

FOREWORD

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This is the final technical report under this contract, covering work performed from June 15, 1963 through November 14, 1964.

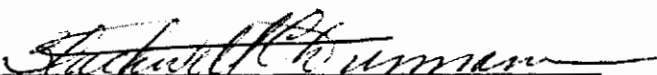
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ABSTRACT

A study has been made of the use of additives to prevent or retard the deterioration of JP-6 fuel thermal stability during storage. Fuel-additive blends were stored at 130°F in sealed 15-gallon steel drums for periods up to 16 weeks. Thermal stability was measured by means of a modified fuel coker developed during this program, using helium pressurization instead of a fuel pump. The standard ASTM-CRC fuel coker and the Eppi-CRC modified fuel coker were found to be unsuitable for this purpose, owing to contamination of the test sections with pump wear debris. A total of 52 additives were submitted by industry for evaluation, from which 19 were selected by screening tests for inclusion in the hot-room storage program. Three JP-6 fuels were used as the base stocks for additive evaluation. Under storage conditions giving a 100°F decrease in base fuel thermal stability, four additives or combinations of additives were found to be effective in maintaining thermal stability. These results indicate that the use of additives is a feasible approach to solving current problems in this area.

This report has been reviewed and is approved.

  
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## SECTION I

### INTRODUCTION

This program was directed toward the comparative evaluation of additives for their ability to retard deterioration of hydrocarbon fuels during storage, this deterioration being measured in terms of decrease in thermal stability. Such deterioration has become a practical problem in connection with JP-6 (MIL-J-25656B) and with "thermally stable fuel" (MIL-F-25524A). These fuels are required to pass thermal stability tests at temperatures 100-150°F higher than the level required for current large-volume jet fuels, and quality maintenance at this higher level becomes critical. Attention has been directed primarily to the JP-6 problem, in view of the projected large-volume use of such fuel in the supersonic transport and other high-performance aircraft.

There are several possible means of improving the thermal and storage stability of jet fuels, particularly the use of special refining processes and the use of inert-gas blanketing during storage and/or service. However, the use of additives offers potentially a means of improving thermal and storage stability at low cost and without any capital expenditures in new treating capacity or special handling equipment. The questions of cost and facility requirements are especially important in connection with any projected large-volume use of high-temperature jet fuels. The use of additives in jet fuels for other purposes has been well established for many years.

As in any use of special-purpose fuel additives, consideration must be given to the possible adverse effects of thermal and storage stability additives on other fuel properties. In particular, it is known that many of the materials evaluated in the past as thermal stability additives have been harmful to the demulsibility or water-separating properties of jet fuels. In order to avoid such effects, it was considered necessary in the present program to screen candidate additives to eliminate materials with pronounced effects on the water-separating properties.

In the early stages of the program, a detailed review was made of the past and current efforts in the field of thermal and storage stability testing and the use of additives for improving the stability of jet fuels. The findings of this review were published as a special report<sup>(1)</sup>.

The experimental program as originally conceived was to screen a very large number of candidate additives, using a small-scale test method for

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evaluating thermal and storage stability. However, it became apparent at the start of the program that no small-scale method had been developed to the point of proven validity. Since the scope of the original program did not include any test method development, it was decided to base the evaluations on hot-room storage at a moderately elevated temperature, determining the thermal stability by means of a modified fuel coker test, run over a range of temperatures to establish breakpoints. This decision limited the number of additives that could be evaluated, since even with the modified coker the test time and sample requirements are considerable.

Using the Eppi-CRC modified fuel coker, certain problems appeared in operation and in interpretation of results that necessitated test method development. A large part of the work in the early stages of the program was concerned with developing a valid method of thermal stability evaluation.

All storage tests were conducted on fuel-additive blends stored at 130° F in 15-gallon steel drums for periods up to 16 weeks. Storage conditions with regard to availability of moisture and air were varied in the early stages of the program in order to obtain the desired level of deterioration. Three JP-6 base fuels were used in evaluating the additives. Out of a total of 52 additives that were obtained for evaluation, 19 were subjected to storage stability tests; the remainder were eliminated because of poor performance in preliminary screening tests or, in a few cases, because of lack of any background information from the suppliers.

## SECTION II

### BASE FUELS AND ADDITIVES

#### A. Base Fuels

The base fuels used in this program consisted of three 20-drum batches of JP-6 fuels furnished by the RTD Aero Propulsion Laboratory. Two of the 20-drum batches were supplied by Ashland Oil and Refining Company and one 20-drum batch by Standard Oil Company of California. These fuels were all additive-free except that each contained 0.10-0.15 volume percent methoxyethanol anti-icing additive, as required in current JP-6 procurements.

In addition to the JP-6 fuels, a single drum identified as Base Oil No. 1 was supplied by Phillips Petroleum Company, originally intended for purposes of correlation between thermal stability programs in progress at Phillips and SwRI. It is understood to be an additive-free paraffinic alkylate; the viscosity and boiling range are considerably higher than those of JP-6 fuels. This drum of fuel was later found to be nonrepresentative of the batch used in the Phillips program and so could not be used for correlation. Only limited use was made of this fuel in the program.

The base fuels were placed in 32°F cold storage immediately on receipt at SwRI and were held in cold storage throughout the program. Code numbers assigned to the base fuels were as follows:

G-1060 - JP-6, Ashland, received Oct 63  
H-1008 - JP-6, Ashland, received Feb 64  
H-1010 - JP-6, SOCal, received Feb 64  
H-1019 - Base Oil No. 1, Phillips, received Mar 64

Suppliers' and SwRI inspection data on these base fuels are presented in Table 1.

The initial thermal stability ratings on the JP-6 base fuels were somewhat contradictory, since difficulty was encountered in obtaining reproducible results in the standard ASTM-CRC fuel coker. This situation is discussed in detail in Section III. In view of this confused situation, the thermal stability of the base fuels will be discussed here only in general terms.

The G-1060 base fuel, which was used in the first two series of hot-room storage tests, was originally intended to be a fuel of marginal thermal and storage stability as measured by the standard ASTM-CRC fuel coker. However, the supplier's data on this fuel showed a clear-cut pass at 450/550°F, as did a subsequent check at SwRI. The SwRI data showed a preheater break-point at 475/575°F, which is somewhat suspect as these conditions are

TABLE 1. INSPECTION DATA ON BASE FUELS

	G-1060 (Ashland)		H-1008 (Ashland)		H-1010 (SOCal)		H-1019 (Phillips)		JP-6 Specs	
	Supplier	SwRI	Supplier	SwRI	Supplier	SwRI	Supplier	SwRI	Supplier	SwRI
Distillation: IBP, °F . . . . .	326	317	314	308	309	307	362	307	280 Min	
10%, °F . . . . .	332	330	325	324	319	320	372	320	350 Max	
50%, °F . . . . .	346	344	340	339	327	329	394	329	425 Max	
90%, °F . . . . .	368	369	364	365	341	344	480	344	500 Max	
EP, °F . . . . .	412	385	384	396	367	370	552	370		
IBP + 50%, °F . . . . .	672	661	654	647	636	636	756	636	600 Min	
Residue, vol % . . . . .	1.0	1.3	1.0	0.9	1.2	1.0	2.0	1.0	1.5 Max	
Loss, vol % . . . . .	1.0	0.1	1.0	0.3	0.8	0.2	0.0	0.0	1.5 Max	
Gravity, API/60 . . . . .	47.6	47.7	49.3	49.3	49.1	49.2	52.5	49.2	37-50	
Existing gum, mg/100 ml . . . . .	0.2	-	0.4	<1.0	0.1	<1.0	1.1	0.1	5.0 Max	
Total potential gum, mg/100 ml . . . . .	1.2	-	1.2	1.0	1.8	4.0	6.6	4.0	10.0 Max	
Insol potential gum, mg/100 ml . . . . .	-	-	-	-	0.0	-	1.1	-		
Sulfur, total, wt % . . . . .	0.016	0.00	0.037	0.00	0.01	0.01	0.0003	0.01	0.4 Max	
Mercaptan sulfur, wt % . . . . .	0.0006	-	0.0006	-	0.0001	-	<0.0002	-	0.001 Max	
Doctor test . . . . .	-	Sweet	-	Sweet	Sweet	Sweet	-	Sweet	Sweet	
Freezing point, °F . . . . .	<-80	-	<-80	<-66	<-76	<-66	-78	<-66	-65 Max	
Aniline point, °F . . . . .	136.4	137.8	138.2	140.0	144.4	145.8	189.2	145.8		
Aniline-gravity product . . . . .	6,493	6,573	6,813	6,902	7,090	7,173	-	7,173	5,250 Min	
Net heat of combustion, BTU/lb . . . . .	-	-	-	-	18,835	-	18,950	-	18,400 Min	
Viscosity, cs at 100°F . . . . .	-	1.05	-	1.00	-	1.00	-	1.00		
at -40°F . . . . .	5.39	5.13	4.43	-	4.53	-	21.34	-	7.0 Max	
Aromatics, vol % . . . . .	10.0	-	8.8	8.5	2.2	1.5	3.4	1.5	25.0 Max	
Olefins, vol % . . . . .	1.5	-	2.3	3.5	1.6	1.5	*	1.5	5.0 Max	
Smoke point, mm . . . . .	27.0	-	29	34	32	40	41	40	20 Min	
Corrosion, copper strip . . . . .	1-A	-	1-B	1	1-B	1	1-A	1	1-B Max	
Water reaction . . . . .	1	1	1	1	1-b	1	1	1	1-b Max	
Thermal stability at 450/550°F										
Filter ΔP, in. Hg . . . . .	0	**	1.0	**	0.55	**	-	**	10 Max	
Preheater rating . . . . .	1	**	3	**	3	**	-	**	3 Max	
Water separator index, WSI . . . . .	96	-	100	100	-	100	-	100	-	
Water separator index, WSIM . . . . .	-	72	-	94	96	99	-	99	-	
Anti-icing additive, vol % . . . . .	0.134	0.127	0.143	0.142	0.107	0.109	None	0.109	0.10-0.15	

\*Bromine number 1.8.

\*\*See text.

\*\*\*Stable above the temperature limit of the standard coker.

†Additional data furnished by supplier: Water content 20 ppm; Saybolt color t27; neutralization no. 0.05 mg KOH/g; flash point 144° F; naphthalenes 0.04 wt %; indenes less than 5 ppm; total nitrogen less than 0.1 ppm; pyrrole nitrogen 0.1 ppm; basic nitrogen less than 1 ppm; trace copper 8 ppb; soluble iron less than 1 ppm; soluble lead 7 ppb; phenols less than 1 ppm; peroxides less than 1 ppm; total oxygen 0.079 wt %.

crowding the operating limit of the standard coker. Subsequent tests in modified fuel cokers indicated that the true breakpoint was well over 500/600° F.

The H-1008 base fuel, which was used in the third and fifth series of hot-room storage tests, showed a marginal thermal stability at 450/550° F according to the supplier's data (pressure drop 1.0 in. Hg, preheater rating No. 3). Attempts to rate this fuel at 450/550° F in the standard ASTM-CRC coker at SwRI gave highly variable results - pressure drops from 0.7 to 25 in. Hg and preheater ratings from No. 0 to No. 4. Subsequently, this fuel was tested over a range of temperatures from 400/500 to 475/575° F, with failing filter pressure drops at all temperatures and a preheater breakpoint of 475/575° F, but with wipable deposits. These erratic results were attributed to pumping difficulties in the standard coker, and it is concluded that no valid standard-coker rating had been obtained. Subsequent tests in modified fuel cokers indicated that the true breakpoint was 500/600° F or higher.

The H-1010 base fuel, which was used in the fourth and sixth series of hot-room storage tests, gave a marginal thermal stability at 450/550° F according to the supplier's data (pressure drop 0.55 in. Hg, preheater rating No. 3). In SwRI standard-coker tests, this fuel was similar to the H-1008; i. e., failing filter pressure drops were observed over the entire range from 400/500 to 475/575° F, and a preheater breakpoint (wipable deposits) was observed at 475/575° F. Again, it was concluded that no valid standard-coker rating had been obtained. Subsequent tests in modified fuel cokers indicated that the true breakpoint was above 500/600° F.

The problem of rating base fuel thermal stability is tied up with the problem of developing a suitable test method, which is discussed in detail in Section III.

In addition to the base fuels that have been discussed, use was made of a commercial turbo kerosine, procured locally from one of the major oil companies. This fuel as received contained 5 lb/1000 bbl of Santolene C corrosion inhibitor. It was used as a base stock for initial screening tests on additives in the CRC water separator. For this purpose, 0.1% methoxy-ethanol was added to the fuel. The kerosine was also used in thermal stability tests to establish a correlation between test methods.

## B. Additives

Candidate additives were solicited from industry by the RTD Aero Propulsion Laboratory. Certain information was requested along with the additive samples. This information included data on the effect of the additive on fuel properties, preferably in JP-6 fuels, but alternatively in JP-4, JP-5,

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or ASTM kerosines (turbine fuels). Specific data were requested on thermal stability (ASTM-CFR fuel coker breakpoint analysis) and water-separating properties (standard and modified water separometer tests). Information was also requested on the recommended concentration of additive in the fuel, and it was indicated that concentrations of 30 lb/1000 bbl or lower were preferred. However, consideration was also given to the use of certain additives that were effective only at higher concentrations, so that the 30 lb/1000 bbl level was not to be regarded as an absolute limit.

For screening purposes, both the suppliers' data and confirmatory water separometer tests run at SwRI and RTD were used. The SwRI data were obtained on blends of each additive in a turbo kerosine containing 0.1% methoxyethanol. The RTD data were obtained on blends in JP-4 fuel. The data furnished by the suppliers of the candidate additives are presented in Tables 2-8. A summary of the water separometer results is given in Table 9.

In evaluating these results for selecting additives to include in the hot-room storage tests, it was understood that thermal stability breakpoint data do not necessarily give information on the ability of a given additive to maintain fuel thermal stability during storage. However, other things being equal, there was considerable interest in evaluating additives that gave good initial thermal stability.

Most of the additives included in the first two series of hot-room storage tests were those on which thermal stability data had been furnished by the respective suppliers and which did not show severe effects on the water separometer index. However, one antioxidant, designated E-1, had been included without any background of thermal stability data. This additive was found to have a pronounced adverse effect on fuel thermal stability, which became even worse during the storage period. Therefore, there was some question regarding the inclusion of certain additives in the third and fourth series of tests without first running some thermal stability screening tests. Preliminary tests were run for this purpose in H-1010 base fuel in the modified coker at 500/600° F (50° F below the breakpoint of the base fuel). All additives failing under these conditions were eliminated from the program.

A summary of the additive screening results for the entire program is given in Table 10, showing the list of all additives received, reasons for eliminating some of them from the storage program, and the series in which the remaining additives were tested. Many additives were eliminated because of adverse effects on water separometer results (WSIM), some of them because of adverse effects on thermal stability (TS), and one additive because it was fuel-insoluble. In general, preference in selecting additives for storage tests early in the program was given to those additives for which the suppliers had furnished background data.



TABLE 2. SUPPLIER'S DATA ON ADDITIVE A-1

Base Fuel:	JP-4*	JP-4*
Additive A-1 concn, lb/1000 bbl:	<u>0</u>	<u>12.5</u>
ASTM-CFR fuel coker tests:		
300/400 °F	0.0"; #0	- -
350/400 °F	0.2"; #3	0.0"; #0
375/400 °F	13.0"/223; #4	0.0"; #2-
400/400 °F	13.0"/230; #4+	13.0"/266; #4+**
Water separator:		
WSI	98	96
WSIM	45	55
Existent gum, mg/100 ml	0.8	1.4
Potential gum (16-hr), mg/100 ml	5.0	6.2

Complete inspection data were furnished on fuel with and without additive; at this concentration, no significant effects on fuel properties were observed except those listed.

\*The JP-4 fuel used for these tests contained:

Ethyl 733 antioxidant	5.0 lb/1000 bbl
Santolene C corrosion inhibitor	5.0 lb/1000 bbl
Metal deactivator	1.2 lb/1000 bbl

\*\*Smaller area than in corresponding test on fuel without A-1.

TABLE 3. SUPPLIER'S DATA ON B-SERIES ADDITIVES

Additive and Concn, lb/1000 bbl*	Fuel Coker Test Results at Conditions Shown				Water Separator		
	400/500/6	425/525/6	450/550/6	475/575/6	500/600/2.5	WSI	WSIM
None (JP-6 base fuel)	0.1"; #1	0.1"; #2	1.2"; #3	25"/212; #4	--	100	98
B-1; 20	0.1"; #1	0.2"; #1	1.3"; #2	25"/226; #3	21"; #4	100	98
50	0.1"; #1	0.3"; #1	1.1"; #1	25"/275; #1	15"; #4	100	96
200	0.1"; #1	0.5"; #1	0.5"; #1	25"/261; #1	18"; #4	100	95
2000	0.0"; #1	0.2"; #1	0.4"; #1	25"/230; #1	13"; #4	100	44
B-2; 20	0.1"; #1	0.1"; #1	0.6"; #2	19"; #2	16"; #4	100	94
50	0.1"; #1	0.2"; #1	0.3"; #1	0.8"; #1	5.6"; #4	100	93
200	0.1"; #1	0.1"; #1	0.2"; #1	0.4"; #1	2.3"; #4	91	67
2000	0.0"; #1	0.1"; #1	0.1"; #1	0.2"; #1	1.2"; #4	65	18
B-3; 20	0.0"; #1	0.0"; #1	0.3"; #2	1.4"; #2	13"; #4	99	97
50	0.0"; #1	0.0"; #1	0.1"; #1	0.2"; #1	2.8"; #4	96	81
200	0.0"; #1	0.0"; #1	0.0"; #1	0.1"; #1	1.7"; #4	85	43
2000	0.0"; #1	0.0"; #1	0.0"; #1	0.0"; #1	0.9"; #4	47	26

Other inspection data given for each additive blend included water tolerance, corrosion, existent and potential gum, and heating value. There were no significant effects on these properties except an increase in the water tolerance test result for the 2000 lb/1000 bbl blends of Additives B-2 and B-3 (these gave 2 ml, vs 1 ml for the other blends and the base fuel).

\* Concentrations given in table refer to material as received at SwRI, which consisted of a kerosine concentrate (50% active ingredient). Supplier's data originally gave fuel blend concentrations in terms of active ingredient.

TABLE 4. SUPPLIER'S DATA ON C-SERIES ADDITIVES

Base fuel:	JP-4*	JP-4*	JP-4*
C-series additive:	None	C-1	C-2
Concentration, ppm**	--	10	10
CFR fuel coker tests:			
300/400°F	0.7"; #1	---	---
350/450°F	1.3"; #1	---	---
400/500°F	6.0"; #2	0.2"; #3	0.3"; #4
425/525°F	25.0"/126; #4	0.8"; #4	2.8"; #4
Water separator index:			
WSI	100	56	55
WSIM	94	36	35
Existent gum, mg/100 ml	0.7	1.1	0.8
Potential gum, mg/100 ml	2.5	2.8	3.0

Existent and potential gum values for 40 ppm additive were as follows:

Existent gum	1.8	1.8
Potential gum	3.0	3.2

Copper corrosion rating was 1A for blends of 40 ppm additive.

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\*The JP-4 fuel used for these tests contained 8 lb/1000 bbl of antioxidant (N, N'-di-sec-butyl-p-phenylenediamine) and 2 lb/1000 bbl of metal deactivator (N, N'-disalicylidene-1, 2-propanediamine).

\*\*In this fuel, 10 ppm = approx. 2.7 lb/1000 bbl.

TABLE 5. SUPPLIER'S DATA ON D-SERIES ADDITIVES

Additive	Concn, lb/1000 bbl	High-Temperature Research Coker Test Results*						WSIM
		400/500	450/550	500/600	550/650	600/700	600/700	
None (JP-6 type fuel)**		0.0"; #2	0.0"; #4	0.0"; #8	-----	-----	-----	100
D-1	20	-----	-----	0.0"; #2	0.1"; #3	0.6"; #5	-----	85.5
D-2	20	-----	-----	0.0"; #2	0.0"; #2	0.0"; #5	-----	71.3
D-3	30	-----	-----	0.0"; #2	0.0"; #4	-----	-----	85
D-4	30	-----	-----	0.0"; #2	0.0"; #4	-----	-----	90
D-5	30	-----	-----	2.0"; #2	2.6"; #3	-----	-----	88.6
D-6	30	-----	-----	2.3"; #2	3.0"; #3	-----	-----	92.1

\*Reservoir temperature 300° F; preheater and filter temperatures as shown.

\*\*Distillation range 347-418° F; aromatics 1.2%; luminometer no. 100; net heat of combustion 18,850 btu/lb.

# *Contrails*

TABLE 6. SUPPLIER'S DATA ON ADDITIVE F-1

<u>Additive Concentration</u>		<u>WSI</u>	<u>Preheater Rating (Max)</u>	<u>Filter Plugging</u>	
<u>ppm</u>	<u>lb/1000 bbl (approx)</u>			<u>ΔP, in. Hg</u>	<u>Minutes</u>
<u>Base Fuel WT, coker tests at 425/525°F</u>					
0	0	100	3	5	300
500	135	-	4	0	300
750	202	100	2	1	300
1000	270	100	0	0	300
<u>Base Fuel EC, coker tests at 400/500°F</u>					
0	0	-	4	25	206
250	67	-	4	25	257
500	135	-	4	0	300
750	202	-	3	0	300
1000	270	-	3	0	300
1250	337	-	0	0	300
<u>Base Fuel GC, coker tests at 400/500°F</u>					
0	0	-	4	25	140
1000	270	-	0	0	300
<u>Base Fuel WC, coker tests at 400/500°F</u>					
0	0	-	4	25	17
1000	270	-	0	0	300
<u>Base Fuel GC, coker tests at 400/500°F</u>					
0	0	-	4	1	300
500	135	-	1	2	300
1000	270	-	1	0	300

TABLE 7. SUPPLIER'S DATA ON ADDITIVE K-1

ASTM-CFR Fuel coker, 6 lb/hr, 300-minute test:

	<u>Test Temperature, °F</u>		<u>Filter</u>	<u>Preheater</u>
	<u>Preheater</u>	<u>Filter</u>	<u>ΔP,</u> <u>in. Hg</u>	<u>Rating</u>
Base fuel	450	550	0.13	0000000011444
Base fuel	425	525	0.09	0000000011111
Base fuel + Additive K-1, 16.8 lb/1000 bbl	425	525	0.10	0000000000111

TABLE 8. SUPPLIER'S DATA ON ADDITIVE M-1

Modified Water Separometer:

	WSIM
Base fuel	87
Base fuel + Additive M-1, 30 lb/1000 bbl	81

Thermal Stability:

(ASTM D1660-61T, Modified, ASTM-CFR Coker Break Point Analysis)

	Test Temperature, °F		Filter ΔP, in. Hg	Preheater Rating
	Preheater	Filter		
Base fuel (kerosine with 20% cat cracked stock)	375	475	5.0	0000000111222
Base fuel (kerosine with 20% cat cracked stock)	400	500	25.0 (235 min)	0000000122333
Base fuel (kerosine with 20% cat cracked stock) + Additive M-1, 15 lb/ 1000 bbl	400	500	4.9	0000000133223
Base fuel (kerosine with 20% cat cracked stock) + Additive M-1, 30 lb/ 1000 bbl	400	500	0.5	0000000111313

Proposed ASTM Accelerated Stability Test for Distillate Fuel Oil

Data are cited on the use of Additive M-1 in a No. 2 burner fuel at concentrations of 2.5-10.0 lb/1000 bbl, which effected reductions in filterable sludge (8.0 mg down to 0.20-1.08 mg), ASTM color (7 down to 2.0-3.5), and visual staining (medium down to none).

**TABLE 9. SEPAROMETER SCREENING TESTS ON ADDITIVES**

Additive and Concentration, lb/1000 bbl	WSI (SwRI)*	WSIM (SwRI)*	Additive and Concentration, lb/1000 bbl	WSIM (RTD)**
None	99	73	None	95
A-1, 12.5	83	41	E-8, 33.6	25
15	81	34	E-9, 33.6	29
B-1, 60	98	68	E-10, 33.6	20
200	98	68	E-11, 33.6	16
B-2, 50	98	76	E-12, 33.6	95
60	98	62	E-13, 33.6	34
B-3, 20	98	80	G-1, 33.6	28
60	91	24	H-1, 33.6	19
C-1, 1	96	63	H-2, 8.0	92
C-2, 1	90	40	J-1, 33.6	93
D-1, 20	99	72	J-2, 33.6	91
D-2, 20	98	60	K-1, 33.6	81
D-3, 30	97	76	L-1, 33.6	92
D-4, 30	98	66	L-2, 33.6	88
D-5, 30	98	61	M-1, 33.6	88
D-6, 30	98	74	N-1, 33.6	92
E-1, 30	98	81	N-2, 33.6	27
E-2, 30	100	75	N-3, 33.6	93
E-3, 30	99	72	N-4, 8.0	85
E-4, 30	97	76	N-5, 33.6***	98
E-5, 30	98	79	P-1, 33.6	95
E-6, 30	98	67	P-2, 33.6	54
E-7	Insoluble		P-3, 33.6	91
F-1, 270		96	P-4, 33.6	93
S-1, 30		85	P-5, 33.6	60
S-2, 30		85	P-6, 33.6	95
			Q-1, 33.6	92
			Q-2, 33.6	63
			Q-3, 33.6	62
			R-1, 33.6	56

\*Base fuel for the SwRI tests was a commercial turbo kerosine plus 0.1% (vol) methoxyethanol.

\*\*Base fuel for the RTD tests was a JP-4 fuel.

\*\*\*Base fuel was Bayol D with WSIM of 96.



TABLE 10. SUMMARY OF ADDITIVE SCREENING TEST RESULTS

Code	Data from Supplier	Eliminated Because of	Included in Series	Code	Data from Supplier	Eliminated Because of	Included in Series
A-1	Yes	WSIM	-	H-1	No	WSIM	-
B-1	Yes	-	3, 4, 5, 6	H-2	No	-	3, 4, 6
B-2	Yes	-	1	J-1	No	-	-
B-3	Yes	-	3, 4, 5, 6	J-2	No	-	3, 4
C-1	Yes	WSIM	-	K-1	Yes	-	2
C-2	Yes	WSIM	-	L-1	No	-	1, 2, 3, 4, 5, 6
D-1	Yes	-	1	L-2	No	-	2
D-2	Yes	WSIM	-	M-1	Yes	-	2, 3, 4, 5, 6
D-3	Yes	-	1	N-1	No	-	3, 4, 6
D-4	Yes	-	3, 4, 5, 6	N-2	No	WSIM	-
D-5	Yes	WSIM	-	N-3	No	-	-
D-6	Yes	-	-	N-4	No	-	-
E-1	No	-	1	N-5	No	-	-
E-2	No	-	-	P-1	No	TS	-
E-3	No	-	3, 4, 5, 6*	P-2	No	WSIM	-
E-4	No	TS	-	P-3	No	-	3, 4
E-5	No	-	3, 4, 5, 6*	P-4	No	-	-
E-6	No	TS	-	P-5	No	WSIM	-
E-7	No	Insol	-	P-6	No	-	-
E-8	No	WSIM	-	Q-1	No	TS	-
E-9	No	WSIM	-	Q-2	No	WSIM	-
E-10	No	WSIM	-	Q-3	No	WSIM	-
E-11	No	WSIM	-	R-1	No	WSIM	-
E-12	No	TS	-	S-1	No	TS	-
E-13	No	WSIM	-	S-2	No	TS	-
F-1	Yes	-	1, 2, 3, 4, 5, 6				
G-1	No	WSIM	-				

\*Additives E-3 and E-5 were tested in combination.

# *Contrails*

In addition to the additives submitted by industry for evaluation, a commercial antioxidant, DBPC (2,6-di-tert-butyl-4-methylphenol) was included in some of the storage test series as a reference point.

SECTION III

FUEL COKER EQUIPMENT AND PROCEDURES

A. General

In order to investigate the deterioration of fuel thermal stability in normal or accelerated storage, it is necessary to have a reliable method of determining thermal stability. For the purposes of the present program, it was originally intended to use some small-scale accelerated method, developed in other Air Force-sponsored research programs, for the evaluation of thermal and storage stability. Since no satisfactory and proven small-scale method became available in time for the present program, it became necessary to use an existing test method. The one that was chosen was the modified fuel coker developed by Eppi under CRC sponsorship<sup>(2)</sup>. Using this method, severe problems were encountered with wipable deposits on the preheater tube, as described in the following sections. Therefore, it was necessary to develop a revised method of thermal stability testing, although no such development work had been contemplated in the program as originally formulated. Since the problem of thermal stability ratings was central to the whole program, this work will be discussed in some detail.

