.0	2.0	5.0	2.0	---	3.0	1.0
290			0.0	0.0	2.0	1.0	0.0	1.0	0.0	1.0	---	0.0	0.0
Average dev at 60 min = 1.00			Average dev at 180 min = 1.40					Average dev at 290 min = 0.50					
Average dev at 120 min = 3.50			Average dev at 240 min = 2.20										

TABLE 42. DEVIATION DATA FROM MULTIPLE TEMPERATURE PROFILES IN ALCOR JFTOT (Cont'd)

Test time, min	Test no.	Coker no.	Deviation values from average tube temperature at indicated distance from fuel outlet, °F												
			Inches from fuel outlet												
			0.85	0.20	0.40	0.60	1.10	1.40	1.70	2.00	2.20	2.35	0.85		
<i>T_{max} 700°F (cont'd)</i>															
60	6399	8	0.0	0.0	3.0	3.0	1.0	3.0	3.0	3.0	---	1.0	1.0		
120			0.0	1.0	0.0	1.0	0.0	1.0	0.0	1.0	0.0	1.0	---	0.0	2.0
180			0.0	1.0	2.0	1.0	2.0	2.0	2.0	2.0	2.0	2.0	---	1.0	1.0
240			0.0	2.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	2.0	---	1.0	2.0
290			0.0	0.0	1.0	2.0	1.0	1.0	1.0	2.0	0.0	---	1.0	0.0	
Average dev at 60 min = 1.80			Average dev at 180 min = 1.40					Average dev at 290 min = 0.80							
Average dev at 120 min = 0.60			Average dev at 240 min = 0.80												
60	6403	8	0.0	2.0	1.0	1.0	0.0	3.0	3.0	4.0	---	2.0	0.0		
120			0.0	4.0	4.0	3.0	2.0	2.0	2.0	3.0	---	4.0	1.0		
180			0.0	3.0	2.0	4.0	0.0	1.0	0.0	4.0	---	1.0	1.0		
240			0.0	5.0	1.0	2.0	5.0	3.0	1.0	3.0	---	6.0	1.0		
290			0.0	5.0	1.0	0.0	0.0	2.0	2.0	2.0	---	2.0	1.0		
Average dev at 60 min = 1.60			Average dev at 180 min = 1.60					Average dev at 290 min = 1.50							
Average dev at 120 min = 2.50			Average dev at 240 min = 2.70												
60	6408	8	0.0	1.0	4.0	1.0	2.0	2.0	2.0	2.0	---	5.0	1.0		
120			0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	---	1.0	1.0	
180			0.0	3.0	1.0	2.0	1.0	1.0	1.0	3.0	---	2.0	1.0		
240			0.0	1.0	5.0	2.0	1.0	2.0	1.0	2.0	---	1.0	2.0		
290			0.0	3.0	4.0	1.0	1.0	0.0	1.0	1.0	---	2.0	1.0		
Average dev at 60 min = 2.00			Average dev at 180 min = 1.50					Average dev at 290 min = 1.40							
Average dev at 120 min = 0.30			Average dev at 240 min = 1.80												
60	6451	8	0.0	1.0	1.0	0.0	0.0	1.0	0.0	0.0	---	1.0	0.0		
120			0.0	2.0	1.0	0.0	1.0	0.0	1.0	1.0	---	2.0	2.0		
180			0.0	3.0	2.0	1.0	0.0	2.0	1.0	0.0	---	2.0	1.0		
240			0.0	0.0	0.0	1.0	0.0	1.0	1.0	1.0	---	3.0	0.0		
290			0.0	0.0	0.0	0.0	1.0	2.0	1.0	1.0	---	0.0	1.0		
Average dev at 60 min = 0.40			Average dev at 180 min = 1.20					Average dev at 290 min = 0.60							
Average dev at 120 min = 1.00			Average dev at 240 min = 0.70												

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