B. Fuel Coker Configurations

1. Basic Equipment

Two standard ASTM-CRC fuel cokers were modified for use in this program. One was originally an automatic (03FC) and the other a manual (01FC) unit, but both had previously been converted to operate as semiautomatic (02FC) units, having automatic temperature control but hand regulation of flow.

2. Eppi-CRC Modification, Coker Configuration (A)

The two ASTM-CRC fuel cokers were modified by replacing the standard test sections with Eppi-CRC modified test sections as described in Reference 2. These test sections are insulated to permit high-temperature operation and are designed for a relatively low flow rate to keep the heat input requirement within the capabilities of the fuel coker electrical system. In addition to the installation of modified test sections, the fuel cokers were equipped with high-range temperature control instruments. The other component of the Eppi modification, a heated fuel reservoir, was not used in the present program; all tests were run with ambient-temperature fuel feed.

# Contrails

The configuration of the flow system using the Eppi-CRC modified test section is shown as Configuration (A) in Figure 1. This is essentially the configuration of the standard ASTM-CRC fuel coker, using the same Eastern Model 1201-198A or 1201-198B pump. The only major change introduced in the present program was the addition of two Whatman No. 12 filter papers in the bypass return line, to reduce the buildup of pump wear debris in the recirculated fuel. The standard 10-micron in-line filters were used before and after the test section, as in the standard ASTM-CRC coker. It should be noted that the use of an in-line filter ahead of the test section is not permissible in tests with a heated fuel reservoir, but no such tests were run in this program. One minor change made in the plumbing was the use of a Grove Model 155 back-pressure regulator to replace the standard Republic regulator, which was found to be marginal for the 250-psig service required in modified fuel coker operation. Another minor change was the installation of a purge line for the manometer pot, which permitted much more efficient flushing.

The original Eppi modified test sections that were installed did not give an adequate fuel residence time in comparison with that in the standard and research fuel cokers. Experimental modifications of the original preheaters involved turning down the preheater inner tube or honing out the preheater outer tube to provide a larger flow area:

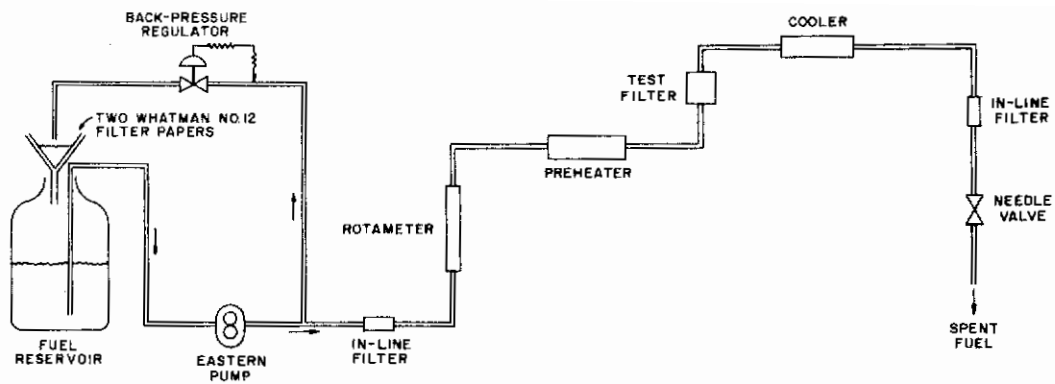
	<u>Diameter, mils</u>			<u>Radial Clearance, mils</u>	<u>Residence Time, seconds*</u>
	<u>Outer ID</u>	<u>Inner OD</u>	<u>Difference</u>		
As furnished by Eppi	638	623	15	7.5	8.0
With turned-down inner tube	638	615	23	11.5	12.3
With honed-out outer tube	647	623	24	12.0	12.8

The residence times using the reworked tubes compare favorably with the 10-13 seconds in the standard and research cokers. The original, as-furnished test sections were used in the first set of storage stability tests, and the honed-out preheater tubes were used in all subsequent tests. The turned-down inner tubes were used only in some experimental studies of fuel coker variables. The honed-out outer tubes represent the current design as approved by the CRC Modified Standard Coker panel.

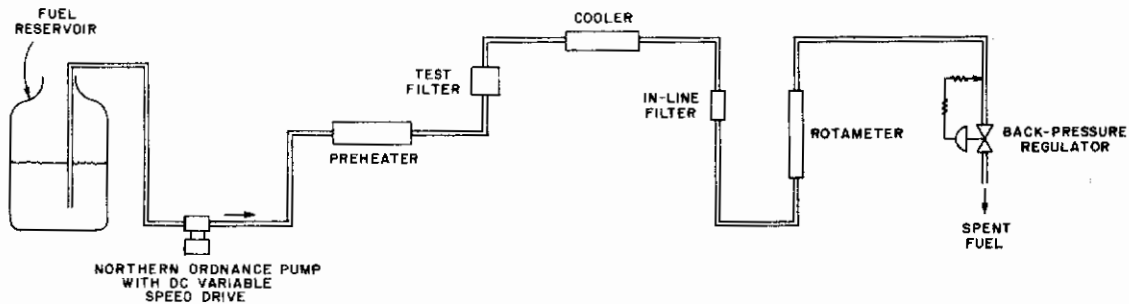
One operating problem with the modified test sections had to do with a rapid decrease in diameter of the aluminum inner tubes in service. This was first thought to be caused by removal of metal during polishing, and

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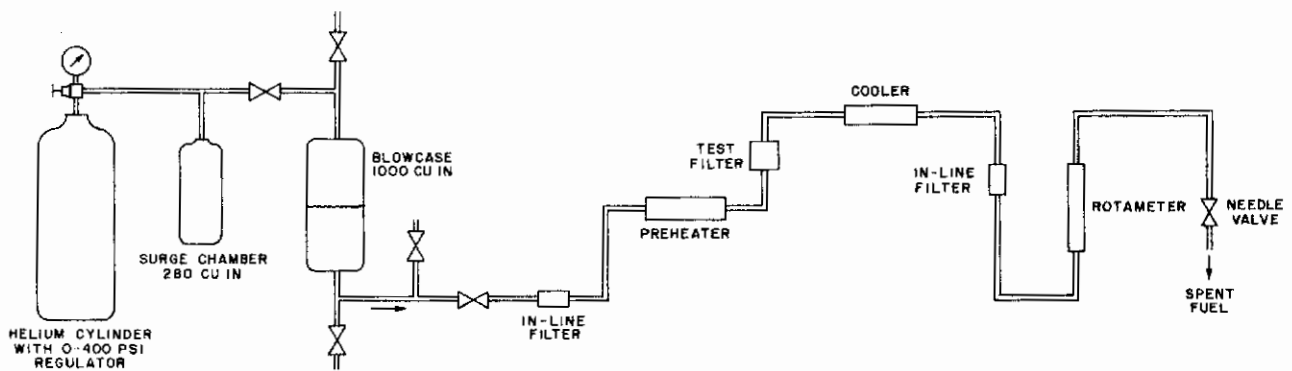
\*Residence times are based on 2.5 lb/hr cold flow of a fluid with 0.8 specific gravity.



CONFIGURATION (A)-EPPI-CRC MODIFIED COKER (EASTERN PUMP)



CONFIGURATION (B)-PROPOSED BY CRC PANEL ON MODIFIED STANDARD COKER (NORTHERN ORDNANCE PUMP)



CONFIGURATION (C)-HELIUM DRIVE SYSTEM DEVELOPED BY SwRI

92429 K

FIGURE 1. MODIFIED FUEL COKER CONFIGURATIONS

it was considered necessary to replace the tubes whenever they lost 5 mils in diameter. However, it became apparent that the decrease in diameter occurred very rapidly, sometimes within four or five days of service, and was accompanied by a lengthening of the tube. The aluminum has very little strength at the service temperatures involved, and apparently the difference in thermal expansion coefficients of the aluminum tube and the internal heater results in a drawing and necking down at the hot end. After about 25 tests, the aluminum tube will increase 1/2 in. in length, and on one occasion it pushed loose the electrical connector located on the cold end of the tube. In view of this rapid necking down of the aluminum tube at the hot end, a limit of 8-9 mils was set on diameter decrease before discarding the tube and heater. While it is recognized that allowing this much change is undesirable, any real solution would require complete preheater section redesign, which was outside the scope of the program. It should be noted that this problem existed in all modified fuel coker testing in the present program, regardless of what fuel drive system was used.

### 3. Northern Ordnance Pump, Coker Configuration (B)

Difficulties with the standard Eastern pump centered around poor service life and the appearance of pump wear debris in the test fuel. During the course of the program, the CRC Modified Standard Coker Panel was considering the use of a different pump, a Northern Ordnance Model 4347-02-A-72. Some work was done with this pump during the course of the program; the configuration, which is shown as Configuration (B) in Figure 1, is essentially the same as that being considered at the time by the CRC Panel. This arrangement provides direct delivery of the fuel to the test section without any external bypass; flow rate is controlled by the use of a variable-speed pump drive. In the present program, a 1/3-hp compound-wound DC motor with a General Radio No. 1700-B speed control was used, providing accurate speed control at a cost that is relatively low compared to mechanical variable-speed drives. The system pressure was controlled by means of a back-pressure regulator or needle valve at the outlet. With this configuration, no in-line filter was used ahead of the test section, and the rotameter was moved downstream from the test section. Pump cooling was found to be necessary because of the high rate of internal fluid slippage; such cooling was provided either by air jets or by applying ice water to the top of the pump in an improvised tank.

This configuration using the Northern Ordnance pump was not used in any of the storage stability evaluations, owing to poor operating experience with the pump.

#### 4. Helium Drive System, Coker Configuration (C)

In order to eliminate pump wear problems, a system was worked out in the present program whereby the fuel was pressurized with helium to move it through the test system. This is shown as Configuration (C) in Figure 1. The fuel reservoir or blowcase is a 1000-in.<sup>3</sup> stainless steel tank, originally designed to hold breathing oxygen, as is the 280 in.<sup>3</sup> surge chamber. A single helium cylinder, regulator, and surge chamber are connected to two fuel coker blowcases. The fuel coker plumbing is standard except that the pump and bypass line are missing and the rotameter has been moved downstream from the test section. Standard 10-micron filters are used before and after the test section; the filter ahead of the test section is changed every time a new test fuel is introduced, in order to avoid or minimize additive carryover effects. All aluminum lines ahead of the test section were replaced by stainless steel. This change, which was being considered at the time by the CRC Modified Standard Coker Panel, was directed primarily at heated-tank operations, where thorough cleaning of lines between tests is mandatory. Although this was not a consideration in the present program, the use of stainless steel was considered desirable merely to keep the plumbing similar to that in other modified cokers.

General views of the fuel blowcases and associated equipment are shown in Figure 2.

The helium-drive system, Configuration (C), was used in all tests on storage program samples with the exception of the first series. All helium-drive tests in this program used the new honed-out preheater section.

The helium used for pressurizing is commercial cylinder helium, which is of high purity (99.99%) and readily available. One cylinder of helium, costing \$34.00 from local suppliers, suffices for about 17 fuel coker tests, making the cost per test about \$2.00. This cost is considerably less than the prorated pump replacement costs that were experienced in the early stages of the program.

One minor but significant advantage of the helium drive over pump drive was demonstrated during a momentary power failure. Since the helium drive was unaffected by the power failure, the fuel continued to flow, and the only effect was a momentary drop in fuel temperature. Such a power failure when pumping fuel through the coker will result in fuel flow stoppage, local overheating, voiding of the test result, and possible damage to equipment. This advantage of the helium drive is believed to be most significant in tests in the higher temperature range.

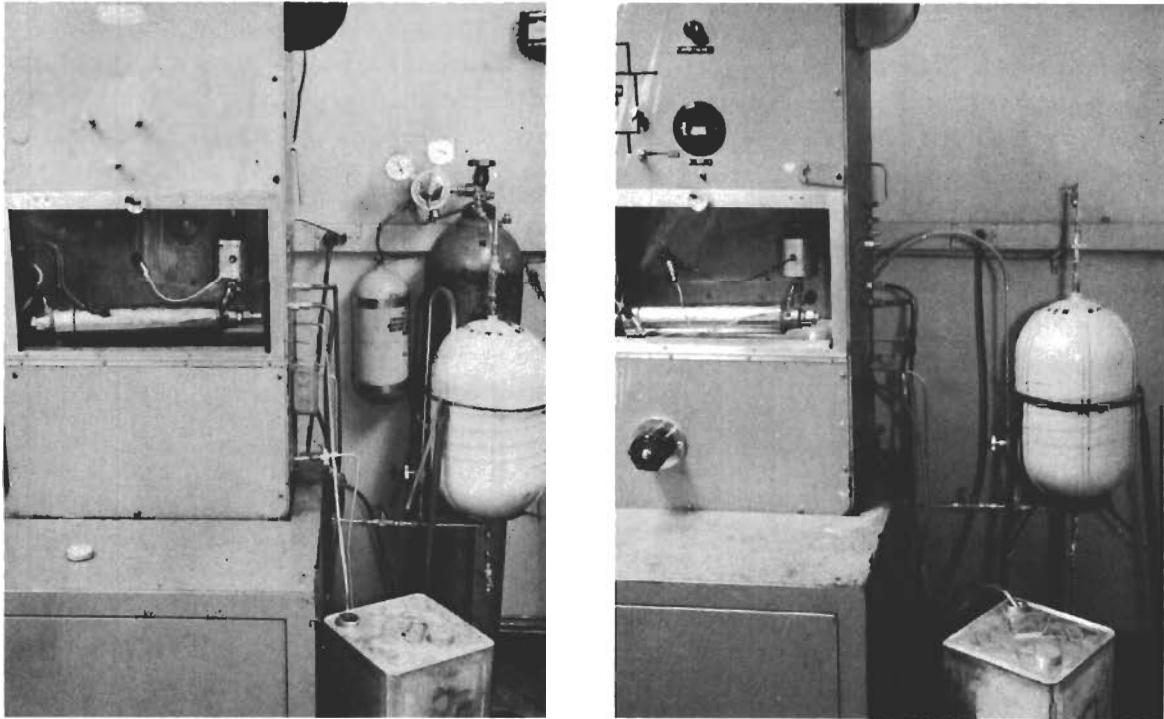


FIGURE 2. HELIUM DRIVE AND TEST SECTIONS IN  
TWO MODIFIED FUEL COKERS



## C. Fuel Coker Operating Procedures

The operation of the modified fuel cokers in all configurations followed in general the standard procedure ASTM D1660-61T, which was the current standard method at the start of the program. The use of the modified test section and high test temperatures necessitated some changes, as outlined in the following paragraphs.

All modified fuel coker tests were run at a fuel flow rate of 2.5 lb/hr. The fuel pressure was held at 250 psig, rather than the 150 psig used in standard tests. Warmup wattages were selected experimentally to reach test temperatures in about 20 minutes. All tests were run for a full 300-minute period, bypassing the test filter in the rare cases in which excessive pressure drop built up. The test temperatures were selected with the filter 100° F higher than the preheater, except for preheater temperatures above 600° F, in which case the filter was held at 700° F maximum to prevent boiling.

All preheater ratings were based on full 300-minute tests. The standard Tuberator was used in all ratings except in the very early portion of the program. After rating the tube "as is," it would be wiped with one pass of a cleansing tissue and then rerated; both unwiped and wiped ratings were reported. The standard ASTM-CRC color code (ratings of 0 to 4) and the Eppi extended scale (ratings of 5 to 8) were used.

The criteria of failure were a preheater rating of 3 or higher and a filter pressure drop of 2.0 in. Hg or more. A pressure drop of 2 in. Hg in these tests at 2.5 lb/hr is roughly equivalent to a pressure drop of 12 in. Hg in standard tests at 6.0 lb/hr, assuming that the degree of filter plugging is the same and that the pressure drop varies as the square of the flow rate. The preheater ratings on the tubes before wiping were used as the criterion of failure in the entire storage program. In the first series, using Configuration (A) with the Eastern pump, this use of unwiped ratings complicated the interpretation of results. At that time, the severity of the wipable deposit problem had not been fully recognized, and test schedules and ratings were based primarily on unwiped ratings. After going over to the helium drive, the problem of disagreement between unwiped and wiped ratings (i. e., the problem of wipable deposits) largely disappeared. The method of establishing breakpoints continued to be based on unwiped ratings.

Using either the original Eastern pump or the Northern Ordnance pump, the operating procedures were as described in the preceding paragraphs. When using the helium drive in the system shown as Configuration (C) in Figure 1, certain procedures had to be observed in starting up the tests in order to maintain repeatable conditions. For example, the blowcase must be charged with the same amount of fuel for each run in order to keep the starting

ratio of gas to liquid volume constant. Using a 10-liter fuel charge, this ratio is 0.64. It is also necessary to avoid the use of pressure or suction in transferring the fuel to the blowcase after it has been aerated, since this could affect the concentration of dissolved oxygen. The intermediate flush with Stoddard solvent was eliminated, since it was felt that this would simply contribute contamination by a material inferior to the test fuel. As indicated previously, the installation of a purge line for the manometer pot increased the flushing efficiency considerably. The simplicity of the fuel plumbing, without any pump or recycle system, also contributed to more efficient flushing.

The start-up procedure for tests with helium drive, Configuration (C), is given in detail in Appendix I.

#### D. Study of Fuel Coker Equipment and Operating Variables

The most serious problem encountered in early tests in the program, using the original Eppi-CRC modified test section in Configuration (A), was the presence of wipable deposits on the preheater tube. As this problem had been recognized only recently, there was no background to use in interpreting the results or eliminating the problem. These wipable deposits were ordinarily rust-colored with a yellowish tinge, were concentrated at the hot end of the tube, and could be removed readily by a single wipe with a cleansing tissue, usually leaving a No. 0 or No. 1 rating on the metal beneath. The original, unwiped ratings on such tubes could vary from No. 1 to No. 4 or higher, depending on the amount and color of the wipable deposits.

Since these wipable deposits interfered seriously with rating and interpretation of test results, considerable time and effort had to be expended in correcting the situation.

It was first established that insoluble particles were accumulating in the fuel in the reservoir during the course of the run; these particles were largely iron and consisted of wear debris from the Eastern gear pump. To alleviate this situation, a double thickness of Whatman No. 12 paper was placed in the bypass return line, filtering the fuel before returning it to the reservoir. It should be noted that this arrangement provides continuous but not positive cleanup of the fuel; i. e., the fuel fed to the coker will contain whatever wear debris it has picked up on its last trip through the pump. Also, it was evident that the bypass filters were not removing all of the finer wear particles, since some brown stain was evident even on the second (bottom) filter paper. It was found that this bypass filtration reduced the amount of wipable deposits and also eliminated the slight but appreciable plugging of the fuel coker test filters that had been observed without bypass filtration. This is illustrated by the following test data on fresh G-1060 base fuel (Ashland,

# Contrails

first batch). Maximum preheater ratings are shown in the form of unwiped/wiped ratings, which is the convention used throughout this report.

Test Temp, °F	Preheater Rating		Pressure Drop, in. Hg	
	With Filter	Without Filter	With Filter	Without Filter
425/525	-	2/0	-	0.10
450/550	1+/0	2/0	0.08	0.18
475/575	2/0	4/0	0.05	0.43
500/600	2/0	4/0	0.05	0.50
525/625	3/0	-	0.04	-

It will be observed that installation of the bypass filter raised the apparent breakpoint of the fuel, based on unwiped ratings, from 475 to 525° F.

As the program continued, it became increasingly apparent that a true picture of fuel thermal stability was not being obtained, since the unwiped ratings reflected the lubricity of fuels and additives and their effect on pump wear. Accordingly, several series of tests were run on base fuels to explore possible means of eliminating the wipable deposits. Much of this work was concerned with possible replacement of the standard Eastern gear pump by a Northern Ordnance gear pump, which had been reported to give better service and less problem with wipable deposits. The Eastern pump has an aluminum body and steel gears and shafts. The body and major working parts of the Northern Ordnance pump are Nitralloy or other alloy steels. Based on nominal delivery ratings, both of these pumps are considerably oversize for supplying the amount of fuel required by the modified coker, as demonstrated by the following rated capacities with no back pressure:

Eastern	0.10 gpm
Northern Ordnance	0.61 gpm
Coker requirement at 2.5 lb/hr	0.0062 gpm

However, in service on kerosine-type fuels against back pressure of 250 psig, both pumps show so much internal slippage as to be marginal for the service.

The Eastern pumps, when new, will usually put up the required 250 psig with some external bypassing back to the reservoir. However, as the pump gears are worn, the amount of external bypassing gradually decreases. The internal slippage is aggravated by temperature rise and is cumulative in nature: A slight temperature rise in the pump caused by internal bypassing

# Contrails

will lower the viscosity of the fuel within the pump, leading to more slippage and consequently higher temperatures. Thus, during the course of a run, the effects pile up and render the pump unserviceable. External cooling of the pump can break this chain of events and enable the pump to finish the run. The most effective cooling of the Eastern pump was obtained simply by wrapping a wet cloth around the pump body. Other expedients tried were the installation of an ice-cooled coil in the pump suction line and isolating the pump from the motor with a plastic bushing to cut down on heat transferred from the motor. Even with all of these expedients, the Eastern pump serviceability was very poor on JP-6 fuels.

The Northern Ordnance pumps were said to be superior to the Eastern pumps for this type of service, but no such advantage was apparent in our tests. The same heating and slippage phenomena were observed, and these appeared to be aggravated by the slower pump speeds required to use these pumps in Configuration (B), i. e., without external bypass. Operating the pump at 1420 rpm with optimum cooling (direct cooling of the pump body with ice), the pump output was just sufficient to feed the coker, and it was calculated that the volumetric efficiency under these conditions was only 1.2%, with internal bypassing accounting for 98.8% of the nominal delivery.

Preliminary studies of the effect of pump type and other operating variables were made on the G-1060 base fuel (Ashland, first batch). The results of these studies are shown in Table 11. The first series of these tests compared the effect of different filtering systems. As stated previously, the use of Whatman No. 12 filter papers in the bypass return line reduced but did not eliminate the wipable deposits. The use of a 2-micron filter ahead of the test section, in place of the standard 10-micron filter, likewise gave some reduction but not elimination of the wipable deposits. Evidently some of the smaller particles of wear debris would pass even through the 2-micron filter.

The next series shown in Table 11 is a study of the effect of fuel residence time, using the original (0.623") and turned-down (0.615") preheater inner tubes. The increased residence time with the turned-down tubes gave heavier deposits, again completely wipable.

The difficulties encountered in Eastern pump operation led to a study of one of these pumps in various stages of wear. As shown in Table 11, about the same amount of wipable deposit was obtained whether the pump was running satisfactorily at 250 psig, whether it was worn to the point where it would deliver only 230 psig with little or no external bypassing, or whether the pressure regulator was backed off to 150 psig to lighten the load on the worn pump.

**TABLE 11. EFFECT OF EQUIPMENT VARIABLES  
ON PREHEATER RATINGS**

Base fuel: Ashland JP-6, first batch (Code G-1060), from cold storage.

Unless otherwise indicated, all tests were run with Configuration (A) (Eastern pump), using the original inner and outer tubes supplied for the modified coker (inner tube OD 0.623"), with a double Whatman No. 12 filter in the bypass return line and the standard 10-micron filter in-line.

		Preheater Ratings (Unwiped/Wiped) at Temperature Shown						
		425	450	475	500	525	550	600
<u>Effect of Filters Used</u>								
<u>Bypass</u>	<u>In-line</u>							
None	10-micron	2/0	2/0	4/0	4/0			
Whatman	10-micron		1+/0	2/0	2/0	3/0		
Whatman	2-micron			2/1*			4/0**	
<u>Effect of Tube diameter</u>								
	0.623" tube (original)		1+/0	2/0	2/0	3/0		
	0.615" tube (reworked)		3+/0		3+/0		4/0***	
<u>Effect of Pump Condition (all with 0.615" heater)</u>								
	OK at 250 psi		3+/0		3+/0			
	Down to 230 psi		3+/0					
	Run at 150 psi		3/0					
<u>Effect of Fuel Drive Method</u>								
	Standard pump (0.615" heater)		3+/0		3+/0		4/0***	
	Helium drive (0.615" heater)		0/0		1/1		1/1	1/1
	N. Ord pump, bypass‡				4/1			
	ditto, after cleaning rig				4/1			
	N. Ord pump, variable drive‡‡		3+/0					

\*Very little and very light No. 2 deposit.

\*\*With 0.615" heater tube.

\*\*\*With 2-micron in-line filter.

‡Flow rate too high to use filter in bypass; used 2-micron line filter. The first run with this pump was made after rig had accumulated 136 runs; the high flow rate with the Northern Ordnance pump apparently dislodged sediment that had accumulated in the lines. Before making the next run, the plumbing and components ahead of the test section were dismantled and cleaned or replaced.

‡‡DC motor drive with no bypass. Pressure was reduced to 150 psi because of excessive pump heating. This run was made immediately after the two initial runs with the Northern Ordnance pump.

# Contrails

Comparative data on the standard Eastern pump, the Northern Ordnance pump, and the helium drive are also shown in Table 11. The helium drive gave no wipable deposits at all and indicated the G-1060 fuel to be stable under 600/700°F conditions\*. The Northern Ordnance pump gave much more extensive deposits (area and density) than the Eastern pump, although this is not reflected in the maximum color ratings. The first two runs on the Northern Ordnance pump were made with direct drive at 1725 rpm and external bypassing, i. e., in Configuration (A), since no speed control device was on hand at that time. Evidently wear debris accumulated in the lines from previous runs was broken loose by the high flow rate and brought into the preheater, almost choking it with deposits that were, however, completely wipable. Complete cleanup and replacement of lines gave somewhat similar results in a subsequent run, although the amount of debris was somewhat less. The pump was subsequently used with variable-speed drive control, i. e., in Configuration (B), and again heavy wipable deposits were obtained; the pump delivery was so poor that the back pressure had to be reduced to 150 psig.

These early tests with the Northern Ordnance pump were subject to some question in view of the rather severe conditions imposed on the pump in its initial runs - high speed, lack of pump cooling, and the presence of deposits loosened from the lines. Therefore, a second pump was obtained and broken in very carefully by running several hours on a mixture of fuel and lubricating oil, with no back pressure. This new pump and the original pump were then tested in Configuration (B) with variable speed control, using effective ice-pack cooling. Comparative runs with these pumps in Configuration (B) and with an Eastern pump in Configuration (A) were made on H-1008 base fuel (Ashland, second batch), testing at 475/575°F, with the following results:

	<u>Preheater Rating</u> <u>(Unwiped/Wiped)</u>	<u>Filter ΔP,</u> <u>in. Hg</u>
Northern Ordnance No. 1	4+/0	0.6
Northern Ordnance No. 2	4+/0	0.7
Eastern	2/0	0.0

---

\*Stability of G-1060 at 600/700°F was not confirmed in later tests with helium drive. In the early tests with helium drive, a poor pressure regulator led to helium pressure fluctuations, which would tend to strip oxygen out of the fuel and make the test unduly mild.

# Contrails

Thus, both of the Northern Ordnance pumps gave heavier wipable deposits and more test filter pressure drop than did the Eastern pump. Since the Configuration (B) used with the Northern Ordnance pumps does not include an in-line filter ahead of the test section, it is logical to expect that any wear debris formed in the single pass of the fuel through the pump will enter the test section and contribute to wipable preheater deposits and possibly to filter plugging.

Another point that was checked with the No. 2 Northern Ordnance pump was the effect of the purity of the flushing solvents. The use of all-reagent-grade trisolvent vs commercial trisolvent\* did not affect the amount of deposit obtained, nor did the use of a thermally stable fuel as the flushing solvent in place of Stoddard solvent.

In various tests with both the Eastern and Northern Ordnance pumps on different base fuels, it was observed that the H-1019 (Phillips Base Oil No. 1) gave somewhat less wipable deposit than did the other base fuels under comparable conditions. The important properties of the fuels in question are shown in the following tabulation:

	<u>G-1060</u>	<u>H-1008</u>	<u>H-1010</u>	<u>H-1019</u>
Viscosity, cs at 100° F	1.05	1.00	1.00	1.83
at -40° F	5.39	4.43	4.53	-
Aromatics, vol %, FIA	10	9	2	3.4
Olefins, vol %, FIA	2	2	2	**
Total sulfur, wt %	0.016	0.037	0.010	0.0003
Mercaptan sulfur, wt %	0.0006	0.0006	0.0001	<0.0002
Anti-icing additive, vol %	0.127	0.142	0.109	None

The H-1019 is a highly refined product with low contents of sulfur and aromatics, so its apparently better lubricity cannot be attributed to these sources. It is significantly higher in viscosity than the other fuels, which would be expected to contribute to better lubrication. The other difference in the H-1019 is the absence of methoxyethanol anti-icing additive. It had been suggested that possibly the presence of methoxyethanol in JP-6 fuels

---

\*The "commercial" trisolvent used in the early stages of the program consisted of commercial-grade benzene with reagent-grade isopropanol and acetone. Although no adverse effect of the commercial-grade benzene could be demonstrated, it was replaced by reagent-grade benzene for the later stages of the program.

\*\*Bromine number 1.4.

# Contrails

was contributing to the problem of wipable deposits. In order to check this point, batches of H-1008 and H-1010 fuels were washed five times with distilled water to remove all methoxyethanol (confirmed by analysis). Modified fuel coker tests were run on the washed and unwashed fuels, using the Eastern pump in Configuration (A) with honed-out preheater:

	H-1008		H-1010	
	<u>Washed</u>	<u>Unwashed</u>	<u>Washed</u>	<u>Unwashed</u>
Preheater ratings (wiped/ unwiped) at following temperatures (° F):				
475/575		2/0		1+/0
500/600	2/0		2/0	
525/625	2+/0		3/0	
550/650	4/0		3/0	

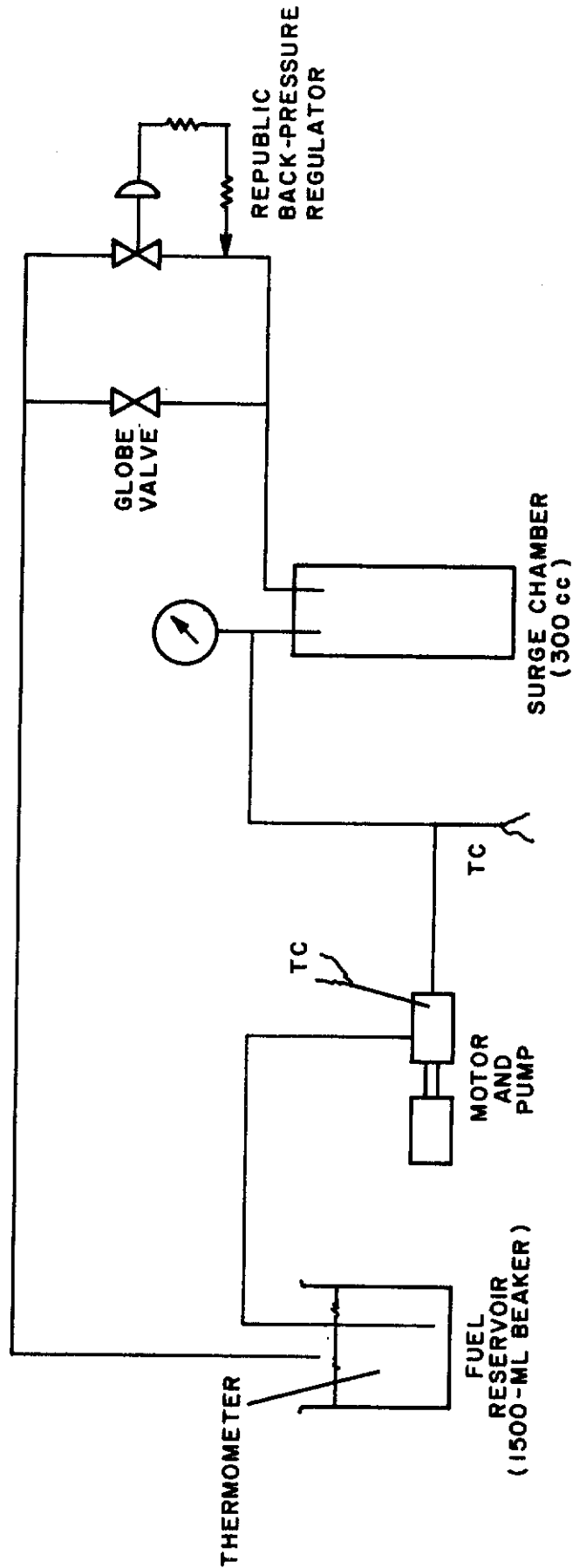
Although comparative ratings at the same temperatures are not available, the data do show clearly that removal of the methoxyethanol did not eliminate the problem of wipable deposits with either of the base fuels. Therefore, it appears that the deposits are a function of poor lubricity of the fuel itself and that this lubricity is not affected markedly by the presence or absence of methoxyethanol.

## E. Pump Stand Tests

Before abandoning completely the idea of using the Northern Ordnance pump, it was considered desirable to set up comparative pump evaluation tests in a breadboard system, which is shown schematically in Figure 3. In each test, one liter of fresh, filtered fuel was charged to the reservoir. All tests were scheduled for 120 minutes of fuel recirculation, with the exception of one that was extended to 300 minutes and two that were terminated early because of excessive heating or pump tie-up. The Eastern pumps were tested at 1725 rpm, which is their operating speed in the fuel coker. A single attempt to test a Northern Ordnance pump at 1725 rpm, without cooling, resulted in pump tie-up; subsequent runs were made at 1420 rpm, which on JP-6 fuels gives a flow rate somewhere on the order of magnitude of the 23.7 ml/min required for fuel coker operation. The fuel temperatures, pressures, and flow rates were recorded throughout each run, and the amount of pump wear debris was determined by filtering the total fuel sample after the run through a 0.45-micron Millipore filter. The results of these tests, using different pumps and methods of pump cooling, are shown in Table 12.

The Eastern pump with H-1008 fuel ran satisfactorily at 150 psig back pressure without cooling, but against 250 psig the run had to be terminated after 90 minutes because of excessive heating and zero delivery. With ice cooling, the Eastern pump completed a 120-minute run against 250 psig, with





92511K

FIGURE 3. FLOW PLAN OF PUMP EVALUATION TEST STAND

TABLE 12. SUMMARY OF PUMP STAND TEST DATA

	1	2	3	4	5	6	7	8	9
Run No.									
Base fuel	-----H-1008----- H-1019 H-1010								
Pump	-----Eastern----- Northern Ordnance -----								
RPM	1725	1725	1725	1725	1420	1420	1420	1420	1420
Cooling	None	None	Ice	None	Ice	None	Air	Air	Air
Nominal pressure, psi	150	250	250	250	250	250	250	250	250
Length of run, min	300	90	120	40*	120	120	120	120	120
Pump temperature, °F									
Start	77	78	67	80	70	82	77	80	83
Finish	108	163	116	149	101	155	105	90	103
Discharge pressure, psi									
Start	150	250	250	250	250	250	250	250	250
Finish	161	150	250	250	250	170	210	250	225
Flow rate, ml/min**									
Start	315	230	210	345	70	40	40	560	120
Finish	210	0	62	*	25	0	3	480	5
Total wear debris, mg	7.7	10.2	4.3	7.2	6.1	15.3	4.2	1.4	5.0

\*Pump tied up.

\*\*Nominal flow rates at no back pressure: Eastern (1725 rpm) - 380 ml/min  
 NoOrd (1725 rpm) - 2300 ml/min  
 NoOrd (1420 rpm) - 1900 ml/min  
 Flow rate corresponding to 2.5 lb/hr in fuel coker - 23.5 ml/min

some evidence of pump heating and fall-off in flow rate. The Northern Ordnance pump when run on H-1008 and H-1010 fuels at 1420 rpm against 250 psig back pressure would not maintain pressure or delivery with air cooling (Runs 7 and 9). With ice cooling (Run 5), pressure and delivery were maintained with H-1008. The sole example of satisfactory performance with air cooling was on H-1019 fuel (Run 8). This run was outstanding in the high delivery rates, lack of any significant temperature rise, and the small amount of wear debris formed. The H-1019 fuel after test was clear, whereas in all tests on other fuels there were suspended particles that were clearly visible as a cloud or haze. Thus, the only really satisfactory result in this series of tests was obtained with the relatively viscous H-1019. These results confirmed early experience indicating that neither the Eastern nor the Northern Ordnance pump is satisfactory for pumping JP-6 fuels against 250 psig back pressure. Even with ice cooling, both pumps yielded considerable amounts of wear debris.

## F. Oxygen Content of Fuel with Helium Drive

In view of the severe pump problems associated with operation on JP-6 fuels against 250 psig, there are obvious advantages in the use of inert gas pressurization instead of pumping. However, when using direct gas pressurization, there is always some concern over the effects of dissolved gas on the test results. For example, when pressurizing kerosine-type fuels with nitrogen at 250 psig and 100°F, approximately 2.3 volumes of nitrogen (NTP) will dissolve in one volume of fuel at equilibrium conditions. This amount of dissolved nitrogen can introduce some control problems in a flow system, since at the point of depressurization the nitrogen will be liberated as bubbles. Also, the presence of dissolved nitrogen would create some concern as to possible effects on thermal stability at very high temperatures, both through displacement of oxygen from the fuel and actual reaction with hot metal surfaces. Attempts to devise gas-pressurized thermal stability test systems in which the pressurizing gas is physically separated from the test fuel have been confined mostly to very small systems. With a fuel sample of 2.5 gallons, as in the modified coker, such separation would require rather massive, expensive, and hard-to-clean equipment.

Helium has been used previously as a pressurizing gas in fuel thermal stability work, but its advantages seem never to have been brought out in the literature. In addition to essentially complete inertness, helium has the advantage of very low solubility in hydrocarbons. Solubilities of helium and other gases in hydrocarbons are shown in Table 13. The values for helium in this table represent the only solubility data found in searching the literature from 1937 to date. Without attempting to evaluate these data critically, it can be seen that the solubility of helium is about one-fourth to one-sixth that of nitrogen in terms of Ostwald coefficient (the volumes of gas dissolved per volume of fuel, the gas volume being referred to actual equilibration temperature and partial pressure).

TABLE 13 SOLUBILITY OF GASES IN HYDROCARBONS

Solvent	Temp., °F*	Ostwald Coeff × 10 <sup>-2</sup>	Temp., °F*	Ostwald Coeff × 10 <sup>-2</sup>	Reference
<u>Nitrogen</u>					
Cyclohexane	68	16.2	104	20.1	(3)
Benzene	68	11.6	104	13.7	(3)
JP-4 fuels			90	14-16**	(4)
JP-5 fuels			90-100	12-14**	(4)
Kerosine	68	10.9	104	11.5	(3)
RJ-1 fuel			100	10.0	(5)
<u>Oxygen</u>					
Cyclohexane	68	22.8	104	22.8	(3)
Benzene	68	22.0	104	24.0	(3)
Kerosine	64	17.0			(3)
<u>Air</u>					
Benzene			100	12.7	(3)
Gasolines			100-104	20-29**	(3)
JP-4 fuels			100	18-20**	(4)
JP-5 fuels			100	15**	(4)
Kerosines	60-68	12-15**	108-120	14-17**	(3)
<u>Helium</u>					
Cyclohexane	68	2.53	100	3.33	(3)
	59	2.32	107	3.51	(6)
Benzene	68	1.93	100	2.51	(3)
	59	1.77	107	2.69	(6)
Toluene	59	1.89	104	2.77	(7)
n-Hexane	59	4.28	107	5.97	(6)
n-Heptane	59	3.65	107	5.07	(6)
n-Octane	59	3.17	107	4.44	(6)
3-Methylheptane	59	3.29	107	4.59	(6)
2,3-Dimethylhexane	59	3.37	106	4.54	(6)
2,4-Dimethylhexane	59	3.53	106	5.13	(6)
2,2,4-Trimethylpentane	59	3.97	107	5.53	(6)
n-Nonane	59	2.70	107	4.07	(6)
n-Decane	59	2.48	107	3.48	(6)
n-Dodecane	59	2.09	107	2.87	(6)
n-Tetradecane	59	1.86	106	2.44	(6)
Paraffin base oil***			100	1.64	(8)
Aromatic base oil***			100	0.55	(8)

\*Where a range of temperature is shown, this indicates range over which measurements were made and does not imply inaccuracy of temperature control in individual measurements.

\*\*Range of average of measurements on a number of different samples.

\*\*\*Lubricating oils, 150 SUS at 100° F, not further identified. The paraffinic base oil contained additives (types and amounts not specified).

# Contrails

In the helium drive system used in the current program, 10 liters of test fuel is charged to a 16.4-liter blowcase. The air present in the blowcase is left undisturbed, and the fuel has been presaturated with air before charging to the blowcase. Thus, the pressurizing consists of applying 250 psi of helium pressure over the 1 atm air pressure, giving a total pressure of 264.7 psi absolute. There is no theoretical basis for assuming that the helium will displace any air from the fuel at this time, since the gases are near-ideal under these conditions and will maintain equilibrium independently, the concentration of each in solution being determined by the respective partial pressures in the gas phase. A small amount of the helium will move from the gas phase into solution to attain this equilibrium. Additional helium will be supplied automatically by the pressure regulator to make up for this small transfer, which would not affect the amounts of air in the gas and liquid phases. This helium equilibrium would be attained very slowly in this quiescent system.

As the fuel is fed to the coker during the run, additional helium will be fed into the gas space to maintain the total pressure, diluting the original helium-air mixture with more helium and lowering the partial pressure of the air in this mixture. There will be a tendency for air to move out of the liquid phase into the gas phase to reestablish equilibrium. The problem of calculating this new equilibrium is complicated by the continuous removal of liquid phase (containing both air and helium) throughout the run; it has not been thought worthwhile to work out a rigorous mathematical solution, particularly since it is by no means certain that equilibrium will ever be established in this dynamic system. The most important question to be resolved is whether there is a significant decrease in the dissolved oxygen content of the fuel being fed to the coker throughout the run, since such a decrease would almost surely have some effect on the severity of the fuel coker test. This question has been approached experimentally by means of a gas chromatographic technique. The procedures used, which were adapted from information kindly supplied by Phillips Petroleum Company, are given in Appendix II.

The first experimental work on oxygen contents of fuel coker feed streams was performed with an open-beaker sampling technique, since pressurized sampling equipment was not yet available. In open sampling, fuel was drawn from the fuel coker feed line into an open beaker, and a sample taken into a syringe while the fuel was flowing into the beaker. This syringe sample was then injected immediately into the chromatographic column. The open sampling technique introduces some error due to oxygen loss by stripping as the excess helium desorbs at the time of sampling.

Samples taken by the open sampling technique from the fuel in the blowcase after air saturation but before pressurization with helium showed an average oxygen content of 0.0479 cc oxygen per cc of fuel, the oxygen being measured at atmospheric pressure of 743 mm Hg. If converted to cc of oxygen measured at the oxygen partial pressure of 156 mm (actual partial

pressure during air saturation procedure), the average oxygen content after saturation was found to be 0.228 cc/cc at 78° F, which is in good general agreement with the published values for Ostwald coefficient (see Table 13). This indicates that the method gives reliable results and that the fuel after the standard air saturation procedure is fully saturated with oxygen.

Samples taken by the open sampling technique from the blowcase after initial pressurization with helium indicated no significant decrease in oxygen content, which is in agreement with theoretical considerations. However, samples taken from the coker feed stream throughout 300-minute runs showed significant decreases in oxygen content starting at about 200 minutes and reaching nearly half of the original oxygen content at the end of 300 minutes.

With the introduction of the pressurized sampling technique, it was demonstrated that the apparent decreases in oxygen content were not real and that the fuel retained essentially all of its original oxygen content almost all of the way through the 300-minute coker runs. Averaged data from a number of runs are plotted in Figure 4, showing that the oxygen content is substantially unchanged for the first 270 minutes of the run and then decreases only by about 8% during the remaining 30 minutes. The points plotted beyond 300 minutes were obtained by continuing the fuel flow and the sampling after the fuel coker runs were completed. These indicate that the oxygen content of the fuel being fed from the blowcase falls off rapidly as the blowcase becomes nearly empty. This probably results from stratification of the fuel with regard to oxygen content, which would give a low content of oxygen in the layers of the fuel in the immediate proximity of the gas-fuel interface.

The important conclusion from this work on dissolved oxygen content is that there is no significant depletion of oxygen in the fuel being fed to the coker throughout the 300-minute run. Therefore, it may be assumed that the only real differences introduced by using helium drive are the elimination of pump wear debris and possibly the elimination of fuel oxidation caused by repeated pumping and recycling.

#### G. Comparison of Helium Drive Results with Standard Coker Results

It was considered desirable to establish a correlation between helium drive results and those obtained in either the standard ASTM-CRC coker or in the Eppi-CRC modified coker, in order to determine whether the introduction of helium pressurization could have caused any major change in severity level. The Eppi-CRC modified coker was unsuitable for this purpose because of the distorting effects of pump wear debris. It was considered that the standard coker could be used, within its operating limit of 475/575° F, to establish correlation with the helium drive.

Attempts to correlate the helium drive with standard coker results on the JP-6 base fuels in this program led to some very interesting information.

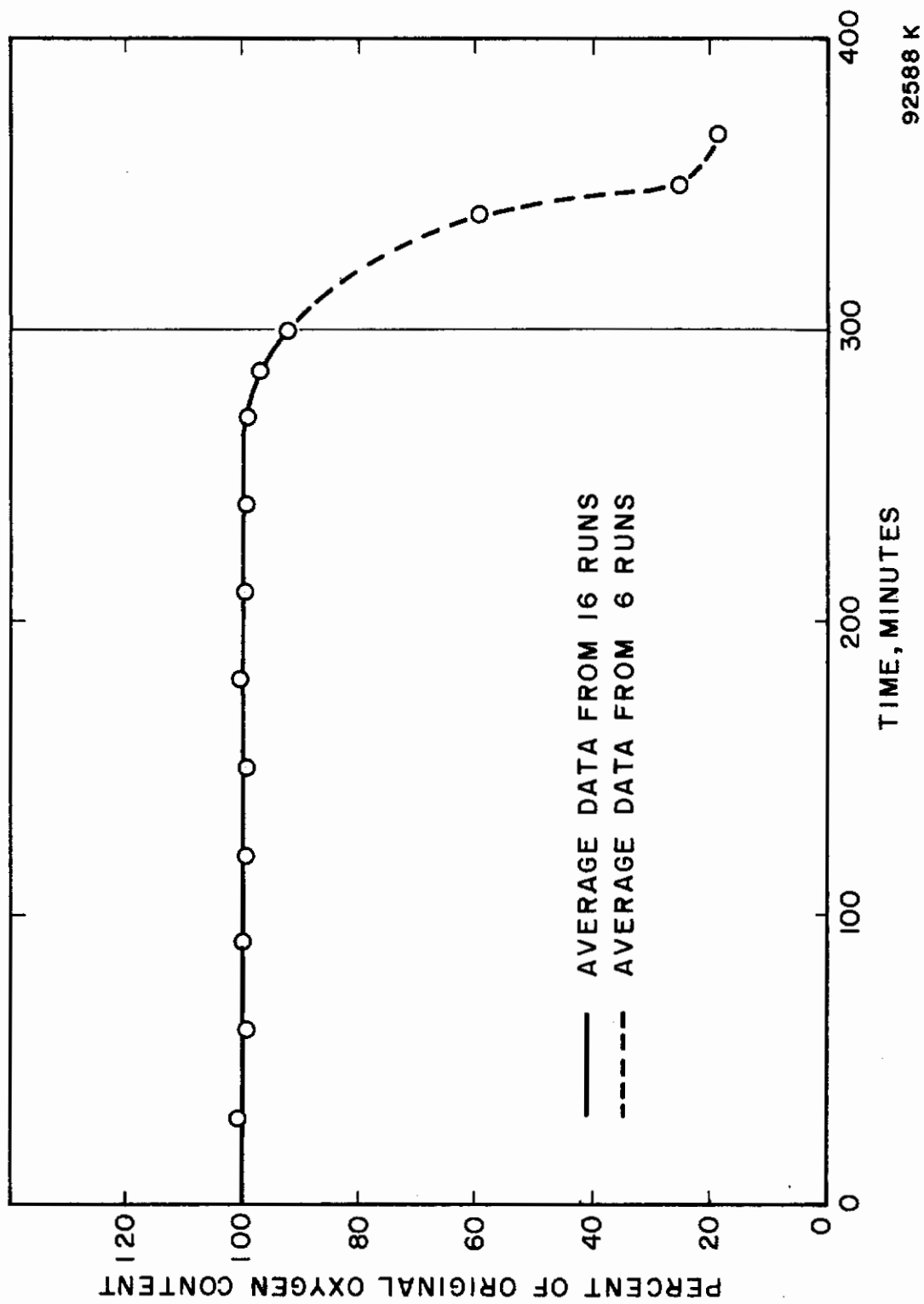


FIGURE 4. OXYGEN CONTENT OF FUEL COKER FEED STREAM DURING AND AFTER RUN

# Contrails

Earlier, it had been found that H-1008 base fuel at 450/550° F in the standard coker gave extremely erratic results on both preheater deposits and filter plugging. Subsequent standard-coker and helium-drive test results on H-1008 and H-1010 base are shown in Table 14. Here it was found that both base fuels in the standard coker tests gave failing filter pressure drops over the entire range of 400/500 to 475/575° F. Further, most of these standard tests showed wipable deposits, which are not normally expected with the standard coker. In contrast, the tests with helium drive gave nonwipable deposits with little or no filter pressure drop, showing true preheater breakpoints in the range of 500/600 to 550/650° F. The standard coker test data, if accepted at face value, would give false (wipable) preheater breakpoints at 475/575° F and filter breakpoints below 400/500° F.

It was suspected that the standard coker test results were being influenced by the presence of pump wear particles, even though the relatively low pump pressure (150 psig) in the standard coker has been thought to minimize such problems. To confirm this suspicion, a check test was run on H-1010 fuel at 450/550° F using the standard ASTM-CRC coker with a double thickness of Whatman No. 12 paper in the bypass line and with a 2-micron in-line filter ahead of the test section (replacing the 10-micron filter normally used). The extra filtration produced a marked reduction in filter plugging and a significant reduction in preheater deposits:

	<u>Filter <math>\Delta P</math>, in. Hg</u>	<u>Preheater Rating (Unwiped/Wiped)</u>
Standard coker	21.0	2+/0
Standard coker with extra filtration	0.8	1/0

From these data, it must be concluded that standard coker data on JP-6 or other high-temperature fuels should be regarded with considerable suspicion. It is believed that the presence or absence of significant quantities of pump wear debris is a somewhat random phenomenon related to the past service history of the fuel coker pump and the lubricity of the particular fuel being tested. This question of the validity of the standard fuel coker test as applied to JP-6 and other high-temperature fuels obviously needs more investigation.

Since it was not possible to obtain a valid comparison of the standard and helium-drive test results on JP-6 fuels, a series of correlation tests was set up on a commercial turbo kerosine, with the following results:



TABLE 14. COMPARISON OF STANDARD COKER AND HELIUM DRIVE RESULTS

Test Temp, °F	Standard ASTM-CRC Coker		Modified, Helium Drive	
	Filter ΔP, in. Hg (minutes)	Preheater Rating (Unwiped/Wiped)	Filter ΔP, in. Hg (minutes)	Preheater Rating (Unwiped/Wiped)
	<u>H-1008 Base Fuel</u>			
400/500	18.0 (300)	0	0.0 (300)	0
425/525	25.0 (238)	0	0.0 (300)	0
450/550	25.0 (261)	2/0	0.0 (300)	2/2
475/575	25.0 (270)	3/0	0.0 (300)	2+/2+
500/600			0.7 (300)	4/4
	<u>H-1010 Base Fuel</u>			
400/500	25.0 (200)	1/0	0.0 (300)	0
425/525	25.0 (253)	2+/0	0.0 (300)	0
450/550	21.0 (300)	2+/0	0.0 (300)	1/1
475/575	13.0 (300)	4/0	0.0 (300)	1/1
500/600			0.0 (300)	1/1
525/625			0.0 (300)	1/1
550/650			0.0 (300)	3/3

# Contrails

Test Temp, °F (Preheater/Filter)	Standard Coker		Helium Drive	
	Filter ΔP, in. Hg*	Preheater Rating**	Filter ΔP, in. Hg*	Preheater Rating**
350/450	0	0000000000000		
375/475	0.2	0000000022211	0	0000000000000
400/500	3.8	0000000142450	0.1	0000000011111
425/525	8.5	0000000055556	20.7 (195)	0000000001333
450/550	25.0 (193)	0000000677755	25.0 (293)	0000000000055
475/575			20.1	0000000000055

From these data, it can be seen that the helium-drive breakpoints are within 25° F of the standard-coker breakpoints. The helium-drive coker data show both preheater and filter breakpoints at 425/525° F. In the standard coker, the preheater breakpoint is 25° F lower and the filter breakpoint is 25° F higher. Thus, for this one conventional kerosine-type fuel, the helium-drive results showed a good correlation with the standard-coker results.

## H. Repeatability and Reproducibility of Helium-Drive Coker Test Results

Check tests were run on a single 55-gallon drum sample of H-1008 base fuel, using the two rigs and three operators that were involved in the evaluation program. The use of a single drum of test fuel precluded the possibility of any drum-to-drum variation. The individual samples for each test were drawn into precleaned 5-gallon cans while the fuel was still in cold storage; each individual sample was warmed to room temperature shortly before filtering and testing in the helium-drive fuel coker.

The following schedule was used for each series of tests at a given test temperature:

	<u>Rig No. 1</u>	<u>Rig No. 2</u>
Flush and Runs 1 & 2	Operator A	Operator A
Runs 3 & 4	Operator B	Operator B
Runs 5 & 6	Operator C	Operator C
Runs 7 & 8	Operator A	Operator A

The flushing procedure (used whenever test fuel is changed) was performed only by Operator A. The operator scheduled for each run would start and finish the run, including precleaning the test section, assembling, flushing if

---

\*After 300 minutes unless otherwise noted.

\*\*Unwiped ratings are shown; wiped ratings were same in all cases.

# Contrails

required, making the run, disassembling, and rating the preheater tube. One preheater tube and one test section were used in Rig No. 1 throughout this work, and a different preheater tube and test section were used in Rig No. 2. Thus, the sequence of tests gave a full indication of both repeatability (Operator A, same apparatus) and reproducibility (different operators and apparatus).

This sequence of eight runs, four in each rig, was first made at the previously determined breakpoint of the fuel, 550/650° F. One week later, the same sequence was repeated at 525/625° F, i. e., 25° F below the breakpoint. The results of these tests are listed in Table 15. It will be observed that the repeatability and reproducibility were perfect in the runs at 525/625° F. Not only did all tests show a zero pressure drop and a 1 or 1+ preheater rating, but also the extent of the No. 1 or 1+ ratings was quite comparable in all tests. At the preheater breakpoint conditions of 550/650° F, all tests showed clearcut preheater failure, with the maximum preheater ratings ranging from 3 to 5+. This is actually a very narrow spread in colors, since the two scales (CRC and Eppi extended) do not have the same color shades and tend to overlap. Thus, it is considered that the agreement of results is extremely good in this difficult rating region. Even the extent of the observed deposits was quite comparable in all tests. At 550/650° F, all filter pressure drops were zero except for one 0.1-inch reading, which is not considered high enough to be significant.

These test results indicate that both reproducibility and repeatability of the helium-drive modified coker results are better than the 25° F temperature increments that are used in establishing breakpoints.

TABLE 15. REPEATABILITY AND REPRODUCIBILITY OF MODIFIED COKER WITH HELIUM DRIVE  
 Test fuel: Single drum of H-1008 (Ashland JP-6, second batch)

Operator	Rig No. 1		Rig No. 2		
	Filter ΔP, in. Hg	Preheater Rating* Unwiped      Wiped	Filter ΔP, in. Hg	Preheater Rating* Unwiped      Wiped	
		Temperature 525/625° F			
A**	0.0	0000000011111    0000000011111	0.0	0000000011111    0000000011111	
B	0.0	000000001111111    000000001111111	0.0	000000001111111    000000001111111	
C	0.0	000000000111111    000000000111111	0.0	000000000111111    000000000111111	
A	0.0	000000000111111    000000000111111	0.0	000000000111111    000000000111111	
		Temperature 550/650° F			
A**	0.0	0000000024442    0000000024442	0.0	000000023332    0000000223442	
B	0.1	0000000113451    0000000113451	0.0	0000000124443    0000000124443	
C	0.0	0000000112331    0000000113321	0.0	0000000012441    0000000012441	
A	0.0	0000000023331    0000000012331	0.0	0000000223321    0000000113311	

\*Preheater ratings are maximum color code observed in each inch of effective length, reading from cold end to hot end (left to right). The underlined numbers indicate a rating slightly higher than the number shown; e.g., 2 indicates a rating between the No. 2 and No. 3 standards.

\*\*First run on each rig by operator A involved flushing the rigs with trisolvent. Subsequent runs by operators B, C, and A did not require flushing.

## SECTION IV

### HOT-ROOM EQUIPMENT AND STORAGE PROCEDURES

#### A. General

At the start of the program, it was decided to adopt a storage temperature of 130° F to investigate the deterioration of high-temperature fuels. The choice of this temperature was dictated by the desire to obtain reasonably fast deterioration without the distorting effects that can be introduced at temperatures far above the range of normal, practical storage conditions. The use of the modified fuel coker as the thermal stability evaluation method required the storage of fairly large quantities of fuel. In order to run a complete break-point analysis in the modified fuel coker, some 10 - 12 gallons of test fuel is required. Therefore, 15-gallon drums were chosen as the storage containers, permitting the use of a considerable air space above the fuel samples.

#### B. Hot-Room Equipment

A 10 x 13-foot hot room for operation at 130° F was provided by SwRI at the start of the program. The building is a standard steel Butler unit on a concrete slab. Walls and roof are insulated with 1.5 inches of glass fiber. Angle-iron racks provide storage for up to 120 fifteen-gallon drums in upright position, with ample space between the drums for air circulation. Heat is supplied by a 10-kw explosion-proof electric heater with fan, mounted on the floor at one end of the room. The unusual feature of the equipment is the use of a high-volume overhead fan rated at 6200 cfm, which provides very vigorous and continuous air circulation throughout the room. This vigorous air circulation is believed to be the key to the temperature uniformity and constancy that have been obtained, as described in the next paragraph. A small blower mounted outside the building near the roofline gives a continuous input of fresh air, with vents provided at floor level to bleed off any accumulation of hydrocarbon vapors. All electrical equipment and wiring within the building is explosion-proof.

Temperature is controlled by means of an on-off controller located outside the building, with the sensing element located in front of the heater fan. This arrangement shortens the on-off cycles of the heater and provides close temperature differential. When the hot room was first put into operation, temperatures were checked by means of thermocouples attached to drums placed at remote corners of the room (top and bottom); it was indicated that there were no detectable differences due to location. The air temperature is recorded continuously at one point in the room and has shown a variation of only  $\pm 1^\circ$  F over an extended period. The 10-kw heater is ample for operation at outside temperatures down to +25° F, and probably much lower.

## C. Fuel Blending and Storage Procedures

Each fuel-additive blend for hot-room storage was made up from a single 55-gallon drum of base fuel, which was removed from cold storage one day before use. The blends were made in the original 55-gallon drums, using a mechanical stirrer. Additives were blended into a small quantity of the base fuel, and the concentrates were added to the drums. After blending, each sample was transferred to the required number of 15-gallon drums, which had been precleaned by thorough rinsing first with trisolvant and then with the test fuel. Next, each 15-gallon drum was aerated for 10 minutes at 1-2 liters/minute, using a diffuser stone. Distilled water (if used) was added to each drum at this point. Each drum was then bunged tight and placed in hot-room storage, except for the one drum retained for determining initial thermal stability. From each set of hot-room storage samples, one drum was normally removed for thermal stability testing after each four-week interval. In some of the hot-room storage series, reaeration of each drum remaining in storage was performed at four-week intervals. For reaeration, each drum was removed from the hot room, allowed to cool to ambient temperature, aerated using the same procedure as in the original aeration, and replaced in the hot room.

At the time any given drum was removed from the hot room for thermal stability testing, it was first agitated while still hot by inverting several times and then allowed to cool to ambient temperature without further disturbance. Then the drum was opened for inspection and removal of part of the sample for the first fuel coker test. No particular difficulty was encountered with stir-up or suspension of water or rust particles in the "wet" drums. These materials settled to the bottom during the cooling period, and subsequent sample draws were made through a tube positioned off the bottom. Slight water hazes, present in some samples, were removed by the filtration through No. 12 Whatman paper which is a part of the fuel coker procedure.

In the first two series of hot-room storage tests in this program, a small amount of free water was added to each drum in order to provide realistic amounts of water and iron rust. Since completely dry and rust-free storage containers are the exception rather than the rule in practice, it was felt that the addition of free water would provide a better simulation of actual storage conditions. In these tests, 0.05% by volume of distilled water was added to each drum prior to hot-room storage. This amount of water is sufficient to provide a small amount of free water even at the 130° F storage temperature but is not large enough to extract any major portion of the anti-icing additive from the fuel. The addition of free water did cause rusting of the drum interiors, primarily the bottoms, as shown in Figure 5.



FIGURE 5. INSIDE BOTTOM OF DRUM AFTER  
HOT-ROOM STORAGE TEST  
WITH FREE WATER IN FUEL

# Contrails

It is not believed that any substantial microbial growth occurred in the "wet" storage samples, in view of the high temperature, the limited amount of water, the presence of anti-icing additive, and the absence of essential mineral nutrients.

Although it would have been desirable to base the entire program on one given set of storage conditions, the first set of conditions chosen did not give adequate deterioration of the base fuel itself and hence did not provide a good evaluation of the effect of additives. Subsequent modifications made to the original storage conditions included eliminating the free water, adopting a 4-week reaeration schedule, and increasing the air/fuel ratio in the storage drums. The storage conditions for the different series were as follows:

Series:	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Base fuel	G-1060	G-1060	H-1008	H-1010	H-1008	H-1010
Fuel sample, gal	12.5	12.5	10.0		10.0	
Air/fuel volume ratio	1/5*	1/5	1/2		1/2	
Storage temperature, °F	130	130	130		130	
Water added, vol %	0.05	0.05	None		None	
Reaeration frequency	None	4-wk	4-wk		4-wk	
Sampling frequency	4-wk	4-wk	4-wk		8-wk	
Storage period, weeks	16*	12	16		8	
Fuel coker configuration	A	C	C		C	

## D. Effect of Storage Conditions on Fuel Deterioration

While the first and second series of hot-room storage tests were in progress, a special series was set up on the same G-1060 base fuel under different storage conditions to explore the effects of storage temperature, presence of free water, and reaeration at 4-week intervals. The air/fuel ratio was 1/5 in all samples. The thermal stability evaluations were made with helium drive, Configuration (C). The following results show the change in preheater breakpoint compared to the fresh-fuel breakpoint of 525°F:

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\*In the first series, a portion of each 12-week sample was placed back in hot-room storage for an additional 4 weeks; the air/fuel ratio during this additional period was approximately 2/1.



# Contrails

	<u>8 weeks</u>	<u>12 weeks</u>
Ambient*, 0.05% water	0	+75
130°F, 0.05% water	+50	+50
130°F, dry	0	+25
130°F, dry, reaerated	0	-25

The very considerable increases in breakpoint during wet storage indicate that small amounts of free water have a definite beneficial effect, which in the present context is undesirable. Even without the presence of water, the base fuel increased 25°F in breakpoint during storage at 130°F. Reaeration at 4-week intervals gave the only instance of a decrease in thermal stability.

It was concluded that the G-1060 fuel is a rather unsatisfactory stock for evaluating the effect of additives, since no substantial deterioration of the base fuel itself could be obtained with any of the storage conditions that were evaluated. The data indicated that dry storage with reaeration was the most severe of the conditions investigated, and this was adopted for the later series of storage stability evaluations. With a view toward increasing further the severity of storage conditions, the air/fuel ratio was increased from 1/5 to 1/2 for the later series, and the storage period was increased to 16 weeks.

The apparent ameliorating effect of free water during storage is somewhat puzzling, since it is commonly supposed that free water and iron rust are detrimental to storage stability. However, one possible explanation is related to the presence of anti-icing additive (methoxyethanol) in the test fuels. If a small amount of free water is present in a drum of fuel during storage, it will extract a certain amount of the methoxyethanol from the fuel, so that a water-methoxyethanol mixture will be present as a free phase in the bottom of the drum. In the presence of only a very limited amount of water, the equilibrium concentration of methoxyethanol in the water phase is relatively high - on the order of 10 - 30%, depending on water/fuel ratio and storage temperature. It appears reasonable to suppose that some of the most polar of the nonhydrocarbon constituents of the fuel will be extracted from the fuel by this water-methoxyethanol mixture. If this occurs, such constituents are effectively removed from the scene with respect to any participation in the interactions that lead to degradation in storage. In the case of storage at 130°F, a further removal of polar nonhydrocarbon constituents may occur at the time the fuel is removed from hot storage and cooled to ambient temperature. At this time, additional water and methoxyethanol are released from solution in the fuel and settle to the bottom; quite conceivably, additional extraction of polar constituents could occur during this process.

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\*Stored indoors in winter months and in open shed in spring months.

There was not any time in the present program to explore these possibilities experimentally. However, it can be regarded as an established fact that the presence of traces of free water in a fuel containing methoxyethanol can have an ameliorating effect with regard to deterioration of thermal stability during storage.

## E. Equilibrium Relationships among Water, Methoxyethanol, and Fuel

In considering the effect of the presence of free water on the deterioration of fuels during storage, a brief study was made of the phase relationships of fuel, methoxyethanol, and water.

It was reported by Midwest Research Institute<sup>(9)</sup> in 1960 that the partition coefficient of methoxyethanol between water and JP-4 fuel at room temperature was 167 and that this value was independent of concentrations and phase volume ratio; i. e., this represented the room-temperature concentration ratio. In a later report from Midwest<sup>(10)</sup>, it was stated that the partition coefficient is not independent of concentration. It was indicated that at 75°F, with 0.001 g/ml methoxyethanol in the JP-4 fuel phase, the equilibrium concentration in the water phase was 0.2 g/ml, or a concentration ratio of 200. At 140°F, this concentration ratio decreased to approximately 90. Recent data from Phillips Petroleum<sup>(11)</sup> indicated that distribution coefficients may vary from 180 to 800, depending on temperature, concentrations, and other factors. Referring to anti-icing additive PFA 55MB, which is principally methoxyethanol, at 0.09 vol % in the fuel, the methoxyethanol contents of the water phase at equilibrium were said to fall within the range of 18 to 22 vol %, i. e., concentration ratios of 200 - 250, which are in general agreement with the room-temperature data in Reference 10.

With this background, a series of tests was set up to determine the phase relationships among water, methoxyethanol, and JP-6 fuel. Distilled water samples containing 0, 5, 10, 20 and 30 wt % methoxyethanol were equilibrated for two days at 77 and 130°F with equal volumes of H-1010 fuel, originally containing 0.108% methoxyethanol. Owing to the relatively large amount of aqueous phase present, its methoxyethanol concentrations were not changed sensibly by equilibration with the fuel. Samples of the fuel phase after equilibration were analyzed for water content by Karl Fischer titration and for methoxyethanol content by the standard dichromate titration, FTMS-791a Method 5327-T. The results of these analyses are shown graphically in Figures 6 and 7.

The data indicate that, with 0.1% methoxyethanol in the fuel phase, the distribution coefficients are approximately 205 at 77°F and 100 at 130°F, which are in general agreement with the literature data cited previously. It should be noted that the use of weight percent methoxyethanol in the water phase, rather than volume percent or g/ml, does not

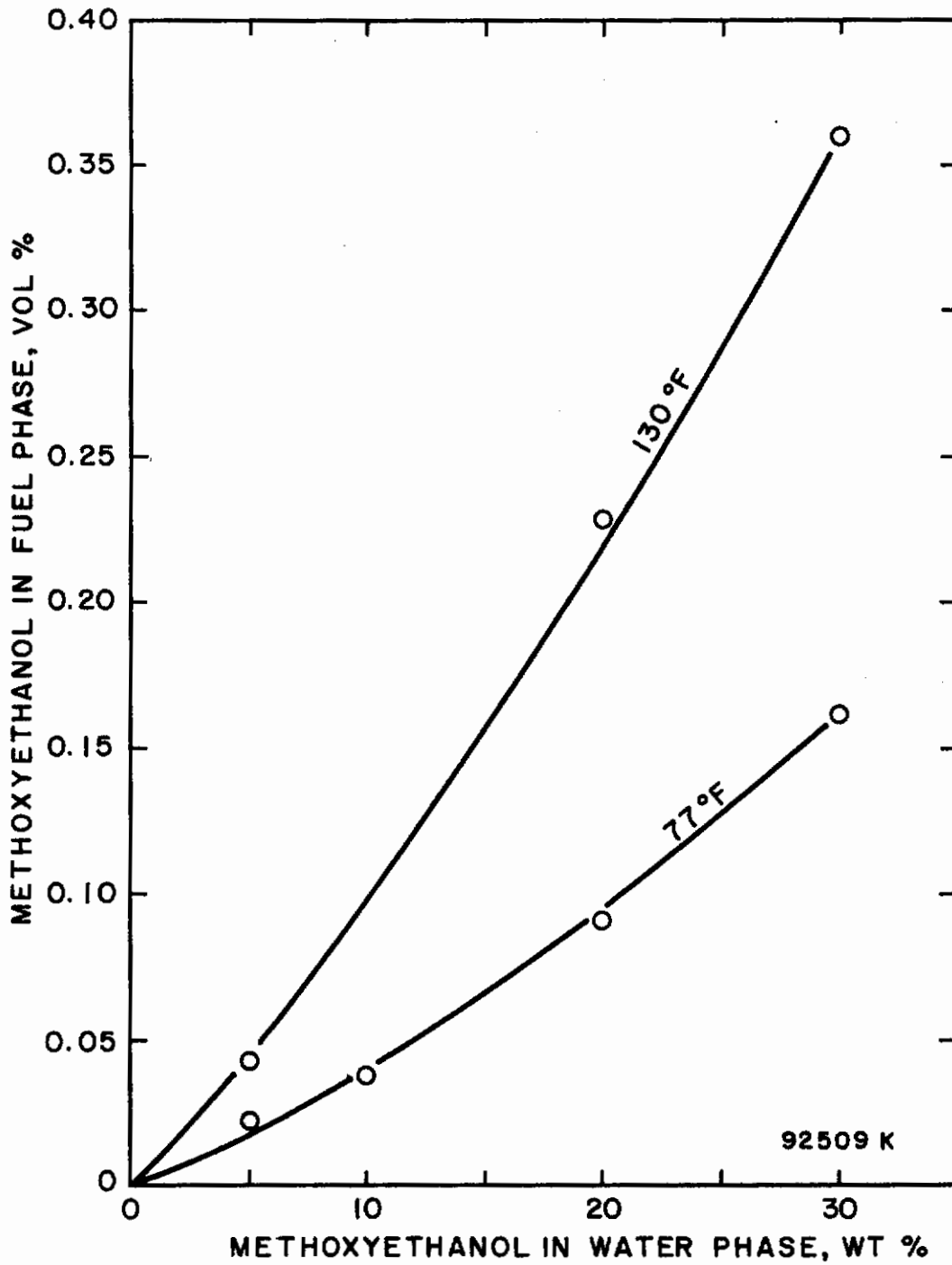


FIGURE 6. DISTRIBUTION OF METHOXYETHANOL BETWEEN WATER AND FUEL PHASES (H-1010 FUEL)

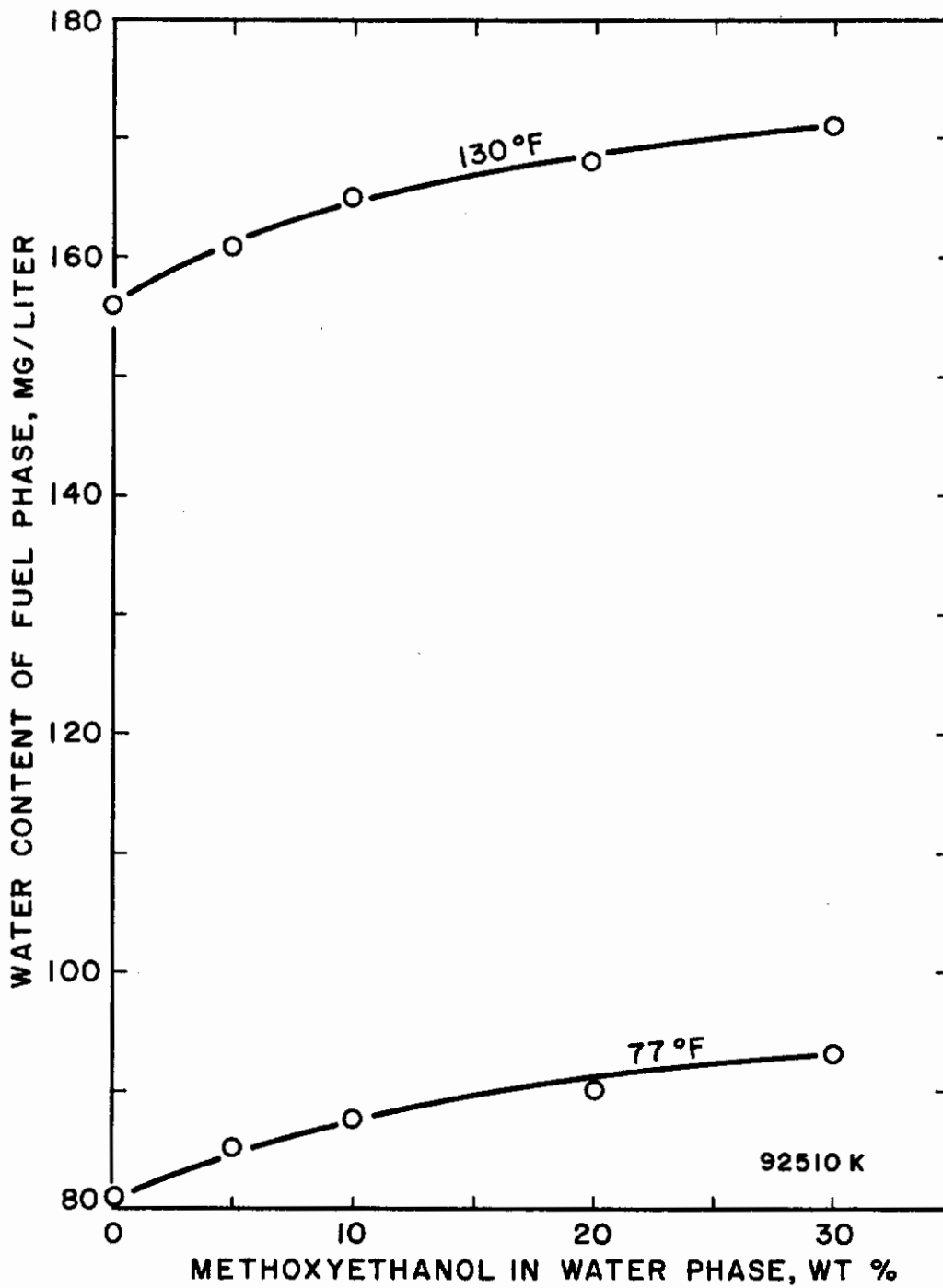


FIGURE 7. WATER CONTENT OF FUEL PHASE (H-1010 FUEL) AS A FUNCTION OF METHOXYETHANOL CONTENT OF WATER PHASE

distort the results seriously, as the methoxyethanol density is close to 1.0 g/ml.

Using these data, the equilibrium relationships in the "wet" storage drums can be calculated by trial solutions for the case of adding 0.05% water to H-1010 fuel, originally containing 0.108% methoxyethanol. It was assumed that the fuel initially contained 40 mg/liter of dissolved water; variations in this value do not affect the over-all relationships to any appreciable extent. This trial solution gave the following values for equilibration at 130° F:

Methoxyethanol content of fuel phase, vol %	0.1035
Methoxyethanol content of water phase, wt %	10.4
Water content of fuel phase, mg/liter	165

Thus, it can be seen that the fuel retains about 96% of its original methoxyethanol content under the 130° F storage condition when 0.05% water is added. It will also be noted that the water content of the fuel under these conditions is quite high, being about twice the maximum saturation value obtainable at 77° F. Therefore, the addition of a trace of free water has three effects: (1) The free water remaining under 130° F storage conditions acquires a methoxyethanol content of about 10%; (2) there is an insignificant decrease in the methoxyethanol content of the fuel phase; (3) the water content of the fuel phase at 130° F is about twice that attainable under 77° F conditions.

Although unrelated to the present work, it is of some interest to note that the water content of a fuel in equilibrium with free aqueous phase increases slightly with increasing methoxyethanol content of the water (Fig 7). This point is of interest in connection with the establishing of accurate "saturation values" for comparison with water contents of fuel samples taken in the field. In order to establish a saturation value that is representative of field conditions, the fuel sample should be equilibrated over a water bottom containing a near-equilibrium amount of methoxyethanol.

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## SECTION V

## AUXILIARY TESTS - EQUIPMENT AND PROCEDURES

A. Water Separometer Tests

CRC water separometer tests were run using the standard equipment and procedures. The two procedures employed were the "standard" procedure, FTMS-791a Method 3255-T, and the "modified" or "Severity 15" procedure, Method 3256. These tests were used primarily in screening candidate additives so as to eliminate those that had obviously undesirable effects on water-separating properties. The modified procedure was also used to obtain additional information on fuel property changes during hot-room storage.

The modified water separometer (WSIM) values are said to be very sensitive to minor amounts of contaminants such as may be derived from new, **uncleaned metal cans**. Therefore, for the purposes of this program, the one-gallon cans used for separometer samples were solvent-cleaned before initial use and then were recleaned and reused.

Another possible source of variation in water separometer results on additive blends may be aging effects in relation to fuel-additive interactions. Since previous experience had indicated that such interactions occurred largely in the first day or two after blending, all blends in the present program were aged at least two days before running the water separometer tests.

The water separometer test methods as defined in FTMS-791a call for filtration of the test fuel if it contains any visible sediment or insoluble matter. Test fuel filtration was adopted as a routine procedure, in view of possible contamination by iron rust in the "wet" storage samples. It should be noted that test fuel filtration has been specifically prohibited by current amendment to the JP-4 and JP-5 fuel procurement specification (MIL-J-5624F Amendment 2). However, for the purposes of the present program, test fuel filtration was performed prior to all tests.

In considering possible minimum limits to be set on WSI and WSIM of additive blends, it may be noted that the present specification requirements for JP-4 and JP-5 fuels are a minimum WSIM of 55 and 85, respectively. A former requirement for JP-4 fuel was a minimum WSI of 95. In the case of JP-6 fuels, it appears that no firm specification requirements have been established. For purposes of additive screening tests, it was considered that the WSI of the fuel-additive blends should be no lower than 90. It was not feasible to set a minimum value for the WSIM, in view of the lack of field data on JP-6 fuels. The turbo kerosine that was used as a base stock in the

# Contrails

additive screening tests had a WSI of 99 and a WSIM of 73. Any additives that had pronounced adverse effects on either the WSI or WSIM were eliminated from the program.

The WSIM values that were obtained on samples from the hot-room storage program were quite erratic, particularly in the case of those involving G-1060 base fuel. This may be illustrated by the following data on the G-1060 base fuel and one additive blend in the first and second storage series:

	<u>WSIM after Weeks at 130°F</u>			
	<u>0</u>	<u>4</u>	<u>8</u>	<u>12</u>
G-1060 base fuel - first series	72	98	68	83
- second series	49	74	76	75
Additive L-1 (30 lb) - first series	82	82	82	75
- second series	57	64	68	84

In each of the series, the "0-week" base fuel represents pseudo-fresh fuel from cold storage. The results on both the base fuel and the additive blend showed so much variation, apparently random, that it was impossible to draw any conclusions as to whether or not the additive had an adverse effect on WSIM in storage. It should be noted that each pseudo-fresh base fuel or additive blend represented a different drum of fuel, so it is quite possible that drum-to-drum variations had a significant effect.

The sort of erratic results illustrated above led to a cross-check on new samples of pseudo-fresh G-1060 base fuel (from a different drum) and L-1 additive blend. Both sets of samples were placed in recleaned one-gallon cans. One set of separometer tests was run immediately, and another set was run after holding the samples for one week at indoor ambient temperatures in the gallon cans. The following results were obtained:

	<u>WSIM (Fresh)</u>	<u>WSIM (1 week)</u>
G-1060 base fuel	96	88, 81
L-1 (30 lb)	88	84, 85

The repeatability was quite good on the one-week samples, and there was no apparent effect of aging on the L-1 blend. The "fresh" G-1060 base fuel from cold storage gave a high value of 96 in these tests. It will be observed that WSIM values all the way from 49 to 96 have been obtained on pseudo-fresh G-1060 base fuel in this program; when first received at SwRI, the WSIM value was 72.



There are several possible sources of erratic WSIM values in the present program. One of these is the sensitivity of results to minor variations in the test procedure. It is known, for example, that traces of isopropanol (used in flushing the rig) can affect test results. It is also considered possible that the prefiltration of test fuels employed in this program may have had a nonreproducible effect on test results. Another possible source of erratic results is drum-to-drum variations in the base fuel as received. Supposedly all drums of a given base fuel were filled from the same batch of fuel, and it is understood that dry (i. e., unoiled) drums are used for JP-6 fuels. However, it is still quite possible that the contaminants in the drums would vary in amount and type and that different drums would behave differently with regard to WSIM values after prolonged cold storage. Finally, it is possible that the individual 15-gallon drums in hot-room storage will show differing behavior with regard to WSIM. Here all of the drums had been thoroughly precleaned, but it is still considered possible that differences in interior contamination may exist.

Base fuel G-1060, which ran generally lower in WSIM than the other two base fuels, also showed the widest variation in WSIM values. Significant drum-to-drum variations were suspected, but the supply of base fuel was exhausted before any detailed checks could be made.

A brief study was made of possible drum-to-drum variations in the WSIM values of base fuels H-1008 and H-1010. These base fuels were pseudo-fresh, from the original 55-gallon drums in cold storage; the WSIM tests were run immediately after sampling and after one week in one-gallon cans at indoor ambient temperatures. The H-1008 base fuel gave WSIM values from 84 to 98, i. e., a possibly significant drum-to-drum variation. The H-1010 base fuel gave values from 95 to 100, which is not considered as a significant variation. There was no regular trend toward higher or lower values on the one-week aged samples.

It was not considered within the scope of the present contract to investigate separator test repeatability and reproducibility. Therefore, it cannot be stated with certainty just what factors were most important in causing the erratic results that were observed.

## B. Light Transmittance Measurements

In order to accumulate additional data for possible correlation with storage and thermal stability test results, the light transmittance was measured on all fresh and aged samples. Light transmittance was measured at 350 and 375 m $\mu$ , using a Bausch and Lomb "Spectronic 20" photoelectric colorimeter. All measurements were expressed as percent transmittance referred to spectroanalyzed isooctane.

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SECTION VI

STORAGE TEST RESULTS AND DISCUSSION

A. General

The hot-room storage tests in this program were run on six series of fuel-additive blends. As discussed in Section IV, storage conditions were varied in the early series of tests in order to obtain significant breakdown of the base fuels. Also, the method of fuel coker evaluation was changed after the first series of tests, since the modified coker with pump was not giving meaningful results.

Complete data on the samples from the hot-room storage tests are given in Appendix III, including fuel coker ratings, water separator values (WSIM), and light transmittance values.

B. First Series of Hot-Room Storage Tests

The initial conditions chosen for hot-room storage were:

Fuel sample, gal	12.5
Air/fuel volume ratio	1/5
Storage temperature, °F	130
Water added, vol %	0.05
Reaeration frequency	None

Four samples of each fuel-additive blend were prepared and were intended for thermal stability evaluations at 0, 4, 8, and 12 weeks. The absence of any deterioration of the base fuel during this period led to the replacement of a portion of the 12-week samples in storage for 4 weeks longer. During this additional storage period, the air/fuel ratio was approximately 2/1.

The base fuel used in this series was G-1060 (Ashland, first batch). The original modified coker, Configuration (A), with original preheater tube and Eastern pump, was used in the thermal stability evaluations.

Complete test data are given in Appendix III, including thermal stability ratings (Table 17), water separator values (Table 23), and light transmittance values (Table 29).

Major difficulty was encountered in rating the thermal stability of this series because of the presence of wipable preheater deposits, as has been discussed in detail in Section III. Both unwiped and wiped ratings were obtained, but the choice of temperatures for establishing breakpoints was

based on unwiped ratings. Therefore, the wiped ratings, which with this apparatus are probably more significant, in most cases did not define the breakpoint temperature. A summary of the breakpoints (preheater temperatures at failure) is given in Table 16 for both unwiped and wiped ratings.

The fresh base fuel had a preheater breakpoint of 525° F based on unwiped ratings, and its true breakpoint (wiped ratings) was above 525° F and undetermined. Several of the additives gave an improvement in initial "thermal stability" as measured by unwiped ratings, but it is not clear how much of this apparent improvement was due to improvement of fuel lubricity rather than true improvement of thermal stability. The greatest improvement in initial "thermal stability" was given by Additive F-1, which gave tests with no deposits at 600/700° F. Additive E-1 caused an immediate decrease in thermal stability that showed up in filter plugging as well as preheater deposits.

During the first 12 weeks of storage, the base fuel did not deteriorate at all in thermal stability, and in fact improved by 25° F, based on unwiped ratings. None of the additive blends showed any improvement in comparison with base fuel performance after 12 weeks. The additional 4 weeks of storage was exceptionally severe, probably because of the opening of the sample drums and the introduction of a large relative volume of air during the additional 4 weeks. In any case, the 16-week samples all showed true breakpoints (based on wiped ratings) of 475° F or lower, with the exception of the L-1 blend, which passed at 475° F. The D-3 additive, which had shown some tendency to lower breakpoints during the 12-week period, gave a filter breakpoint at 450° after 16 weeks of storage. The E-1 additive showed a deleterious effect on thermal stability throughout the storage period. This additive is an approved antioxidant for use in jet fuels at a maximum concentration of 8.4 lb/1000 bbl; in the present series of tests, it was used at 30 lb/1000 bbl. The unfavorable results on this additive indicate that it probably decomposes at temperatures above 400° F and thus contributes to poor thermal stability.

The indeterminacies in this first series of storage tests prevented any overall comparison of additive efficiencies. However, it was demonstrated that Additives D-3 and especially E-1 had harmful effects on storage and thermal stability, and that Additive F-1 had an initially marked beneficial effect that was not maintained over 12 weeks of storage at 130° F.

## C. Second Series of Hot-Room Storage Tests

The storage conditions for the second series of hot-room storage tests were:

TABLE 16. PREHEATER BREAKPOINTS IN FIRST SERIES OF STORAGE TESTS

<u>Additive</u>	<u>Concn,</u> <u>lb/1000 bbl</u>	<u>Preheater Breakpoint (°F)</u> <u>after Weeks of Storage as Shown:</u>				
		<u>0</u>	<u>4</u>	<u>8</u>	<u>12</u>	<u>16</u>
<u>Based on Unwiped Ratings</u>						
None (G-1060 base fuel)		525	500	>525	550	≤475
DBPC	30	550	500	575	550	≤475
B-2	50	575	525	550	475	≤450
D-1	20	575	500	500	500	≤475
D-3	30	500	475	475	475	≤450*
E-1	30	450*	450*	450*	425*	≤425*
L-1	30	525	500	450	450	>475
F-1	270	>600	>600	600	≤500	475
<u>Based on Wiped Ratings</u>						
None (G-1060 base fuel)		>525	>525	>525	>550	≤475
DBPC	30	550	>500	575	550	≤475
B-2	50	>575	>525	>550	525	≤450
D-1	20	>575	>500	>500	>500	≤475
D-3	30	500	475	475	>475	≤450*
E-1	30	450*	450*	450*	425*	≤425*
L-1	30	>525	500	>450	>450	>475
F-1	270	>600	>600	600	≤500	475

\*Filter breakpoint.

# Contrails

Fuel sample, gal	12.5
Air/fuel volume ratio	1/5
Storage temperature, °F	130
Water added, vol %	0.05
Reaeration frequency	4-wk

These were the same as in the first series, except for the 4-week reaeration, which was adopted in an attempt to increase the severity of storage and to induce deterioration of the base fuel. The total storage period was 12 weeks.

The base fuel used in the second series was G-1060 (Ashland, first batch), the same as the first series. However, for the second series, the modified fuel coker with helium drive, Configuration (C), was adopted for the thermal stability evaluations. This permitted an objective evaluation of thermal stability changes without the distortions of test results created by fuel pump wear debris.

Complete test data are given in Appendix III, including thermal stability ratings (Table 18), water separator values (Table 24), and light transmittance values (Table 30). It will be observed from a comparison of the unwiped and wiped preheater ratings of the thermal stability data that the problem of wipable deposits had disappeared and that the test results could be interpreted unambiguously. Comparison of results in the following paragraphs is based on unwiped ratings, but the wiped ratings would have yielded the same comparisons. There were no instances of filter plugging in this second series, hence all comparisons are based on preheater breakpoints. The changes in preheater breakpoints, in comparison with that of fresh base fuel G-1060, are plotted against storage time in Figure 8.

First of all, it will be observed that the G-1060 base fuel did not deteriorate during 12 weeks of storage and in fact improved by 25°F over its original breakpoint of 525°F. Significant improvements in initial thermal stability amounting to 50°F or more were given by DBPC (270 lb), L-1 (30 lb), L-1 (270 lb), F-1 (270 lb), L-2 (30 lb), and M-1 (30 lb). However, of all these additives, only L-1 (30 lb) and F-1 (270 lb) showed any advantage over the base fuel itself after 12 weeks of storage, and this was a mere 25°F advantage. It will be observed that the beneficial effect of the L-1 at a dosage of 30 lb was not observed at 270 lb, and in fact the 270-lb dosage was apparently harmful at intermediate storage periods. The K-1 additive showed harmful effects after 12 weeks of storage.

Thus, this second series of storage tests indicated that the G-1060 base fuel was not a good choice for evaluating the effect of additives on storage stability, since it did not itself deteriorate after 12 weeks of accelerated storage. However, it should be kept in mind that this series was run

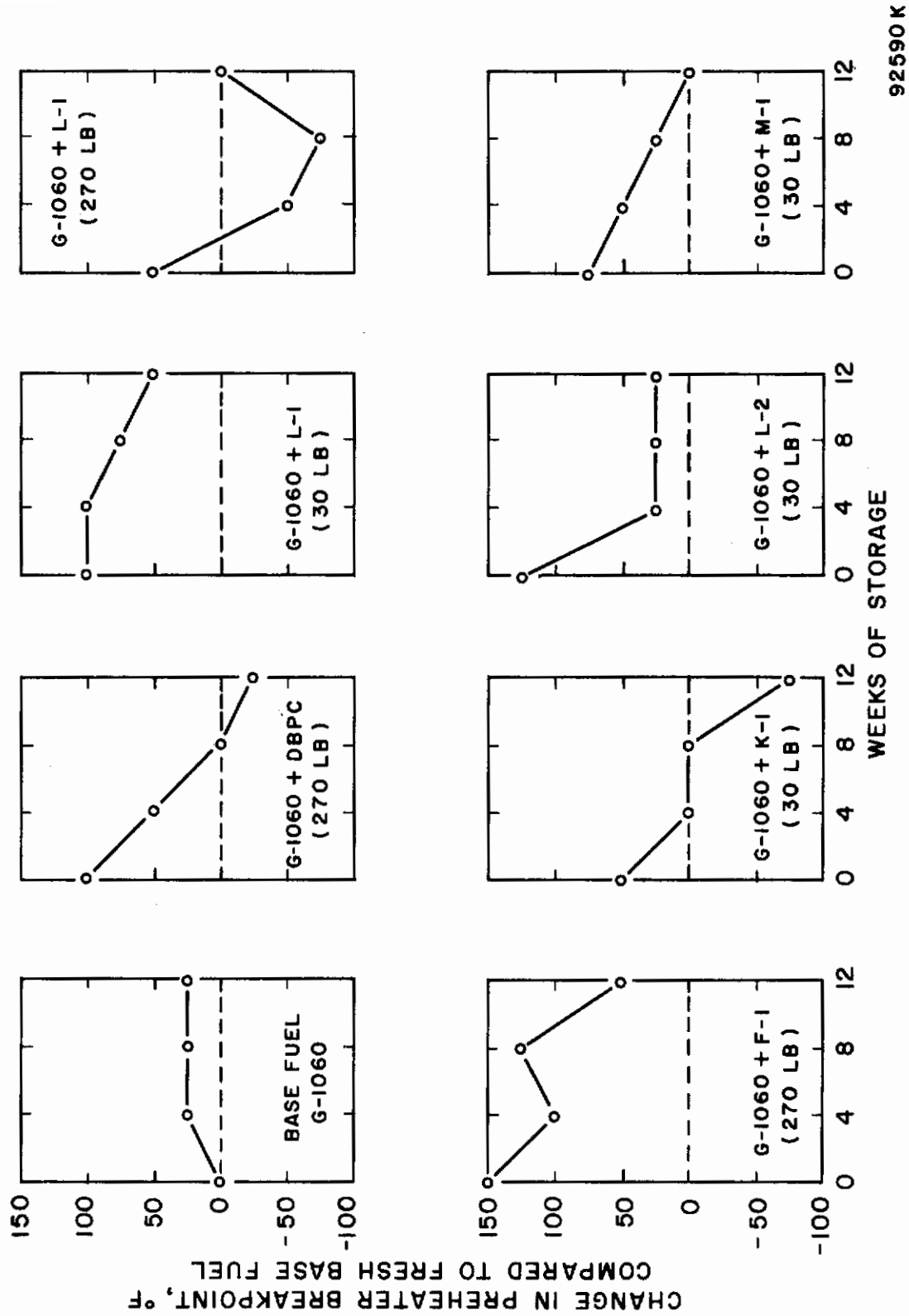


FIGURE 8. PREHEATER BREAKPOINT VARIATION FOR SECOND SERIES OF STORAGE TESTS

with the presence of a trace of free water, which was found to have an ameliorating effect on the deterioration of thermal stability.

D. Third, Fourth, Fifth, and Sixth Series of Hot-Room Storage Tests

1. General

The remaining series of hot-room storage tests in the present program were run under the following conditions:

Fuel sample, gal	10.0
Air/fuel volume ratio	1/2
Storage temperature, °F	130
Water added	None
Reaeration frequency	4-wk

In comparison with the previous series, the storage conditions were made more severe by increasing the air/fuel ratio and eliminating the use of free water, as well as retaining the reaeration procedure. The total storage period was 16 weeks in the third and fourth series, with recheck tests of 8 weeks' storage comprising the fifth and sixth series.

The base fuels used in these series were H-1008 (Ashland second batch) and H-1010 (Standard Oil of California). The modified fuel coker with helium drive, Configuration (C), was used for all thermal stability evaluations.

The third and fourth series were run on additives that had appeared promising in previous test series, as well as a number of previously untried additives. The fifth and sixth series were run primarily as check tests on the third and fourth series, to explore the possibility of any distorting effect of drum-to-drum base fuel variations on the results of the additive evaluations.

2. Selection of Additives

For the earlier series of tests, the selection of additives had been based primarily on suppliers' data and on water separometer test results. In order to expand the coverage of the additive field, it was desired to include in the later series of evaluations a number of additives for which no suppliers' background data were available. It was recognized that many of the materials submitted could be detrimental to thermal stability. For example, in the first series of hot-room storage tests, Additive E-1, a recognized antioxidant, had a seriously detrimental effect on thermal stability. Therefore, it was considered desirable to screen unknown additives



for possible deleterious effects on thermal stability before including them in the later series of hot-room storage evaluations.

For this purpose, additive blends in H-1010 base fuel were tested for thermal stability at 500/600°F, which represented a temperature level 50°F below the breakpoint established for this fuel. Tests were run with helium drive, Configuration (C), with the following results:

<u>Additive and Concentration, lb/1000 bbl</u>	<u>Coker Results</u>
E-4 (30)	Fail
E-6 (30)	Fail
E-12 (30)	Fail
E-3 (30) and E-5 (8)	Pass
H-2 (30)	Pass
J-2 (30)	Pass
N-1 (30)	Pass
P-1 (30)	Fail
P-3 (30)	Pass
Q-1 (30)	Fail
S-1 (30)	Fail
S-2 (30)	Fail

All additives failing under these conditions were eliminated from the program, and those passing were used to round out the third and fourth storage series.

For the fifth and sixth storage series, the amount of base fuel was limited, and it was necessary to restrict the number of additives and to eliminate some of those used in the third and fourth series. The selection was based on performance data available at that time from the third and fourth series.

### 3. Drum-to-Drum Variations

The method of preparation of the additive blends for the first four series of hot-room storage tests was predicated on the assumption that the base fuel as received was uniform from drum to drum and that it remained uniform during its retention in cold storage. This assumption cannot be considered as correct per se.

While the third and fourth storage tests were in progress, the remaining base fuel in cold storage was checked for drum-to-drum variations. Each drum was removed from cold storage overnight and then stirred for 20 minutes, after which 8 gallons was removed for thermal stability testing. In

# Contrails

the following tabulation of breakpoints, the "A" drums were partial drums remaining in cold storage; the other drums were full and undisturbed prior to this sampling.

	<u>Preheater Breakpoint, °F</u>
<u>H-1008</u>	
Previous data	550
Drum A	500*
Drum B	500**
Drum C	500
Drum D	475*
Drum E	575
Drum E, top	Above 575
Drum E, bottom	Above 575
Homogenized, A-E	575
<u>H-1010</u>	
Previous data	550
Drum A	625
Drum B	550
Drum C	575
Homogenized, A-F	575

These variations in base fuel from drum to drum were naturally a cause of considerable concern. In the case of the H-1008 fuel, the range of variation in preheater breakpoint was 100°F. Also, it is noteworthy that samples drawn from the top and bottom of Drum E gave passing tests at 575°F, whereas the regular sample from Drum E after stirring gave a breakpoint at 575°F. Even more surprising was the performance of the homogenized five-drum mixture, for which the breakpoint of 575°F was considerably above the average of the breakpoints of the component drums.

The situation was somewhat similar for H-1010, except that only three of the remaining six drums were investigated. A variation of 75°F in preheater breakpoint was found. Here the homogenized six-drum mixture rated 575°F, which in this case is within the range of the breakpoints of the three drums tested.

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\*Also appreciable filter pressure drop, but less than 2.0 in. Hg.

\*\*Also failing filter pressure drop, more than 2.0 in. Hg.

The homogenization of the base fuels was accomplished in order to prepare uniform base stocks for the fifth and sixth series of hot-room storage tests. For this purpose, the drums of each base fuel were emptied into a clean aluminum tank and mechanically agitated for 40-50 minutes prior to drawing off base fuel for making the additive blends. All additive blends were made on the same day for these series and were retained in cold storage until the hot-room schedule could be started.

As will be described in the following section, the results of the fifth and sixth series, using homogenized base fuels, gave good checks on the third and fourth series. Therefore, it appears that the relative ratings of additives in the third and fourth series are valid in spite of the known drum-to-drum variations in the base fuels. It is quite likely that very slight variations in base stock quality or drum contaminants, which may cause significant changes in thermal stability of the base stock, will be overridden by the effects of additives.

#### 4. Comparative Performance of Additives

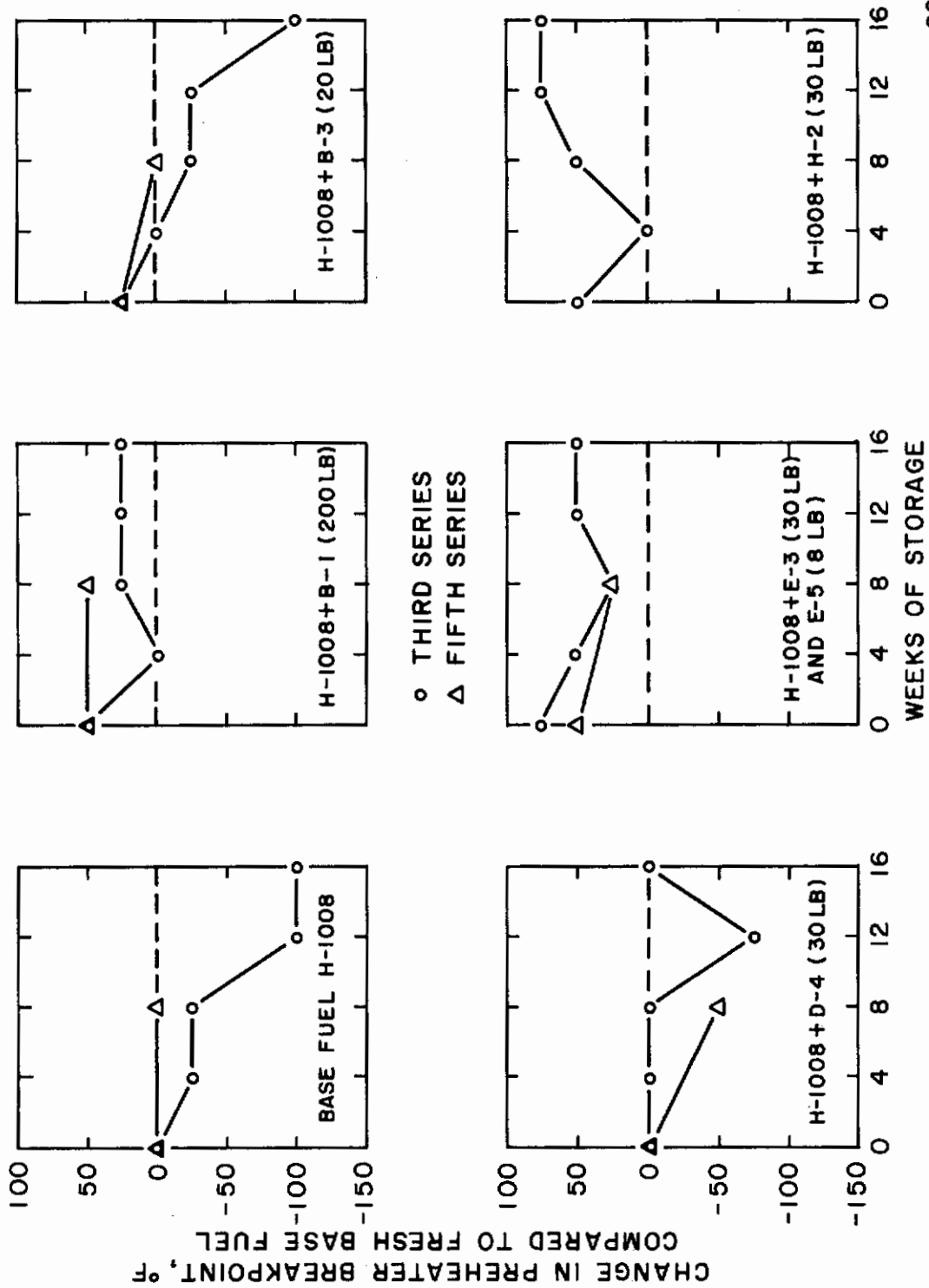
Complete test data for the third, fourth, fifth, and sixth series of storage tests are given in Appendix III, including thermal stability ratings (Table 19-22), water separator ratings (Tables 25-28), and light transmittance values (Tables 31-34). The thermal stability ratings in terms of preheater breakpoints\*, are plotted in Figure 9 (H-1008, third and fifth series) and Figure 10 (H-1010, fourth and sixth series). From these plots, it will be observed that the rechecks using homogenized base fuels (fifth and sixth series) reproduced closely the results of the earlier third and fourth series. Therefore, it appears that these combined results provide a valid comparison of the effects of the various additives in the two base fuels.

It will be further observed that both of the base fuels showed significant deterioration after 16 weeks, amounting to 100° F loss in preheater breakpoint from the original values.

In the H-1008 base fuel (Figure 9), all of the additives either improved initial thermal stability or had no effect. However, after the full 16 weeks of storage, only B-1, E-3/E-5 combination, H-2, J-2, and F-1 maintained thermal stability equal to or higher than that of fresh base fuel. If the results are judged only on the basis of reducing the degradation of the base fuel, then D-4 and L-1 would be added to the list of beneficial additives.

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\*With H-1010 base fuel in the fourth series, a few filter breakpoints were observed. However, these coincided in temperature with the preheater breakpoints in all cases except one, where there was only a 25° F difference.



92579 K

FIGURE 9. PREHEATER BREAKPOINT VARIATION FOR THIRD AND FIFTH SERIES OF STORAGE TESTS

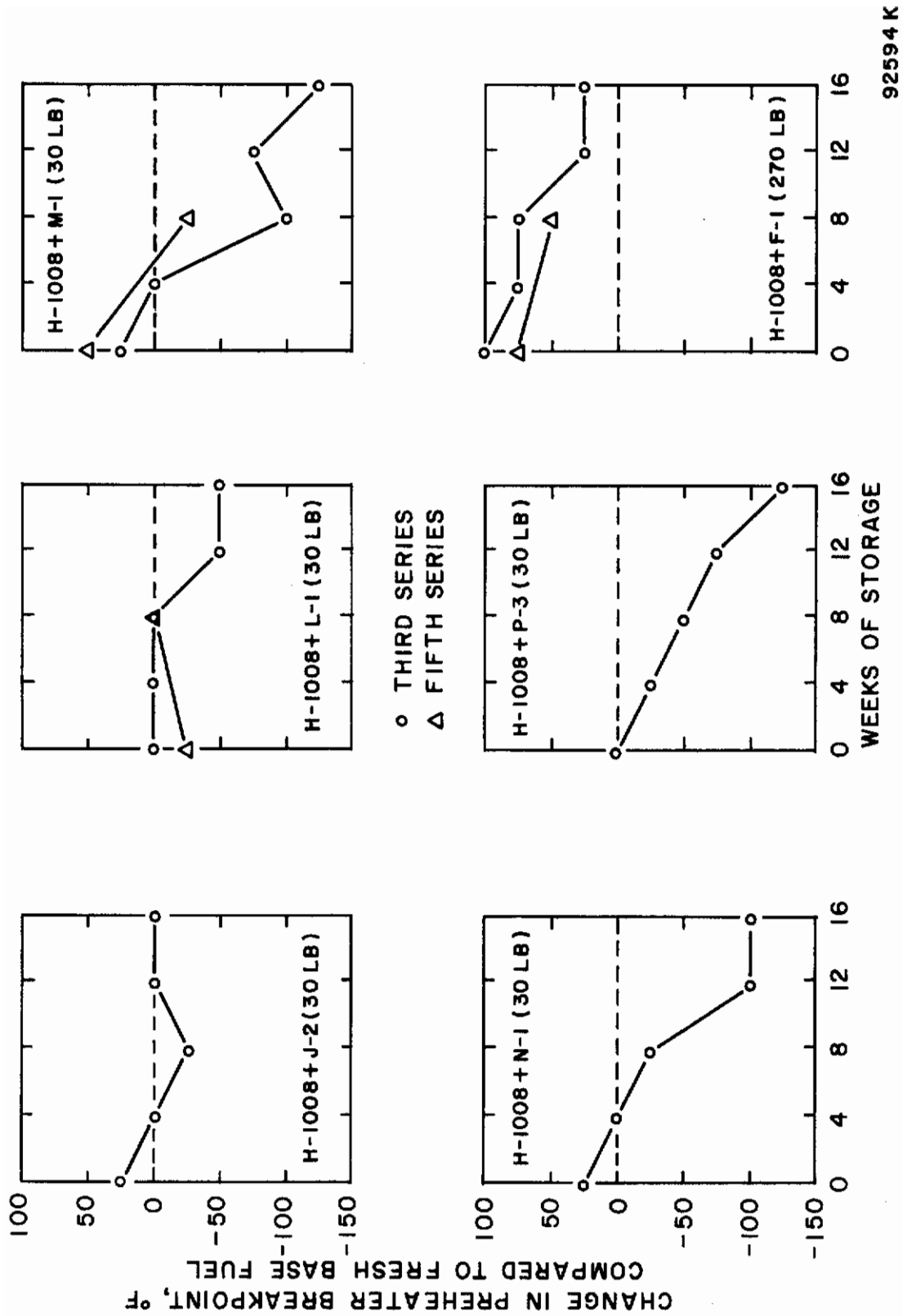


FIGURE 9. PREHEATER BREAKPOINT VARIATION FOR THIRD AND FIFTH SERIES OF STORAGE TESTS (Cont'd)

92594 K

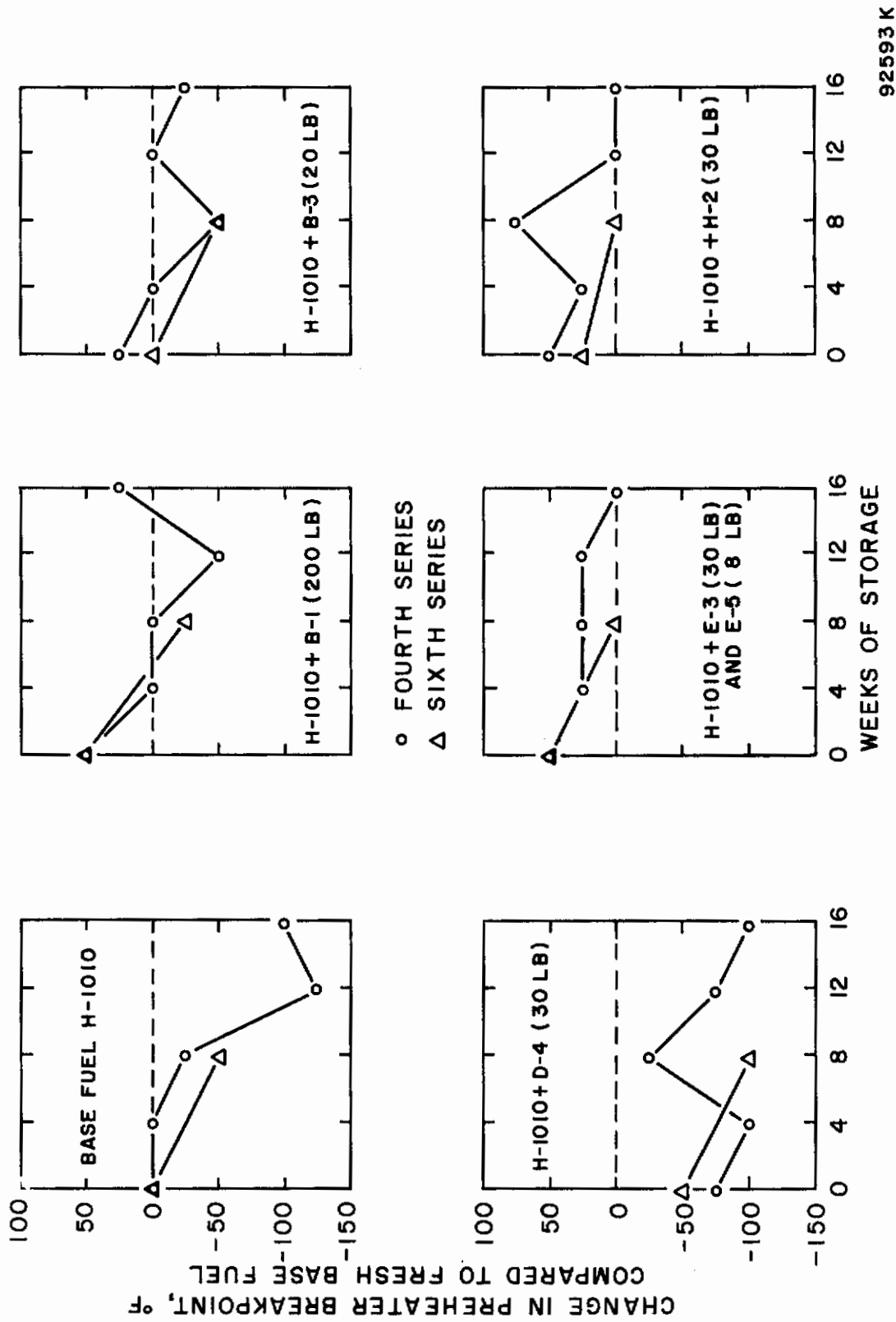


FIGURE 10. PREHEATER BREAKPOINT VARIATION FOR FOURTH AND SIXTH SERIES OF STORAGE TESTS

92593 K

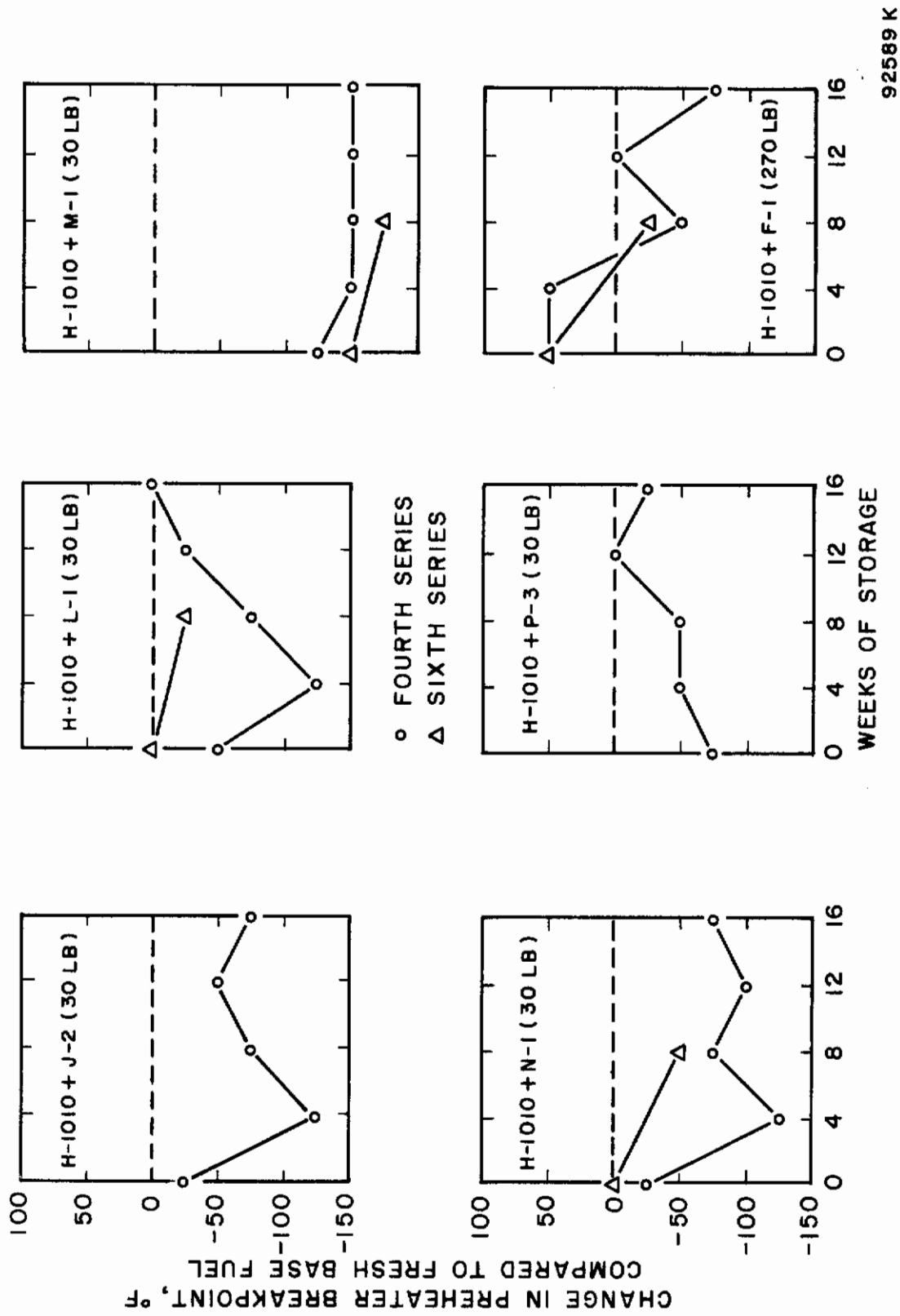


FIGURE 10. PREHEATER BREAKPOINT VARIATION FOR FOURTH AND SIXTH SERIES OF STORAGE TESTS (Cont'd)

# Contrails

In the H-1010 base fuel (Figure 10), many of the additives harmed the initial thermal stability - notably D-4, M-1, and P-3, and possibly J-2 and L-1. Also, the improvements in initial thermal stability given by some of the other additives were relatively minor when compared with performance in H-1008 base fuel. Thus, it appears that the H-1010 base fuel is definitely less susceptible than H-1008 to improvement of thermal stability by means of additives. After 16 weeks of storage, the only additives maintaining thermal stability of H-1010 equal to or higher than that of the fresh base fuel throughout the test period were E-3/E-5 combination and H-2. If the results are judged only on the basis of reducing the degradation of the base fuel, then B-1 and B-3 and possibly F-1, L-1, and P-3 would be added to the list. The latter two additives showed poor thermal stabilities in the early stages of the test but subsequent improvement.

It is of interest to note that the two best additive packages in these series are conventional materials. The E-3/E-5 combination consists of a conventional antioxidant and metal deactivator, both approved for use in jet fuels. For the hot-room storage tests, the concentrations of each were approximately four times the normal maximum for jet fuels. The H-2 additive is a conventional metal deactivator approved for use in jet fuels; here its working concentration was 15 times the normal maximum for jet fuels.

One feature of most of the preheater breakpoint variations shown in Figure 9 and 10 is the somewhat irregular nature of the changes in thermal stability during storage. These cannot be ascribed to drum-to-drum variations in the base fuel, as a single drum of base fuel was used in preparing each fuel-additive blend for the storage series. Erratic or cyclic changes in fuel properties during storage seem to be the rule rather than the exception; this has been amply demonstrated in the case of gum content of fuels in ambient or accelerated storage. The appearance of erratic variations in thermal stability during storage may be inherent in the behavior of the fuels, or it may be simply a reflection of the impossibility of achieving absolute reproducibility of conditions within the individual 15-gallon drums. Although the drums were all new and precleaned and were taken from a single batch of drums, it cannot be guaranteed that they were all exactly the same with regard to steel composition or the amount of oxide present on the surfaces. Another possible variable is the amount of air leakage out through the bung seals during storage at 130° F.

What has been demonstrated by these series of storage tests is that base fuels that suffer thermal stability deterioration during storage at 130° F can be improved by the use of certain additives.

## E. Water Separometer Ratings on Storage Samples

The WSIM values on the samples from the storage programs showed erratic variation that did not correlate with thermal stability ratings.



# Contrails

Although there were some instances in which the WSIM followed the thermal stability changes, these were the exception rather than the rule. Complete water separator ratings are given in Appendix III, Tables 23-28.

Of the three base fuels, G-1060 showed the lowest average WSIM values on the additive-free fuel, ranging from 49 to 98 on the storage samples. The H-1008 base fuel gave generally higher values, except for one value of 50 obtained after 16 weeks of storage, at which time the thermal stability had also deteriorated significantly. The H-1010 base fuel gave the highest WSIM values of the three base fuels; it ranged from 84 to 96 during the entire 16-week storage period, despite a significant deterioration in thermal stability. The relative ratings of the base fuel WSIM values were in general reflected in the corresponding values for the additive blends, but the individual variations were great.

One point of particular interest is the performance of Additive F-1 with regard to WSIM values. This additive had originally been checked in turbo kerosine at 270 lb/1000 bbl and was found to improve the WSIM, giving a value of 96 in comparison with 73 on the kerosine. However, when blended in JP-6 fuels for the storage tests, the trend in WSIM was downward, and a number of very low values were obtained:

		WSIM				
		<u>0-wk</u>	<u>4-wk</u>	<u>8-wk</u>	<u>12-wk</u>	<u>16-wk</u>
First series:	Base fuel	72	98	68	83	
	F-1 blend	48	47	62	39	
Second series:	Base fuel	49	74	76	75	
	F-1 blend	54	42	54	67	
Third series:	Base fuel	93	97	86	86	50
	F-1 blend	80	51	48	33	20
Fourth series:	Base fuel	92	91	84	89	96
	F-1 blend	76	76	71	62	72
Fifth series:	Base fuel	96		88		
	F-1 blend	62		59		
Sixth series:	Base fuel	100		82		
	F-1 blend	97		80		

The relatively poor performance in WSIM of the F-1 additive is undoubtedly influenced by the high concentration of 270 lb/1000 bbl that is recommended for this additive. The rather consistent downward trend in WSIM ratings indicates that this additive could introduce some difficulty in fuel handling.

Of the other additives that were evaluated in storage tests, there were instances noted of adverse effect on WSIM, but not to the extent noted for Additive F-1.

As was discussed in Section V, the erratic behavior of the WSIM values may be due to actual variations in the samples, or it may be largely a reflection of sensitivity to minor and uncontrolled variables in the separator test procedure. In any case, the erratic results that were obtained make it difficult to put any weight on the WSIM values as an index to behavior in storage.

## F. Light Transmittance Values on Storage Samples

Complete data on the light transmittance values of the storage samples, at 350 and 375  $m\mu$ , are given in Appendix III, Tables 29-34. All attempts to establish any overall correlation between light transmittance values and thermal stability ratings were unsuccessful. For the third and fourth series of hot-room storage tests, the light transmittance values of the base fuels followed the deterioration of thermal stability, but the results on additive blends were completely noncorrelative. This is exemplified by the following data from the third series of storage tests, in which changes in preheater breakpoint are referred to the fresh base fuel and light transmittance values are given in % of isooctane at 350  $m\mu$ :

		<u>Fresh</u>	<u>4-wk</u>	<u>8-wk</u>	<u>12-wk</u>	<u>16-wk</u>
Base fuel:	$\Delta$ breakpoint	0	-25	-25	-100	-100
	Transmittance	89.8	87.2	85.6	72.1	71.6
B-1/200 lb:	$\Delta$ breakpoint	+50	0	+25	+25	+25
	Transmittance	85.8	84.2	78.2	74.2	71.4
N-1/30 lb:	$\Delta$ breakpoint	+25	0	-25	-100	-100
	Transmittance	92.6	86.6	82.4	92.3	90.1

It will be noted that the B-1 additive, which maintained thermal stability equal to or above that of fresh base fuel, gave the same loss in light transmittance during storage as did the deteriorating base fuel. On the other hand, the N-1 additive, which allowed thermal stability deterioration about the same as in the base fuel, maintained light transmittance at quite high values throughout the storage period.

Since poor correlations of this type were encountered throughout the data, it was impossible to establish even any limited relationships between thermal stability changes and light transmittance. It was concluded that measurements of light transmittance at 350 or 375  $m\mu$  have no value in predicting thermal stability changes of fuel-additive blends.

## G. Comparison of Effects of Additives

The performance of the various additives in storage tests is summarized qualitatively in the following table, in which improvement is indicated by "+" and "++", poorer performance by "-" and "--", and no effect by "0", all comparisons being made with the base fuel performance at the corresponding storage periods. The columns headed "I" indicate effects on initial thermal stability, and the columns headed "S" indicate effects over the entire storage period. Additive concentrations are given in lb/1000 bbl.

Storage series:	1		2		3 & 5		4 & 6	
Base fuel:	G-1060		G-1060		H-1008		H-1010	
Base fuel final $\Delta T$ :	*		+25		-100		-100	
	<u>I</u>	<u>S</u>	<u>I</u>	<u>S</u>	<u>I</u>	<u>S</u>	<u>I</u>	<u>S</u>
DBPC (30)	*	*						
DBPC (270)			++	-				
B-1 (200)					+	++	+	+
B-2 (50)	*	-						
B-3 (20)					+	0	0	+
D-1 (20)	*	*						
D-3 (30)	-	-						
D-4 (30)					0	+	--	0
E-1 (30)	--	--						
E-3/E-5 (30/8)					+	++	+	++
F-1 (270)	++	0	++	+	++	++	+	0
H-2 (30)					+	++	+	++
J-2 (30)					+	+	-	0
K-1 (30)			+	--				
L-1 (30)	*	*	++	+	0	+	-	+
L-1 (270)			+	-				
L-2 (30)			++	0				
M-1 (30)			+	0	+	-	--	-
N-1 (30)					+	0	-	0
P-3 (30)					0	-	--	+

From this summary, it can be seen that the most promising additives are B-1, E-3/E-5 combination, F-1, and H-2. It should be emphasized, however, that other effective additives may have been overlooked simply because of lack of time for their evaluations. For example, Additive D-1 did not receive a firm rating in the first series of storage tests, but it was not found feasible to include it in later series. Also, it should be emphasized that time did not permit evaluation of different concentrations of the additives, so many of them

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\*Breakpoints indeterminate because of wipable deposits.

# *Contrails*

were undoubtedly tested at concentrations far from optimum. This was particularly true in the case of those additives for which the suppliers furnished no data or inadequate test data.

Considering the four additives or additive combinations found to be most effective, it may be noted that the E-3/E-5 combination is a conventional antioxidant-metal deactivator combination, used at approximately four times the normal maximum concentrations used in jet fuels. The H-2 is a conventional metal deactivator, used at approximately 15 times the normal maximum jet fuel concentration. The B-1 and the F-1 were experimental additives, both used at very high concentrations, in line with the suppliers' data.

The present program has demonstrated that certain additives are effective in retarding or eliminating the thermal stability deterioration of JP-6 fuels in 130°F storage. If time had permitted a thorough storage-test evaluation of all additives over a range of concentrations, as well as in combination, it is quite possible that many other effective additives would have been found.

## SECTION VII

## SUMMARY AND CONCLUSIONS

A modified fuel coker test using helium pressurization instead of a fuel pump has been developed for the evaluation of JP-6 fuel deterioration. The original Eppi-CRC modified fuel coker was unsatisfactory for this purpose because of severe problems with pump wear debris and wipable pre-heater deposits. The helium-drive fuel coker test has been demonstrated to give excellent repeatability and reproducibility of results. The service experience with the helium-drive test has been very good, and there is no significant depletion of oxygen content in the fuel fed to the coker throughout the test. The level of severity at a given test temperature for the helium-drive modified fuel coker is approximately the same as for the standard ASTM-CRC fuel coker, based on limited results. The standard ASTM-CRC fuel coker has been demonstrated to be unsatisfactory for evaluating the JP-6 fuels used in this program, owing to the same problems with pump wear debris that appear with the Eppi-CRC modified fuel coker.

The deterioration of thermal stability of JP-6 fuels and fuel-additive blends has been measured by means of 130°F hot-room storage tests in 15-gallon steel drums. In the early stages of the program, storage conditions were varied with respect to moisture and air availability in order to achieve significant deterioration of the base fuels. The final conditions chosen were an air/fuel ratio of 1/2 in sealed drums, reaerating at four-week intervals, with no free water present.

A total of 52 additives were obtained for evaluation, and data on effects of the additives on fuel thermal stability and water-separating properties were furnished by some of the suppliers. Further tests of these properties were run to screen out some of the more undesirable materials. A total of 19 additives were selected for evaluation in hot-room storage tests.

The first series of hot-room storage tests did not yield any valid comparisons in most cases because of difficulties with the original modified fuel coker test. Subsequent series of hot-room storage tests were evaluated using the helium-drive modified coker. In the second series of hot-room storage tests, significant deterioration of the base fuel itself was not achieved, so again the results were somewhat inconclusive. In the remaining four series of hot-room storage tests, the selection of base fuels and storage conditions permitted the realization of a 100°F deterioration of base fuel thermal stability during 16 weeks of storage, thus giving a good evaluation of the effect of additives in preventing or lessening this deterioration. Four additives, one of them a combination of two additives, were found to be most effective in maintaining fuel thermal stability during hot-room storage.

# Conclusions

Time did not permit a thorough evaluation of all promising additives over a range of concentrations nor any exploration of synergistic effects with combinations of additives. Therefore, it is quite probable that such additional studies would reveal other effective additives or combinations of additives among the group tested.

The water separometer (WSIM) results on the fresh fuel-additive blends and on the samples from the hot-room storage program were quite erratic and could not be related to thermal stability variations. It is not known to what extent these erratic results represented actual variations in the samples themselves and to what extent they represented difficulty with the separometer procedure.

Measurements of light transmittance on the samples from the hot-room storage program did not show any correlation with thermal stability test results, and it is concluded that such measurements are of no value in predicting the performance of fuel-additive blends.

The principal contribution of this program has been the demonstration that certain additives can produce marked improvements in maintenance of fuel thermal stability during storage at 130°F. It remains to be seen whether such improvements can also be realized in normal ambient-temperature storage. The choice of the relatively mild temperature of 130°F and the use of steel drums as storage containers are believed to increase the probability of correlation of the results of the hot-room tests with those of normal ambient-temperature storage. Further work is needed to demonstrate whether such a correlation does exist and whether the same additives will be effective under normal storage conditions.

At the other end of the scale, it is evident that a true small-scale method to evaluate thermal stability deterioration will be necessary before any rapid progress can be made in selecting optimum additives and concentrations. The number of combinations of fuels and additives that could be evaluated in the present program was very strictly limited by the operating time and sample requirements of the modified fuel coker.

Finally, it should be noted that most of the history of deterioration of JP-6 thermal stability during normal storage has been based on standard ASTM-CRC fuel coker tests. In the light of the poor experience with the standard coker in the present program for rating JP-6 fuels, it appears that the whole question of deterioration of thermal stability in normal storage should receive serious reexamination. This is not to suggest that all previous work based on the standard coker is valueless or that the standard coker will encounter operating problems on all JP-6 fuels. However, further investigation of the role of pump wear debris in creating wipable preheater deposits and false filter plugging in the standard coker is definitely in order. As a first step in this direction, it would be desirable to check for wipable preheater deposits as a part of the standard coker procedure whenever it is

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applied to JP-6 or other high-temperature fuels. In the event that pumpwear debris is found to be a frequent factor influencing ratings, serious consideration should be given to the use of a test such as the modified helium-drive coker.

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APPENDIX I

START-UP PROCEDURE FOR MODIFIED FUEL  
COKER WITH HELIUM DRIVE

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## APPENDIX I

### START-UP PROCEDURE FOR MODIFIED FUEL COKER TEST WITH HELIUM DRIVE

NOTE: Where not otherwise stated, follow the standard fuel coker procedure, ASTM D1660-61T.

#### Sequence for Use When Changing Test Fuel

1. Start to filter new test fuel into calibrated carboy, using standard Whatman No. 12 filters. The total amount required is 11.2 liters, which includes 1 liter for flushing blowcase, 200 ml for flushing fill line, and 10 liters for charging blowcase.
2. After emptying previous test fuel and venting blowcase, remove it from the system by disconnecting fittings at top and bottom.
3. Double rinse blowcase with filtered trisolvant, 700 ml each rinse (shake blowcase thoroughly with rinse solvent).
4. Reconnect blowcase to system and fill with 2000 ml of filtered trisolvant.
5. Pressurize blowcase to 20 psig with helium and flush system in standard manner.
6. When blowcase has been emptied, close valve from helium source and vent blowcase to atmosphere.
7. After depressurizing, install a new 10-micron element in the in-line filter ahead of test section.

NOTE: The other in-line filter (after the test section) is to be changed only as needed. Check its condition at least weekly and also after any run in which (a) the test filter pressure drop was 0.5 inch or more, (b) the test filter bypass line was opened during the run, or (c) the test filter was absent during the run.

8. Remove blowcase from the system, then double rinse it with filtered test fuel, 500 ml each rinse. Flush blowcase filling line with 200 ml of filtered test fuel.
9. Blow filtered air through the blowcase for 2 minutes at a high rate, supplying air through the regulator at 3 psig.

# Contrails

10. The remaining amount of filtered test fuel (in the carboy) should be exactly 10 liters; if not, adjust to 10 liters. Then aerate with diffuser stone in standard manner (3 minutes at 1 to 2 liters/minute).
11. Replace blowcase in system and fill with 10 liters of filtered and aerated test fuel. Fill blowcase by siphoning; do not use pressure or vacuum.
12. Pressurize slowly with helium, taking about 5 minutes to bring to 250 psig.
13. Flush with test fuel for several minutes while setting flow rate, and then commence run according to standard procedure, using warmup wattages as required for 2.5 lb/hr flow rate.

## Sequence for Use When Same Test Fuel Is Used

1. Start to filter test fuel into calibrated carboy, using standard Whatman No. 12 filters. The total amount required is 10 liters.

NOTE: Test fuel should not be left in glass carboys any longer than absolutely necessary, since even the diffused light in the laboratory can cause chemical changes in the fuel. If fuel must be left in a glass carboy longer than 8 hours, it should be protected from light and dust (for example, by an aluminum foil dust cover over the neck and a cloth over the entire carboy).

2. Empty test fuel from previous run from the blowcase. This may be added to the material being filtered.
3. Check and change in-line filters as needed.

NOTE: The in-line filter ahead of the test section does not need to be checked between runs on the same fuel, and it is in fact preferable to leave it alone unless contaminated fuel is suspected. The in-line filter after the test section must be checked at least weekly and also after any run in which (a) the test filter pressure drop was 0.5" or more, (b) the test filter bypass line was opened during the run, or (c) the test filter was absent during the run.

4. Blow filtered air through the blowcase for 2 minutes at a high rate, supplying air through the regulator at 3 psig.
5. Aerate the 10 liters of test fuel with diffuser stone in standard manner (3 minutes at 1 to 2 liters/minute).

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6. Fill blowcase with 10 liters of filtered and aerated test fuel. Fill blowcase by siphoning; do not use pressure or vacuum.
7. Pressurize slowly with helium, taking about 5 minutes to bring to 250 psig.
8. Flush with test fuel for several minutes while setting flow rate, and then commence run according to standard procedure, using warmup and running wattages as required for the 2.5 lb/hr flow rate.

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APPENDIX II

METHOD FOR DETERMINATION OF DISSOLVED  
OXYGEN CONTENT OF FUELS

# *Contrails*

APPENDIX II

METHOD FOR DETERMINATION OF DISSOLVED  
OXYGEN CONTENT OF FUELS

General

A gas chromatographic method is described for the analysis of dissolved oxygen in kerosine-type fuels. The basic analytic method was adapted by Southwest Research Institute from a method supplied by Phillips Petroleum Company. The sampling methods described herein are intended for application to analysis of fuel streams being fed to a fuel coker apparatus from a helium-pressurized blowcase.

Apparatus and Materials

The apparatus used is an Aerograph Gas Chromatographic Instrument, Model A-90 (Wilkins Instrument and Research, Inc.). This unit utilizes thermal conductivity cells as the sensing elements. Gas separation is effected by a five-foot length of coiled 1/4-in. copper tubing packed with 13X molecular sieves. Helium is used for the carrier gas. The column is unheated.

A small fore-column is connected to the main column by means of Tygon tubing. This fore-column, located between the injection port and the main column, is packed with molecular sieves and is changed before each test series. The purpose of the fore-column is to protect the main column from contamination by the injected fuel.

A 50-microliter gas-tight syringe is used for injecting air or oxygen samples into the column for calibration runs. Fuel samples are injected either by a 0.250-ml syringe or by a 0.250-ml pressurized sampling device.

Calibration

Initial calibration is accomplished by injecting 50 microliters of pure oxygen into the column and recording the peak height. This procedure may be repeated using pure nitrogen if it is also desired to analyze fuels for nitrogen content. A calibration factor or factors are obtained by counting the chart lines for the peak heights:

$$\text{Factor} = \frac{50}{\text{no. of chart lines}} = \text{microliters oxygen per chart line}$$

Air may be used for this calibration, using a 22:78 oxygen to nitrogen ratio for determining chart line values. Argon is included in the oxygen peak when calibrating with air.

## Open Sampling Method

A sampling tee, needle valve, and sampling line are provided in the fuel feed line between the helium-pressurized blowcase and the fuel coker. After purging the sampling line, the fuel is drawn into an open 250-ml beaker. With the fuel flowing slowly, and with the end of the sampling line well below the surface of the fuel in the beaker, a 0.250-ml syringe is filled with fuel drawn from the immediate vicinity of the end of the sampling line, thus obtaining fresh representative fuel before it has had time to absorb oxygen from the atmosphere. The sample is injected immediately into the injection port of the analyzer. The resultant peak heights are used to determine the oxygen content, and nitrogen content if desired, by multiplying the peak height (number of chart lines) by the previously obtained calibration factors:

$$\text{cc oxygen/cc fuel} = \frac{\text{Factor} \times \text{no. of chart lines}}{250}$$

This calculation gives cc oxygen/cc fuel with the oxygen measured at whatever partial pressure of oxygen was used in the calibration - atmospheric pressure if pure oxygen was used.

## High-Pressure Sampling Method

A 0.250-ml "Mikro-Tek" high-pressure sampler is connected into the fuel coker feed line (between the blowcase and the coker) and into the gas chromatographic analyzer. Using this arrangement, fuel being fed from the blowcase to the coker passes continuously through the sampler. Samples may be injected into the analyzer carrier gas stream at any time simply by pushing the plunger on the sampler. Determinations of oxygen (and nitrogen if desired) are made as described previously for open sampling.

## Discussion

The open sampling method is subject to some inherent error due to stripping of dissolved oxygen from the fuel during the evolution of dissolved helium at the instant of depressurization. This error becomes greater in proportion to increases in the dissolved helium content. The error can be minimized by drawing the sample into the syringe from the end of the sampling line and by rapid injection from the syringe into the analyzer. However, such errors do exist and are known to become serious toward the end of the coker run, i. e., when the dissolved helium content of the fuel feed stream is beginning to approach equilibrium with the 250 psi helium pressure in the blowcase. Therefore, the open sampling method cannot be recommended for accurate determination of dissolved oxygen content throughout the course of fuel coker runs. The pressurized sampling method is accurate and has presented no operational problems.

APPENDIX III

COMPLETE TEST DATA FROM HOT-ROOM STORAGE TESTS

# *Contrails*

**TABLE 17. COMPLETE FUEL COKER RATINGS FOR FIRST SERIES OF STORAGE TESTS**

Base Fuel: G-1060                      Air/Fuel volume ratio: 1/5 (2/1 after 12 weeks)  
 Storage temperature: 130° F  
 Water added, vol %: 0.05  
 Reaeration frequency: None

Fuel coker configuration (A) - Modified fuel coker with Eastern pump, using original preheaters.

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Base Fuel (G-1060)</u>				
0	425/525**	0.10	0000001122222	-
0	450/550**	0.18	0000011212222	-
0	450/550**	0.06	0000011222222	0000000000000
0	475/575**	0.43	0000111324444	0000000000000
0	500/600**	0.50	0000111424444	0000000000000
0	450/550	0.08	0000000000011	0000000000000
0	475/575	0.05	0000000001122	0000000000000
0	500/600	0.05	0000000011222	0000000000000
0	525/625	0.04	0000001223333	0000000000000
4	425/525	0.0	0000000000000	0000000000000
4	475/575	0.0	0000000012222	0000000000000
4	475/575	0.0	0000000112222	0000000000000
4	500/600	0.0	0000000012333	0000000000000
4	525/625	0.0	0000000123333	0000000000000

\*Preheater ratings are maximum color code observed in each inch of effective length, reading from cold end to hot end (left to right). The underlined numbers indicate a rating slightly higher than the number shown; e. g., 2 indicates a rating between the No. 2 and No. 3 standards.

\*\*This series of tests was run without any filter in the bypass return line to the reservoir.

TABLE 17. COMPLETE FUEL COKER RATINGS FOR FIRST  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Base Fuel (G-1060) (Cont'd)</u>				
8	450/550	0.05	000000000222	000000000000
8	475/575	0.0	000000001222	000000000000
8	500/600	0.0	000000001222	000000000000
8	500/600	0.0	000000001222	000000000000
8	525/625	0.0	000000222222	000000000000
12	450/550	0.0	000000001222	000000000000
12	500/600	0.0	000000001222	000000000000
12	550/650	0.0	000000012333	000000001000
16**	475/575	0.0	000000022233	000000000033
16	525/625	0.0	000000011444	000000000444
<u>Additive DBPC, 30 lb/1000 bbl</u>				
0	425/525	0.0	000000000000	000000000000
0	450/550	0.0	000000000000	000000000000
0	475/575	0.0	000000000000	000000000000
0	500/600	0.02	000000000111	000000000000
0	550/650	0.0	0000001223333	0000001223333
4	425/525	0.0	000000000000	000000000000
4	475/575	0.0	0000000011222	000000000000
4	500/600	0.0	0000000001333	000000000000
4	500/600	0.02	0000000123333	000000000000
8	450/550	0.0	000000000022	000000000000
8	475/575	0.0	000000000022	000000000000
8	525/625	0.0	0000000012221	000000000000
8	550/650	0.0	0000000002222	0000000001210
8	575/675	0.0	0000000013333	0000000013322
12	500/600	0.0	0000000001222	000000000000
12	525/625	0.0	0000000011222	000000000000
12	550/650	0.0	0000000012443	0000000001332

\*See note on first page of table.

\*\*Test terminated at 240 minutes; ran out of fuel.



TABLE 17. COMPLETE FUEL COKER RATINGS FOR FIRST  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive DBPC (Cont'd)</u>				
16	475/575	0.0	0000000000233	0000000000033
16	525/625	0.0	0000111114444	0000000114444
<u>Additive B-2, 50 lb/1000 bbl</u>				
0	425/525	0.0	0000000000000	0000000000000
0	475/575	0.0	0000000000000	0000000000000
0	525/625	0.05	0000000002222	0000000000000
0	550/650	0.08	0001111222222	0000000000000
0	575/675	0.0	0000012333333	0000000000000
4	450/550	0.0	0000000000122	0000000000000
4	475/575	0.0	0000000001222	0000000000000
4	500/600	0.0	0000000011222	0000000000000
4	525/625	0.0	0000000122333	0000000000011
8	500/600	0.1	0000000012221	0000000000000
8	525/625	0.0	0000000001222	0000000000000
8	550/650	0.0	0000000013332	0000000000000
12	450/550	0.1	0000000112222	0000000001100
12	475/575	0.2	0000001123333	0000000012211
12	525/625	0.3	0000023334444	0000000033333
16	400/500	0.2	0000000011222	0000000000222
16	450/550	0.6	0000000124444	0000000024444
<u>Additive D-1, 20 lb/1000 bbl</u>				
0	450/550	0.0	0000000000000	0000000000000
0	525/625	0.10	0000000112222	0000000000000
0	550/650	0.0	0000000022222	0000000001221
0	575/675	0.0	0000011233333	0000000000122
4	475/575	0.0	0000000012222	0000000000000
4	500/600	0.0	0000000133322	0000000000000

\*See note on first page of table.

TABLE 17. COMPLETE FUEL COKER RATINGS FOR FIRST  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive D-1 (Cont'd)</u>				
8	475/575	0.0	000000001222	000000000000
8	475/575	0.0	0000000011222	0000000000000
8	500/600	0.0	0000000001332	0000000000000
8	500/600	0.0	0000000011333	0000000000210
12	475/575	0.1	0000000001222	0000000000111
12	475/575	0.1	0000000011222	0000000000111
12	500/600	0.0	0000000013333	0000000002200
16	425/525	0.1	0000000000000	0000000000000
16	475/575	0.0	0000000001144	0000000000044
<u>Additive D-3, 30 lb/1000 bbl</u>				
0	475/575	0.0	0000000111222	0000000111222
0	500/600	0.0	0000000022333	0000000022333
0	525/625	0.05	0000001184444	0000001184444
4	450/550	0.0	0000000000011	0000000000000
4	450/550	0.0	0000000001122	0000000001122
4**	475/575	0.12	0000000011224	0000000011114
4	500/600	0.0	0000000001222	0000000000000
4	500/600	0.0	0000000112244	0000000111122
4	525/625	0.0	0000001144444	0000001144444
8	450/550	0.0	0000000001222	0000000000111
8	450/550	0.0	0000000000122	0000000000011
8	475/575	0.1	0000000001333	0000000000033
8	500/600	0.0	0000000113333	0000000000133
12	450/550	1.4	0000000001222	0000000000011
12	450/550	0.4	0000000012222	0000000001222
12	475/575	0.3	0000000023333	0000000000000
16	450/550	3.1	0000000000122	0000000000000
16	475/575	0.7	0000000011333	0000000000000

\*See note on first page of table.

\*\*Test terminated at 281 minutes; ran out of fuel.

# Contrails

TABLE 17. COMPLETE FUEL COKER RATINGS FOR FIRST  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive E-1, 30 lb/1000 bbl</u>				
0	425/525	0.20	000000000000	000000000000
0	450/550	2.45	000000000011	000000000000
0	475/575	0.60	0000000466888	0000000466888
0	475/575	0.10	0000000011666	0000000011666
0	475/575	0.28	0000000001666	0000000001666
4	425/525	0.10	000000000000	000000000000
4	450/550	26/230	000000000011	0000000000011
4	475/575	1.25	0000000001166	0000000001166
4	500/600	0.58	0000000011888	0000000011888
8	400/500	1.0	000000000000	000000000000
8	425/525	0.8	000000000000	000000000000
8	450/550	25/160	0000000011222	000000000000
8	450/550	25/225	0000000000144	0000000000044
12	400/500	0.1	000000000000	000000000000
12	425/525	5.7	0000000001111	000000000000
12	475/575	25/190	0000000011144	0000000000044
16	425/525	2.0	0000001111111	000000000000
16**	475/575	25/174	0000000000114	0000000000004
<u>Additive L-1, 30 lb/1000 bbl</u>				
0	500/600	0.0	0000000012222	000000000000
0	500/600	0.04	0000011002222	000000000000
0	525/625	0.0	0000000011333	000000000000
4	450/550	0.0	0000000000222	000000000000
4	475/575	0.0	0000000002222	0000000002200
4	500/600	0.0	0000000223322	0000000003300
4	500/600	0.0	0000000001122	000000000000
4	525/625	0.0	0000000001322	0000000001311

\*See note on first page of table.

\*\*Test terminated at 180 minutes; ran out of fuel.

TABLE 17. COMPLETE FUEL COKER RATINGS FOR FIRST  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive L-1 (Cont'd)</u>				
8	400/500	0.0	000000000000	000000000000
8	425/525	0.0	000000000000	000000000000
8	450/550	0.0	0000000001233	000000000000
8	450/550	0.0	0000000000133	000000000000
12	425/525	0.1	0000000011222	000000000000
12	425/525	0.2	0000000011122	000000000000
12	450/550	0.1	0001112211333	000000000000
16	425/525	0.0	000000000000	000000000000
16**	475/575	0.0	0000000000111	000000000000
<u>Additive F-1, 270 lb/1000 bbl</u>				
0	475/575	0.0	0000011100000	000000000000
0	575/675	0.0	0000000000011	000000000000
0	600/700	0.0	0000000011222	000000000000
4	450/550	0.0	0000000000011	000000000000
4	500/600	0.0	0000000000011	000000000000
4	550/650	0.0	0000000001111	000000000000
4	600/700	0.0	0000000000111	000000000000
8	550/650	0.0	0000000000011	000000000000
8	575/675	0.0	0000000011111	000000000000
8	575/675	0.0	0000000121111	0000000020000
8	600/700	0.0	0000000232332	0000000020330
8	600/700	0.0	0000004321111	0000004300000
12	500/600	0.0	0000000012311	0000000000300
12	525/625	0.0	0000000023222	0000000023111
12	575/675	0.1	0000000042211	0000000042111
16	450/550	0.0	0000000000122	0000000000022
16***	475/575	0.0	0000000002444	0000000000444

\*See note on first page of table.

\*\*Test terminated at 270 minutes; ran out of fuel.

\*\*\*Test terminated at 235 minutes; ran out of fuel.

TABLE 18. COMPLETE FUEL COKER RATINGS FOR  
SECOND SERIES OF STORAGE TESTS

Base Fuel: G-1060

Air/fuel volume ratio: 1/5  
Storage temperature: 130° F  
Water added, vol %: 0.05  
Reaeration frequency: 4-week

Fuel coker configuration (C) - Modified fuel coker with helium drive, using honed-out preheaters.

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Base Fuel (G-1060)</u>				
0	500/600	0.0	000000000111	000000000111
0	525/625	0.0	000000013432	000000013432
0	550/650	0.0	0000000124442	0000000124442
4	525/625	0.0	000000000111	000000000111
4	550/650	0.0	000000000333	000000000333
4	575/675	0.0	0000000124551	0000000124551
8	500/600	0.0	000000001111	000000001111
8	525/625	0.0	000000011111	000000011111
8	550/650	0.0	0000000111233	000000000233
12	525/625	0.0	0000000111111	0000000111111
12	550/650	0.0	0000000112334	0000000112334
12	575/675	0.0	0000000112244	0000000112244
<u>Additive DBPC, 270 lb/1000 bbl</u>				
0	550/650	0.0	0000000111110	0000000111110
0	600/700	0.0	000000002222	000000002222
0	625/700	0.0	000000014240	000000014140
4	525/625	0.0	0000000011111	0000000011111
4	550/650	0.0	000000000211	000000000211
4	575/675	0.0	0000011111141	0000011111141
8	500/600	0.0	0000000001111	0000000001111
8	525/625	0.0	0000000001332	0000000001332
8	550/650	0.0	0000000003123	0000000003023
12	475/575	0.0	0000000001122	0000000001122
12	500/600	0.0	0000000115541	0000000115541
12	525/625	0.0	000000004431	000000004431

\*Preheater ratings are maximum color code observed in each inch of effective length, reading from cold end to hot end (left to right). The underlined numbers indicate a rating slightly higher than the number shown; e.g., 2 indicates a rating between the No. 2 and No. 3 standards.

# Contrails

TABLE 18. COMPLETE FUEL COKER RATINGS FOR  
SECOND SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130 °F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive L-1, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000011111	0000000011111
0	600/700	0.0	0000112222111	0000112222111
0	625/700	0.0	0000000023521	0000000022511
4	575/675	0.1	0000000000000	0000000000000
4	600/700	0.0	0000000000111	0000000000111
4	625/700	0.0	0000000023433	0000000023433
8	550/650	0.0	0000000011111	0000000011111
8	575/675	0.0	0000000001111	0000000001111
8	600/700	0.0	0000001144335	0000001144335
12	550/650	0.0	0000000000111	0000000000111
12	575/675	0.0	0000000244332	0000000244332
12	600/700	0.0	0000000114345	0000000113345
<u>Additive L-1, 270 lb/1000 bbl</u>				
0	550/650	0.1	0000000001222	0000000001222
0	575/675	0.0	0000000023441	0000000023441
0	600/700	0.0	0000003333441	0000003333441
4	425/525	0.0	0000000000000	0000000000000
4	475/575	0.0	0000000344443	0000000344443
4	525/625	0.4	0000000114444	0000000114444
4	550/650	0.3	0000000004443	0000000004443
4	575/675	0.0	0000000022322	0000000022322
8	400/500	0.0	0000000000000	0000000000000
8	425/525	0.0	0000000000000	0000000000000
8	450/550	0.0	0000000000043	0000000000043
8	450/550	0.0	0000000000005	0000000000005
12	450/550	0.1	0000000000111	0000000000111
12	450/550	0.0	0000000000000	0000000000000
12	500/600	0.0	0000000001111	0000000001111
12	525/625	0.0	0000000123322	0000000123322
12	550/650	0.0	0000000555411	0000000555411

\*See note on first page of table.

TABLE 18. COMPLETE FUEL COKER RATINGS FOR  
SECOND SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive F-1, 270 lb/1000 bbl</u>				
0	550/650	0.0	0000000011111	0000000011111
0	600/700	0.0	0000000011111	0000000011111
0	650/700	0.0	0000000012222	0000000012222
0	675/700	0.0	0000011124225	0000011124225
4	550/650	0.0	0000001222222	0000001222222
4	600/700	0.0	0000001122222	0000000012222
4	625/725	0.0	0000011124442	0000011124442
4	650/700	0.0	0001113344334	0001111122214
8	600/700	0.0	0000000011111	0000000000000
8	625/700	0.0	0000000112222	0000000112222
8	650/700	0.0	0000000122225	0000000122225
12	550/650	0.0	0000000000111	0000000000111
12	575/675	0.0	0000001432221	0000001432221
12	600/700	0.0	0000000222332	0000000222221
<u>Additive K-1, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000002222	0000000002222
0	575/675	0.1	0000000143334	0000000143334
0	600/700	0.0	0000000114444	0000000114444
4	500/600	0.0	0000000001112	0000000001112
4	525/625	0.0	0000000013321	0000000013321
4	550/650	0.0	0000000344321	0000000344300
8	500/600	0.0	0000000011222	0000000011222
8	525/625	0.0	0000000011322	0000000011322
8	550/650	0.0	0000000013222	0000000013222
12	425/525	0.0	0000000000111	0000000000111
12	450/550	0.1	0000000001144	0000000001144
12	500/600	0.1	0000000014522	0000000014522

\*See note on first page of table.

TABLE 18. COMPLETE FUEL COKER RATINGS FOR  
SECOND SERIES OF STORAGE TESTS(Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Rating*	
			Unwiped	Wiped
<u>Additive L-2, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000011111	0000000011111
0	600/700	0.0	0000000012210	0000000012210
0	625/700	0.0	0000000222222	0000000222222
0	650/700	0.0	0000000036554	0000000036554
4	500/600	0.0	0000000000111	0000000000111
4	525/625	0.0	0000000002222	0000000002222
4	550/650	0.0	0000000002541	0000000002540
8	525/625	0.0	0000000011222	0000000011222
8	550/650	0.0	0000000223333	0000000223333
8	575/675	0.0	0000002444443	0000002444443
12	500/600	0.0	0000000001221	0000000001221
12	525/625	0.0	0000000012110	0000000012110
12	550/650	0.0	0000000014331	0000000014331
<u>Additive M-1, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000012211	0000000012211
0	575/675	0.0	0000001122221	0000001122221
0	600/700	0.0	0000000133332	0000000133332
4	550/650	0.0	0000000122221	0000000022220
4	575/675	0.0	0000000355542	0000000355542
4	600/700	0.0	0000013344488	0000013344488
8	500/600	0.0	0000000011111	0000000011111
8	525/625	0.0	0000000011111	0000000011111
8	550/650	0.0	0000000112234	0000000112234
12	500/600	0.0	0000000001111	0000000001111
12	525/625	0.0	0000000013222	0000000013222
12	550/650	0.0	0000000112332	0000000112332

\*See note on first page of table.



TABLE 19. COMPLETE FUEL COKER RATINGS FOR THIRD  
SERIES OF STORAGE TESTS

Base Fuel: H-1008

Air/fuel volume ratio: 1/2  
Storage temperature: 130° F  
Water added, vol %: None  
Reaeration frequency: 4-week

Fuel coker configuration (C) - Modified fuel coker with helium drive, using  
honed-out preheaters.

Weeks at 130° F	Test Temp, ° F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Base Fuel (H-1008)</u>				
0	500/600	0.0	000000 <u>111111</u>	000000 <u>111111</u>
0	525/625	0.3	00000000 <u>11222</u>	00000000 <u>11222</u>
0	550/650	0.0	0000000012331	0000000002330
4	500/600	0.0	0000000000111	0000000000111
4	525/625	0.0	0000000002332	0000000002332
4	550/650	0.0	0000000113345	0000000113345
8	500/600	0.0	0000000111111	0000000011111
8	525/625	0.0	0000000223444	0000000223444
8	550/650	0.0	0000000114555	0000000113555
12	425/525	0.0	0000000000222	0000000000222
12	450/550	0.0	0000000000555	0000000000555
12	500/600	0.0	0000000055555	0000000055555
16	425/525	0.0	0000011111122	0000011111122
16	450/550	0.0	0000000024443	0000000024443
16	475/575	0.0	0000000015555	0000000015555
<u>Additive B-1, 200 lb/1000 bbl</u>				
0	550/650	0.0	0000000011111	0000000011100
0	575/675	0.0	0000000001111	0000000001111
0	600/700	0.0	0000000011123	0000000011113
4	525/625	0.0	0000000001111	0000000000000
4	550/650	0.0	0000000000141	0000000000141
4	600/700	0.0	0000000155554	0000000155554

\*Preheater ratings are maximum color code observed in each inch of effective length, reading from cold end to hot end (left to right). The underlined numbers indicate a rating slightly higher than the number shown; e.g., 2 indicates a rating between the No. 2 and No. 3 standards.

TABLE 19. COMPLETE FUEL COKER RATINGS FOR THIRD  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive B-1 (Cont'd)</u>				
8	525/625	0.0	0000000001221	0000000001221
8	550/650	0.0	00000000011121	00000000011121
8	575/675	0.0	00000000122332	00000000122342
12	550/650	0.0	0000000001111	0000000001111
12	575/675	0.0	0000000001233	0000000001133
12	600/700	0.0	0000000023444	0000000013344
16	550/650	0.0	0000000022222	0000000022222
16	575/675	0.0	0000000011133	0000000011133
16	600/700	0.0	0000000023333	0000000023333
<u>Additive B-3, 20 lb/1000 bbl</u>				
0	550/650	0.0	0000000001111	0000000001111
0	575/675	0.0	0000000002442	0000000002442
0	600/700	0.0	00000000122344	00000000112244
4	500/600	0.0	0000000000111	0000000000111
4	525/625	0.0	0000000001111	0000000001111
4	550/650	0.0	00000000113355	0000000012355
8	500/600	0.0	0000000001211	0000000001211
8	525/625	0.0	0000000011243	0000000011243
8	550/650	0.0	0000000045555	0000000045566
12	500/600	0.0	0000000001111	0000000001111
12	525/625	0.0	0000000001443	0000000000443
12	550/650	0.0	0000000005555	0000000005555
16	425/525	0.0	0000000011111	0000000011111
16	450/550	0.1	0000000011143	0000000011143
16	500/600	0.0	0000000023444	0000000023444

\*See note on first page of table.

TABLE 19. COMPLETE FUEL COKER RATINGS FOR THIRD  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive D-4, 30 lb/1000 bbl</u>				
0	500/600	0.9	000000000111	000000000111
0	525/625	1.3	000000000111	000000000000
0	550/650	0.2	0000000113441	0000000111241
4	500/600	0.0	000000000111	000000000111
4	525/625	0.0	0000000001111	0000000001111
4	550/650	0.0	0000000023443	0000000012342
8	525/625	0.0	0000000002221	0000000002221
8	550/650	0.0	0000000114555	0000000114555
8	575/675	0.0	0000000114455	0000000114455
12	450/550	0.0	0000000012222	0000000012221
12	475/575	0.0	0000000011433	0000000011433
12	525/625	0.0	0000001144444	0000001155444
16	450/550	0.0	0000000000000	0000000000000
16	500/600	0.0	0000000001111	0000000001111
16	525/625	0.0	0000000001222	0000000001222
16	550/650	0.0	0000000114444	0000000114444
<u>Additive E-3, E-5; 30.8 lb/1000 bbl</u>				
0	550/650	0.0	0000000011111	0000000011111
0	600/700	0.0	0000000011222	0000000000111
0	625/700	0.0	0000000128218	0000000128218
4	550/650	0.0	0000000000111	0000000000111
4	575/675	0.0	0000000000111	0000000000111
4	600/700	0.0	0000000011321	0000000001321
8	525/625	0.0	0000000111111	0000000111111
8	550/650	0.0	0000000011111	0000000011111
8	575/675	0.0	0000000012244	0000000012244

\*See note on first page of table.

# Contrails

TABLE 19. COMPLETE FUEL COKER RATINGS FOR THIRD  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive E-3, E-5 (Cont'd)</u>				
12	550/650	0.0	0000001111111	0000001111111
12	575/675	0.0	0000000001222	0000000001222
12	600/700	0.0	0000001112321	0000001112211
16	575/675	0.0	0000000001111	0000000001111
16	600/700	0.0	0000000111433	0000000111433
16	625/700	0.0	0000002355777	0000002355777
<u>Additive H-2, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000000111	0000000000111
0	575/675	0.0	0000000001111	0000000001111
0	600/700	0.0	0000000001141	0000000001131
4	500/600	0.0	0000000111111	0000000111111
4	525/625	0.0	0000000001222	0000000001222
4	550/650	0.0	0000000000133	0000000000133
8	525/625	0.0	0000000000111	0000000000111
8	575/675	0.0	0000000001122	0000000001122
8	600/700	0.0	0000000000032	0000000000032
12	575/675	0.0	0000000011222	0000000011122
12	600/700	0.0	0000000001222	0000000001122
12	625/700	0.0	0000000014334	0000000015335
16	600/700	0.0	0000000001122	0000000001112
16	600/700	0.0	0000000011222	0000000011222
16	625/700	0.0	0000000154455	0000000154455
<u>Additive J-2, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000011222	0000000011111
0	575/675	0.0	0000000002555	0000000002555
0	600/700	0.0	0000000001244	0000000001244

\*See note on first page of table.

# Contrails

TABLE 19. COMPLETE FUEL COKER RATINGS FOR THIRD  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive J-2 (Cont'd)</u>				
4	525/625	0.2	0000000111111	0000000111111
4	550/650	0.0	0000000012342	0000000012342
4	575/675	0.0	0000000001243	0000000001243
8	475/575	0.0	0000000011111	0000000011111
8	500/600	0.0	0000000011222	0000000011222
8	525/625	1.1	0000000011124	0000000011114
12	500/600	0.0	0000000111111	0000000111111
12	525/625	0.0	0000000112211	0000000112211
12	550/650	0.0	0000000001143	0000000001143
16	525/625	0.0	0000000000000	0000000000000
16	550/650	0.0	0000000123444	0000000123444
16	575/675	0.0	0000000114455	0000000114455
<u>Additive L-1, 30 lb/1000 bbl</u>				
0	500/600	0.7	0000000011122	0000000011122
0	525/625	1.1	0000000122222	0000000122222
0	550/650	0.0	0000000001234	0000000000234
4	500/600	0.0	0000001111111	0000001111111
4	525/625	0.0	0000000011111	0000000011111
4	550/650	0.0	0000000012242	0000000002242
8	525/625	0.0	0000000011111	0000000011111
8	550/650	0.0	0000000001144	0000000001144
8	575/675	0.0	0000000012555	0000000012555
12	475/575	0.0	0000000011211	0000000011111
12	500/600	0.0	0000000011344	0000000011344
12	525/625	0.0	0000001114454	0000001114555

\*See note on first page of table.

TABLE 19. COMPLETE FUEL COKER RATINGS FOR THIRD  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive L-1 (Cont'd)</u>				
16	475/575	0.0	0000000111111	0000000111122
16	500/600	0.0	0000000011144	0000000011144
16	525/625	0.0	0000000114544	0000000114544
<u>Additive M-1, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000000122	0000000000011
0	575/675	0.0	0000000112332	0000000112332
0	600/700	0.0	0000000113445	0000000113445
4	500/600	0.0	0000000222222	0000000222222
4	525/625	0.0	0000000012222	0000000012222
4	550/650	0.0	0000000002344	0000000002344
8	425/525	0.0	0000000002222	0000000002222
8	450/550	0.0	0000000001444	0000000001444
8	475/575	0.0	0000000114454	0000000114454
8	525/625	0.2	0000000125543	0000000125543
12	425/525	0.0	0000000011111	0000000011111
12	450/550	0.0	0000000011111	0000000011111
12	475/575	0.0	0000000011333	0000000011333
16	400/500	0.0	0000000111111	0000000111111
16	425/525	0.0	0000000011555	0000000011555
16	450/550	0.0	0000000045555	0000000045555
<u>Additive N-1, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000011221	0000000001221
0	575/675	0.0	0000000012332	0000000001331
0	600/700	0.0	0000000011322	00000000011311

\*See note on first page of table.

TABLE 19. COMPLETE FUEL COKER RATINGS FOR THIRD  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive N-1 (Cont'd)</u>				
4	525/625	0.0	0000000111221	0000000111221
4	550/650	0.0	0000000022441	0000000022441
4	575/675	0.0	0000000024455	0000000024455
8	500/600	0.0	0000000000111	0000000000111
8	525/625	0.0	0000000023344	0000000023344
8	550/650	0.0	0000000012334	0000000012334
12	425/525	0.0	0000000001111	0000000001111
12	450/550	0.0	0000000001144	0000000001144
12	500/600	0.0	0000000114444	0000000115555
16	425/525	0.1	0000000111111	0000000111111
16	450/550	0.0	0000000011554	0000000011554
16	475/575	0.0	0000000115555	0000000115555
<u>Additive P-3, 30 lb/1000 bbl</u>				
0	500/600	0.2	0000000011111	0000000011111
0	525/625	0.0	0000000000111	0000000000111
0	550/650	0.0	0000000011241	0000000011141
4	500/600	0.0	0000000122222	0000000122222
4	525/625	0.0	0000000011333	0000000011333
4	550/650	0.0	0000000001245	0000000001245
8	450/550	0.0	0000000011111	0000000011111
8	475/575	0.0	0000000002222	0000000002222
8	500/600	0.0	0000000001333	0000000001333
12	425/525	0.0	0000000001111	0000000001111
12	450/550	0.0	0000000000110	0000000000110
12	475/575	0.0	0000000022455	0000000022455

\*See note on first page of table.

# Contrails

TABLE 19. COMPLETE FUEL COKER RATINGS FOR THIRD  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive P-3 (Cont'd)</u>				
16	400/500	0.0	0000000001111	0000000001111
16	425/525	0.0	0000000000114	0000000000114
16	450/550	0.0	0000000013444	0000000013444
<u>Additive F-1, 270 lb/1000 bbl</u>				
0	600/700	0.0	0000000000111	0000000000111
0	625/700	0.0	0000000111112	0000000000112
0	650/700	0.0	0000000014345	0000000014245
4	600/700	0.0	0000000001111	0000000001122
4	625/700	0.0	0000000113355	0000000113355
4	650/700	0.0	0000000226656	0000000226656
8	600/700	0.0	0000000001111	0000000001111
8	625/700	0.0	0000000006666	0000000004666
8	650/700	0.0	0000000018227	0000000016215
12	525/625	0.0	0000000011111	0000000011111
12	550/650	0.0	0000000011111	0000000011111
12	575/675	0.0	0000000011333	0000000011333
16	550/650	0.0	0000000011111	0000000011111
16	575/675	0.0	0000000013322	0000000013322
16	600/700	0.0	0000000023333	0000000023333

\*See note on first page of table.



TABLE 20. COMPLETE FUEL COKER RATINGS FOR FOURTH SERIES OF STORAGE TESTS

Base Fuel: H-1010

Air/fuel volume ratio: 1/2  
Storage temperature: 130° F  
Water added, vol %: None  
Reaeration frequency: 4-week

Fuel coker configuration (C) - Modified fuel coker with helium drive, using honed-out preheaters.

Weeks at 130° F	Test Temp, ° F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Base Fuel (H-1010)</u>				
0	500/600	0.0	0000000000000	0000000000000
0	525/625	0.0	0000000001122	0000000001122
0	550/650	0.0	0000000011353	0000000011353
4	500/600	0.9	0000000012222	000000002222
4	525/625	4.0	0000000112221	000000001110
4	550/650	0.0	0000001144444	0000000114444
8	475/575	0.0	0000000011221	000000000220
8	500/600	0.0	0000000002211	000000002200
8	525/625	0.0	0000000013331	0000000013331
12	400/500	0.0	0000000012222	0000000000000
12	425/525	0.0	0000000003333	0000000000000
12	450/550	0.0	0000000444444	0000000000000
12	500/600	0.0	0000001444444	0000000111144
16	400/500	0.0	0000000111111	0000000111111
16	425/525	0.0	0000000011111	0000000011111
16	450/550	0.0	0000000001233	0000000000000
<u>Additive B-1, 200 lb/1000 bbl</u>				
0	550/650	0.0	0000000000000	0000000000000
0	575/675	0.0	0000000000111	0000000000111
0	600/700	0.0	0000000011566	0000000011566
4	500/600	0.0	0000000000011	0000000000011
4	525/625	0.5	0000000001122	0000000001111
4	550/650	0.0	0000000123343	0000000000000

\*Preheater ratings are maximum color code observed in each inch of effective length, reading from cold end to hot end (left to right). The underlined numbers indicate a rating slightly higher than the number shown; e.g., 2 indicates a rating between the No. 2 and No. 3 standards.

TABLE 20. COMPLETE FUEL COKER RATINGS FOR FOURTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive B-1 (Cont'd)</u>				
8	525/625	0.0	0000000001111	0000000001111
8	550/650	0.0	0000000001233	0000000000000
8	575/675	0.0	0000000001133	0000000001133
12	475/575	0.0	0000000002222	0000000001111
12	500/600	0.0	0000000002233	0000000000000
12	525/625	0.8	0000000001444	0000000001111
16	475/575	0.0	0000000011111	0000000011111
16	525/625	0.0	0000000011111	0000000011111
16	550/650	0.0	0000000011111	0000000011111
16	575/675	0.0	0000000011232	0000000000000
<u>Additive B-3, 20 lb/1000 bbl</u>				
0	550/650	0.0	0000000001211	0000000001211
0	575/675	0.0	0000000012244	0000000012244
0	600/700	0.0	0000001134444	0000001134444
4	500/600	0.0	0000000001111	0000000001111
4	525/625	0.0	0000000001121	0000000001121
4	550/650	0.0	0000001123354	0000001123354
8	475/575	0.0	0000000011222	0000000000222
8	500/600	0.0	0000001144444	0000001122222
8	525/625	0.0	0000000134334	0000000113344
12	450/550	0.0	0000000000111	0000000000111
12	500/600	0.0	0000000001111	0000000001111
12	525/625	0.0	0000000011222	0000000011222
12	550/650	0.0	0000000334442	0000000223332
16	475/575	0.0	0000000111111	0000000111111
16	500/600	0.3	0000000122222	0000000122222
16	525/625	2.2	0000000133333	0000000122233

\*See note on first page of table.

TABLE 20. COMPLETE FUEL COKER RATINGS FOR FOURTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive D-4, 30 lb/1000 bbl</u>				
0	450/550	0.8	0000000001111	0000000001111
0	475/575	0.0	000000000 <u>4444</u>	000000000 <u>3444</u>
0	500/600	0.0	000000011 <u>4444</u>	000000011 <u>4444</u>
0	550/650	0.0	0000000 <u>444555</u>	0000000 <u>334555</u>
4	400/500	0.0	0000000011111	0000000011111
4	425/525	0.0	0000000001111	0000000000111
4	450/550	0.0	0000000012333	0000000011333
4	500/600	0.0	000000001 <u>4333</u>	000000000 <u>4333</u>
8	450/550	0.0	0000000001111	0000000001111
8	500/600	0.0	0000000001221	0000000001221
8	525/625	0.0	00000001 <u>33222</u>	00000001 <u>33222</u>
12	450/550	0.0	0000000011222	0000000011222
12	475/575	0.0	0000000112332	0000000111331
12	500/600	0.0	0000011133443	0000011133554
16	400/500	0.0	0000000000000	0000000000000
16	425/525	0.0	0000000000111	0000000000111
16	450/550	0.1	0000000011133	0000000011133
<u>Additive E-3,E-5; 30,8 lb/1000 bbl</u>				
0	550/650	0.0	0000000000111	0000000000111
0	575/675	0.0	0000000021111	0000000021111
0	600/700	0.0	000000000 <u>3443</u>	000000000 <u>3443</u>
4	550/650	0.0	0000000000122	0000000000122
4	575/675	0.0	0000000012233	0000000012233
4	600/700	0.0	0000000000233	0000000000111
8	525/625	0.0	0000000000111	0000000000111
8	550/650	0.0	0000000001122	0000000001122
8	575/675	0.0	00000000 <u>34222</u>	00000000 <u>34222</u>

\*See note on first page of table.

TABLE 20. COMPLETE FUEL COKER RATINGS FOR FOURTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive E-3, E-5 (Cont'd)</u>				
12	550/650	0.0	0000001122222	0000001122222
12	575/675	0.0	0000000001444	0000000001444
12	600/700	0.0	0000000033444	0000000022222
16	500/600	0.0	0000000000111	0000000000111
16	525/625	0.0	0000000000222	0000000000222
16	550/650	0.0	0000000001133	0000000001111
<u>Additive H-2, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000000111	0000000000111
0	575/675	0.0	0000000011222	0000000011222
0	600/700	0.0	0000000000116	0000000000116
4	525/625	0.2	0000000000111	0000000000111
4	550/650	0.0	0000000000122	0000000000022
4	575/675	0.2	0000000012333	0000000011111
8	550/650	0.0	0000000000111	0000000000111
8	600/700	0.0	0000000011222	0000000000000
8	625/700	0.0	0000000000336	0000000000006
12	525/625	1.4	0000000112222	0000000112222
12	550/650	0.0	0000000011333	0000000011333
12	600/700	0.0	0000000224555	0000000112444
16	525/625	0.0	0000000000222	0000000000111
16	550/650	0.0	0000000012333	0000000011111
16	575/675	0.0	0000000114444	0000000111111
<u>Additive J-2, 30 lb/1000 bbl</u>				
0	500/600	0.0	0000000012221	0000000011221
0	525/625	0.0	0000000234443	0000000234443
0	550/650	0.0	0000000022346	0000000022336

\*See note on first page of table.

# Contrails

TABLE 20. COMPLETE FUEL COKER RATINGS FOR FOURTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive J-2 (Cont'd)</u>				
4	375/475	0.0	000000000000	000000000000
4	400/500	0.1	000000011111	000000011111
4	425/525	0.0	000000000133	000000000133
4	475/575	0.0	000000014544	000000014554
8	425/525	0.5	000000001111	000000001111
8	450/550	0.0	000000001222	000000001222
8	475/575	0.0	000000003333	000000003333
12	450/550	0.1	000000000011	000000000011
12	475/575	0.0	000000001122	000000001122
12	500/600	0.0	000000011333	000000011333
16	425/525	0.0	000000001111	000000001111
16	450/550	0.0	000000001222	000000001222
16	475/575	0.0	000000000145	000000000145
<u>Additive L-1, 30 lb/1000 bbl</u>				
0	450/550	0.0	000000001211	000000001211
0	475/575	0.0	000000012111	000000012111
0	500/600	0.0	000000022421	000000022421
0	550/650	0.0	000002334334	000002334334
4	400/500	0.0	000000001111	000000001111
4	425/525	0.0	000000012331	000000012331
4	450/550	0.0	000000012332	000000002332
8	425/525	0.0	000000000000	000000000000
8	450/550	0.0	000000000111	000000000111
8	475/575	0.0	000000000144	000000000144
12	450/550	0.0	000000000011	000000000011
12	500/600	0.0	000000000111	000000000111
12	525/625	0.0	000000011333	000000011333

\*See note on first page of table.

TABLE 20. COMPLETE FUEL COKER RATINGS FOR FOURTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive L-1 (Cont'd)</u>				
16	500/600	0.0	0000000011111	0000000011111
16	525/625	0.0	0000000011222	0000000011222
16	550/650	0.0	0000000011333	0000000011333
<u>Additive M-1, 30 lb/1000 bbl</u>				
0	425/525	0.1	0000000004651	0000000004651
0	450/550	0.0	0000000003443	0000000003443
0	500/600	0.0	0000000005542	0000000005542
0	550/650	0.0	0000000154441	0000000154441
4	375/475	0.0	0000000001111	0000000000111
4	400/500	1.2	0000000003444	0000000003444
4	400/500	0.0	0000000003444	0000000003444
8	375/475	0.0	0000000000111	0000000000111
8	400/500	0.0	0000000013454	0000000011664
8	400/500	25/240	0000000012333	0000000011354
12	375/475	0.2	0000000011111	0000000011111
12	400/500	4.8	0000000001233	0000000000000
12	400/500	4.8	0000000001333	0000000000000
16	375/475	0.0	0000000011222	0000000011111
16	400/500	25/220	0000000012343	0000000000343
16	400/500	10.5	0000000011444	0000000011444
<u>Additive N-1, 30 lb/1000 bbl</u>				
0	500/600	0.0	0000000012222	0000000011222
0	525/625	0.0	0000000013221	0000000013221
0	550/650	0.0	0000000134665	0000000134665

\*See note on first page of table.

TABLE 20. COMPLETE FUEL COKER RATINGS FOR FOURTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, ° F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive N-1 (Cont'd)</u>				
4	400/500	0.0	000000000111	000000000111
4	425/525	0.0	0000000001232	0000000001232
4	475/575	0.0	0000000014553	0000000014553
8	400/500	0.0	000000000111	000000000111
8	450/550	0.0	000000000111	000000000111
8	475/575	0.0	0000000001332	0000000001332
8	500/600	0.0	0000000004321	0000000004321
12	400/500	0.0	0000000011111	0000000011111
12	425/525	0.0	0000000011122	0000000011122
12	450/550	0.0	0000000011333	0000000011333
16	425/525	0.0	0000000111122	0000000111122
16	450/550	0.0	0000000022222	0000000022222
16	475/575	0.0	0000000011333	0000000011333
<u>Additive P-3, 30 lb/1000 bbl</u>				
0	450/550	0.0	000000000111	000000000111
0	475/575	0.0	0000000003222	0000000003211
0	500/600	0.0	0000000034432	0000000034432
0	550/650	0.0	0000000344442	0000000344442
4	425/525	0.0	0000000011111	0000000011111
4	475/575	0.0	0000000022211	0000000022211
4	500/600	0.0	0000000133222	0000000133222
8	475/575	0.0	0000000001222	0000000001222
8	500/600	0.0	0000000001223	0000000001223
8	525/625	0.0	0000000012223	0000000012223
12	475/575	0.0	0000000011111	0000000011111
12	525/625	0.0	0000000021111	0000000021111
12	550/650	0.0	0000000011333	0000000011222

\*See note on first page of table.

TABLE 20. COMPLETE FUEL COKER RATINGS FOR FOURTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive P-3 (Cont'd)</u>				
16	475/575	0.0	0000000001222	0000000001222
16	500/600	0.0	0000000112222	0000000112222
16	525/625	0.0	0000000001332	0000000001332
<u>Additive F-1, 270 lb/1000 bbl</u>				
0	550/650	0.0	0000000011121	0000000001121
0	575/675	0.0	0000000001221	0000000001221
0	600/700	0.0	0000000011231	0000000011231
4	550/650	0.0	0000000011122	0000000011122
4	575/675	0.0	0000000011111	0000000011111
4	600/700	0.0	0000000011344	0000000011344
8	475/575	0.0	0000000012222	0000000012222
8	500/600	0.0	0000000534333	0000000423222
8	525/625	0.0	0000000543333	0000000543333
8	575/675	0.0	0000001442222	0000000411111
12	475/575	0.0	0000000001111	0000000001111
12	525/625	0.0	0000000022221	0000000011111
12	550/650	0.0	0000001142222	0000001142222
16	450/550	0.0	0000000000222	0000000000111
16	475/575	0.0	0000000013322	0000000003311
16	525/625	0.0	0000000054332	0000000054332

\*See note on first page of table.



TABLE 21. COMPLETE FUEL COKER RATINGS FOR FIFTH SERIES OF STORAGE TESTS

Base Fuel: H-1008

Air/fuel volume ratio: 1/2  
 Storage temperature: 130° F  
 Water added, vol %: None  
 Reaeration frequency: 4-week

Fuel coker configuration (C) - Modified fuel coker with helium drive, using honed-out preheaters.

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Base Fuel (H-1008)</u>				
0	550/650	0.0	0000001222222	0000001222222
0	575/675	0.0	0000000013341	0000000013341
0	600/700	0.0	0000000012441	0000000012441
8	550/650	0.0	0000000011222	0000000011222
8	575/675	0.0	0000000011344	0000000011344
8	600/700	0.0	0000000223444	0000000223444
<u>Additive B-1, 200 lb/1000 bbl</u>				
0	575/675	0.0	0000000001111	0000000001111
0	600/700	0.0	0000000011222	0000000011222
0	625/700	0.0	0000000000144	0000000000144
8	575/675	0.0	0000000111111	0000000111111
8	600/700	0.0	0000000001111	0000000001111
8	625/700	0.0	0000000112266	0000000112266

\*Preheater ratings are maximum color code observed in each inch of effective length, reading from cold end to hot end (left to right). The underlined numbers indicate a rating slightly higher than the number shown; e. g., 2 indicates a rating between the No. 2 and No. 3 standards.

TABLE 21. COMPLETE FUEL COKER RATINGS FOR FIFTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive B-3, 20 lb/1000 bbl</u>				
0	550/650	0.0	0000000012221	0000000012221
0	575/675	0.0	0000000111222	0000000011222
0	600/700	0.0	0000000011333	0000000011222
8	525/625	0.0	0000000111111	0000000111111
8	550/650	0.0	0000000111222	0000000111222
8	575/675	0.0	0000000113355	0000000113355
<u>Additive D-4, 30 lb/1000 bbl</u>				
0	550/650	0.0	0000000011222	0000000011222
0	575/675	0.0	0000000002321	0000000002321
0	600/700	0.0	0000000023333	0000000012233
8	500/600	0.0	0000000001111	0000000001111
8	525/625	0.0	0000000003333	0000000003333
8	550/650	0.1	0000000003354	0000000003354
<u>Additive E-3, E-5; 30, 8 lb/1000 bbl</u>				
0	575/675	0.0	0000000001111	0000000001111
0	600/700	0.0	0000000011111	0000000011111
0	625/700	0.0	0000000027726	0000000027726
8	575/675	0.0	0000000112222	0000000112222
8	600/700	0.0	0000000122232	0000000122232
8	625/700	0.0	0000001122266	0000001122266
<u>Additive L-1, 30 lb/1000 bbl</u>				
0	500/600	0.4	0000000111111	0000000111111
0	525/625	0.7	0000000112222	0000000111221
0	550/650	0.0	0000000001324	0000000001324
8	525/625	0.0	0000000000111	0000000000111
8	550/650	0.0	0000000011222	0000000011222
8	575/675	0.0	0000000122332	0000000122332

\*See note on first page of table.

TABLE 21. COMPLETE FUEL COKER RATINGS FOR FIFTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive M-1, 30 lb/1000 bbl</u>				
0	550/650	0.0	000000001111	000000001111
0	600/700	0.0	000000001122	000000001122
0	625/700	0.0	0000000065114	0000000065114
8	525/625	0.0	000000001111	000000001111
8	550/650	0.0	0000000123333	0000000123333
8	575/675	0.0	0000000133342	0000000133342
<u>Additive F-1, 270 lb/1000 bbl</u>				
0	600/700	0.0	000000001111	000000001111
0	625/700	0.0	0000000022222	0000000022222
0	650/700	0.0	0000000012234	0000000012234
8	575/675	0.0	000000001111	000000001111
8	600/700	0.0	0000000112222	0000000112222
8	625/700	0.0	0000000114455	0000000114455

\*See note on first page of table.

TABLE 22. COMPLETE FUEL COKER RATINGS FOR SIXTH SERIES OF STORAGE TESTS

Base Fuel: H-1010

Air/fuel volume ratio: 1/2  
Storage temperature: 130° F  
Water added, vol %: None  
Reaeration frequency: 4-week

Fuel coker configuration (C) - Modified fuel coker with helium drive, using honed-out preheaters.

Weeks at 130° F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped

Base Fuel (H-1010)

0	550/650	0.0	0000000001111	0000000001111
0	575/675	0.0	0000000001244	0000000001244
0	600/700	0.0	0000000011155	0000000011155
8	500/600	0.0	0000000122222	0000000112222
8	525/625	0.0	0000000334432	0000000224432
8	550/650	0.0	0000011444444	0000011445544

Additive B-1, 200 lb/1000 bbl

0	575/675	0.0	0000000001111	0000000001111
0	600/700	0.0	0000000001111	0000000001111
0	625/700	0.0	0000000028886	0000000028886
8	525/625	0.0	0000000001111	0000000001111
8	550/650	0.0	0000000032222	0000000022221
8	575/675	0.0	0000000001144	0000000001144

Additive B-3, 20 lb/1000 bbl

0	550/650	0.0	0000000001111	0000000001111
0	575/675	0.0	0000000001444	0000000001444
0	600/700	0.0	0000000000144	0000000000144

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\*Preheater ratings are maximum color code observed in each inch of effective length, reading from cold end to hot end (left to right). The underlined numbers indicate a rating slightly higher than the number shown; e. g., 2 indicates a rating between the No. 2 and No. 3 standards.

TABLE 22. COMPLETE FUEL COKER RATINGS FOR SIXTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130° F	Test Temp, ° F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive B-3 (Cont'd)</u>				
8	500/600	0.0	000000002222	000000002222
8	525/625	0.0	0000000011332	0000000011332
8	550/650	0.0	0000000135544	0000000115555
<u>Additive D-4, 30 lb/1000 bbl</u>				
0	500/600	0.0	0000000012111	0000000012111
0	525/625	0.0	0000002223222	0000002223222
0	550/650	0.0	0000000122244	0000000112244
8	450/550	0.0	0000000012222	0000000012222
8	475/575	0.0	0000000113333	0000000113333
8	500/600	0.0	0000000003343	0000000003343
<u>Additive E-3, E-5; 30, 8 lb/1000 bbl</u>				
0	575/675	0.0	0000000111111	0000000111111
0	600/700	0.0	0000000011111	0000000011111
0	625/700	0.0	0000000556226	0000000556226
8	525/625	0.0	0000000011122	0000000011122
8	550/650	0.0	0000000012222	0000000011222
8	575/675	0.0	0000000011244	0000000011111
<u>Additive H-2, 30 lb/1000 bbl</u>				
0	575/675	0.0	0000000011222	0000000011222
0	600/700	0.0	0000000122333	0000000122333
0	625/700	0.0	0000000128666	0000000128666
8	525/625	0.0	0000000001111	0000000001111
8	550/650	0.0	0000000012222	0000000011111
8	575/675	0.0	0000000000444	0000000000112

\*See note on first page of table.

TABLE 22. COMPLETE FUEL COKER RATINGS FOR SIXTH  
SERIES OF STORAGE TESTS (Cont'd)

Weeks at 130°F	Test Temp, °F (Preheater/Filter)	Filter ΔP, in. Hg	Preheater Ratings*	
			Unwiped	Wiped
<u>Additive L-1, 30 lb/1000 bbl</u>				
0	525/625	0.0	0000000001111	0000000001111
0	550/650	0.1	0000000002211	0000000001111
0	575/675	0.0	0000000011144	0000000011144
8	500/600	0.0	0000000001111	0000000001111
8	525/625	0.0	0000000011222	0000000000122
8	550/650	0.0	0000000011322	0000000011322
<u>Additive M-1, 30 lb/1000 bbl</u>				
0	375/475	0.0	0000000000000	0000000000000
0	400/500	0.0	0000000001122	0000000001122
0	425/525	0.0	0000000004555	0000000004555
8	375/475	0.0	0000000001111	0000000001111
8	400/500	0.0	0000000111144	0000000111144
8	400/500	0.0	000000002243	0000000002243
<u>Additive N-1, 30 lb/1000 bbl</u>				
0	525/625	0.0	0000001122111	0000001122111
0	550/650	0.0	0000000112221	0000000111111
0	575/675	0.0	0000000111332	0000000111332
8	500/600	0.0	0000000112222	0000000111122
8	525/625	0.0	0000001333333	0000001222332
8	550/650	0.0	0000000124443	0000000124443
<u>Additive F-1, 270 lb/1000 bbl</u>				
0	575/675	0.0	0000000001111	0000000001111
0	600/700	0.0	0000000112222	0000000001222
0	625/700	0.0	0000000133334	0000000133334

\*See note on first page of table.

# Contrails

TABLE 22. COMPLETE FUEL COKER RATINGS FOR SIXTH  
SERIES OF STORAGE TESTS (Cont'd)

<u>Weeks</u> at 130° F	<u>Test Temp, ° F</u> (Preheater/Filter)	<u>Filter ΔP,</u> <u>in. Hg</u>	<u>Preheater Ratings*</u>	
			<u>Unwiped</u>	<u>Wiped</u>
<u>Additive F-1 (Cont'd)</u>				
8	525/625	0.0	0000001112222	0000001112222
8	550/650	0.0	0000000014111	0000000014111
8	600/700	0.0	0000000266554	0000000266554

\*See note on first page of table.

TABLE 23. WATER SEPAROMETER DATA FOR FIRST SERIES OF STORAGE TESTS

<u>Additive</u>	<u>Concn,</u> <u>lb/1000 bbl</u>	<u>WSIM after Weeks of Storage</u>			
		<u>0</u>	<u>4</u>	<u>8</u>	<u>12</u>
None (G-1060 Base Fuel)		72	98	68	83
DBPC	30	87	92	81	74
B-2	50	94	65	72	71
D-1	20	97	97	66	74
D-3	30	79	-	75	60
E-1	30	83	97	85	76
L-1	30	82	82	82	75
F-1	270	48	47	52	39

TABLE 24. WATER SEPAROMETER DATA FOR SECOND SERIES OF STORAGE TESTS

<u>Additive</u>	<u>Concn,</u> <u>lb/1000 bbl</u>	<u>WSIM after Weeks of Storage</u>			
		<u>0</u>	<u>4</u>	<u>8</u>	<u>12</u>
None (G-1060 Base Fuel)		49	74	76	75
DBPC	270	68	74	65	81
L-1	30	57	64	68	84
L-1	270	89	56	66	73
F-1	270	40	45	54	42
K-1	30	51	48	43	94
L-2	30	89	99	98	98
M-1	30	54	42	54	67



TABLE 25. WATER SEPAROMETER DATA FOR THIRD SERIES OF STORAGE TESTS

<u>Additive</u>	<u>Concn,</u> <u>lb/1000 bbl</u>	<u>WSIM after Weeks of Storage</u>				
		<u>0</u>	<u>4</u>	<u>8</u>	<u>12</u>	<u>16</u>
None (H-1008 Base Fuel)		93	97	86	86	50
B-1	200	85	90	89	82	48
B-3	20	75	79	87	71	37
D-4	30	97	88	84	72	47
E-3, E-5	30, 8	85	94	96	89	74
H-2	30	78	93	88	65	64
J-2	30	92	96	89	81	70
L-1	30	88	91	86	80	83
M-1	30	86	92	--	57	76
N-1	30	94	82	73	78	77
P-3	30	82	91	95	55	82
F-1	270	80	51	48	33	20

TABLE 26. WATER SEPAROMETER DATA FOR FOURTH SERIES OF STORAGE TESTS

<u>Additive</u>	<u>Concn,</u> <u>lb/1000 bbl</u>	<u>WSIM after Weeks of Storage</u>				
		<u>0</u>	<u>4</u>	<u>8</u>	<u>12</u>	<u>16</u>
None (H-1010 Base Fuel)		92	91	84	89	96
B-1	200	92	82	88	51	89
B-3	20	92	93	77	97	67
D-4	30	99	98	92	96	97
E-3, E-5	30, 8	97	85	90	73	67
H-2	30	97	78	98	65	51
J-2	30	92	98	99	94	89
L-1	30	96	92	99	93	87
M-1	30	--	73	83	77	63
N-1	30	94	87	98	57	88
P-3	30	82	100	98	86	95
F-1	270	76	76	71	62	72

TABLE 27. WATER SEPAROMETER DATA FOR FIFTH SERIES OF STORAGE TESTS

<u>Additive</u>	<u>Concn,</u> <u>lb/1000 bbl</u>	<u>WSIM after Weeks</u> <u>of Storage</u>	
		<u>0</u>	<u>8</u>
None (H-1008 base fuel)		96	88
B-1	200	95	89
B-3	20	98	55
D-4	30	85	76
E-3, E-5	30, 8	100	90
L-1	30	96	90
M-1	30	96	92
F-1	270	62	59

TABLE 28. WATER SEPAROMETER DATA FOR SIXTH SERIES OF STORAGE TESTS

<u>Additive</u>	<u>Concn,</u> <u>lb/1000 bbl</u>	<u>WSIM after Weeks</u> <u>of Storage</u>	
		<u>0</u>	<u>8</u>
None (H-1010 base fuel)		100	82
B-1	200	94	68
B-3	20	92	88
D-4	30	94	98
E-3, E-5	30, 8	98	84
H-2	30	99	74
L-1	30	100	98
M-1	30	95	86
N-1	30	94	86
F-1	270	97	80

TABLE 29. LIGHT TRANSMITTANCE VALUES FOR FIRST SERIES OF STORAGE TESTS

Additive and Concentration*	Light Transmittance, % of Isooctane									
	Fresh Blend		4 Weeks at 130° F		8 Weeks at 130° F		12 Weeks at 130° F			
	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ		
None	87.6	88.4	-	-	93.2	94.0	87.2	89.9		
DBPC, 30	91.4	95.6	-	-	90.2	92.2	86.1	86.9		
B-2, 50	91.2	94.0	-	-	83.4	90.2	79.6	86.4		
D-1, 20	86.2	89.9	-	-	67.0	77.8	64.8	77.2		
D-3, 30	93.8	94.2	83.0	84.9	77.8	78.0	84.4	81.2		
E-1, 30	37.2	90.0	35.2	83.8	34.2	80.4	31.5	76.6		
L-1, 30	89.8	92.0	83.2	75.1	59.9	47.6	71.0	57.8		
F-1, 270	87.2	89.0	91.8	93.6	86.8	87.8	89.8	94.0		

\*In JP-6 base fuel G-1060; concentrations in lb/1000 bbl.

TABLE 30. LIGHT TRANSMITTANCE VALUES FOR SECOND SERIES OF STORAGE TESTS

Additive and Concentration*	Light Transmittance, % of Isooctane											
	Fresh Blend		4 Weeks at 130°F		8 Weeks at 130°F		12 Weeks at 130°F					
	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ				
None	98.2	97.2	94.8	96.8	92.4	94.6	91.0	93.4				
DBPC, 270	96.4	86.0	96.3	97.8	92.8	95.4	91.4	94.4				
L-1, 30	88.2	82.4	81.5	70.8	69.2	55.8	68.4	54.2				
L-1, 270	47.2	23.8	26.4	11.4	22.4	8.2	24.8	10.2				
F-1, 270	95.6	95.2	94.8	96.4	95.0	97.5	93.4	97.1				
K-1, 30	87.4	94.1	75.8	87.2	77.5	90.0	71.2	88.6				
L-2, 30	98.2	96.8	87.3	91.8	82.1	88.1	78.1	84.3				
M-1, 30	96.4	98.2	95.0	96.2	92.0	95.4	90.4	93.8				

\*In JP-6 base fuel G-1060; concentrations in lb/1000 bbl.

TABLE 31. LIGHT TRANSMITTANCE VALUES FOR  
THIRD SERIES OF STORAGE TESTS

Additive and Concentration*	Light Transmittance, % of Isooctane											
	Fresh Blend		4 Weeks at 130° F		8 Weeks at 130° F		12 Weeks at 130° F		16 Weeks at 130° F		16 Weeks at 130° F	
	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ
None	89.8	94.8	87.2	93.6	85.6	87.8	72.1	72.8	71.6	75.2		
B-1, 200	85.8	96.8	84.2	95.6	78.2	80.4	74.2	75.6	71.4	73.2		
B-3, 20	91.6	93.8	90.4	91.8	88.2	89.8	82.4	84.9	80.0	82.8		
D-4, 30	89.8	92.4	84.6	88.6	80.6	82.1	78.6	79.2	77.2	78.2		
E-3, E-5; 30, 8	93.1	95.6	86.4	89.8	80.4	82.2	79.9	80.4	78.6	79.9		
H-2, 30	96.5	97.1	88.8	92.4	85.0	86.6	81.6	83.3	79.2	80.4		
J-2, 30	95.2	96.6	86.4	89.6	86.2	87.5	85.6	87.2	73.5	75.2		
L-1, 30	88.6	89.8	88.4	90.4	85.7	87.9	80.4	82.6	79.6	80.4		
M-1, 30	89.4	92.3	86.2	88.6	84.3	85.5	79.1	82.4	77.5	79.6		
N-1, 30	92.6	95.8	86.6	88.0	82.4	84.6	92.3	91.5	90.1	92.4		
P-3, 30	89.2	92.8	86.2	87.6	83.6	86.6	94.8	97.0	92.6	93.5		
F-1, 270	96.5	98.0	94.6	96.8	92.5	94.0	89.2	95.8	85.4	87.6		

\*In JP-6 base fuel H-1008; concentrations in lb/1000 bbl.

TABLE 32. LIGHT TRANSMITTANCE VALUES FOR FOURTH SERIES OF STORAGE TESTS

Additive and Concentration*	Light Transmittance, % of Isooctane											
	Fresh Blend		4 Weeks at 130°F		8 Weeks at 130°F		12 Weeks at 130°F		16 Weeks at 130°F		375 mμ	
	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ	350 mμ	375 mμ
None	95.0	97.5	93.6	95.1	88.8	89.6	61.5	62.2	60.5	63.2		
B-1, 200	86.2	97.0	85.6	91.2	83.6	85.0	71.0	72.2	69.9	71.2		
B-3, 20	87.4	89.6	86.2	88.4	83.6	85.5	80.2	82.6	79.6	81.4		
D-4, 30	94.2	96.4	74.1	78.6	71.2	75.3	74.2	75.7	72.1	73.5		
E-3, E-5; 30, 8	90.4	93.8	84.1	86.1	82.4	84.0	78.8	79.4	76.5	78.3		
H-2, 30	93.4	95.8	82.1	84.8	79.8	80.4	72.4	74.9	71.6	73.1		
J-2, 30	86.5	88.3	82.0	86.5	79.3	81.6	75.4	79.2	72.1	73.9		
L-1, 30	85.2	87.4	82.6	89.6	80.4	82.9	78.2	79.6	76.4	78.8		
M-1, 30	85.1	86.3	84.6	86.8	81.3	84.6	68.5	70.2	67.2	70.4		
N-1, 30	90.3	94.0	82.8	84.4	78.8	82.4	88.0	90.8	86.7	87.5		
P-3, 30	85.6	87.3	90.4	92.4	88.5	89.6	93.5	97.2	90.2	95.7		
F-1, 270	95.4	97.8	87.6	89.9	84.6	85.3	91.7	95.2	89.7	90.2		

\*In JP-6 base fuel H-1010; concentration in lb/1000 bbl.

TABLE 33. LIGHT TRANSMITTANCE VALUES FOR FIFTH  
SERIES OF STORAGE TESTS

Additive and Concentration*	Light Transmittance, % of Isooctane			
	Fresh Blend		8 Weeks at 130° F	
	350 mμ	375 mμ	350 mμ	375 mμ
None	94.4	96.5	92.6	93.4
B-1, 200	95.5	97.5	93.1	95.7
B-3, 20	95.2	97.6	94.1	96.6
D-4, 30	91.0	93.4	84.6	87.2
E-3, E-5; 30, 8	92.6	94.9	89.5	90.2
L-1, 30	92.4	94.0	88.6	90.2
M-1, 30	91.0	92.8	89.2	92.3
F-1, 270	96.4	98.8	94.4	96.2

\*In JP-6 base fuel H-1008; concentrations in lb/1000 bbl.

TABLE 34. LIGHT TRANSMITTANCE VALUES FOR SIXTH SERIES OF STORAGE TESTS

Additive and Concentration*	Light Transmittance, % of Isooctane			
	Fresh Blend		8 Weeks at 130° F	
	350 m $\mu$	375 m $\mu$	350 m $\mu$	375 m $\mu$
None	95.6	97.8	90.1	93.6
B-1, 200	96.4	98.4	93.7	94.8
B-3, 20	96.6	97.2	90.4	93.5
D-4, 30	92.5	94.8	88.9	90.4
E-3, E-5; 30, 8	93.5	94.7	87.6	88.4
H-2, 30	95.7	96.8	91.8	94.4
L-1, 30	93.2	95.8	89.4	91.3
M-1, 30	93.4	95.6	91.0	93.5
N-1, 30	94.6	96.2	92.5	93.7
F-1, 270	96.8	99.2	95.7	97.1

\*In JP-6 base fuel H-1010; concentrations in lb/1000 bbl.